

## Final Publishable JRP Summary for IND15 SurfChem Traceable Quantitative Surface Chemical Analysis for Industrial Applications

### Overview

The chemical and topographical metrology of surfaces is one of the most rapidly growing fields of metrology. This is driven by the need for reliable chemical measurements and accurate and traceable surface analysis measurements in European industry for product development, for quality control in production and for the protection of intellectual property. To address this need, this project aimed to provide traceable measurement methods and certified reference materials (CRM) for surface chemical analysis in order to support next generation technologies developed by European manufactures (e.g. organic solar cells, printed organic electronics, organic light emitting diodes, biochips). The project provided prototypes of well-characterised reference materials and traceable methods to enable the comparability of results between laboratories; as this is currently a limiting factor. In addition, new quantitative methods for measuring surfaces were developed as Good Practice Guides and, based on them; New Work Item Proposals for industrial standards ISO TC 201 "Surface Chemical Analysis" and ISO 202 "Microprobe Analysis" have been initiated.

### Need for the project

Surface chemical analysis has made major contributions to industry with the development of new products and the solution to production problems. Surface chemical analysis has been important for example in both the science and technology of microelectronics, coatings for optical, wear and other property enhancements, bonding and corrosion for aerospace and transport, protein adhesion and toxicity for body implants, polymer surface changes for construction work, drug delivery etc. In these fields of technology the functionality of surfaces, thin films and interfaces plays an important role and substantially depends on chemical composition. In recent years, with the growing awareness of the importance of nanotechnology both surface and nanoanalysis have featured even more widely. The applications mentioned above are integral to the development of advanced technologies in Europe's industry. In such sectors, the analysis and control of the amount of substance at surfaces by quantitative surface chemical analysis is vital not only for product development but also for trade and the protection of intellectual property, for research and development, and for quality control.

The composition of solid material surfaces and interfaces, at the atomic level, is usually very different from that of the bulk material and is often key to the way that the material behaves in its service environment in industrial applications. For example the bonding, wettability, cell adhesion and reactivity of components are all radically affected by this composition which may only represent one part in  $10^9$  of the atoms present at the surface. Consequently the goal of surface chemical analysis is to deliver quantitative elemental, chemical state and functional group information from the surface of materials.

However, prior to the start of this project, the uncertainty of measurement related to the methods of surface chemical analysis most often used in industrial environments was not accurately known and measurements from different testing laboratories were not comparable. This meant that the analytical results of such methods could only be interpreted in a qualitative, subjective manner and limited their value and comparability. There was an urgent need to determine the uncertainty of methods of surface chemical analysis and to develop methods with reduced uncertainty enabling comparability of analytical results. In some methods of surface chemical analysis, theoretical predictions could be used to calculate quantities, however these were not verified. Furthermore, only a few samples of known surface and in-depth composition were available for validations prior to the start of this project. The use of reference materials is the most convenient way to specify or calibrate instrumentation for surface chemical analysis and to

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establish traceability to the SI and enable comparability of analytical data. Therefore certified reference materials (CRMs) with certified surface chemistries, composition and morphology i.e. defined thickness and lateral structure are needed for valid and comparable surface chemical analysis at industrial end users.

A recent trend related to nanotechnology activities in Europe is that industrial materials show more and more intended (and even non-intended) structures at scales down to the low nanometre range and chemical imaging of surfaces has become a new challenge for the development of instrumentation. This increasing activity in industrial analysis suffered from a lack of metrologically underpinned methodology and appropriate reference materials for a traceable specification of resolution power and calibration for such instruments.

There are specific needs for non-destructive methods of surface chemical analysis for fast in-line quality control in industry. One example is the identification of unwanted organic contamination at the surface of high end products used in space technology or as medical implants (even the food industry requests such analysis tools). Furthermore, new metrologically-underpinned techniques of surface chemical analysis for real time and in-situ measurements of working catalysts are needed by European industry for the development of more efficient, selective and cost-effective catalysts. Mass-market examples here are the oil and gas industry and car makers developing new catalytic exhaust cleaning systems. However, these new techniques of surface chemical analysis needed development as the metrology related to their measurements was poor prior to the start of this project.

### Scientific and technical objectives

This project was focused on the provision of reference materials for instrument calibrations and metrological methods to improve the capability of methods (EPMA, XPS, AES, SIMS, XRR) widely used in industry for quantitative surface chemical analysis. It also addressed metrology issues of industrial in-line quality control and real time in-situ measurement of catalysts all of major interest commercially and under research.

The objectives of the project were:

- The provision of new CRM with known and stable surface chemistries as well as with defined thickness and lateral structure for instrument development and calibration, and the verification of industry-relevant surface chemical measurements.
- The provision of new, fast, non-destructive methods of quantitative surface chemical analysis for industrial in-line quality control. In particular, this will include the development of advanced techniques for real time, in-situ measurement of catalyst structure and activity on a localised scale to underpin the development of more efficient, selective and cost-effective catalysts.
- The provision of metrological methods including the development of new CRM to improve the capability and traceability of technologies widely used in industry for surface analysis such as electron and fluorescence spectroscopy, X-ray reflectometry, electron probe microanalysis or ion mass spectrometry.

### Results

*Objective 1: The provision of new CRM with known and stable surface chemistries as well as with defined thickness and lateral structure for instrument development and calibration, and the verification of industry-relevant surface chemical measurements*

*Objective 3: The provision of metrological methods including the development of new CRM to improve the capability and traceability of technologies widely used in industry for surface analysis such as electron and fluorescence spectroscopy, X-ray reflectometry, electron probe microanalysis or ion mass spectrometry.*

Objectives 1 and 3 were achieved via (i) the development of certified reference materials (CRMs) for instrument calibration and (ii) metrological methods to improve the capability of a wide range of instruments and analytical methods used in a number of important technology driven industries.

#### *CRMs for Electron Probe Micro Analysis (EPMA) by Energy Dispersive X-Ray Spectroscopy (EDS)*

Quantitative EPMA by EDS analysis is highly important for industry. ISO/TC 202 "Microbeam analysis" estimated there are 35,000 installations worldwide, 2,500 instruments sold per year and 40 millions of

samples analysed annually. The project delivered a new universal reference material for quantitative analysis by EPMA using EDS enabling a chain of traceability by absolute calibration of detector efficiency. An innovative and efficient Silicon wafer based technology to provide a film with the elements C, Al, Mn, Cu, Zr at optimized composition has been developed as a certified reference material and its energy dispersive (ED) spectrum certified<sup>3,8</sup>. The CRM is available to end-users through BAM's webshop (<https://www.webshop.bam.de>) and can be used to calibrate ED spectrometers to ISO 15632:2012 with only one measurement<sup>8</sup>. With the latest detector generation quantitative EDS analysis of low Z elements (i.e. low atomic number) is enabled and strongly requested e.g. by the ceramics industry. Therefore a new prototype CRM for quantitative EPMA analysis of light elements was developed with B, C, N and O as constituents in a film which helps to reduce the uncertainty of quantitative analysis by 50% (relative).

#### *2D test structures for the determination of the field of view (FOV) of surface analytical instruments*

The ability to unequivocally identify and control the real FOV of an instrument at specific settings is central for quantitative surface chemical analysis of structured samples because signal contributions from outside the nominal FOV would falsify results. For this application new prototype 2D test structures prepared as inlays of Al in a Cr surroundings with structure sizes ranging from 10 µm down to 50 nm have been developed for XPS, AES and SIMS instruments. For optimal performance of the "Al-in-Cr" test structures height mismatches at the boundaries between the two materials have to be minimized. In the project such step heights were reduced down to only 1 nm by development of an innovative technology (patent pending) by PTB.

#### *CRMs for chemical in-depth analysis of layered organic materials*

Driven by recent developments in molecular electronics, energy harvesting by organic solar cells and production of devices for medical diagnostics there is a need for specific certified organic reference materials suitable for the calibration of methods for quantitative surface chemical in-depth analysis of layered organic materials and thin films. Two reference materials have been developed by the project prepared as layer stacks of different organic molecules (Irganox family<sup>2</sup>) with different mixtures and layer thicknesses. They can be used to optimise and validate quantitative chemical depth-profiling using argon cluster ion beams for in-depth sputtering and SIMS<sup>7,11</sup> or XPS instruments for analysis with uncertainties below 10 %. Results were validated by traceable XRR at partner PTB<sup>12</sup>. The new CRMs have been commercialized by project partner NPL.

#### *New diagnostic method based on valid liposome labelling of tissue and imaging ToF-SIMS analysis*

Direct and laterally resolved determination of molecular entities on tissue is an important task in surface analysis for drug testing or finding biomarkers related to a specific disease, but pose a real challenge for existing analytical methods. As a workaround the project developed functional liposomes to be used for specific binding (labelling) of molecular entities at biological surfaces<sup>9</sup> to enable identification and analysis. The project focused on amyloid-β peptide deposits - the major component of senile plaques in Alzheimer's disease brain tissues - which were imaged by time-of flight (ToF) SIMS. Analytical procedures for the selective labeling of amyloid-β peptide with liposomes were developed and demonstrated to be capable of quantitative analysis down to the single liposome regime (single analyte binding events) simultaneously with several endogenous molecules that cannot be detected using conventional immune-histochemistry imaging<sup>10</sup>.

#### *Traceable fluorescence measurements for biodiagnostic device platforms*

Functional silane-modified glass slides are popular platforms in the biodiagnostic device industry as they are chemically and mechanically robust, comparatively cheap and versatile in adaption and handling. Their quality substantially defines the performance of such devices (because the efficiency of immobilization of probe biomolecules depends thereon). Direct determination of amines is important but practically impossible and the solution is to label them. Therefore the project developed a method for a traceable quantification of the number of surface aminosilane molecules on glass using a new dual-mode boron-dipyrromethene-type fluorescence label which allows parallel quantification by fluorescence and XPS<sup>1</sup>. Fluorescence spectroscopy is the standard qualitative method used in industry for quality management but the lack of test samples and methodology was making it difficult to manage quantitatively. However calibration is now

possible with the new dual mode label and quantitative XPS. Quantitative fluorescence measurements of surface amine groups on industrial glass slides are made traceable to the SI via XPS and synchrotron-based reference-free total reflection (TXRF) in course of the project<sup>15</sup>. For XPS and TXRF, the limits of detection (LOD) were determined to be 0.2 aminosilane molecules per nm<sup>2</sup>.

*Objective 2: Industrial in-line quality control of technologically relevant surfaces and real time in-situ measurement of catalysts all of major interest commercially and under research.*

The project developed the metrology for new methods of surface chemical analysis by optical, mass spectroscopic and scanning probe techniques requested by important technology driven industries.

*New methods of quantitative surface chemical analysis for industrial in-line quality control*

The project developed a validated FTIR analysis methodology for fast non-destructive quantitative in-line monitoring of ambient organic surface contaminants in industrial clean rooms and to test the cleanliness of optical components and other hardware for aerospace applications. Those contaminants would have severe impact on the functionality of products. Method development was accompanied by the preparation of well-defined organic reference films for the calibration of IR and Raman equipment which is commonly used in industry<sup>4</sup>. Industrial stakeholders supported project partners INRIM and METAS in this work by providing samples from real production lines. Further to this, a quantitative surface enhanced Raman scattering method for fast detection of melamine, a toxic molecule frequently found in milk and dairy products, has been developed and validated by INRIM<sup>6</sup>. Food industry and trade needs such fast optical methods for regular quality control in order to avoid health hazards related to food mass products.

*Metrology for ambient mass spectrometry of surface contaminants on medical device surfaces*

The project has also developed the metrology for ambient mass spectrometry based on desorption electrospray ionisation (DESI)<sup>16</sup> and plasma assisted desorption ionisation (PADI). The latter method has been shown to enable quick analysis of small contaminant molecules on industrially relevant surfaces of medical devices. Contaminants such as polymers, additives and lubricants were successfully detected by a validated PADI mass spectrometry method. The lack of reference samples for new ambient mass spectrometric techniques was an important issue and to address this NPL developed reference sample candidates. A Rhodamine-B film reference sample turned out to be very useful.

*New methods for in-situ measurement of catalyst structure and activity*

There are new cutting-edge measurement techniques for real-time, geometrically localised characterisation of heterogeneous catalyst properties and activity such as scanning probe microscopy (SPM), atomic force microscopy and indirect nanoplasmonic sensing on the horizon and requested, e.g., by the chemical (including oil and gas) industry or the car makers (exhaust gas treatment) developing new and more efficient catalysts. The relationship between catalyst structure and activity is commonly poorly understood due to measurement barriers that preclude the determination of local surface behaviour under relevant reaction conditions. The project aimed for the development of new metrologically underpinned methods for different applications. In each case the goal was to address the major challenges associated with the widespread uptake of these techniques and ultimately present case studies demonstrating their application to characterizing relevant nanostructured surfaces. New SPM based measurement approaches were developed for simultaneous imaging of surface topography of a working catalyst whilst mapping its electroactivity in an electrolyte solution with high spatial resolution<sup>17</sup> and for spatially resolving (~20 nm) chemical mapping of chemical reactions on the catalyst's surface beyond the diffraction limit by using a near-field spectroscopic approach<sup>18</sup>. Furthermore the metrology has been developed for a simple but innovative optical method based on nanoplasmonic sensors for in situ studies of sintering phenomena of catalytically active platinum nanoparticles on SiO<sub>2</sub> supports in atmospheres both highly relevant for mass market industrial applications<sup>5</sup>. Sintering is the most important poisoning mechanism of catalysts with extremely high economic impact.

## Actual and potential impact

*Dissemination:*

- The project generated 18 media articles in highly ranked peer reviewed scientific journals such as ACS Analytical Chemistry and its European counterpart, Analytical and Bioanalytical Chemistry.



- The project organized a special session on Metrology in Surface Chemical Analysis as part of the scientific program of the ECASIA13 conference which traditionally attracts many stakeholders from European industry.
- BAM hosted a training course on traceable EDS analysis for industrial stakeholders and a workshop on surface chemical analysis of organic surfaces with participation of external stakeholders.
- Partners BAM, NPL, SP and INRIM regularly attended meetings of the Surface Analyses Working Group at the International Meter Convention (2012-2014) and introduced results of the project.

#### Standards:

- During the project the partners participated in regular VAMAS TWA 2, ISO TC 201, 202 and 229 meetings (2012-2014) and introduced results of the project at these meetings.
- Partner BAM and collaborator Bruker Nano will contribute new knowledge on CRM based calibration and function control of EPMA using ED spectrometers to the upcoming regular update of ISO 15632:2012 *Microbeam analysis -- Selected instrumental performance parameters for the specification and checking of energy-dispersive X-ray spectrometers for use in electron probe microanalysis* under ISO TC 202.

To launch a formal standardization procedure such as a New Work Item Proposal that will lead to a new or improved standard, ISO technical committees issue Resolutions which specific technical requests. In addition they often formally request pre-normative technical work, in this area via the VAMAS infrastructure. Results of the project presented to TC 201 subcommittees have already led to such resolutions and related pre-normative inter-laboratory comparisons and will, ultimately, result in new ISO standards at a later date:

- ISO TC 201 SC7 “Electron spectroscopies” requested work on an industrial standard for identification and control of the field of view (FOV) of instruments using new 2D structured references developed by the project. ISO TC 201 SC7 further requested an inter-laboratory comparison to be launched under VAMAS TWA2 before drafting an ISO New Work Item Proposal.
- ISO TC 201 SC6 “Secondary ion mass spectrometry” requested work on an industrial standard for the use of static SIMS for organic depth profiling of mixed materials using metrologically underpinned methods and CRMs developed by the project. ISO TC 201 SC6 further requested\* an inter-laboratory comparison to be launched under VAMAS TWA2 before drafting an ISO New Work Item Proposal.
- ISO TC 201 SC6 “Secondary ion mass spectrometry” requested work on an industrial standard on the use of ambient mass spectrometry using new metrologically underpinned methods and NPL’s Rhodamine B reference sample. ISO TC 201 SC6 further requested an inter-laboratory comparison on *Desorption electrospray ionisation: intensity repeatability and constancy* to be launched under VAMAS TWA2 before drafting an ISO New Work Item Proposal.

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#### Industry Impact:

- The CRMs, other reference materials and metrological procedures developed by the project were tested by interested European industrial partners and stakeholders including instrument manufacturers, calibration and testing laboratories and instrumentation users in a range of sectors (ION-TOF, SPECS, KRATOS, Focus, Scienion, Bruker Nano, TASCAN, Omicron NanoTechnology, Mettler-Toledo, Thermo Fisher Scientific, Thales Alenia Space, Insplorion, Ionoptika, 3M Germany). Companies delivered feedback on usability and appropriateness. The reference materials also received global attention from ULVAC-PHI and Panasonic (Japan).
- So far 15 of the CRM for ED spectrometer calibration have been sold to European companies, testing laboratories and universities (and one university from Canada).
- Partners PTB and BAM intend to commercialise the CRMs for the determination of field of view (FOV) of instruments. Stakeholders such as instrument makers and testing laboratories will use these materials for the specification and calibration of instruments for quantitative surface chemical analysis of structured samples, e.g., in the microelectronics, sensors and display industries.
- Layered organic CRMs delivered by partner NPL have received much interest from European and global industry for R&D and quality management of new organic displays, electronic devices and solar cells. In response to this NPL has started commercialisation activity.

- Methods of quantitative surface chemical analysis for industrial in-line quality control were developed and validated in collaboration with industrial stakeholders and accompanied by the preparation of organic reference films for the calibration of IR and Raman supporting the implementation of the standard ECSS-q70-05a (European Cooperation for Space Standardization) in the industry.
- The technology developed by the project to study nanoparticle sintering by INPS sensor chips in industrial applications is being commercialised by Insplorion AB. Insplorion expects substantial interest from the oil and gas industries.
- The new optical method for detection of melamine contamination in milk matrix has been implemented in food control covering the European legal limits published by Codex Alimentarius Commission.

*Policy:*

- Almost all European manufacturers of surface analytical instrumentation and private test laboratories offering services in surface analysis are Small and Medium Enterprises (SME) that cannot afford the financial and metrological infrastructure required for the development of specific CRMs, establishment of chains of traceability and pre-standardisation work. Therefore, by delivering reference materials and traceable methods, the project will directly impact European SMEs, who themselves deliver services to large European industries.
- Dissemination of the traceability concept to field laboratories through the metrology infrastructure put in place is a major outcome of the project. This will be achieved through certified reference materials enabling comparable measurement results delivered by test laboratories. The development of references for surface chemical analysis will contribute to the harmonization of measurement and documentary standards across borders, which is essential for confidence in international quality assurance, needed to facilitate global trade.

The uptake of project outputs will lead to environmental, financial and social impacts:

The environmental impact of this project comes from the development of a consistent metrological base for quantitative surface chemical analysis which supports innovation and will, on this route, promote the development of new sustainable industrial processes cleaner and more energy and material efficient thus avoiding environmental loads. One important example is the new analytical in situ methodology to study deactivation of supported metal catalysts developed by the project. These catalysts are used e.g. to convert car exhaust gases. So the project will have longer term impact on the reduction of air pollution by supporting the development of more stable and efficient exhaust catalysts. This will indirectly underpin Directive 1998/69/EC relating to measures to be taken against air pollution by emissions from motor vehicles.

The project's results will also enable cost reductions in industrial testing as the need for costly internal validations of specific analytical methods (for example for accreditation under ISO 17025) should no longer exist because appropriate CRMs and written international standards are available now or in the near future. Furthermore, this project already delivered and will deliver reference materials and traceable methods having a direct impact on the future market strength of European SMEs, specifically the European instrument makers in the field of surface chemical analysis but also the testing laboratories who deliver services to the large European companies and the public. The shares of European instrument makers on the world market had been 45 % for AES, 60 % for XPS and 60 % for SIMS instruments with total sales of US\$195M in 2003. The output of the project will strengthen their competitiveness vs. players located in Asia and Northern America by added values of products as e.g. better daily performance control based on the project's CRMs for calibrations supported by standardisation initiated through project partners. Sales numbers and turnover at European instrument makers will be at least stabilised, most probably increased.

### List of publications

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6. M. Giovannozzi, F. Rolle, M. Sega, M.C. Abete, D. Marchis, A. M. Rossi, *Rapid and sensitive detection of melamine in milk with gold nanoparticles by surface enhanced Raman scattering*, Food Chemistry **2014**, 159, 250-256, DOI: 10.1016/j.foodchem.2014.03.013.
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10. A. Gunnarsson, S. Solé Domènech, B. Johansson, V. Vukojevic, L. Terenius, A. Codita, B. Winblad, M. Schalling, F. Höök, P. Sjövall, *Simultaneous Mapping of Amyloid- $\beta$  and Lipids in Brain Tissue using Antibody-coupled Liposomes and Time-of-Flight Secondary Ion Mass Spectrometry*, The Journal of the American Chemical Society **2014**, 136, 9973-9981. DOI: 10.1021/ja5019145.
11. M. Holzweber, A. G. Shard, H. Jungnickel, A. Luch and W. E. S. Unger, *Dual beam organic depth profiling using large argon cluster ion beams*, Surface and Interface Analysis, ECASIA special issue **2014**, 46, 936-939. DOI: 10.1002/sia.5429
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JRP-Coordinator: Dr Wolfgang Unger, BAM                      Tel: + 49 30 8104 1823                      E-mail: wolfgang.unger@bam.de JRP website address: <a href="http://www.emrp-surfchem.bam.de">http://www.emrp-surfchem.bam.de</a>	
JRP-Partners: JRP-Partner 1 METAS, Switzerland, JRP-Partner 2 INRIM, Italy JRP-Partner 3 NPL, UK JRP-Partner 4 PTB, Germany JRP-Partner 5 SP, Sweden JRP-Partner 6 Chalmers, Sweden JRP-Partner 7 ION-TOF, Germany	JRP-Partner 8 Kratos, United Kingdom JRP-Partner 9 Scienion, Germany JRP-Partner 10 SPECS, Germany JRP-Partner 11 FOCUS, Germany
REG-Researcher (associated Home Organisation):	Elin Larsson, Francesco Mazzotta Chalmers, Sweden
ESRMG-Researcher (associated Home Organisation):	Tobias Fischer BAM, Germany

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