National Physical Laboratory

NPL REPORT
DQL-AS 025

Proposals for the 2006-2009 Valid Analytical Measurement (VAM) - Physical Programme

Version for Public Comment

March 2006

dti
FUNDED BY THE DTI

dti

The DTI drives our ambition of 'prosperity for all' by working to create the best environment for business success in the UK. We help people and companies become more productive by promoting enterprise, innovation and creativity.

We champion UK business at home and abroad. We invest heavily in world-class science and technology. We protect the rights of working people and consumers. And we stand up for fair and open markets in the UK, Europe and the world.

The National Physical Laboratory is operated on behalf of the DTI by NPL Management Limited, a wholly owned subsidiary of Serco Group plc

NPL REPORT DQL-AS 025

Proposals for the 2006-2009 Valid Analytical Measurement (VAM) - Physical Programme

Version for Public Comment

March 2006

ISSN 1744-0602

National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

We gratefully acknowledge the financial support of the UK Department of Trade and Industry (National Measurement System Policy Unit)

Approved on behalf of the Managing Director, NPL
by Dr Stuart Windsor, Business Leader, Analytical Science Team

EXECUTIVE SUMMARY

This document is a summary of the proposals for the 2006-2009 VAM–Physical Programme. The proposals have been developed following an extensive consultation exercise to identify and validate the key drivers and requirements for the work.

The Programme is presented in four Technical Themes, each containing a number of proposed projects, which are described in Sections 4 to 7 of this document. Comments received in response to this document will be considered prior to the prioritisation of the Programme through a decision conference in May 2006.

CONTENTS

Proposals for the 2006-2009 VAM-Physical Programme

1. Introduction to the VAM & VAM-Physical Programmes

1.1. Overview

The Valid Analytical Measurement (VAM) Programme supports UK industry to ensure that analytical measurements are carried out competently and accurately. These measurements have a major impact on important areas of UK life such as innovation and competitiveness, air quality (emissions and pollutant monitoring), pharmaceuticals, vehicle exhaust testing, breath alcohol testing, medical devices and personal care.

Analytical measurements are vital to the UK economy as they are required to ensure product quality, to monitor impact on the environment and to satisfy legal requirements. It is estimated that the UK spends approximately £8 billion on analytical measurements every year. The VAM Programme underpins the reliability and integrity of these analytical measurements by supporting the provision of capability of the reference methods and standards required for traceable, quantitative measurement. It enables the UK to demonstrate the comparability of analytical measurements with those of its trading partners.

The Programme has two main technical themes: Physical measurement (‘The VAM-Physical Programme) and Chemical measurement (‘The VAM-Chemical Programme’). The VAM-Chemical Programme (operated chiefly by LGC) consists of an integrated set of projects designed to develop the methods, capabilities and facilities to provide reference materials and standards to enable laboratories to make measurements that are traceable to internationally recognised standards.

The VAM-Physical Programme supports accurate physical analytical measurements. These are fundamental in a wide range of industrial and legislative processes and are the crucial measurement issues in scientific fields such as gas, particulate, trace chemical, environmental, surface chemical, electroanalytical, single molecule and nanotechnological analysis. They are particularly important in a number of emerging scientific areas where analytical measurements currently lack sufficient traceability (e.g. measurement of the physical characteristics of nanoparticles, and the characterisation of novel, miniaturised sensing devices). A recent report on nanotechnologies by the Royal Society and Royal Academy of Engineering [1] highlighted the need for standardisation in nanoscience by recommending that the Department of Trade and Industry (DTI):

“...supports the standardisation of measurement at the nanometre scale required by regulators and for quality control in industry through the adequate funding of initiatives under its National Measurement System Programme…”

A Government research report on the potential risks posed by engineered nanoparticles [2] specified a key research objective as *“To identify the most suitable metrics and associated methods for the measurement and characterisation of nanoparticles”,* and highlighted NPL as the key laboratory developing such methods and techniques.

The Programme also supports measurement requirements for a number of issues of increasing public concern such as greenhouse gases, and detection and identification of pollutants in ambient and indoor air. Air quality has a large effect on quality of life and health – in the UK, it is estimated that airborne particles contribute to the premature death of 32,000 people per year.

A recent independent review of the long-term measurement needs across the National Measurement System (NMS) Programme portfolio [3] revealed that the VAM-Physical Programme had the largest number of 'measurement needs' per unit cost (see Section 2.3.5 for more details). In addition, the most recently published Government review of the NMS [4] revealed the VAM-Physical Programme to have the highest innovation content of any Programme in the NMS Portfolio. The Programme is therefore the ideal vehicle to support the Government's stated aim [5] of increasing UK innovation, resulting in 'prosperity for all'.

This document describes the proposals for the 2006-2009 VAM-Physical Programme, which aims to advance the work carried out in previous Programmes. The National Measurement System Directorate (NMSD) of the DTI have advised that the Programme should have an increased focus on research-based projects, and that the benefits of the Programme should continue to be disseminated through a series of successful knowledge transfer routes.

1.2 Aim and objectives of the VAM-Physical Programme

The aim of the Programme is to provide the technical and administrative infrastructure required to ensure valid and traceable analytical measurements in the UK and to establish the equivalence of analytical measurements with our trading partners.

The objectives of the Programme are:

- To enhance wealth creation, prosperity, innovation and industrial competitiveness by improving analytical measurements of input, process and output parameters and to provide leadership amongst the UK analytical science community in the application of leading-edge science and technology.

- To underpin the requirements for stable and accurate analytical measurements to support fair regulation and to support the process of developing national and international regulations on a sound and cost-effective measurement basis.

- To support measurements in the healthcare and environmental sectors intended to improve the quality of life throughout the UK and to contribute towards the monitoring and control of climate change.

- To carry out a range of activities to transfer the outputs of the programme in terms of knowledge and technology to UK users, including industry, SMEs, regulators and trade bodies.

- To support trade and the comparability of UK measurements by participation in international comparisons and the development of an infrastructure for the global acceptance of measurement results.

2. FORMULATION OF THE 2006-2009 VAM-PHYSICAL PROGRAMME

2.1. Overview of the formulation process

The formulation process is formed of the following key phases:

- *Orientation* (March – June 2005). Defining the scope and direction of the new Programme through processes including a 'trend spotting forward look' review of measurement requirements and an Orientation Meeting (June 2005). Orientation aims to determine the direction and strategy of the Programme, and ensure that all formulation contractors and NMSD have a common vision of the direction of the new Programme.
- *Consultation* (June 2005 – May 2006) and *Programme development* (January – August 2006). Engaging with UK industry and other key beneficiaries to obtain and record the detailed technical needs of the user community. Input is obtained by mechanisms such as focus groups, questionnaires, interviews and discussion forums. The outline proposals for the new Programme were presented to the VAM Working Group at their Annual Review meeting on 6-7 December 2005 and steer from the working group was used to focus the scope of the Programme. A summary of the proposals was also made available for comment through the VAM-Physical Formulation website. The projects that passed this initial review stage have been developed further and are presented in this document.
- *Prioritisation* (May - June 2006). Following the public consultation exercise, the proposed projects in the new Programme undergo Prioritisation (May 2006). The meeting uses decision conferencing to score all of the proposed projects against pre-set criteria - the output from the meeting is a prioritised list of projects.
- *Programme approval* (June – September 2006). Development of the Programme in its final form for Ministerial approval. If approved, the new VAM Programme will commence at the beginning of October 2006.

This document is therefore published in the latter part of the Consultation phase of formulation. All comments received in response to the proposals detailed here will be considered and acted upon prior to the prioritisation of the Programme.

2.2 Orientation

2.2.1 Foresight documents

A fundamental part of the Orientation stage of formulation is to undertake a 'trend spotting forward look' review of available literature in order to identify trends in technology, market requirements and measurements needs. The study undertaken as part of VAM-Physical formulation studied over 90 highly relevant documents forming a wide range of sources including:

- DTI and other government departments
- Office for science & technology Foresight reports
- Research Councils
- Royal Society of Chemistry

- Institute of Physics
- Institute of Materials, Minerals and Mining
- Royal Society / Royal Academy of Engineering
- Roadmaps (e.g. from the Micro and Nanotechnology Network and the NanoRoadMap project)
- European Commission (strategy documents and directives)
- Nanotechnology reports (from, for example, The Institute of Nanotechnology, The National Science Foundation, Swiss Re and Cientifica)
- UK and European air quality expert groups
- The national and scientific press
- Health and Safety Executive / Environment Agency
- Independent reports commissioned as part of the 2003-6 VAM-Physical Programme

In order to give an indication of the vast number of relevant documents applicable to each scientific theme, Figure 1 shows some of the key foresight and policy documents relevant to gas analysis.

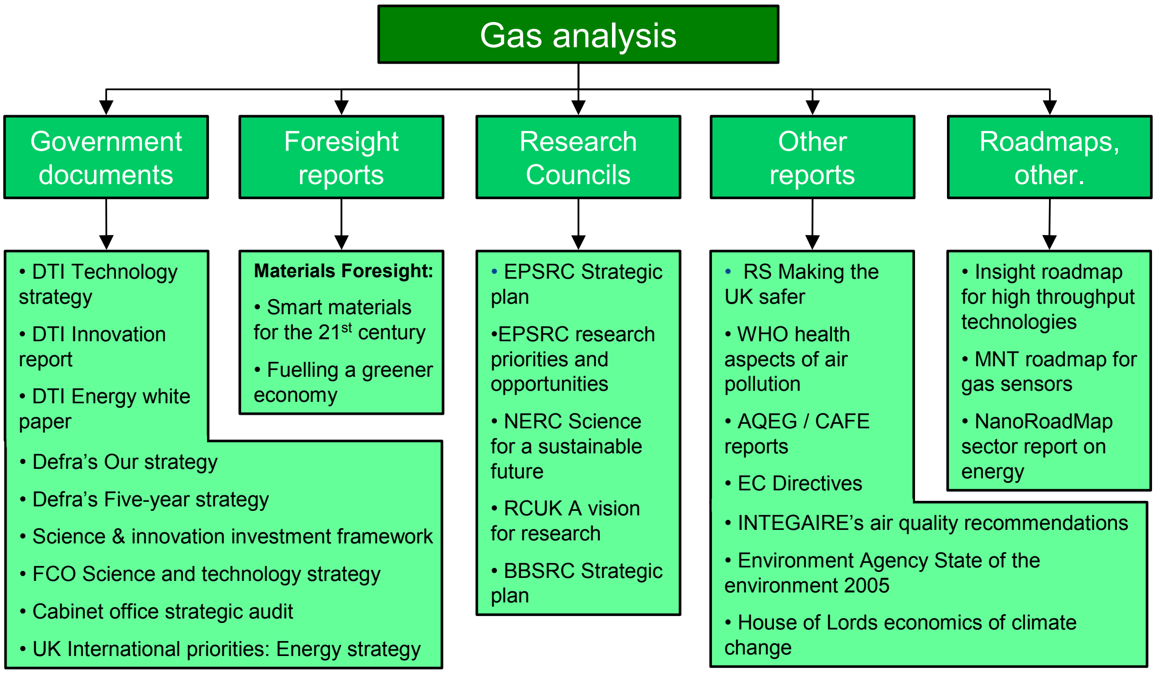

Figure 1: Figure showing the key foresight and policy documents applicable to the field of gas analysis

2.2.2 Key drivers for the VAM-Physical Programme

The review of independently produced reports has identified four high-level drivers for physical analytical measurements. These drivers, which are closely linked to the proposed objectives of the VAM-Physical Programme, are detailed below:

(a) Industrial competitiveness and trade

Industrial production and innovation depends on the capability to measure, characterise and understand new processes and novel devices, as well as to control efficiency in manufacture. This is only possible when access is available to valid measurement methods and a comparable basis for measurements. Innovative new technologies pose requirements that are at the cutting edge of measurement capability. The optimisation of process performance depends on the ability to monitor control parameters on a stable basis. Import and export of manufactured goods depends on the acceptance of a uniform basis for measurements.

(b) Regulation

Regulations developed at both European and UK level ensure that emissions from industry are controlled within agreed limits. Such regulations are most effective when they define performance levels that are both achievable by industry with available technology and operate at levels that provide adequate protection for society. In cases where protection is required that goes beyond that which can be achieved with available technology, regulation can act to stimulate innovation in the development of improved technology. The imposition of such regulation on a basis that is fair to regulated industry and society requires an infrastructure for traceable measurements at a level of uncertainty appropriate for the application.

(c) International representation of the UK's interests

The effective achievement of the objectives of the VAM Programme requires the UK's interests to be represented and defended on the various international bodies where international documentary standards are developed (e.g. ISO and CEN) and where the validity of the UK's national measurement standards must be demonstrated (e.g. CCQM). Activities of this type have become more intense as a result of the drive to provide a single set of standards across the EU and to provide a measurement infrastructure that can support global trade.

(d) Environmental protection and quality of life

The continued protection of human health and the environment depends on the capability to monitor accurately the amounts of harmful species in the environment. These measurements can be used to determine whether the most damaging species are below acceptable levels, and subsequently whether a reduction in their concentrations is required. These objectives can only be achieved if both regulators and regulated industry have access to measurement standards that are valid at the very low levels appropriate for environmental monitoring.

2.2.3 Ensuring international comparability through traceability

The VAM-Physical Programme ensures that stakeholders in the UK have direct access to traceable measurement standards for gases, particulates and surface analysis. Much of the demand for traceability of these measurements is driven by two requirements:

- The internationally-accepted standard for test and calibration laboratories (ISO 17025) requires that they make use of traceable measurement standards where available. Whilst it is not possible to provide traceable measurement standards for every analytical quantity that is measured, the range of measurement standards with the greatest applicability across sectors may be provided.

- Fair and free international trade requires that the results of measurements be accepted around the world without additional measurement. This can only be met by ensuring that measurements made in the UK are made on a basis that is transparent and recognised internationally.

These two drivers are the justification for the UK's involvement in the international measurement system based on traceability to the international system of units (SI), demonstrated through international comparisons. This is organised under the CIPM Mutual Recognition Arrangement (MRA) [6] agreed between 45 nations around the world. The MRA is based on the technical capability of individual laboratories to disseminate measurements that are traceable to the SI and is underpinned through a series of international intercomparisons [7].

2.2.4 Technology strategy priorities

Following on from the publication of its Innovation Report [5], the DTI has developed a Technology Strategy [8] which has the vision "for the UK to be seen as a global leader in innovation and a magnet of technology-intensive companies, where new technology is applied rapidly and effectively in creating wealth and addressing major societal challenges such as global warming and affordable healthcare for an ageing population".

The Technology Strategy Board has recently reported its progress [9] in each of seven key technology areas:

- Electronics and photonics
- Advanced materials
- Information and communication technologies
- Bioscience and healthcare technologies
- Sustainable consumption and production
- Emerging energy technologies
- Design engineering and advanced manufacturing

The key technology areas were used by PA Consulting to develop nine needs-based groupings for their Needs Analysis study. These groupings, listed below, were used to present the proposed Programme to the VAM Working Group in December 2005 under a series of 'Consultation Themes':

- Sustainable consumption and production
- Sustainable energy
- Transport
- The built environment
- Secure environment
- The intelligent connected world
- Healthcare

- Underpinning metrology
- Management and knowledge transfer

2.3 Consultation

2.3.1 Introduction

In order to formulate the project proposals presented in this document, the requirements of the Programme have been evaluated through a substantial consultation exercise. The following methods have been used to gain input from key stakeholders:

- A one-day gas and environmental analysis focus group held in the Midlands and attended by invited representatives of industry, trade associations, academia, Government organisations and UKAS. A large number of draft project ideas were presented to, and discussed by, the group.

- A one-day Surface and nano-Analysis Measurement Steering (SAMS) Workshop at NPL. The workshop, attended by key experts in the field of surface analysis used responses to a in-depth questionnaire to discuss and prioritise a number of key measurement issues and proposed projects.

- A one-day conference, ‘The Measurement of Engineered Nanoparticles’, held at NPL under the auspices of the Micro and Nanotechnology Measurement Club. The conference, with over 90 attendees, allowed formal and informal discussion of issues relating to the physical measurement and sampling of nanoparticulates.

- Development of a VAM-Physical formulation website to allow interested parties to input their views and download relevant documents (see Section 2.3.2).

- Feedback through three on-line questionnaires (gas and environmental analysis, surface analysis, and nanoparticulate analysis), available through the formulation website (see Section 2.3.3).

- Continuous consultation with collaborative partners in existing VAM projects, other NMS Programmes, the Measurement for Innovators Programme and contacts established through knowledge transfer activities in previous VAM Programmes.

- One-to-one discussions with key individuals through invitations to meet key staff at NPL, visits to the contacts’ organisations, and telephone and e-mail discussions.

Representatives from the following groups of stakeholders have contributed to consultation:

- Government departments
- Industrial trade associations
- Major UK companies in appropriate sectors (e.g. gas companies, motor vehicle industry)
- UK and European regulatory bodies (e.g. the Environment Agency)
- BSI
- UKAS

- UK SMEs and instrument manufacturers in appropriate scientific sectors
- Analytical laboratories
- UK Regional Development Agencies
- Experts within other Teams at NPL involved in the delivery of other NMS Programmes
- Other UK Laboratories delivering NMS Programmes and projects (LGC, NEL & AEA Technology)
- Other National Metrology Institutes
- Members of NPL clubs
- Contacts on NPL mailing lists
- Members of the Royal Society of Chemistry, Institute of Physics, and Institute of Materials, Minerals and Mining
- Serco Assurance
- EPSRC Programme Managers
- Representatives of the UK food industry
- UK electrochemistry experts

To support these consultation activities, NPL has also:

- Developed a series of roadmaps (Section 2.3.4)
- Reviewed and critiqued the results of the NMSD's Needs Analysis study (Section 2.3.5)
- Undertaken a patent mapping study (Section 2.3.6)
- Held on-going discussions with LGC staff formulation and delivering the VAM-Chemical Programme (Section 2.3.7)
- Promoted the formulation process via a number of wide-reaching mechanisms (section 2.3.8)

A brief summary of each of the consultation activities is presented below.

2.3.2 VAM-Physical formulation website

A key tool developed to assist the formulation of the VAM-Physical Programme is a formulation website hosted by NPL: www.npl.co.uk/formulation/vam (see Figure 2).

The website has played an important role in all stages of the formulation process to date. It has allowed visitors to view relevant source documents and details of the Orientation meeting, obtain background information about the VAM Programme, register for focus groups meetings, fill in on-line questionnaires and comment on the outline proposals for the Programme (or any other aspects of formulation). The website will be continue to be updated throughout the formulation process.

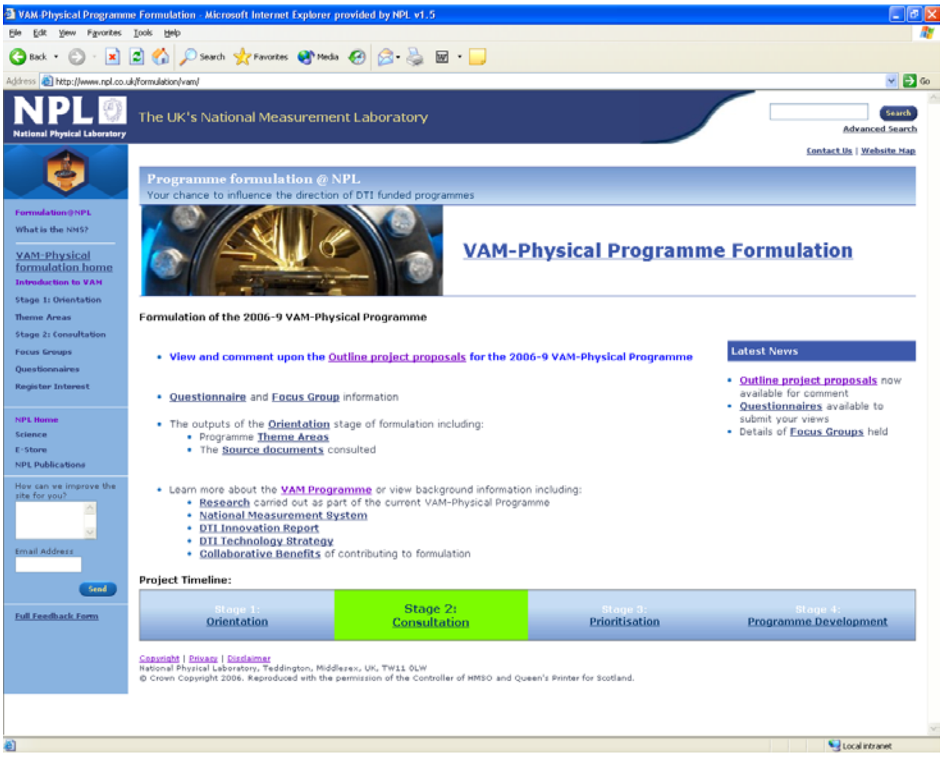

Figure 2: Screenshot of the VAM-Physical formulation website homepage (February 2006)

Between its launch (July 2005) and the end of February 2006, the website received over 9,200 hits. A breakdown of the number of hits per month is shown in Figure 3 – the large increases in people accessing the website in October and December / January can be ascribed to increased interest generated by successful promotional campaigns.

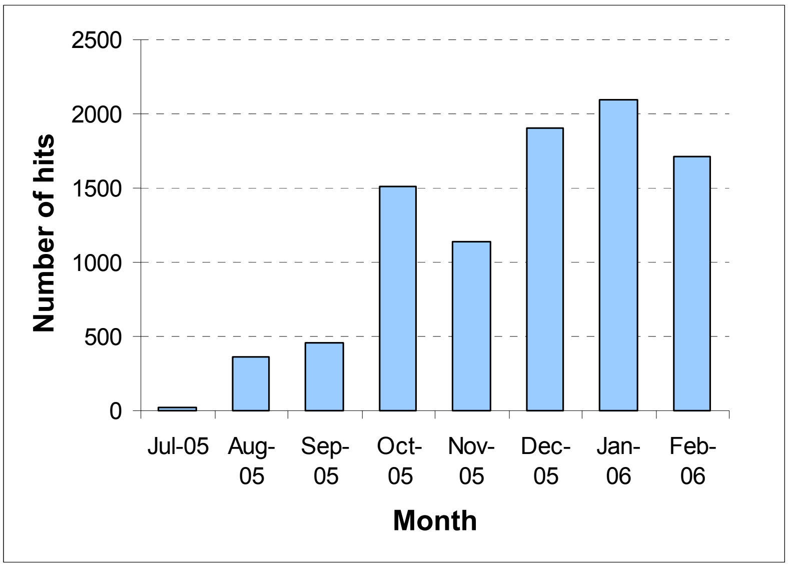

Figure 3: Bar chart showing the number of hits per month to the VAM-Physical formulation website

2.3.3 Questionnaires

To assist in gauging as many stakeholders' views as possible during the formulation process, three web-based questionnaires were produced, covering the areas of the VAM-Physical programme listed below:

- Gas & environmental analysis
- Nanoparticulates
- Surface and nano-analysis

Each questionnaire comprised a list of activities for possible inclusion in the Programme, and respondents were asked to rate each activity according to its importance to organisations in their sector. Five responses (on a scale from lowest priority to high priority) were possible, along with a further option of 'no view'.

Each response was recorded and allocated a score from 1 (lowest priority) to 5 (highest priority) and a 'percentage of maximum score' calculated (after excluding the responses of 'no view'). For each project, the five highest scoring technical activities and the two highest-scoring knowledge transfer (KT) activities are summarised in Table 1 below.

Gas and environmental analysis questionnaire – Top five technical activities		
1	Development of 'variable gas standard' technology, allowing multiple concentrations of a gas mixture to be delivered from a single cylinder	**80%**
2	Development of measurement techniques to monitor gaseous emissions from industrial sources	**79%**
3	In situ measurement of gaseous pollutants using portable field mass spectrometry and FTIR	**79%**
4	Development of methods to speciate particulate and vapour phase metal compounds in ambient air, particularly those potentially hazardous to human health	**79%**
5	Validation of the measurement of climate-change priority pollutants in ambient air	**77%**

Gas and environmental analysis questionnaire – Top two KT activities		
1	Represent the UK's interests on European and International Standardisation bodies	**80%**
2	Support Industry and Government through the provision of services to validate or certify gas and particulate analysis instrumentation	**79%**

Nanoparticles questionnaire – Top five technical activities		
1	Production and validation of nanoparticulate reference materials	**93%**
2	Measurement of micro and nanoparticles distributed in liquids or solids	**90%**
3	Formulation of internationally-valid standardisations for nanotechnological substances and materials	**90%**
4	Development of documentary standards for nanoparticulate measurement techniques	**84%**
5	Determination of nanoparticulate size distributions with high detection efficiencies	**80%**

Nanoparticles questionnaire – Top two KT activities		
1	Represent the UK's interests on European and International Standardisation bodies	**90%**
2	Publication of reports and best-practice guides via web-based media	**84%**

Surface and nano-analysis questionnaire – Top five technical activities		
1	Validation and quantification of AFM nanomechanical measurements	85%
2	Development of surface analysis techniques for the analysis of non-atomically flat and textured surfaces at the sub-micron scale	84%
3	Accurate measurement techniques for the analysis of compounds at surfaces and in thin layers	84%
4	Development of methods to identify the chemical structure of contamination layers on solid surfaces	82%
5	Development of techniques for the reliable analysis and quantification of complex molecules at surfaces, insulators and organic layers	82%

Surface and nano-analysis questionnaire – Top two KT activities		
1	Represent the UK's interests on European and International Standardisation bodies	90%
2	Develop and maintain the Surface and nano-analysis website	88%

Table 1: Summary of the highest-scoring questions from the three on-line questionnaires

2.3.4 Roadmaps

As part of the VAM formulation process, a series of three roadmaps have been developed, one for each Technical Theme of the new Programme. These roadmaps, covering the Themes of gas analysis, particulate and trace chemical analysis, and surface and nano-analysis, are presented as part of the introduction to each Theme (Sections 4 to 6).

2.3.5 Needs analysis

In early 2005, PA Consulting, in association with DTI NMSD, undertook a review of the long-term measurements need across the projects in the NMS portfolio. The study reviewed over 400 documents from a range of sources (DTI, Foresight, Research Councils, etc.), assigning the stated or implicit measurement needs contained within to one or more Programmes across the portfolio.

A subsequent review of the Needs Analysis study at NPL revealed that a large quantity of relevant documentation was overlooked, and a number of the measurement needs identified by the study had been assigned to too few Programmes within the portfolio. These (new and already identified) needs were allocated to all the relevant Programmes across the NMS portfolio. The results of this are presented in Figure 4, where it can clearly been seen that the VAM-Physical and VAM-Chemical Programmes address a large number of important measurement issues.

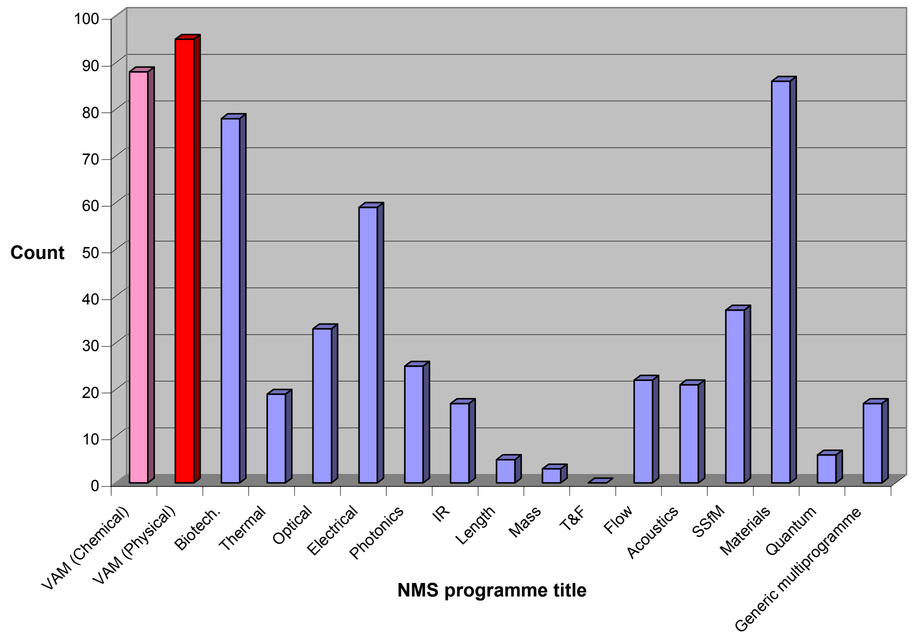

Figure 4: Results of the review of NMSD's needs analysis study. The number of measurement needs assigned to VAM-Physical and VAM-Chemical are shown by the red and pink bars respectively.

An indication the increasing pressure on the VAM-Programme to deliver results on a limited budget is shown in Figure 5. Here, the number of needs per Programme has been divided by the Programme budget; this give an indication of the number of measurement needs to be met per unit Programme cost.

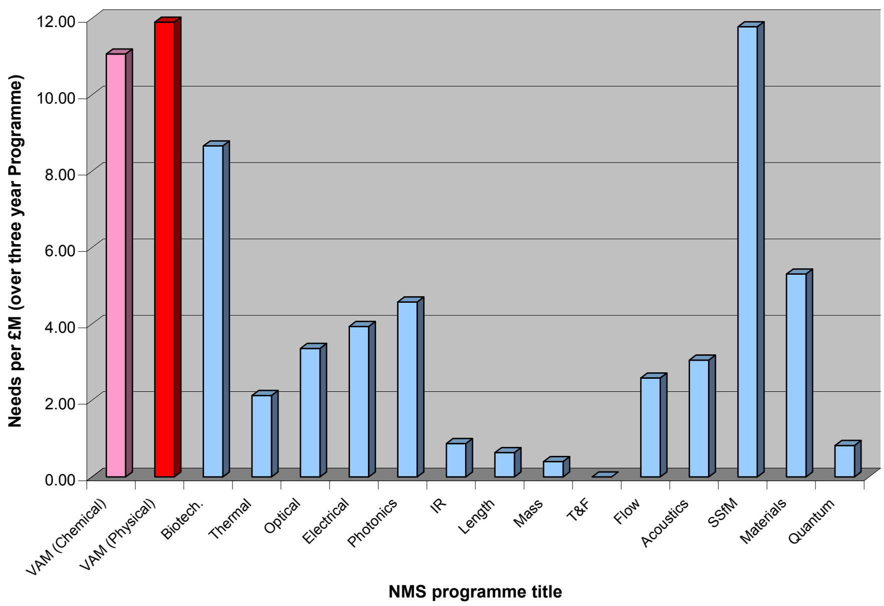

Figure 5: Calculation of the number of measurement needs per million pounds of Programme budget. (Results from the review of NMSD's Needs Analysis study; budgetary information taken from the latest three-year Programme cycle.)

2.3.6 Patent mapping

A patent mapping study has been undertaken by Scientific Generics in order to identify the areas of developing technology related to the VAM Programme, especially those areas where UK industry is particularly strong or weak. Some provisional results from the study are presented here.

Hundreds of thousands of patents (filed in the USA, with the European Patent Office, or under the Patent co-operation treaty) were examined. The study looked at the patents filed in 47 technical categories relating to both the VAM-Chemical and VAM-Physical Programmes and identified key tends, such as the number of patents per year and the international profile of these patents. The results are shown schematically in 6 and 7. In both Figures, a colour coding systems from black (lowest) to red (highest) has been applied (see the scale on each Figure), and the purple bar charts at the top and sides of the Figures represent the total number of patents recorded in each category.

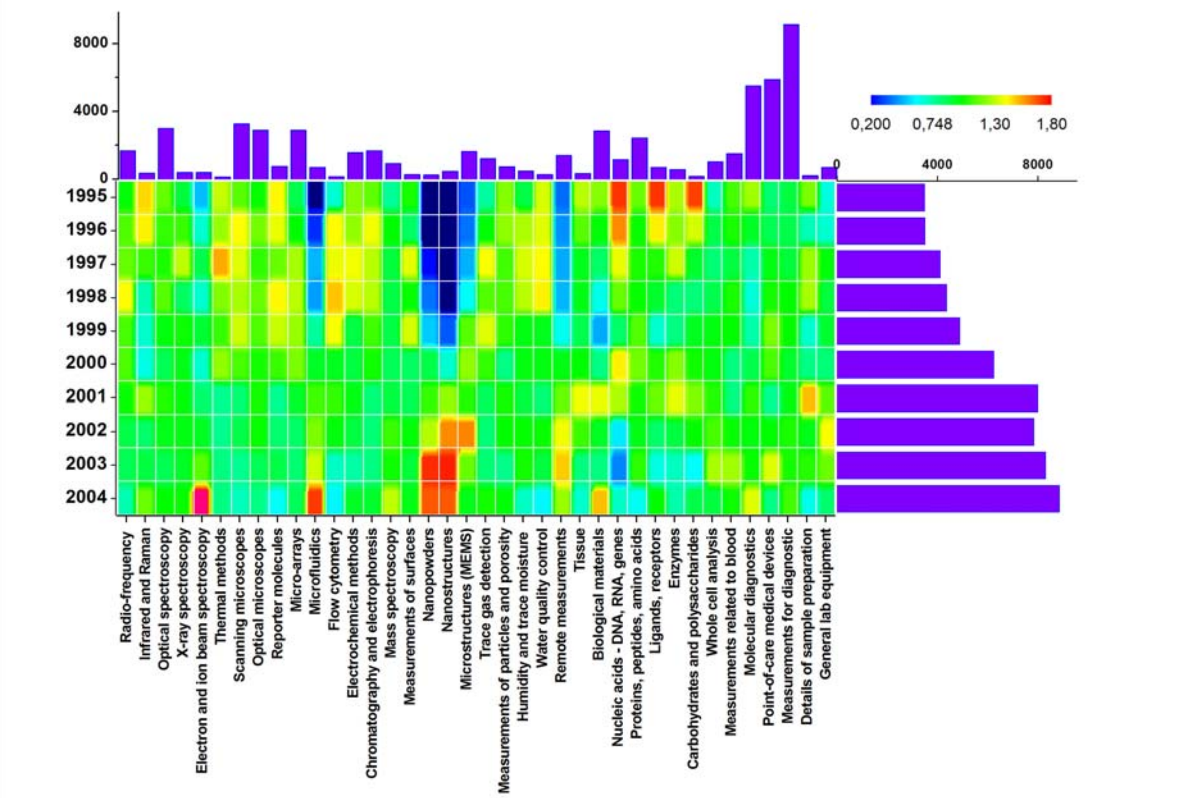

Figure 6: Results from patent mapping study – timeline (technical category v year).

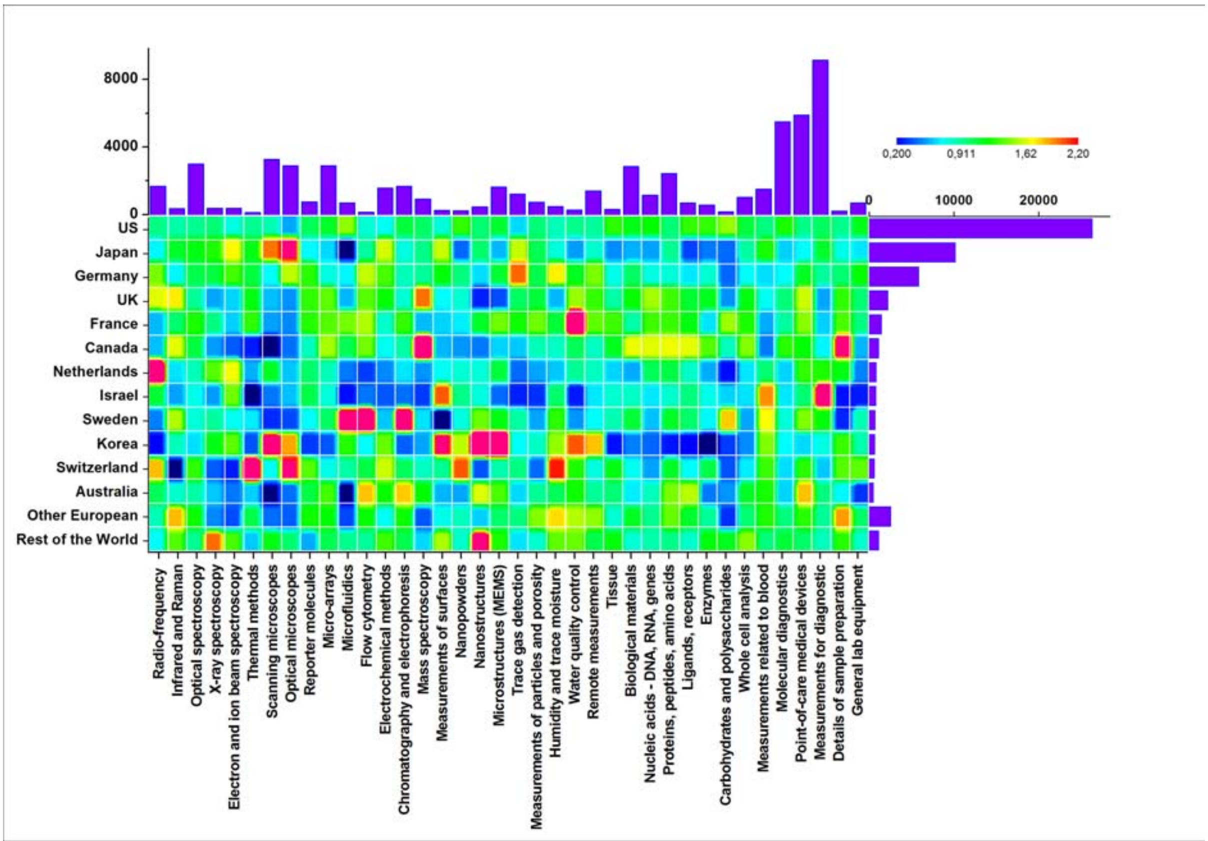

Figure 7: Results from patent mapping study – geographical (technical category v country).

The key findings of the study include:

- The technical categories of nanopowders, nanostructures, microfluidics and electron and ion beam spectroscopy have all shown strong relative growth within the last three years.
- The UK 'punches above it weight' in mass spectrometry, infra-red and Raman, but needs to develop further in categories such as nanostructures, MEMS, x-ray spectroscopy, scanning microscopes and optical microscopes.

2.3.7 VAM-Physical / VAM-Chemical collaboration

To ensure alignment with the VAM-Chemical Programme, key staff involved in the formulation of each Programme have met at least once a month since the beginning of the formulation process. These meetings have ensured that the formulation of the two halves of the Programme has proceeded on a similar basis. In addition, ideas for collaborative projects in the forthcoming Programme have been discussed at length through:

- Discussions between technical experts. A round-table discussion of key scientific staff in the field of mass spectrometry and elemental analysis identified a number of areas for possible collaboration, The most promising of these, a mass spectrometry project, has been worked up into a project proposal - see project S06 in Section 6 for full details.

- Discussions of areas for collaboration between the two parts of the Programme in the area of knowledge transfer. Ideas proposed by NPL included improvements to the VAM website; staff secondments; co-ordination of CCQM representation; formation of an impact analysis task group and organisation of a web-based staff-industry forum to explore VAM issues in selected areas.

2.3.8 Promotion of the VAM-Physical formulation process

In order the capture the input of as many stakeholders as possible, a number of methods were used to promote the formulation process to a wider audience. These promotional activities targeted key sectors, and, in the main, drew readers' attention to the formulation website.

Examples of the methods used to promote the formulation process include:

- Distribution of an electronic flyer to over 1,100 relevant UK contacts on VAM and targeted NPL mailing lists.
- Promotion through an Institute of Nanotechnology electronic newsletter circulated to over 41,000 contacts in the field of nanotechnology.
- Publication of an article in the VAM Bulletin (Autumn 2005, Issue 33, pp 25-26).
- Production of a formulation 'business card' for distribution to attendees at relevant NPL meetings.
- Inclusion of short articles in the Royal Society of Chemistry's Analytical Division newsletter (February 2006) [10] and the Institute of Physics' Thin Films and Surfaces Group newsletter (December 2005).

2.3.9 Feedback obtained from the outline Programme (December 2005 version)

An outline Programme was presented to the VAM Working Group on 6-7 December 2005, and subsequently made freely available to all interested parties through the formulation website.

The Working Group identified a number of areas that the Programme should focus on. Comments received, both from the Working Group and from feedback via the website, were used to add to the projects selected for further development. These projects are presented in the following Section.

3. PROPOSED TECHNICAL CONTENT OF THE 2006-2009 VAM-PHYSICAL PROGRAMME

3.1 Introduction

The proposed technical content of the 2006-2009 VAM-Physical Programme is presented in this section. The proposals have been developed following the extensive consultation exercise and feedback from the Working Group described in Section 2.

3.2 Programme structure

Following the recommendations of the Working Group at its 2005 Annual Review, the Programme in now presented under four Technical Themes, rather than the 'Consultation Themes' used in December 2005 (and outlined in Section 2.2.4).

The Technical Themes are:

Theme G:	**Gas analysis**
Theme P:	**Particulate and trace chemical analysis**
Theme S:	**Surface and nano-analysis**
Theme M:	**Management and knowledge transfer**

The first three of these Themes consist solely of technical projects (30 in total). The fourth Theme, 'Management and knowledge transfer' covers the proposed management and KT aspects of the 2006-2009 Programme, including formulation of the 2009-2012 Programme and the decant into the new laboratory facilities at NPL.

A full list of proposed projects is given in Table 2. An indication of how each of these projects maps to the eight Consultation Themes (derived from the DTI's Technology Strategy key technology areas) is shown in Table 3.

Theme	PROJECT		
	Number	**Title**	**Original number**
Gas analysis	**G01**	Binary and multi-component gas standards at emission levels	1.1G
	G02	Ambient concentration standards for ozone and its precursors	1.2G
	G03	Improved facilities for the analysis of ultra-pure gases for high-accuracy metrology and the special gas industry	1.3G
	G04	Valid portable mass spectrometry for field applications	†
	G05	Standards and new measurement technologies for the natural gas and fuels industries	2.1G
	G06	Gas standards and primary dynamic facilities for ambient (acid) gases	2.2G
	G07	Stable standards for monitoring greenhouse gases	2.3G
	G08	Gas standards to underpin emissions from road and air transport	3.1G
	G09	Stable cylinder standards for binary and multi-component mixtures	3.2G
	G10	Standards to underpin measurements of gaseous species in indoor air and occupational exposure	4.1G
	G11	Gas sensors and multi-gas data fusion	6.1G
	G12	High accuracy gas analysis	8.1G
	G13	Practical gas measurements by isotope dilution mass spectrometry (IDMS)	8.2G
Particulate and trace chemical analysis	**P01**	Accurate size and number measurements of airborne nanoparticles	‡
	P02	Accurate and traceable mass and chemical composition measurements of airborne particulates	3.4P
	P03	Methodologies for accurate ambient measurements of mercury speciation and particulate chemical compositions	7.1P
	P04	Fundamentals of aerosol measurement	8.3P
	P05	Standards and valid procedures to underpin measurements of pH and ionic content	1.6P
	P06	Ultra trace analytical methods (SERS)	5.1G
Surface and nano-analysis	**S01**	Quantitative analysis of surfaces with sub-micron topography – XPS, SIMS, AFM combined study	1.8S
	S02	Quantitative analysis: In-depth composition for surfaces and fibres with radii of curvature >10nm	1.9S
	S03	Quantitative analysis of major constituents in compounds and compound layered structures at surfaces	3.5S
	S04	Development of measurement infrastructure for QC, accreditation and valid measurements by static SIMS	4.3S
	S05	Micro- and nano-scale measurement of surface chemistry for surface wettability	5.4S
	S06 *	Ambient and imaging mass spectrometry	5.5S
	S07	Quantitative depth profiling and cluster sputtering of thin organic layers at surfaces	6.2S
	S08	Quantitative analysis of organic coatings and molecular layers	7.2S
	S09	Practical analysis of delicate materials	7.3S
	S10	Quantification issues in lateral resolution and lateral force in AFM	8.4S
	S11	Optimisation of operating conditions for high-resolution AFM nanomechanics measurements	8.5S
Mgmnt & KT	**M01**	Programme management and decant	○
	M02	Formulation of the next VAM-Physical Programme	9.2M
	M03	Dissemination and representation	9.3M

Table 2: Table of proposed projects for the 2006-2009 VAM-Physical Programme.

† Formed from a combination of projects previously numbered 1.4G & 5.2G
‡ Formed from a combination of projects previously numbered 1.7P & 3.3P
○ Formed from a combination of projects previously numbered 9.1M & 9.4M

* Project S06 is a proposed collaborative project between NPL and LGC. The equivalent project in the VAM-Chemical Programme is Project O3.

Theme	Project No.	Consultation Theme (from DTI Technology Strategy key technology areas)							
		Sustainable consumption & production	Sustainable energy	Transport	The built environment	Secure environment	The intelligent connected world	Healthcare	Underpinning metrology
Gas analysis	G01	●	●	●	○	○	○	○	○
	G02	●	●	○	○	●	○	●	○
	G03	●	○	○	○	○	●	○	●
	G04	●	●	○	○	●	○	○	○
	G05	○	●	●	○	○	○	○	○
	G06	●	●	○	○	○	○	●	○
	G07	●	●	○	○	○	○	○	●
	G08	○	●	●	○	○	○	○	○
	G09	●	○	●	○	●	○	○	●
	G10	○	○	○	●	○	○	●	○
	G11	○	●	○	○	●	●	○	●
	G12	●	●	○	●	○	○	○	●
	G13	○	○	○	○	○	○	○	●
Particulate and trace chemical analysis	P01	●	○	●	●	○	○	●	●
	P02	●	○	●	●	○	○	●	●
	P03	●	○	○	●	○	○	●	●
	P04	○	○	○	●	○	○	●	●
	P05	○	●	●	●	○	○	○	●
	P06	○	○	○	○	●	○	●	●
Surface and nano-analysis	S01	●	○	○	○	●	○	●	○
	S02	●	○	○	○	●	○	●	○
	S03	●	○	●	○	●	○	●	●
	S04	●	○	○	○	●	○	●	●
	S05	●	○	○	○	●	○	●	○
	S06	○	○	○	○	●	○	●	○
	S07	○	○	○	○	○	●	●	○
	S08	●	○	○	○	○	○	●	○
	S09	○	○	●	○	○	○	●	●
	S10	●	○	●	○	●	○	●	●
	S11	●	○	●	○	●	○	●	●

Table 3: Table indicating how the proposed projects map to the eight original Consultation Themes (derived from the DTI's Technology Strategy key technology areas). The relevant Themes are marked with a blue circle.

4. GAS ANALYSIS THEME

Introduction to Theme

Measurements of gases are fundamental to the control of emissions, the quality of ambient and indoor air, and to many aspects of industrial process control. These measurements are applicable across a wide range of industrial sectors including power generation, chemicals/petrochemicals, oil and other fuels, aerospace, vehicles, electronics, water, waste incineration and landfill, public health, analytical instrumentation (including sensors), metal and non-metal processing, and pharmaceuticals.

The VAM-Physical Programme has carried out work to fulfil the requirements for valid gas and particulate measurements in the UK, which has resulted in the development of:

- Primary gas concentration standards prepared by absolute gravimetric techniques, validated comprehensively and with demonstrated international comparability, covering industrial emissions, natural gas, flammability, occupational exposure, forensic applications, and ambient air quality.
- Absolute gas calibration facilities, which employ dynamic methods where gas standards cannot be prepared gravimetrically with known concentrations.
- National calibration and test facilities to evaluate the performance or type-test analysers for certain industrial emission, process control and ambient air quality applications.

Where the requirements for these are still strong, it will be necessary to replace standards and maintain facilities as they are used. Further, as noted above, the UK is signatory to the Mutual Recognition Arrangement (MRA) between National Measurement Institutes (NMIs) worldwide. As a consequence, key international comparisons have been carried out, that enable the UK to demonstrate acceptable international comparability. These have continuing benefits - by facilitating UK industry's trade with our international partners.

In addition, strong UK inputs have been made to a number of important European (CEN) and worldwide (ISO) standards, some which are now published, and others are advanced drafts. These have major impacts both on UK regulations and on industrial efficiency, and technical input has been made to ensure that the UK's interests are supported and protected.

Theme aims and objectives

The aim of the Theme is to underpin the requirements of UK industry and regulators for traceable measurements of ambient, emission level, forensic, occupational health and indoor air gases.

The objectives of the Theme are:

- To develop and disseminate primary and secondary standard gas mixtures and dynamic measurement facilities to underpin UK industry and regulators.

- To promote the use of new and advanced analytical instrumentation to improve the validity of measurements of process efficiency, air quality, emissions and trace species.
- To provide a focus for the development and application of leading-edge technologies and innovative analytical approaches.
- To transfer knowledge and technology developed in the Theme to UK industry, regulators and trade bodies.
- To represent the UK in CCQM, EUROMET and international standardisation bodies, and to participate in international scientific collaborations in order to ensure the global comparability of UK measurements of gases and particles.

Key drivers

Wide ranging consultations involving UK industry, regulators and government, have been carried out in order to obtain a balanced rationale to address the short and long-term requirements for gas measurements. These have resulted in the identification of four drivers:

(a) Industrial competitiveness and trade

There are continuing requirements to increase the enterprise, innovation, and competitiveness of the UK's manufacturing industries. Gas measurement contributes to this strongly through the optimisation of industrial process efficiency, and by providing cost-effective environmental monitoring technologies. These can both be achieved by the utilisation of advanced on-line instrumentation, calibrated with valid standards. In addition, there is a move towards 'self-calibrating' and 'remotely calibrated' instrumentation, which need to be implemented accurately to be of benefit to industry. The field of analytical instrumentation is highly competitive, and includes many SMEs.

National and international trade is also facilitated by accurate gas measurements, for example, to enable valid natural gas trading between different organisations and countries (towards a single European natural gas market), and for aspects of emissions trading that require measurement.

In addition, there is an increasing emphasis on product certification and type approval in Europe and elsewhere to control emissions from aircraft and other vehicles and to ensure the performance of analytical instrumentation. These activities must be underpinned by valid calibration and measurement techniques, which are acceptable across the single European market to enable international acceptance and facilitate trade.

(b) Regulation

The field of gas measurements is the subject of a large and increasing range of regulations. These include both national regulations, and increasingly, new EU Directives, (requiring Member states to enact them into national law). They include the following:

- EU Directives that limit the emissions of industrial sources to the environment (e.g. Large Combustion Plant);
- EU Directives that seek to control the quality of ambient air in both urban and rural locations, and related CAFE requirements;

- National regulations for the routine measurement of gases covered by international legal metrology regulations (e.g. alcohol-on-breath and vehicle MOT tests);
- European requirements (Euro IV and V) and future ones covering emissions from cleaner vehicles using new technologies;
- EC proposals for a single European market for natural gas;
- EU regulations on trace contamination in the environment, food etc;
- International (ICAO etc) requirements on aircraft emissions;
- International protocols (e.g. Kyoto climate change, UN/ECE trans-boundary pollution, OECD toxicity) to which the UK is a signatory leading to controls on greenhouse gas emissions and pollutant emissions trading;

In addition, new mandatory CEN standards are being drafted to underpin all the EU Directives and provide prescriptive measurement methods that require traceability to national standards. Furthermore, the accreditation standard ISO EN 17025 has now been implemented across Europe – this emphasises the requirement for national traceability.

The VAM-Physical gas analysis Theme enables industry to demonstrate compliance with regulations and statutes in a fair and cost-effective manner, it ensures the acceptability of results by regulators accreditation bodies and the public, and provides support to regulators to enable them to enforce legislation in a technically sound and impartial way.

(c) International representation of the UK's interests

UK measurements of gases must be accepted internationally to enable fair and free trade. The Mutual Recognition Arrangements established by the CIPM between metrology institutes across the world facilitates this. This MRA requires key comparisons and research studies in the gases area, for both the European Research Area and outside, to sustain agreements, and to develop mutual acceptance in further aspects, where needed for trade and for dissemination of results that are traceable to SI units.

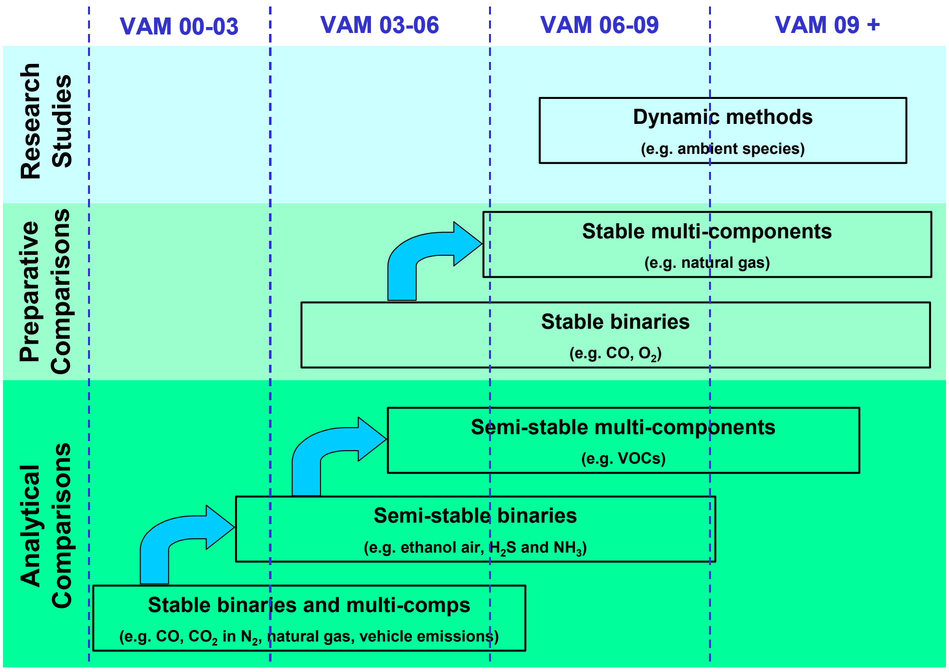

Figure 8: Diagram showing a timescale for CCQM gas analysis international intercomparisons

The recent progress in the organisation of International Key Comparisons within the CCQM Gas Analysis Working Group is shown in Figure 8.

(d) Environmental protection and quality of life

The protection of human health and the natural environment depends strongly on the ability to measure harmful, flammable, toxic and carcinogenic substances cost-effectively, and with satisfactory validity to be fit for purpose. European research studies [11] have estimated that approximately 6% of annual deaths are caused, or precipitated, by outdoor air pollution. A range of measurement applications (including those not covered by current and planned regulations) are involved:

- Emissions (direct and fugitive) from certain industrial and agricultural sources
- Determination of odours and the identification of odorous species
- Air quality in indoor air, vehicles, healthcare establishments, etc.
- The quality of alternative fuels being developed to support sustainable development
- Trace gases, including explosives detection.

Measurement issues

A number of significant advances in gas metrology were developed by NPL to address industrial requirements during the 2003-2006 VAM-Physical Programme. These include:

- Improvements in the verification of the performance of passivated gas cylinders, which have allowed the dissemination of standards for unstable species (e.g. sulphur odorants).
- Development of an automated gas analysis system, which has reduced comparison uncertainties from 1.5% to better than 0.5% with substantially reduced gas usage.
- Implementation of a single-pan balance procedure, which has reduced the time required for weighing to an uncertainty of 0.01% from 2 hours to less than 10 minutes.
- The development of the ‘preparative comparison’ for international comparisons which has improved the uncertainty of comparisons from 0.2% to better than 0.05%.

The 2006-2009 VAM-Physical Programme proposes to implement further technological advances in order to reduce the cost and uncertainty of traceable gas mixtures, including the exploitation of:

- NPL’s IPR covering dynamic dilution methods
- Micro-cylinder technology
- Absolute optical absorption methods
- Improved cylinder passivation chemistry.

The central projects in the gas analysis Theme make use of common facilities and technologies. The underpinning traceability and synergies between the projects are highlighted in Figure 9:

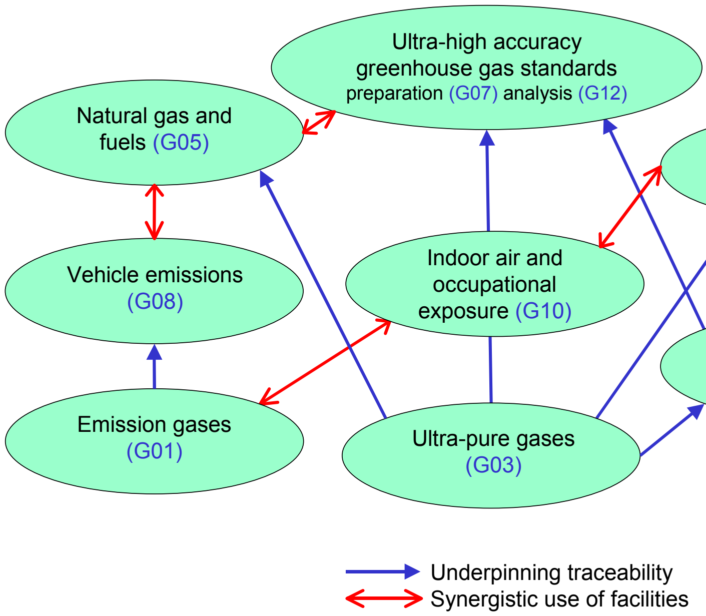

Figure 9: Diagram showing the synergies and underpinning traceability between projects in the gas analysis theme

Theme Roadmap

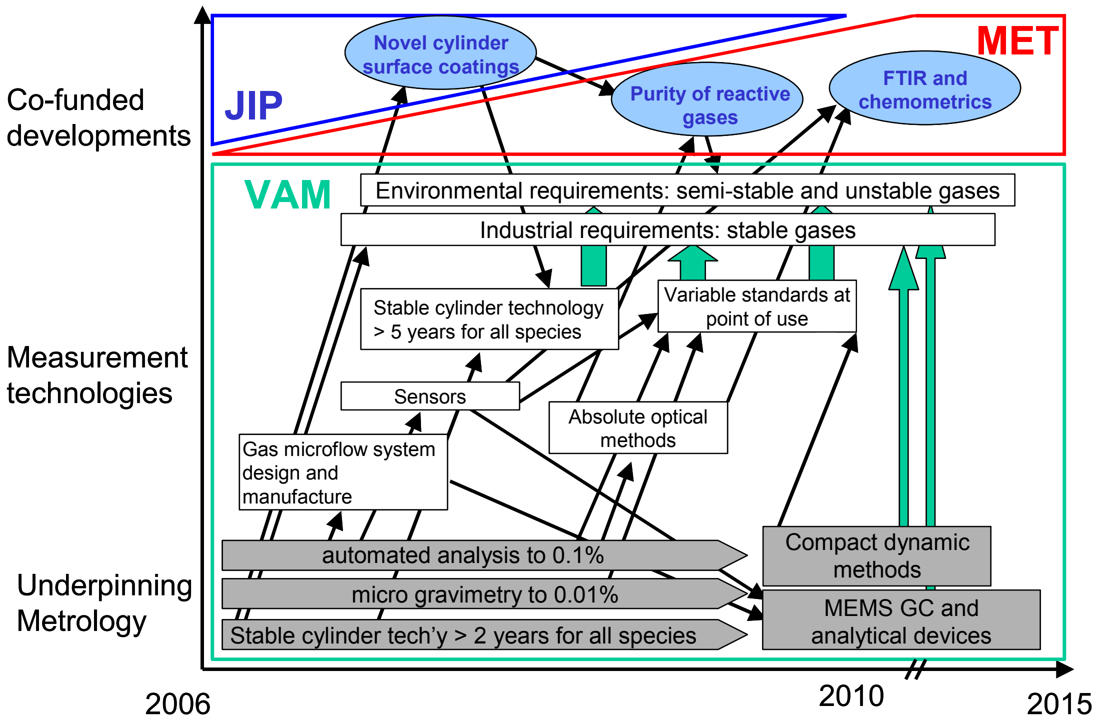

Figure 10: Gas Analysis roadmap showing the strategic development of research within this Theme. It illustrates the feed through of underpinning metrology in VAM through to collaborative industry JIP and MET projects. The timings in the roadmap are indicative and show relative order.

Project G01	Binary and multi-component gas standards at emission levels

Project aim and objectives

The aim of this project is to underpin the requirements of UK industry, the stack testing community and regulators for traceable measurements of emission level gases.

The objectives of the project are:

- To provide capability for existing national standards and facilities for the supply of gaseous emission level standards.
- To extend the scope of NPL's binary gas flow dilutor technology to facilitate the delivery of standards at 'non decade' values.
- To ensure the demonstrable international acceptability of UK standards through the CIPM MRA for emission level gases.

Background and rationale

The control of emissions from industrial processes is essential for the protection of the UK's environment and the health of the population. Continuing reductions in emissions from industrial processes are also a central part of the UK's move towards sustainable development and production.

Defra's five-year strategy published in 2004 highlights the effects of poor air quality on human health. Ensuring the reduction of process emissions will make a significant contribution to the improvement of air quality in the UK.

An important part of the regime for controlling industrial emissions is the accurate measurement of gases from emission sources from specified industries. For example, emissions from combustion plants and waste incineration are limited by EU Directives 2001/80/EC and 2000/76/EC respectively. Monitoring of emission levels is then carried out according to agreed CEN standards that specify the use of traceable calibration standards for each of these sectors.

The proposed project will provide capability for current standards designed to underpin existing measurement requirements as well as extend the range to include new species specified by forthcoming changes in legislation (e.g. HF). The project will also focus on improved automation to reduce the cost and delivery time of standards to UK customers. Further use will be made of NPL's patented binary gas flow dilutor technology to facilitate the delivery of standards at 'non decade' values.

There has been strong support for activities in this area from the Source Testing Association and member organisations.

Summary of technical work

This project will involve the preparation and analysis of binary and multi-component standards containing compounds relevant to process emissions e.g. by-products of combustion and other industrial processes such as NH_3 and HF. Standards could be prepared in high-pressure gas cylinders or dynamically using appropriate dilution technologies.

New gas filling facilities and gas sampling / analysis equipment will be implemented where required in order to support the preparation and analysis of binary and multi-component gas standards. This will include the further development of the Mini Automatic Gas Analysis System.

Deliverables

G01.1 Provision and extension of current gas filling and analysis facilities (including the automated gas analysis system and the binary network).
G01.2 Provision and development of primary gas standards to underpin measurements of stack emission species including CEN hydrocarbon requirements, NH_3 and HF.
G01.3 Development of a primary capability for the certification of HCl and HF standards.
G01.4 Participation in CCQM comparisons to underpin the comparability of UK standards, and dissemination of technical advances to ISO TC158 (Gas analysis and gas standards).

Project G02	Ambient concentration standards for ozone and its precursors

Project aim and objectives

The aim of this project is to provide capability for measuring ambient ozone, and for ambient concentration gases known to play a role in ozone formation and particle formation.

The objectives of the project are:

- To upgrade the UK standard ozone photometer for dissemination and participation in an international comparison.
- To develop and disseminate standards for ozone precursors.
- To validate, or delimit, common manual techniques for ambient hydrocarbon and ammonia measurement.
- To investigate methods for sampling and accurate measurement of semi-volatile organic compounds.
- To organise a comparison for ozone precursors amongst EUROMET laboratories.

Background and rationale

The gaseous pollutant in ambient air that has most effect on human health, in both urban and rural locations, is ozone. It also has significant detrimental effects on agriculture and materials. Ozone is a secondary pollutant that is formed photochemically by the reaction of sunlight, oxygen and nitrogen dioxide in a complex process in which volatile organic compounds (VOCs) play a critical role. Some of these VOCs are carcinogenic, and also give rise to the formation of particles.

Particulate matter in the atmosphere has a greater impact on human health, and it also has a major role in climate change. It has both primary (directly emitted) and secondary components (formed from gases in the atmosphere). The secondary components can be organic, formed from volatile or semi-volatile organic compounds, or inorganic such as ammonium nitrate, formed from ammonia and NO_x. Like ozone formation, secondary particle formation is a complex process that is difficult to model accurately without reliable supporting measurements.

Ozone is regulated by the EC Directive 2002/3/EC, which includes a requirement to measure ambient concentrations of both ozone and 30 specific ozone precursor compounds. Benzene, as a carcinogen, is regulated by the EC Directive 2000/69/EC.

Particulate matter (PM) is regulated by the EC Directive 1999/30/EC, which requires only the measurement of the mass of airborne particles. However, a planned revision of the Directive, issued in September 2005, will introduce a requirement to measure the composition of the PM. The task of apportioning PM to different sources, and of predicting the consequences of measures taken to reduce PM concentrations, will depend on accurate measurements both of particle composition and of gaseous precursors.

In addition to the requirements imposed by the EU, there are also requirements for monitoring across Europe imposed by the UNECE as part of the International Agreement on Trans-boundary Air Pollutants.

This project will underpin measurements made using a variety of techniques for ozone and the most important of the precursor species, including hydrocarbons and ammonia. NPL is the pre-eminent national laboratory in this field and maintained its technical lead under VAM 2003-2006 by the production and dissemination of a new ‘ozone precursor’ mixture. The proposed project includes the coordination of a comparison exercise amongst EUROMET laboratories based on further development of NPL’s ‘ozone precursor’ mixture.

Summary of technical work

The NPL primary ozone photometer will be maintained and upgraded, building on links with the BIPM, which has a suite of similar photometers. This will involve the implementation of changes to the temperature control system that are now recognised to be best practice

A new ‘family’ of cylinder standards of ozone precursor hydrocarbons (at levels below 50ppb) will be made as part of the ongoing validation of these standards. The cryotrapping GC-FID analysis used for ozone precursor mixtures will be refined and documented as a UKAS procedure.

The facility for generating on-line multi-component mixtures will be validated for an extended range of species to include key organic particle precursors. Methods for measurement of ammonia at ambient concentrations (~5ppb), including denuder techniques, will be investigated using on-line generation techniques. Pumped and diffusive sampling techniques will be validated for ambient concentration ozone precursor and particle precursor hydrocarbons.

The issues associated with sampling and analysing semi-volatile organic compounds will be reviewed. An intercomparison for ozone precursors will be completed and the results of the scheme disseminated.

Deliverables

G02.1 Upgrade the UK national Standard Reference Photometer for ozone and disseminate traceability to UK laboratories.
G02.2 Research and development of standards and analytical techniques for ozone and particle precursor VOCs, leading to new cylinder standards and accredited techniques.
G02.3 A review of sampling and analysis issues for semi-volatile organic compounds in ambient air.
G02.4 Validation of common manual techniques for measurement of hydrocarbons and ammonia (e.g. by pumped or diffusive sampling or denuders).
G02.5 Participation in a comparison of ozone precursor standards (through EUROMET in collaboration with the JRC) and dissemination of project outputs through international fora including AQUILA.

Project G03	Improved facilities for the analysis of ultra-pure gases for high-accuracy metrology and the special gas industry

Project aim and objectives

The aim of this project is to extend the capability of the current trace gas analysis and trace water vapour facilities to address the metrology needs of the specialty gas industry and specialty gas users such as the microelectronics industry.

The objectives of the project are:
- To provide capability for the trace gas analysis and primary trace water vapour facilities
- To develop a validated source of zero gas.
- To participate in a comparison of purity analysis with at least two laboratories in Europe.
- To extend the capability of the trace gas facility to alternative matrices (e.g. NF_3).

Background and rationale

The manufacture of semiconductors and microelectronics depends on the availability of pure gas-phase precursors with impurity levels well below 10 ppb. Similar requirements exist for the source gases used for the preparation of standard gas mixtures at ambient levels and of certain reactive species (e.g. NO). Consequently, there is a requirement for measurements of these ultrapure gases to support the special gas industry and their customers. The SEMI Industry Roadmap details requirements for decreasing impurity levels in all key specialty gases including inert purge gases and process gases (including NF_3 and fluorocarbon etching gases). There is a similar requirement for source gases with similar specifications for the manufacture of high-accuracy standard gas mixtures.

As part of the 2003-2006 VAM Programme, NPL developed a unique facility capable of the analysis of inert trace gases (including oxygen, carbon dioxide, methane, hydrogen and carbon monoxide) and water vapour at the ppb level. The increasing requirement for reduced wastage rates in the semiconductor and microelectronics industries is imposing even more stringent requirements on the suppliers of source gases. In particular, for the measurement of trace impurities in reactive process gases and progress towards the measurement of impurities at the sub-ppb level. These will require a validated source of ‘zero gas’ and a complete understanding of the associated sampling and analysis issues.

A further requirement is for the demonstration of the comparability of NPL’s standards with those maintained by other NMIs in Europe. It is proposed that this be investigated by studying the stability characteristics of selected ultra-pure gas mixtures and the scope for using them as transfer standards between laboratories and between companies.

The EPSRC Strategic Plan 2003-2007 discusses developments in chromatography and separation science and spectroscopic techniques under the section entitled ‘The Quantum Realm’. Developments in these areas will have a significant impact on trace gas analysis and associated industries.

Summary of technical work

Provision and development of the trace gas facility and trace water vapour facilities will include the replacement of the current oil circulation bath system on the trace water vapour generator with a Peltier-based unit. This will result in improved transportability and a facility that is easier and cheaper to maintain with a reduced risk of contamination.

New developments will include a validated zero gas generation system which will include the construction of a purifier and gas distribution system. This development will facilitate the preparation of gas standards at parts-per-trillion levels to support the validation of trace gas analysers.

Work will be undertaken to extend current facilities to generate and measure trace impurities in alternative matrices. This part of the project will require the construction of reactive gas handling and abatement facilities or collaboration with a specialty gas supplier who can provide equivalent equipment.

Deliverables

G03.1 Upgrading of the trace gas facility and trace water vapour facility to achieve sub-ppb specification.
G03.2 Development of validated zero gas generation and measurement capability to contain all key species at sub ppb levels.
G03.3 Development of new, low gas consumption FTIR facility for purity analysis of reactive and stable species.
G03.4 Dissemination of project outcomes from a purity analysis inter-comparison including papers, conference presentations and dissemination to SEMI.

Project G04	Valid portable mass spectrometry for field applications

Project aim and objectives

The aim of the project is to validate portable, real-time, thermal desorption mass spectrometry (TD/MS) for environmental monitoring applications.

The objectives of the project are:

- To adapt and extend the calibration transfer strategy delivered under VAM 2003-2006 to be applicable to portable, real-time, mass spectrometers for use in low level VOC monitoring.
- To demonstrate the system in the field.
- To identify and investigate the key scientific challenges to be met for the successful transfer of library calibration spectra between MS instruments in environmental monitoring applications.

Background and rationale

Thermal desorption mass spectrometry (TD/MS) offers portable systems capable of real-time analysis for a wide range of contaminants. This makes such systems attractive for a range of monitoring applications including landfill, land remediation, petrochemical, road-side and purity analysis at breweries. In these last two examples, GC-MS might often be the technique of choice but due to the availability of real-time data, TD/MS is often used. This is important for time indexing pollution events in roadside monitoring and essential in ensuring that CO_2 recovered from brewing for re-sale is not contaminated (as there is a requirement to measure the CO_2 on-line, in near real-time). However, a key limitation affecting the wider use of TD/MS systems in the field is the requirement to take into the field multi-component calibration standards for frequent calibration (often daily) due to the effect of instrumental drift.

This work will underpin the use of TD/MS for the monitoring of accidentally or maliciously released chemicals where the Royal Society report 'Making the UK safer: detecting and decontaminating chemical and biological agents' (April 2004) has highlighted the need to develop analytical techniques for secure environment applications. In addition, Defra's 'Waste and Resources R & D Strategy 2004-2007' has stated the need for policy development built on a sound evidence base. This project would make TD/MS a valuable tool for this objective.

The development of an analysis algorithm incorporating a calibration transfer strategy that reduces the re-calibration frequency would be seen as an innovative step forward by the monitoring community. Consequently, this would facilitate the marketability of such instruments promoting the sales of UK SMEs involved in mass spectrometer manufacture. In addition, validated TD/MS could become an increasingly valuable tool in detecting the release of carcinogenic and hazardous compounds in urban or industrial areas. This will provide regulators with the necessary information to ensure effective abatement strategies, protection of the public and continued improvements in quality of life.

Summary of technical work

A thermal desorber will be interfaced with a portable mass spectrometer system. Calibration of a Standard Test Atmosphere Generator will be carried out for a representative subset of contaminant gases found in many of the applications detailed above. The TD/MS system will then be exposed to multi-component mixtures at the ppb level and a large dataset will be recorded comprising of both training (for calibration) and testing (for validation) measurements. The dataset will be used to characterise the system drift and then test the application of the calibration transfer strategy developed under VAM 2003-2006 (developed for an MS system only). As required, the strategy will be adapted for the TD/MS system and incorporated into an analysis algorithm allowing quantitative analysis of multi-component mixtures with a reduced frequency of re-calibration.

Concurrently, a second TD/MS system will be taken on-loan from a collaborating SME and a sub-set of the dataset will be measured. This will allow an investigation into the key issues affecting the transfer of library spectra between instruments. Proposals will be made towards the eventual solution of the identified issues.

Finally, the TD/MS system with the analysis algorithm will be taken on a suitable field trial to demonstrate and assess its field capability.

Deliverables

G04.1 A comprehensive dataset including training and testing measurements on a TD/MS system of low level VOCs, with a sub-set of measurements on a second system. Application of calibration transfer strategy delivered in VAM 2003-2006 and assessment of performance.

G04.2 Further development of calibration transfer strategy for field application and incorporation into an analysis algorithm to quantify spectra. Identification of key issues in instrument-to-instrument transfer of library spectra with consideration and proposals for eventual solution.

G04.3 Field demonstration of validated TD/MS system employing the analysis algorithm.

G04.4 Dissemination of project outcomes through webpages, the availability of a dataset, and publication of two peer-review articles and a conference paper.

Project G05	Standards and new measurement technologies for the natural gas and fuels industries

Project aim and objectives

The aim of this project is to provide capability for measuring components of natural gas including artificial odorant species and higher molecular weight hydrocarbons.

The objectives of the project are:

- To provide capability to extend the range of primary national standards for natural gas, refinery gases and other fuels.
- To develop new sampling and analytical methods for quantitative measurements of sulphur odorant species and refinery gases.
- To support the UK natural gas industry through a study of the methods used to measure the hydrocarbon and water dewpoints of natural gases.
- To participate in international comparisons in order to support the recognition of UK standards through the CIPM MRA and to organise a PT scheme.
- To disseminate best practice on the measurement of natural gas through ISO TC193, journals and conferences.

Background and rationale

The UK natural gas industry is a multi-billion pound business – the value of natural gas transferred across the UK network is more than £2million per hour. The immense and wide-reaching impact of natural gas on the UK economy has recently been reported in depth in the media due to the increase in wholesale gas prices (up 80% in the twelve-month period to February 2006). The UK is now a net importer of natural gas, meaning that more gas is imported than exported - in fact it is predicted that 75% of gas used in the UK will be imported by 2015. More of the UK's power stations operate on gas than any other fuel, hence any change in natural gas prices impact on the cost of electricity and therefore industry (and the public) as a whole.

The fiscal value of natural gas, along with its physical properties (hydrocarbon dewpoint, density, etc.), is directly calculated from accurate determination of its composition. This necessitates high-accuracy primary standard gas mixtures and analytical methods. Such capabilities have been developed in previous VAM Programmes and there is a pressing need for these to be further improved and expanded.

One particular area of increasing importance to UK industry is the analysis of gas from refineries. Accurate measurement of the composition of the gas emitted is required to determine contributions to the EU Gas Emission Trading Scheme (based on European Directive 2003/87/EC). Support of emissions trading schemes is identified as a key requirement in a number of Government strategy documents including 'Delivering the essentials of life' – Defra's five-year strategy (December 2004) and the DTI Energy white paper – 'Our energy future: creating a low carbon economy' (February 2003).

NPL is committed to continue to support UK natural gas analysis laboratories in upkeeping their world-leading capabilities. To assist with this, it is important that on-going support is given through the continued international recognition of UK standards through the CIPM MRA, organisation of a proficiency testing (PT) scheme.

Summary of technical work

A key growth area will be the development of a new set of traceable standards to support analysis of refinery gases (such as blast furnace, oven and reformer gases). The development of these and other natural gas standards will be supported by the development of two new analytical methods – a GC method to measure accurately the composition of refinery gases, and a GC-MS method for the quantitative analysis of sulphur odorant species.

A study will be undertaken to compare the methods used by the natural gas industry for the determination of hydrocarbon and water dewpoints. Comparison of the most-commonly used instruments and methods will be supported by the preparation of a set of traceable gas standards.

NPL's world-leading position in the field of natural gas analysis will be supported through participation in a CCQM international intercomparison. NPL will also co-ordinate a CCQM study to apply its 'harmonisation' method of natural gas analysis to a range of standards used in a previous intercomparison (CCQM-K23). A proficiency testing scheme will be arranged for UK natural gas analysis laboratories – this will follow on from two highly successful PT schemes undertaken in previous VAM Programmes.

The technical outputs of the project will be disseminated through the publication of peer-reviewed journal articles and presentations at conferences. NPL will also continue to support the UK's interests on relevant National and International standardisation committees such as BSI PTI/015 and ISO TC193/SC1 Working Groups.

Deliverables

G05.1 Expansion of the UK's gas standards facility for natural gas, high hydrocarbons and sulphur odorant species.
G05.2 Development of validated, quantitative methods for the high-accuracy analysis of natural gas by GC, and sulphur odorant species by GC-MS.
G05.3 Development of a robust infrastructure to underpin the traceable measurement of refinery, reformer and blast furnace gases.
G05.4 Rigorous comparison of methods to measure the hydrocarbon and water dewpoints of natural gases in order to support UK industry.
G05.5 Participation in CCQM international intercomparisons to support the recognition of UK standards through the CIPM MRA; organisation of a proficiency testing scheme in order to support UK natural gas analysis laboratories.
G05.6 Dissemination of the project's achievements through peer-reviewed articles and presentations at conferences. Support of the UK's interests through input into international fora.

Project G06	Gas standards and primary dynamic facilities for ambient (acid) gases

Project aim and objectives

The aim of this project is to provide capability for measuring the ambient concentration of the gaseous pollutants CO, SO_2, NO and NO_2

The objectives of the project are:

- To provide and develop facilities for regulated ambient measurements of CO, SO_2, NO and NO_2.
- To investigate the feasibility of generating ambient concentrations of sulphur dioxide by time-division dilution.
- To improve the delivery of calibration to sites through more efficient site procedures and the use of smaller ‘cocktail’ mixture cylinders.
- To provide capability for the international acceptability of UK standards through participation in CCQM and EUROMET comparisons.

Background and rationale

The exposure of the population to carbon monoxide, sulphur dioxide and nitrogen dioxide, primarily originating in combustion sources, has decreased dramatically in recent decades, but it is still a cause for concern in some areas. Because of their direct health effects, ambient concentrations of these gases are regulated by the EU directives 1999/30/EC and 2000/69/EC, which are currently coming into force. Nitrogen monoxide is regulated as a component of NO_x, whose measurement is required as part of the ozone Directive 2002/3/EC because of the role of NO and NO_2 in ozone formation. Accurate measurement of NO is also necessary because it is the species actually measured by a chemiluminescence analyser, the reference instrument for NO_2.

These Directives require routine automatic monitoring to be carried out by networks of stations across each nation in Europe. The harmonisation of this monitoring is achieved through implementing CEN standards that require the use of traceable calibration gas mixtures. In addition to the requirements imposed by the EU, there are also requirements for monitoring across Europe imposed by the UNECE as part of the International Agreement on Trans-boundary Air Pollutants.

The proposed project will build on NPL’s leading role amongst European reference laboratories for ambient air quality monitoring by improving the mechanisms used for providing capability for and disseminating standards to contractors appointed by Defra and to local authorities throughout the UK. Dissemination of standards for sulphur dioxide and nitrogen monoxide (required for calibrations of nitrogen dioxide) is complicated by their limited stability periods in high-pressure cylinders. It is proposed to carry out a feasibility test into the replacement of NPL’s existing primary permeation facility for sulphur dioxide, with a more cost-effective dilution system based on NPL’s patented time-division dilution (TDD) system using stable, higher concentration SO_2 standards. If successful, this will lead to a reduction in maintenance costs and delivery times from this facility.

Summary of technical work

Primary standard cylinders of CO and NO at concentrations necessary for calibrating ambient analysers, typically 10 ppm and 720 ppb respectively will be manufactured and validated against existing mixtures. The stability of cylinders for NO, and also for similar concentrations of NO_2 and SO_2, will be assessed as part of ongoing QA/QC.

The facilities and procedures for certifying cylinders of NO, NO_2, and SO_2 will be refined in the light of internal results and those from external comparisons, leading to revised UKAS documents, and in particular there will be an investigation into replacing the current system of generating primary SO_2 mixtures by an on-line permeation system with a system based on time-division dilution.

There will also be an investigation into the practicality of improving calibration at field monitoring sites through more efficient site procedures and smaller ‘cocktail’ mixture cylinders, in collaboration with manufacturers and users.

International comparisons will take place through, for example, CCQM and EUROMET, and technical experts will directly represent the UK’s interests on, or provide input into National and International standardisation activities.

Deliverables

G06.1 Provision and development of facilities for regulatory ambient measurements of CO, NO_x and SO_2.
G06.2 Investigation of the feasibility of generating ambient concentrations of sulphur dioxide by time-division dilution.
G06.3 Improvement to practical delivery of calibration to sites through more efficient site procedures, smaller ‘cocktail’ mixture cylinders and provision of instrument testing facilities.
G06.4 International comparisons, dissemination of project outputs and support of the UK’s interests through international fora including AQUILA.

Project G07	Stable standards for monitoring greenhouse gases

Project aim and objectives

The aim of this project is to provide stable primary standard gas mixtures of the major components of atmospheric composition (oxygen, carbon dioxide, methane, nitrous oxide and argon) with a gravimetric uncertainty of better than 0.1%.

The objectives of the project are:

- To provide capability for standards of carbon dioxide and methane (with uncertainties of better than 0.1%).
- To develop a new capability for preparing high accuracy standards in micro-cylinders.
- To disseminate standards to the WMO laboratories through participation in a CCQM comparison.

Background and rationale

The monitoring of global climate change and the development of a global consensus as to its causes and consequences presents a major challenge for the metrology community. A key parameter used to indicate the extent of global warming is the concentration of atmospheric gases with strong 'global warming potentials'. The two principal species monitored for this reason are carbon dioxide and methane.

A comparison carried out under the auspices of the CCQM between the leading metrology laboratories in the world and the WMO's Global Atmospheric Watch (GAW) network showed a significant discrepancy of 0.5% for one important component of the atmosphere (methane). This result underlines the requirement for leading NMIs to play a larger role in underpinning the scales used to monitor long-term trends in atmospheric gas composition.

The Government's Chief Scientific Adviser, Sir David King has stated that "climate change is the most severe problem that we are facing today – more serious even than the threat of terrorism" (Science 303 (2004) 176-7). Whilst international scientific consensus on global climate change is being developed by the Inter-governmental Panel on Climate Change (IPCC), much of the data they depend upon is developed by the GAW Programme.

There is also a related requirement from UKAS-accredited laboratories for the supply of more accurate standard gas mixtures. A further output from the work in this project aimed at high-accuracy standards for greenhouse gases will be to meet this wider requirement.

Summary of technical work

The approach proposed by this project will be to improve NPL's gravimetric capability so that the cost-effectiveness of the service can be improved through the preparation of standards with fewer dilution steps that exploit the low mass and volume of micro-cylinders. This will decrease the time taken to prepare standards and also reduce the number of 'parent' standards stored at NPL to maintain dilution hierarchies. A consequence of this will be a reduction in the cost of standards (whilst maintaining the same uncertainty).

The development of a facility for preparing high-accuracy standards using micro-cylinder technology may require manufacture (externally) of pressed aluminium parts and welding (in-house or on contract). It will also involve experimentation with the effectiveness of crimped seals (and other options) and work on passivation of the end products.

The new facility will be used to prepare primary standard gas mixtures of carbon dioxide and methane close to the background atmospheric levels. Theses will be used to participate in a CCQM comparison (of binary carbon dioxide and methane mixtures in air).

Deliverables

G07.1 Provision of capability for standards of carbon dioxide and methane at atmospheric levels (with uncertainties better than 0.1%).
G07.2 Results of the evaluation of gravimetric preparation based on samples in 'micro-cylinders' or balance gas in larger cylinders, leading to single-step gravimetry with uncertainties less than 0.1%.
G07.3 Dissemination of the results of the micro-cylinder developments, and their application to gravimetry and storage of primary gas mixtures.

Project G08	Gas standards to underpin emissions from road and air transport

Project aim and objectives

The aim of the project is to ensure that the UK is able to comply with present and future EU Directives regulating gaseous emissions from road and air transport, and to support the international recognition of UK standards through the CIPM MRA.

The objectives of the project are:

- To provide traceable and internationally-recognised gas standards to underpin the requirements for MOT emission tests and to support the UK vehicle testing industry (through the BTC) in improving their exhaust gas sampling and analysis methods.
- To provide a Proficiency Testing scheme for exhaust and evaporative emissions from vehicles.
- To provide traceable and internationally-recognised gas standards to underpin the requirements for UK breath alcohol measurements to ensure compliance with present and future UK legislation.
- To provide gas standards to enable traceable measurements of emissions, and develop liquid sampling techniques, for engines using alternative fuels (e.g. LPG, LNG and bio-diesel).

Background and rationale

Gaseous emissions from road and air transport continue to be carefully regulated in order to support society's objectives of maintaining sustainable, safe means of transport. In particular, emissions from motor vehicles are regulated by the EU's Euro IV regulations. These are implemented through routine UKAS-accredited MOT testing of vehicles against traceable calibration gas mixtures.

There is a requirement to extend NPL's range of automotive emission standards (for example the inclusion of oxygen or NO), driven by EU Auto Oil II, Euro IV 2005, EU Directive 98/69/EC, EU Emission Standards for Gasoline Cars, and Euro V, EU directive 99/96/EC (to be implemented 2008/2009) for HGVs' diesel engines. The continual demand for a reduction of harmful engine emissions has led to improvements in engine test house sampling methods of exhaust gases, and these are required to be underpinned by traceable gas standards. There has been a request from the British Technical Council for the motor industry (BTC) for VAM to support them in this area.

There is a requirement to underpin present road and marine breath alcohol measurement legislation (35 µg alcohol per 100 ml of breath, Road Traffic Act 1988 (as amended), Transport and Works Act 1992) and future lower levels for safety critical aircraft personnel and air traffic controllers (9 µg alcohol per 100 ml breath, Railways and Transport Safety Act 2003) in the UK, and a new generation of mobile evidential breath analysers will be brought into use during the course of the 2006-2009 VAM Programme.

This project will also investigate industry requirements for gas standards for alternative fuels and develop them where necessary. Government policy is responding to market forces in this area and significant developments are expected during the course of VAM 2006-9. In particular, it is anticipated that although these fuels have lower emissions overall, they may give rise to hydrocarbon and hazardous by-products that are not generated from present fuels.

In addition to the legislation described above, a VAM commissioned NPL report, 'Review of Measurement Requirements for Current Automobile Emissions Technologies' (NPL Report DQL-AS 011), highlighted the changing EU legislation, sampling, analytical, and associated measurement requirements for engine emissions.

The minimum metrological standards of production, verification, and performance of approved evidential breath alcohol instruments are contained in the 'Quality Framework for Evidential Breath Alcohol Analysis Instruments, Version C 2.0 30/09/04', in which references are made to several NPL reports, including the requirement that breath alcohol testing instruments should be calibrated by a UKAS accredited procedure that refers to NPL certified gas standards.

Summary of technical work

A range of gas standards and facilities for the measurement of vehicle exhaust emissions will be provided and extended where necessary. MOT emission gas standards will be prepared to underpin present and future requirements, including the addition of NO or oxygen where necessary, analysed and compared using a micro-GC. Liquified gas standards to underpin sampling and measurements of LPG and LNG fuels will be prepared. A PT scheme to assess capability of UK engine makers test houses' exhaust emission measurements will be organised.

Breath alcohol gas standards and associated 'interfering substances' will be prepared as necessary, to underpin present and future requirements, including new type approval. Analysis and comparison of breath alcohol standards will be carried out using a GC. A gas blending rig for type approval work will be constructed.

Deliverables

G08.1 Provision and preparation of high accuracy gas standards to underpin present and future MOT vehicle exhaust emission testing in the UK.
G08.2 Provision and preparation of high accuracy gas standards to underpin present and future breath alcohol testing in the UK.
G08.3 Preparation of 'interfering substance' and associated ethanol breath alcohol standards, and development of a gas blending rig to underpin type approval of next generation of breath alcohol analysers.
G08.4 Organisation of an engine test house exhaust emission PT scheme through BTC emissions group, and underpin vehicle evaporative emissions measurements.
G08.5 Development and dissemination of gas standards to underpin measurements of emissions from alternative-fuelled (e.g. LPG, LNG and bio-diesel) vehicles. Development of sampling technologies for liquefied gaseous fuels and participation in relevant CCQM and EUROMET intercomparisons.

Project G09	Stable cylinder standards for binary and multi-component mixtures

Project aim and objectives

The aim of this project is to develop stable standard gas mixtures to underpin the requirements of UK industry and regulators for traceable measurements of reactive species at concentrations below 10 parts-per-million.

The objectives of the project are:

- To provide the capability for the preparation, purity analysis and quality control of gas standards supporting measurements of process and aircraft emissions (including ammonia, sulphur dioxide and hydrogen sulphide).
- To trial recent industrial developments in passivated cylinder technology and to report on their application to gas metrology.
- To investigate the potential of micro-fluidic dosing and micro-cylinder technology for gas standard preparation and storage.

Background and rationale

There is an ongoing requirement for standards with improved stability for a range of gases at concentrations below 10 ppm. These include ammonia, hydrogen chloride and sulphur dioxide at stack emission levels as well as formaldehyde at engine emission levels. This project will provide the capability to deliver such standards and extend it to allow standards of new species and at lower concentrations to be produced. It will also enable more cost-effective production methods to be employed.

It is proposed to meet this requirement by investigating the potential for the preparation of binary and multi-component mixtures using micro-fluidic dosing and micro-cylinder technology. The work will include the development of lower concentration standards for species such as NH_3 where both improved package and analytical capabilities are required. Improved and alternative cylinder technologies will also be explored. Work in this area will result in efficiency gains in the preparation and handling of potentially all in-house gas standards and will significantly reduce costs associated with filling activities and storage. There is enormous potential for spinouts to specialty gas companies relating to the storage and handling of pure gases and gas standards. Successful development in this area will have a significant impact on the instrument calibration market (particularly the portable instrument market) and potentially the electronic manufacturing sector relating to the handling of precursor and dopant species.

The ‘EPSRC Research Priorities & Opportunities’ report outlines future requirements for developments in analytical science including miniaturisation including microfluidic chip technology. This project will contribute to the development of applications using such technology.

New standards are required to improve the efficiency of measurements made of key ambient air pollutants. This activity is supported by Defra as outlined in their publication ‘Delivering the Essentials for Life: Defra's Five-year Strategy' published in 2004. The report also highlights the need for better measurement of aircraft exhaust emissions, a topic that is also highlighted in the DTI aviation programme.

Summary of technical work

This project will include the development of new capabilities for the production and analysis of sub-100 ppm NH_3 standards, low concentration high-pressure gas cylinder standards for H_2S and SO_2, and the development of new high-pressure gas cylinder standards of formaldehyde. This work will require advances in cylinder treatment technologies.

In addition to the above, the project will also explore the possibilities for alternative gas standard storage, which will focus on the construction of a micro-cylinder filling facility.

Deliverables

G09.1 Provision and development of the current primary gas standards facility including preparation, purity analysis and quality control to ensure efficient and effective processes including development of improved GUM and ISO Guide 34 compliant software.

G09.2 Development of new standards supporting measurements of process emissions (e.g. agricultural) and aircraft emissions: sub 100 ppm NH_3, and low concentration SO_2 and H_2S in cylinders.

G09.3 Research and development of alternative gas standard preparation and storage technologies including a prototype micro-cylinder filling facility.

G09.4 Dissemination of project outcomes including the introduction of a new work item at ISO TC158.

Project G10	Standards to underpin measurements of gaseous species in indoor air and occupational exposure

Project aim and objectives

The aim of this project is to underpin the accurate measurement of gaseous species encountered in indoor air and through occupational exposure in order to address current and future regulatory and health and safety requirements.

The objectives of the project are:

- To extend the range of nationally available primary standard gas mixtures to underpin measurements of indoor air quality and occupational exposure.
- To develop and validate new methods for monitoring indoor air and occupational exposure.
- To provide a proficiency testing scheme for laboratories working on materials emissions testing.
- To disseminate the outputs of the project through efficient and broad-reaching mechanisms.

Background and rationale

Gaseous pollutants in indoor air have been strongly linked to public health issues: 5% of the UK workforce is estimated to have suffered from illness resulting from indoor air pollution. In addition to adversely affecting the well-being and quality of life of the UK population, this results directly in a loss of productivity, thus harming the economy. Concern over the health effects of these pollutants has lead to the formulation and implementation of wide-ranging EC directives requiring the identification and monitoring of the most prevalent toxic species. Examples include:

- A forthcoming directive on ambient air quality and cleaner air for Europe (COM (2005) 447).
- Directive 1999/13/EC – limiting the emissions of VOCs from the use of organic solvents in a vast range of industrial applications and installations.
- The construction products directive (89/106/EEC) – specifying that the levels of gases emitted from building materials should not result in a threat to health.
- The third daughter directive (2002/3/EC) – setting limits for 30 hydrocarbons and VOCs (including formaldehyde) in air.

The EU's proposed regulatory framework for the Registration, Evaluation and Authorisation of Chemicals (REACH) also necessitates the acccurate measurement of harmful species. This aims to improve the protection of human health and the environment through improved and earlier identification of the properties of chemical substances.

Detection and measurement of toxic pollution in indoor air also supports SCALE (The European environment and health strategy), which aims to reduce diseases caused by environmental factors (including air pollution) in Europe, and CEHAPE (Children's environment and health action plan for Europe), which identifies air pollution as one of the major factors affecting children's health across the continent.

In order to meet this increasing volume of legislation, there is a strong requirement for standards and validated methods to measure accurately the key pollutant species in indoor air, and those experienced through occupational exposure. A report commissioned as part of the 2003-2006 VAM Programme ('The use of gas reference materials in food packaging and the control of indoor air quality') recommended that efforts be focussed on species such as formaldehyde, VOCs and other chlorinates and non-chlorinated hydrocarbons – these are present in indoor air: at home, at work or in vehicles (cars, aircraft cabins and commercial shipping). Brominated hydrocarbons are also of increasing concern due to their use in fire retardants and farming applications.

Summary of technical work

Standards of VOCs and chlorinated (and, where applicable, other halogenated) hydrocarbons at levels down to 10ppb will be developed and supported by GC-FID analysis. This will support work and build on expertise gained in the 2003-2006 VAM Programme where a set of chlorinated and ozone precursor standards were prepared successfully.

A facility for the measurement of formaldehyde and other similar pollutant species at concentrations between 100ppt and 500ppb with improved accuracy will be developed and validated by measurements underpinned with a range of gas standards.

The STAG and CATFAC controlled atmosphere test facilities (developed under previous VAM Programmes) will be used extensively to support the development of these sorbent materials where appropriate. The scope of NPL's time division dilution (TDD) facility will be expanded in order to validate the measurements of chlorinated hydrocarbons.

A proficiency testing scheme will be organised in order to test UK labs' capabilities of performing the analytical measurements required to support the European legislation described above. Dosed diffusion tubes will be used as samples for distribution to participating laboratories.

Deliverables

G10.1 Provision and development of standards and facilities for the accurate measurement of volatile organic compounds and chlorinated hydrocarbon species. Participation in a bilateral compassion with a leading NMI.

G10.2 Development and validation of a capability for the accurate measurement of formaldehyde (and similar species) in ambient and indoor air.

G10.3 Development of the time division dilution facility to cover new species at reduced levels of uncertainty, supported by development and provision of capability of the STAG and CATFAC controlled atmosphere test facilities.

G10.4 Organisation of a proficiency testing scheme to assess the measurement capabilities of UK analytical laboratories.

G10.5 Dissemination of project outputs via the publication of peer-reviewed articles and presentations at conferences. Support of the UK's interests through input to key international fora.

Project G11	Gas sensors and multi-gas data fusion

Project aim and objectives

The aim of this project is to provide the standards and infrastructure necessary for the testing, validation and calibration of gaseous sensors and to develop and disseminate algorithms for 'fusion' of data from sensors and multi-gas instruments.

The objectives of the project are:

- To develop and disseminate a method for combining the results of natural gas analysis using micro-GC, where direct 'bridging' between channels is not possible.
- To explore techniques capable of fusing data from different instruments in environmental monitoring applications.
- To develop a calibration transfer methodology for distributed gas sensor networks.

Background and rationale

Low cost gaseous sensors play an increasingly important role in many parts of industry. The Foresight Sensors Taskforce (www.foresight.gov.uk) have identified the importance of gas sensors to UK sensor manufacturers. The Micro- and Nano-technology (MNT) Roadmap for Gas Sensors developed in 2005, identified key application areas for sensors in monitoring: air quality (including measuring VOCs and benzene), indoor air (including in-cabin and in-car air), odour and the detection of explosives. A key technology required by many sensors is 'separation' along with: sample conditioning, extraction, focussing and transport to the sensor site. Key existing markets for UK sensor manufacturers include: process control, food production, industrial safety, engine emission monitoring and engine management.

The most important sensor applications to be addressed by this project are in the monitoring of indoor air and the monitoring and control of emissions from automobile engines. Such sensors generally have relatively poor stability, and therefore require regular calibration. In addition, they are often deployed in safety-critical applications where it is essential that they operate correctly at, or close to, a specified 'alarm' threshold.

A complementary requirement that the project will address is the general challenge of making best use of analytical data from two or more instruments that measure complementary sets of gases. This challenge arises in many atmospheric monitoring and process control applications. One example of this problem is the fusion of data from two columns on a gas chromatograph. Whilst this has been successfully addressed in the previous VAM Programme for the special case where a 'bridging' compound can be measured with good repeatability on both columns, the strongest requirement is to extend the approach to the general case where a straightforward direct bridging compound is not available. An important application of this is to 'micro-GCs' now used for routine laboratory analysis of natural gas. A more complex problem is to evaluate the potential for 'fusing' the data from different multi-species instruments (e.g. mass spectrometry and FTIR). There is huge potential for this type of approach, for example, overcoming the limitations in calibration stability of a portable mass spectrometer, by 'fusing' its results with those from an FTIR, which does not suffer from this limitation, but generally measures a different suite of gases. This research on sensor fusion will have additional applications to the future deployment of MEMS devices to address odour measurement, homeland security and medical applications.

Summary of technical work

The work supported by this project will involve the acquisition of data sets, the development of algorithms, propagation of uncertainties. In the case of the work on the 'micro-GC', the results will be evaluated in routine applications and disseminated through a new standard published by ISO TC 193 (Natural Gas).

Deliverables

G11.1 Development of a protocol for 'fusing' data from multi-column gas chromatographs where 'direct bridging' is not possible.
G11.2 Algorithm for fusion of multi-species analytical data for mass spectrometer and FTIR in environmental monitoring applications, including a peer-reviewed publication.
G11.3 A validated principle to underpin the calibration transfer methodology for MEMS gas sensor networks.

Project G12	High accuracy gas analysis

Project aim and objectives

The aim of this project is to improve the accuracy of the analysis of primary gas standards in order to support international collaboration on monitoring the composition of the global atmosphere.

The objectives of the project are:

- To investigate the use of analysis methods that can be calibrated with respect to pure materials in order to give ‘absolute’ analysis, in particular a high-accuracy paramagnetic oxygen analyser.
- To develop a new GC method to compare standards of atmospheric air for argon and nitrous oxide.
- To review the scope for preparing standard gas mixtures at uncertainties better than 0.1% through traceability to standards of pressure.
- To develop novel accurate and stable on-chip calibrator systems for use on MEMS gas analysis devices.

Background and rationale

The monitoring of changes in the composition of the background global atmosphere is an essential part of the global effort to assess the potential for global warming. It has driven important requirements for high-accuracy analysis of gases. A recent trend in this area has followed the observation of an anti-correlation between oxygen and carbon dioxide levels in the atmosphere. This has now generated a requirement for measurements of oxygen with an uncertainty approaching 0.01%.

New methods have been developed that have strong potential to meet this requirement for oxygen measurements and to work at levels of uncertainty that bridge the gap between the levels achievable with available process analysers (approximately 0.3%) and the uncertainties achievable in the preparation of standards by gravimetry (typically better than 0.01%).

The proposed project will also address a related requirement for the analysis of nitrous oxide and argon at levels typical of the background atmosphere. These are required to enable accurate comparisons of gravimetrically-prepared standards of ambient air (including the minor components methane, argon and nitrous oxide). These are part of the work of the leading laboratories within the CCQM (including NPL) to underpin the WMO’s Global Atmospheric Watch (GAW) programme. A facility for measuring the argon composition of the atmosphere is also needed to carry out corrections for atmospheric density in high accuracy gravimetry.

The development of MEMS devices capable of carrying out analytical functions on single chips has enormous future potential for analytical science. Although early research focussed on processing and measuring liquids, more recently, important advances have been made in the use of similar technology for measurements in the gas phase. For example, complete systems have been reported capable of VOC measurements by gas chromatography (Lab on a Chip 2005 (5) 1123-1131) and measurements of sulphur compounds by fluorescence (Lab on a Chip 2005 (5) 1374-1379). These systems take advantage of the capability to manufacture a complete system including sample introduction, separation and detection steps on a single platform. However, these systems raise important un-answered questions about calibration, since their compact size render calibration methodologies based on, for example, gas in pressurised containers, unsuitable.

Summary of technical work

This project will involve a characterisation study of a novel oxygen analyser based on the principle of paramagnetism (manufactured by a UK SME). This technique has the unique advantage of a dynamic range that is sufficiently large to support calibration with pure oxygen and analysis at levels down to 100 µmol/mol. The accuracy required for this study will be achieved by using specially-prepared primary standard gas mixtures. This experimental study will be complemented by a review of the scope for preparing standard gas mixtures with traceability to standards of pressure. This will seek to assess whether the very small uncertainties routinely estimated for calibrations of pressure measurement devices can be transferred to corresponding uncertainties for gas composition.

A new GC-TCD method using a low-volume detector and a cooled column will be developed for the analysis of nitrous oxide and argon at ambient levels. This will be used for comparisons of accurate primary standards prepared at levels typical of the background atmosphere as well as for the measurements of argon in pure gases used throughout NPL's work on primary gas standards.

The work on calibration of MEMS GC systems will be carried out in collaboration with a UK university. It will involve specific research into the delivery of on-chip calibrator systems, and the evaluation of their performance.

Deliverables

G12.1 Research to establish the accuracy and stability achievable with on-chip calibrators with MEMS systems for gas analysis.
G12.2 Experimental investigation on the use of analysis methods that can be calibrated with respect to pure materials in order to give 'absolute' analysis.
G12.3 Development of standards, and analytical methods, for oxygen, nitrous oxide and argon (with uncertainties better than 0.1%).
G12.4 Review of the scope for preparing standard gas mixtures at uncertainties better than 0.1% through traceability to standards of pressure.

Project G13	Practical gas measurements by isotope dilution mass spectrometry (IDMS)

Project aim and objectives

The aim of this project is to promote the use for gas measurements of the SI traceable IDMS curve method, developed in previous VAM Programmes at NPL, by extending its application to practical laboratory GC-MS instruments.

The objectives of the project are:

- To demonstrate the SI traceable IDMS curve method with a 'unit mass resolution' (benchtop) quadrupole mass spectrometer (QMS).
- To develop a 'calibration' method for IDMS analysis of selected species such as carbon dioxide and methane.
- To disseminate the findings of the project in order to promote and underpin measurements that are traceable to the SI in gas metrology.
- To publish the results in peer-reviewed journals and through the European 'Virtual Institute for Primary Isotopic Measurements'.

Background and rationale

There is no single general method that gives clear and direct traceability to the SI across analytical science. IDMS is the only analytical method capable of making high-accuracy, traceable analytical measurements of organic, inorganic and gaseous samples and has been recognized as a primary method by the international Consultative Committee for Amount of Substance (CCQM).

Although the conventional IDMS method has been well established for decades, its implementation has proven to be difficult when high accuracy is required. There are at least two aspects that have been hindering its potential wider application in gas metrology: the lack of readily available certified isotope reference materials needed during the process of measuring isotope ratios and the difficulty to measure the absolute isotope ratios accurately due to instrument drift, bias or cross-contamination. In order to overcome these difficulties, a method called the IDMS curve method has been developed at NPL. This method utilises the characteristics of an isotope dilution process and formulates the IDMS measurement equation in such a way that:

- It is not necessary to measure the isotope ratio of the un-diluted spike directly, hence it avoids any risk of contaminating the instrument.
- Its intermediate measurands can be chosen to fall within a limited range of isotope ratios so that the effect of instrumental non-linearity is minimised and can be configured in pairs and arranged in such a way that the final results are insensitive to instrumental drift, systematic error, cross contamination, equilibration and/or mass fractionation process.
- It does not require external calibration hence it fulfils the requirements of being a primary method that can be used to measure unknown samples for which no isotopic reference materials are available.

Although this method has been shown to be capable of measuring stable gases with a comparable accuracy to that of primary gravimetry, it is currently implemented at NPL with a purpose-built four input mass spectrometer, which is difficult to implement outside NMIs. The proposed project will implement the IDMS curve method with a widely-used benchtop GC-MS instrument so that the accuracy of the IDMS curve method and the speed and convenience of pre-separation through GC can be combined. This implementation will provide a convenient method for carrying out traceable gas measurements and make it easier to be adapted in practical analytical measurements. As the principle applies to LC-MS configuration as well, there are potentials that this method could be spread into organic and inorganic sectors alike/

Summary of technical work

It is planned to implement and demonstrate the IDMS curve method via a representative practical laboratory GC-MS instruments such as the Agilent 5973 GC-MS. Its inlet will be modified to enable gas sampling through a multi-port gas-sampling valve and a temperature stabilised sample loop. Its existing software will be then modified accordingly and new programme codes will be written to control the measurement procedure and process data following the principle of the IDMS curve method.

For test and validation purpose, isotopic blends of CO_2 and CH_4 or other suitable stable gas will be prepared through gravimetry and barometry. Gravimetry is a primary method, but it requires a certain minimum amount of precious isotopes to during the process of making the blends. Barometry is not a primary method, but it requires much smaller quantities of these isotopes. A barometric blending system will be constructed for this purpose and will form part of the calibrating facility.

We will collaborate with IRMM of JRC with respect to accurate measurement of CO_2 (the main greenhouse trace gas) and CH_4 (a principal component of natural gas) and to provide capability for the established collaboration with other laboratories in Europe (e.g. PTB, BAM, METAS, EMPA and the EC JRC at Geel) with complementary interests in ‘primary isotopic’ measurements.

Deliverables

G13.1 Demonstration of the implementation of the IDMS curve method with a laboratory GC-QMS instrument leading to a publication.
G13.2 Extension of the curve method by measuring multi-component samples.
G13.3 Construction of a dynamic or barometric gas blending system for the preparation of internal standards.
G13.4 Dissemination of project outputs through the JRC-sponsored ‘Virtual Institute’: the Joint European Programme for primary isotope measurement.

5. PARTICULATE AND TRACE CHEMICAL ANALYSIS THEME

Introduction to Theme

The Particulate and Trace Chemical Analysis Theme is centred on the measurement issues relating to airborne particles, and includes the related areas of Surface Enhanced Raman Spectroscopy (SERS), pH and ionic content. Many of the techniques for handling particle-laden air are similar to those for handling gas mixtures, addressed by the Gas Analysis Theme. The chemical analysis of small particles is also close in concept to some of the work included in the Surface and Nanoanalysis Theme.

Airborne particles are the single most dangerous component in air pollution in the United Kingdom, causing an estimated 32,000 premature deaths here annually. They are also a major factor in climate change, both directly through their absorption and reflection of radiation, and indirectly through their effect on the formation of water droplets. They have a much more local and immediate effect on the climate than greenhouse gases such as carbon dioxide.

Airborne particles are extremely diverse, varying in size, composition and origin. Sooty particles from diesel engines are just a few nanometres in size, while windblown dust particles, typically rich in silicon, can be tens of micrometres. Some particles originate from natural processes, such as sea salt particle formation, while others arise from human activities. Many particles are formed by the reaction of gases in the air, for example the gas ammonia (from agricultural sources) reacts with nitrogen dioxide (from vehicle exhausts) to form particles of ammonium nitrate.

The diverse physico-chemical characteristics of particles lead to many measurement issues. The components and properties most relevant to health have not been determined. Current legislation refers only to total mass of particles below 10 µm in size (PM_{10}), or below 2.5 µm ($PM_{2.5}$). Projects P01 to P04 in this Theme are aimed directly at measuring and characterising airborne particles as a necessary part of understanding their health and climatic effects.

The most common technique for analysing ionic components of airborne particles such as ammonium, sulphate and nitrate is ion chromatography. Project P05 contains underpinning work in pH and related ion analysis topics which builds on existing strengths in these areas.

SERS shows great promise as a tool for ultra-trace analysis. The technique relies on the enhancement of Raman signals from target molecules, sometimes down to single molecule levels, using a nanostructured metallic substrate. The technique has potentially wide applications to the detection and identification of ultra-low levels of chemical components in the atmosphere and the environment especially when in the particulate phase. However, to achieve reproducible enhancement levels to make SERS genuinely useful as a trace analysis tool, more regular and reproducible metallic substrates are needed. Project P06 is a more forward-looking project focussed on achieving these goals.

Theme aims

The aims of the Theme are:

- To provide underpinning traceability for airborne particle number density measurements and develop methods that can measure characteristics of particles (such as surface area) that are not readily measurable with existing technology.
- To provide capability for measurement methodologies, standards and facilities required for robust measurements of airborne particle size and number density.
- To underpin measurements of airborne particle mass, and provide metrological support for measurements of organic and elemental carbon, and for relevant properties such as reflectivity and non-volatile mass.
- To develop novel analytical methods to perform accurate measurements of mercury vapour and of the chemical composition of airborne particles.
- To provide capability for the UK's link to primary standards of pH and solution conductivity, and develop focussed research into practical pH measurement in on-line environments, particularly fuel cells.
- To develop efficient and reproducible nano-structured surfaces to underpin the use of Surface Enhanced Raman Spectroscopy (SERS) as an ultra-trace analysis tool.

Key drivers

Four high-level drivers for physical analytical measurements were identified during the formulation process. These are**:**

(a) Industrial competitiveness and trade

Industrial competitiveness is strongly influenced by the measurement techniques for particles used to enforce the regulation being reliable and cost-effective. The same techniques are also crucial to allowing innovation in production methods or vehicle engines, for example, to be properly evaluated. More specifically, the emerging industrial applications of manufactured nanoparticles, within the field of nanotechnology, require measurement techniques that will be closely related to those developed within these projects, aimed at ambient air and emissions. These issues have led to a strong market for particulate instrumentation, which again requires reliable, internationally recognised validation and calibration.

The determination of pH is the most commonly made analytical measurement throughout the world. An increased understanding, particularly at end-user level, of the measurement science underpinning electrochemical techniques and the need for traceability in valid electro-analytical measurements, would make process pH measurements more accurate, therefore benefiting UK competitiveness reducing barriers to trade.

There is an increasing requirement in the analytical community to detect and characterise target molecules at ultra low and even single molecule levels, especially in environmental matrices. Optimised procedures and analytical techniques would increase the UK's ability to perform accurate low level quantification, thereby improving efficiency and increasing analytical throughput, and providing the UK with a competitive advantage in these areas.

(b) Regulation

Regulations developed at both European and UK level ensure both that particulate emissions from vehicles and industry, and ambient particulate concentrations, are controlled within agreed limits. Such regulations are most effective when they define performance levels that are both achievable by industry with available technology and operate at levels that provide adequate protection for society. The imposition of such regulation on a basis that is fair to regulated industry and society requires an infrastructure for traceable measurements at a level of uncertainty appropriate for the application. The measurement infrastructure for airborne particles is notably immature and requires measurement science to develop.

Many areas of regulation require accurate pH measurements; for example the monitoring of ambient air quality, water quality and food quality). These lead to transparency in measurements used to inform policy making (at local, national and international levels) and to determine compliance with limit values set for pollutants.

The use of optimised trace analysis techniques is a vital step towards the technical capability required to assess compliance with ever more exacting regulatory limits, for example for allowable pollutant levels in environmental matrices. Accurate, traceable measurements at low levels with low uncertainties will enhance the tools available to regulatory authorities for accurately judging compliance against limit values which exist in the environmental field.

(c) International representation of the UK's interests

The above regulatory and standardisation issues above, and the international nature of air pollution and climate change, emphasise the importance of international representation as part of this project. NPL has been very active, in the writing of European standard methods for air pollution by CEN, such as CEN TC264/WG15 (PM_{10} and $PM_{2.5}$) and WG 25 (Mercury). Particle counting and sizing standardization is addressed through ISO TC24/SC4 (Sizing by methods other than sieving). The practical implementation of PM legislation has involved the detailed description of what is meant by 'equivalent' methods, and NPL will continue to play a prominent part as this develops. Issues of PM measurement in national networks are addressed through the European AQUILA group. At the metrological level, representation for particle measurements will take place through EUROMET and CCQM, and for pH through CCQM's Electrochemical Analysis Working Group.

(d) Environmental protection and quality of life

Benefits to environmental protection, through climate change, and quality of life, through both climate change and the health effects of airborne particles, are central to the work of this theme, as described in the Introduction. There would also be less direct but very pervasive benefits through the capabilities to be established through the pH and SERS work.

Measurement issues

Some of the key measurement issues are summarised below:

Airborne particulate sampling
Characteristics of size selective inlets; Flow control and measurement; Sample conditioning effects; Transport losses in pipework; Characteristics of dilution devices; Reactions on and with filters (in polluted air); Treatment of semi-volatile particles.

Measurement of particle number concentration, size distribution, and other properties
Characteristics of condensation particle counters; Characteristics of particle charging methods; Characteristics of electrometer charge counters; Characteristics of electrical mobility size selection; Definition and determination of surface area; Definition and determination of oxidative capacity

Measurement of total mass, and mass of specific components
Factors affecting filter weighing reference methods; Equivalence of on-line or indirect methods for total mass (e.g. oscillating microbalance, beta attenuation, optical); Definition and determination of elemental and organic carbon content; Methods for ionic composition (e.g. ion chromatography); Equivalence of on-line methods for ionic composition; Methods for mercury and other metals.

pH and ionic content
Reducing the uncertainty of the primary Harned cell; Methodologies for on-line measurements in various environments.

SERS
Design and production of optimised nano-structured surfaces; Application of SERS techniques to environmental and other measurements.

Theme Roadmap

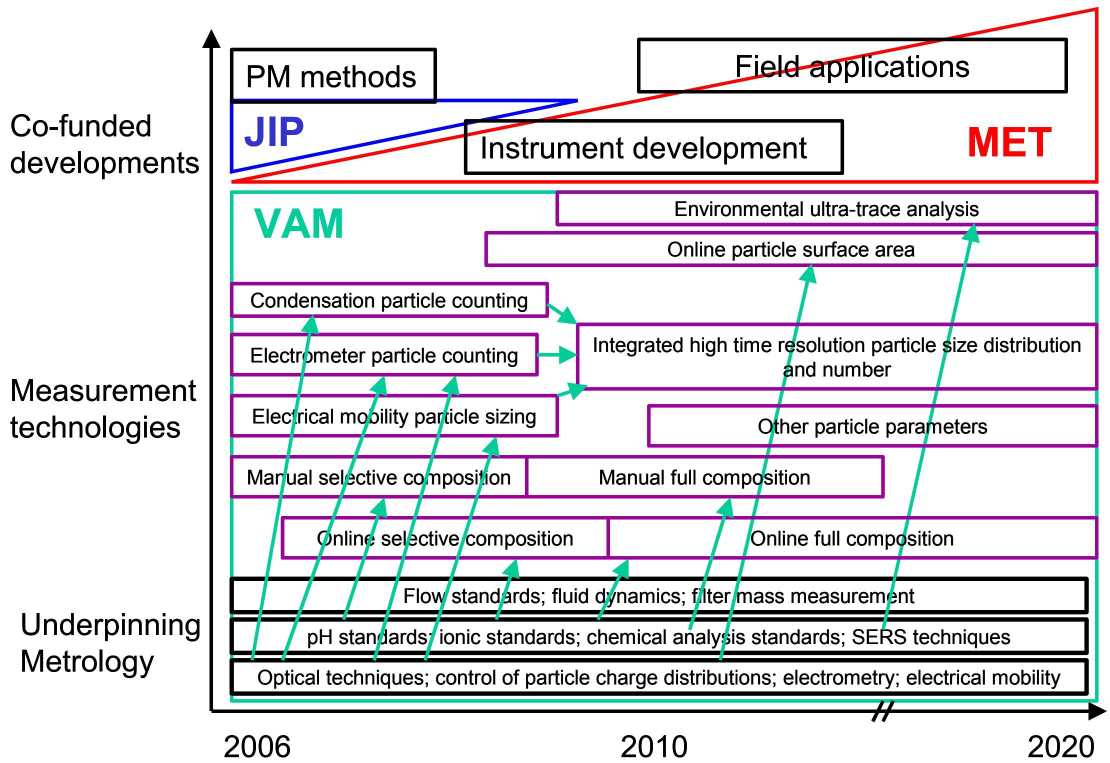

Figure 11: Particulate and trace chemical analysis roadmap showing the strategic development of research within this Theme. It illustrates the feed through of underpinning metrology in VAM through to collaborative industry JIP and MET projects. The timings in the roadmap are indicative and show relative order.

Project P01	Accurate size and number measurements of airborne nanoparticles

Project aim and objectives

The aim of this project is to provide the capability for measurement methodologies, standards and facilities required for robust measurements of airborne particle size and number density.

The objectives of the project are:

- To construct a particle generation mixing chamber for assessing particle monitoring instruments with a variety of size and composition distributions of particles.
- To quantify factors affecting instrument sampling.
- To compare particle counting measurements using different techniques such as condensation particle counting and electrometry.
- To compare size selection parameters for different types of particle using different techniques such as impactors and electrical mobility.
- To investigate the influence of particle charge and size on the performance of Differential Mobility Analysers (DMAs) to assess the validity of physical assumptions and software used in determining size distributions with this method.

Background and rationale

Airborne particles are currently of great scientific interest. They have a major impact on human health, larger than any gaseous pollutant, and they also have a major role in climate change. Primary particles are emitted from natural and anthropogenic sources, notably traffic exhausts, while secondary particles (those formed within the atmosphere) arise from a variety of both natural and anthropogenic precursors. The parameters critical for human health effects are not well established, but emphasis is moving towards smaller particles. For example, European legislation based on total particle mass (Directive 1999/30/EC) is moving from the PM_{10} size fraction (less than 10 micrometers) to $PM_{2.5}$ (less than 2.5 micrometers).

However, it is widely agreed that mass alone is too simple a measure of the presence of particles, and to make useful progress it is also necessary to know the particle size distribution and composition. The emphasis on smaller particles such as nanoparticles (<100nm diameter) means that the number of particles may be a more reliable gauge of health effects than mass, and there are proposals to regulate particle number emissions from vehicles in the Euro V regulations.

Measurement instruments for such particles are commercially available, and in some cases are already deployed at ambient monitoring sites for Defra. However, these measurements are poorly supported within the measurement infrastructure.

This project will create a particle generation facility, versatile both in size range and particle composition, connected to a sampling chamber (rather than a simple outlet tube). This facility will be used to investigate salient measurement issues relating to the robust assessment of particle size, and to develop the production of calibration standards using realistic mixtures of particles in humid air. Best practice will be assessed and disseminated to the relevant communities. The generation facility will be made available for the calibration of instruments.

Common methods to determine the size distribution of nanoparticles, such as Differential Mobility Analysers (DMAs), rely on applying an electric field to the stream of particles. However it is unclear whether assumptions regarding the charge distributions, required for determining sizes, are always accurate. This is especially true at the lowest size ranges. Measurements using these instruments need validation, and accepted measurement protocols. This project aims to investigate the efficiency of DMA columns, particularly at the extremes of their size ranges, considering also how different polarities of particles influence the results.

Drivers for the work in this project can be found in a number of recent, key foresight documents. For example, Defra's current five year strategy ('The essentials of life', 2004) highlights air quality as one of Defra's key priority areas. The document proposes to develop of health-based standards to protect people from the effects of pollutants. Aditionally, the 2004 RS/RAE report, 'Nanoscience and nanotechnologies: opportunities and uncertainties' identified nanoparticles as a priority area for the DTI to support standardisation and measurement through its National Measurement System Programme.

Summary of technical work

A particle generation facility will be developed and validated, providing a mixture of carbonaceous and inorganic particles in air of variable humidity, and at variable concentration, based on equipment procured in the current VAM Programme. This will be incorporated with a sampling chamber so that all aspects of field equipment can be evaluated with laboratory-generated particles similar to those in ambient air. In parallel, operational and metrological aspects of techniques used for ambient particle counting and sizing will be assessed. Specifically, factors affecting the accuracy of counting by both condensation and electrometry techniques, and of sizing by both electrical mobility and impactor techniques, will be quantified, with recommendations for operation and calibration developed.

Common to both electrometry and electrical mobility is an assumed knowledge of the particle charge distribution. This aspect will be specifically investigated for various particle compositions and 'neutraliser' techniques, in collaboration with electrical experts at NPL, with emphasis on the performance of Differential Mobility Analysers.

Deliverables

P01.1 Construction of a particle generation mixing chamber for versatile assessment of particle monitoring instruments.
P01.2 Result of a comparison of sampling, and size and number measurements, for different types of particle using different techniques.
P01.3 Evaluation of the performance of DMA columns and software at extremes of size ranges, and with non-ideal charge distributions.
P01.4 Production of three peer review articles and representation at key European or international conferences.

Project P02	Accurate and traceable mass and chemical composition measurements of airborne particulates

Project aim and objectives

The aim of this project is to underpin measurements of airborne particle mass, and provide metrological support for measurements of organic and elemental carbon.

The objectives of the project are:

- To support UK regulatory measurements of particle mass in the PM_{10} and $PM_{2.5}$ ambient air fractions including standardisation and equivalence procedures.
- To develop valid methods for quantifying the mass of organic and elemental carbon within particulate matter.
- To evaluate reflectivity and transmission based measurements of particles (such as black smoke and non-volatile mass), as low cost indicators for other components of particulate matter, and compare these with existing methodologies.

Background and rationale

Airborne particles are currently of great scientific interest. They have a major impact on human health, but the parameters critical for these health effects are not well established. Emphasis is moving towards smaller particles, for example, European legislation (1999/30/EC) is moving from the PM_{10} to the $PM_{2.5}$ size fraction.

This legislation is based on total particle mass. However, it is widely agreed that mass alone is too simple a measure of the presence of particles, and to make useful progress it is also necessary to know the particle size distribution and composition. The proposed revision to the EU Directives (issued September 2005) includes the requirement to characterise the $PM_{2.5}$ for chemical species including sulphate, nitrate, chloride, ammonium, elemental carbon and organic carbon.

Manual and automatic instruments exist for both the total mass and the speciated composition, and many are deployed in Defra monitoring networks. The issues relating to automatic PM_{10} and $PM_{2.5}$, notably the differences between the widely deployed TEOM instruments and the gravimetric reference method, are well known and reasonably near to a practical resolution. However, the issues are of such importance and the loose ends are sufficiently messy that significant further work is required.

The position regarding speciated measurements is less satisfactory. Elemental and organic carbon is not well defined even at the reference level. This project will address these requirements by underpinning measurements of mass, organic carbon and elemental carbon, and also investigating alternative parameters such as reflectivity and non-volatile mass. Related issues with particulate matter measurements in stacks and from vehicles will also be investigated.

The aim of this project was identified as being of high importance by Defra’s Air Quality Expert Group highlighted in their 2005 report on Particulate Matter in the United Kingdom.

The role of aerosols in climate change has also recently been highlighted in The Intergovernmental Panel on Climate Change 24th Session Report (September 2005).

Summary of technical work

Scientific investigations supporting regulatory measurements of PM_{10} and $PM_{2.5}$ (particle total mass), especially relating to the revision of the CEN standards for these measurands, or to the equivalence of non-reference methods, will be carried out using the existing temperature and humidity controlled facility, or at NPL's monitoring site as required. This is the crucial air quality parameter in Europe, with much current activity, and experience has shown that the ability to respond flexibly to developments is very valuable to UK regulators and manufacturers.

Evaluation of organic and elemental carbon analysis will continue using the reference instrument purchased in the current programme, in collaboration with other interested organisations, using samples taken at NPL and elsewhere, and samples generated at NPL, and making comparisons with Defra Network instruments.

In parallel, methods based on transmission and reflectivity, known to be relevant to elemental carbon, and on non-volatile mass, which removes known uncertainties caused by semi-volatile materials such as ammonium nitrate and some organic compounds, will be evaluated using similar samples.

Deliverables

P02.1 A report detailing research into the measurement science issues surrounding PM_{10} and $PM_{2.5}$ (particle mass), including standardisation and equivalence procedures in support of regulators.
P02.2 Production of collaboratively-developed valid methods for quantifying the organic and elemental carbon components of PM.
P02.3 A report of a study of reflectivity and transmission measurements of particles (e.g. black smoke, and non-volatile mass) as low cost indicators for other PM components.
P02.4 The results of a comparison of Network instruments with EMEP instruments and support of the UK's interest on a relevant CEN TC264 Working Group.

Project P03	Methodologies for accurate ambient measurements of mercury speciation and particulate chemical compositions

Project aim and objectives

The aim of this project is to develop novel analytical methods to perform accurate measurements of mercury vapour and of the chemical composition of ambient air particulates.

The objectives of the project are:
- To develop an accurate measurement methodology for speciated mercury in ambient air to support the UK monitoring network in assessing compliance against forthcoming EU legislation.
- To develop improved methodologies for the measurements of the anionic and cationic composition of ambient particulates.
- To review analytical methodologies for the measurement of oxidative capacity.

Background and rationale

As outlined in the Theme introduction, ambient air particulates are currently of great scientific interest. The metallic composition of particulate matter, covering lead, nickel, arsenic, cadmium and mercury, is required as part of EU Directives 1999/30/EC and 2004/107/EC, but standard methods for mercury are being produced later than for the other metals, and details of its speciation state are required. Data from Defra suggests that particulate mercury emissions from crematoria alone will increase by 65% by 2020. These pollutants are emitted from natural and anthropogenic sources, and are known to be a threat to human health via inhalation, and, especially for mercury, via deposition followed by incorporation into terrestrial and aquatic biosystems.

The proposed revision to the EU Air Quality Directives (issued September 2005) includes the requirement to characterise the $PM_{2.5}$ for chemical species including sulphate, nitrate, chloride, ammonium, elemental carbon and organic carbon. Reference methods for the first four of these (anions and cations) typically involve ion chromatography, but methodologies to characterise fully the chemical composition of ambient air particles and deposited dust are lacking. Improved methods and comparability for these are vital to achieving the broader aim of reliable 'mass closure' of the components of particulate matter, leading to better understanding of particle sources and formation, and more specific assessment of health effects. An understanding of human and environmental exposure to these particles is underpinned by technologies that enable their measurement and characterisation in important exposure pathways.

The need for robust measurements in this area is highlighted by the recent report on Particulate Matter by Defra's Air Quality Expert Group, and the WHO report 'Health Aspects of Air Pollution: Results from the WHO project 'Systematic Review of Health Aspects of Air Pollution in Europe'. Furthermore the Government's Nanotechnology Coordination Research Group's recent report, 'Characterising the potential risks posed by engineered nanoparticles' highlights the 12 areas in which more research is urgently required to better characterised the size, number and composition of small particles and determine the publics likely exposure to them, and the hazards which they pose to human health and the environment.

Summary of technical work

This project will develop and assess novel and accurate analytical methods for the measurement of the soluble fraction, and the oxidative capacity of ambient air particulates and deposited dust. Particular attention will be focussed on the measurement of the anionic and cationic content of ambient air particulates using automatic, and manual, chromatographic methods. This project will also include development of a robust methodology to measure speciated mercury in ambient air. In particular, attention will be focussed on minimising losses of vapour phase mercury in tubing and similar equipment.

Deliverables

P03.1 Production of an accurate measurement methodology for speciated mercury in ambient air

P03.2 Production of a review of analytical methodologies for the measurement of the oxidative capacity of ambient particulates.

P03.3 Development of improved methodologies for the measurements of the chemical composition of ambient particulates.

P03.4 Representation at CEN committee TC264/WG25 (Mercury) and the production of three peer review papers.

Project P04	Fundamentals of aerosol measurement

Project aim and objectives

The aim of this project is to provide underpinning traceability for airborne particle number density measurements and develop methods that can measure characteristics of particles (such as the surface area) that are not readily measurable with existing technology.

The objectives of the project are:

- To provide a framework for traceability of measurements of nanoparticle number concentration.
- To review and investigate methods for measuring the surface area of ambient nanoparticles.
- To develop a prototype device for diluting flows of nanoparticles using NPL's patented 'time division dilution' method.

Background and rationale

It is widely recognised that the biological activity of nanoparticles depends on a range of physical and chemical properties that are challenging to monitor and control during toxicity screening studies [Health effects of particles produced for nanotechnologies, HSE, EH75/6, Dec 2004.]. A priority for research in this field is "to identify suitable metrics and develop the methods which characterise and assess the behaviour and properties of nanoparticles" [Proceedings of the workshop 'Research Needs on Nanoparticles', Brussels, January 2005].

Regulatory work on airborne particles has focussed on gravimetric measurements that report results on a total mass basis (expressed in $\mu g/m^3$). More recently, there has been a strong move towards the use of counting methods that can be used to determine the number density of nanoparticles. These make use of detection systems based on either the condensation particle counter (which measures number density directly) or the electrometer (which measures the charge carried by particles and converts this into a number density). These methods provide the basic detection mechanisms for all types of nanoparticle monitoring, including the detection component in sophisticated instruments capable of sizing sampled nanoparticles. Although these methods provide the basis for measurements used to monitor public exposure, there is no framework for traceability in place for either approach. Consequently, measurements made by these methods are not generally comparable.

Recent research on the toxicology of both engineered and ambient nanoparticles suggests that "biological activity of nanoparticles will depend on physicochemical parameters not routinely considered in toxicity screening studies" [Oberdoester et al, Particle and Fibre Toxicology, 2005 (2) 8]. Progress with research into these issues is limited by a "lack of reliable, affordable and standardised measurement methods for measuring nanoparticle size and shape and characterising nanoparticles (eg composition and surface area)", [Characterising the potential risks posed by engineered nanoparticles, Defra, 2005]. Amongst the most important measurands is surface area (expressed in m^2/m^3) because it most closely represents the capability of particles to catalyse chemical reactions. There are a very small number of instruments in use that are capable of measuring the total surface area of nanoparticles, however, they depend on different physical mechanisms and therefore do not measure the same property. There is therefore a strong requirement for an experimental investigation into the comparability of these techniques.

Summary of technical work

The project will focus on the development of a robust infrastructure providing traceability to the SI system for measurements of numbers of nanoparticles at number densities appropriate to ambient and emission (rather than manufactured) levels. This will involve developing a well-characterised electrometer system with quantifiable efficiency, which can give traceability to the ampere (through a calibrated ultra-low current meter).

The project will extend the range over which traceability is disseminated by developing a novel device for diluting flows of nanoparticles using NPL's patented "time division dilution" (TDD) method. This will provide a new approach to the calibration of laboratory and field sampling equipment, which has the benefits of the "absolute" traceability of the TDD approach.

The second part of this project will review those methods (such as the epiphaniometer) that have the potential for measuring the area of particles. It will also investigate their performance and consider how the requirements for standards for these methods could be met.

Deliverables

P04.1 Facility for disseminating traceable calibration of particle number density.
P04.2 System for providing accurate dilutions of nanoparticle number density for nanoparticles in the size range 2 to 500nm.
P04.3 Report and experimental investigation into the mechanisms for measuring the surface area of nanoparticles and possible methods for providing standards.

Project P05	Standards and valid procedures to underpin measurements of pH and ionic content

Project aim and objectives

The aim of this project is to provide the capability for the UK's link to primary standards of pH and solution conductivity, and develop focussed research into practical pH measurement in on-line environments, particularly fuel cells.

The objectives of the project are:

- To provide the capability for the UK's link to primary standards of pH by undertaking research into the role of the Ag/AgCl electrode in the uncertainty of Harned Cell measurement and to disseminate the results to CCQM's Electrochemical Analysis Working Group.
- To develop suitable methodologies for on-line pH and ion measurement in environments such as the direct methanol fuel cell.
- To participate in a collaborative European project on pH measurement.

Background and rationale

The measurement of pH remains an important and widely undertaken analytical measurement. The development of improved technologies, such as more efficient direct methanol fuel cells, and the production of international standards for on-line water quality monitoring has meant that there is a growing requirement to underpin the validity of these measurements in end-user communities such as the environmental, food, water, nuclear and semi-conductor industries. These industries have pH measurement requirements that are still not fully underpinned by robust measurement procedures and standards. Improved procedures and traceability in these areas will increase process efficiency, improve process control and thus provide substantial economic benefits.

More specifically, accurate pH measurement techniques are required to underpin the measurement of the soluble component of ambient air particulate matter (PM), in order to work towards a full description of the chemical composition and physicochemical properties of these particles. Measurements of pH in this arena are also complementary to ion chromatography measurements of the soluble fraction of PM as they provide a direct indication of the quantity of ammonium salts within the particles. These measurements will also help contribute to a more accurate mass closure model for ambient air particulates.

These pH measurements also require improved underpinning traceability by working to improve the primary method for the measurement of pH. There are still unresolved discrepancies in pH comparisons at the CCQM level and research is required to identify areas for improvement in the methodology.

Improving water quality measurements has been identified as a key priority in Defra's Five Year Strategy, 'Delivering the Essentials of Life'. There is also a pressing requirement from the rapidly expanding fuel cell industry for research into methodologies to perform accurate, diagnostic pH measurements within fuel cell environments, as recognised in the Material

Foresight Report entitled, 'Fuelling a Greener Economy' and prioritised in the EPSRC's 'Research Priorities and Opportunities' document.

Summary of technical work

The technical work in this project will include input into the drafting of a new international standard for the performance of on-line pH measurements. It will also include research into the role of the Ag/AgCl electrode in the uncertainty of Harned Cell measurement by testing the voltage response, drift, and memory effects of alternative Ag/AgCl electrode designs. Additionally, this project will develop suitable methodologies for on-line pH and ion measurement in environments such as the direct methanol fuel cell. This will include development of laboratory-based procedures for measurement, calibration and quality assurance, followed by demonstration in a suitable real environment

Deliverables

P05.1 Participation in Euromet project 843 to develop on-line sensors and analysing equipment for pH measurement in water.
P05.2 A report detailing research into the role of the Ag/AgCl electrode in the uncertainty of Harned cell measurement.
P05.3 The development and publication of suitable methodologies for the on-line pH and ion measurement using glass electrode and ion selective electrodes.
P05.4 Attendance at the Society of Chemical Industry's Electrochemical Technology Group Committee, and the publication of two peer review papers.

Project P06	Ultra trace analytical methods (SERS)

Project aim and objectives

The aim of this project is to develop efficient and reproducible nano-structured surfaces to underpin the use of Surface Enhanced Raman Spectroscopy (SERS) as an ultra-trace analysis tool.

The objectives of the project are:
- To design an optimal metal surface for reproducibly enhancing Raman signals.
- To produce (in collaboration with Imperial College) and test optimised nano-structured surfaces using a suitable micro-/nano-fabrication technique.
- To disseminate the outcomes at meetings of the UK SERS Forum and to produce a peer-reviewed article and a paper at a relevant major European or international conference.

Background and rationale

There is an increasing recognition that ultra-trace measurements that have hitherto only been available in sophisticated laboratory environments may have wide applications to the detection and identification of ultra-low levels of chemical agents released into the atmosphere, and ensuring safe water, food, and medicines, by detecting dangerous or toxic contaminants at ultra-low levels.

SERS shows great promise as an analytical tool for ultra-trace analysis. Despite the availability of a full theoretical description of the SERS process, it is known that more regular and reproducible metallic substrates are needed to achieve reproducible enhancement levels, hence providing a greater possibility of producing meaningful quantitative SERS analysis at the ultra-trace level. There are several drivers for the proposed body of work, including: homeland security (the need to detect and identify ultra-low levels of chemical agents); public safety (ensuring safe water, food, and medicines, by detecting dangerous or toxic contaminants at ultra-low levels); and applications in forensic science where ultra-trace detection is required.

The requirements for these measurements are highlighted in several recent strategy documents, including: Defra's 'Our Strategy 2003-2006', 'Making the UK Safer: Detecting and decontaminating chemical and biological agents' (Royal Society) and also the 'Chemical Vision 2020 Roadmaps' which highlight the need for the development of robust analytical techniques with ultra-high sensitivity, particularly for the analysis of nanomaterials.

Summary of technical work

Successful research at NPL [J. Chem. Phys., 2005 (123) 084702; Chem. Phys. Lett., 2004 (383) 577], and elsewhere, has been based on the use of silver colloids to achieve very large SERS enhancements, but it has not been possible to achieve sufficient stability within colloidal suspensions to make them useful as SERS substrates for trace analysis studies. This work has included the joint organisation of a highly successful Faraday Discussion meeting on SERS with the royal Society of Chemistry. This project will design and develop nanostructured metal surfaces and achieve the correct balance between reproducibility and enhancement to ensure that these surfaces are applicable to quantitative trace analysis using SERS methodologies. These structures will be designed for optimum practical enhancement using computer modelling techniques, fabricated in collaboration, probably with a leading UK university, and then tested for performance in SERS applications. This project will also include the organisation and hosting, by NPL, of a further UK SERS Forum meeting, following on from successful meetings over the last three years, which have provided a focus for UK activities in this area.

Deliverables

P06.1 Design of the optimal practical metals surface for reproducible enhancing Raman signal by electromagnetic modelling procedures.

P06.2 Production of optimum nanostructured surfaces using a suitable micro-/nano-fabrication technique, and initial testing of the surface.

P06.3 Collaboration with relevant academic group to develop and characterise the nanostructured surface for SERS applications.

P06.4 Organisation and hosting of a UK SERS meetings, possibly as a session within an ICORS conference, and also the production of four peer review articles.

6. Surface and nano-analysis Theme

Introduction to Theme

The successful, modern knowledge-based economy is based on growing innovation for the development of high added value products that have a strong competitive edge in the global market place. Central to the correct operation and novel properties of many of these products is the surface chemistry. Surface chemical analytical techniques are key to understanding and characterising these surfaces from the microscale to the nanoscale. The frontline industry-favoured techniques for surface chemical analysis are X-ray photoelectron spectroscopy (XPS), static secondary ion mass spectrometry (SSIMS) and atomic force microscopy (AFM). These complementary techniques provide key quantitative information on atoms and molecules at surfaces, providing high specificity, identification of complex molecules and analytical measurement of materials at the nanoscale.

Work supported under previous VAM Programmes has led to significant benefits in these areas providing revolutionary new methods, major improvements in measurement reproducibility and repeatability, an infrastructure to ensure valid interpretation and fit-for-purpose measurements and frontier research for challenging measurements at the nanoscale. The proposed new programme builds significantly on this, developing key measurements needed by industry and begins the development of strategically important new areas.

Theme aims

The aims of this Theme are:

- To support innovation and competitiveness through the development of reliable leading-edge measurement.
- To develop, strategically, the measurement capabilities and infrastructure to meet the needs of knowledge-based industries and for quality of life.
- To ensure that UK measurements are fit-for-purpose, provide international comparability of measurements and ensure that the measurement infrastructure is in place to underpin systems for accreditation and quality control.
- To provide leadership in frontier issues in surface and nano-analysis measurement in the UK and internationally.
- To conduct key knowledge transfer activities including national and international comparisons, representation on international bodies to ensure international uniformity and traceability of UK measurements and to ensure that knowledge outputs are promulgated effectively and impact the key audiences.

Key drivers

In the recent DTI Innovation Report "Competing in the Global Economy: the Innovation Challenge", Government urges that: “innovation, the exploitation of new ideas is absolutely essential to safeguard and deliver high quality jobs, successful business, better products and services and more environmentally friendly processes".

The DTI's business-led Technology Strategy Board vision [A Call to Action, 2005] is for the UK to be a global leader in innovation and a magnet for technology-intensive companies. UK industry is restructuring from traditional heavy industry, such as processing bulk chemicals etc, to a high added-value, knowledge-based economy. Consequently, there is a major growth in the need for measurement, characterisation and understanding of new complex products.

The UK has major strengths in the new knowledge-intensive areas including, nano- and micro- technology, and emerging technologies. These cover a wide range of industry sectors which rank amongst the highest value-added companies [DTI Value Added Scoreboard 2005]; including, Health and Personal Care (£5.1B), Pharmaceuticals (£22.9B), Chemical Additives (£7.6B) and Aerospace and Defence (£10.4B). The values given above are not sector sizes but the added value. Major sectors in the global economy include Advanced Materials (£200B) and Medical Devices (£115B) [DTI Technology Strategy Board – A Call to Action, 2005]. The annual value for all nanotechnology-related products is estimated, by 2011, to be $1000B [US National Science Foundation].

The innovative technologies in these sectors which rely on surface chemistry are wide-ranging, including drug delivery and controlled release systems, anti-fouling implants, high-efficiency detergents, natural fibre coatings, cosmetics, technical textiles with applications from wound healing to protection from chemical warfare, functionalised microfabricated devices, organic electronics, displays and ink-jet fabrication.

A major goal in medical research is to enhance people's well-being and quality of life. This also reduces the burden on the healthcare system. For many medical devices, the surface molecular chemistry is critical, including drug delivery systems, diagnostic arrays, coronary stents, catheters, artificial implants, wound healing and tissue engineering. The Materials Foresight "Priority topics for biomaterials", highlights the need for surface chemistry, the surface design, molecular orientation and functionality for anti-microbial, anti-thrombogenic and analgesic capabilities. The OST Foresight project "Detection and Identification of Infectious Diseases" highlights the need to characterise surface chemistry for molecular binding and recognition for healthcare applications. The Chemicals Foresight report "A Chemical Renaissance", similarly highlights that the development of pharmaceuticals (including drug delivery) depends on chemistry at the molecular level. This has a major impact on quality of life, over 2.5 million coronary stents and more than 100,000 heart valves are fitted annually but many of these fail to be effective through lack of control of surface aspects. With the age-shift in the population (by 2020 there will be one million more people aged 45 to 64 than in their 20s and 30s [Foresight; "The Age Shift", Dec 2000]), there is a growing and urgent need for long-life implants with improved and new medical devices.

Urgent societal issues are sustainable consumption and production as well as energy efficiency. New materials and processes are enabling end-of-life management and product recycling to be built into product design to reduce waste. Advanced materials and their surface properties are key to many renewable and clean energy technologies including, hydrogen storage, carbon sequestration, fuel cell membranes and photovoltaics. Innovative modern products, for example those using nanotechnology, use less raw materials, have lower energy consumption and produce less waste. The requirement for work in these areas is brought out in the vision for key technology areas, such as Advanced Materials and

Sustainable Production and Consumption, identified in a recent DTI Technology Strategy report [A Call to Action, 2005].

The opportunities and areas for action in Nanotechnology have also been identified in the recent independent Royal Society and Royal Academy of Engineering report "Nanoscience and nanotechnologies: opportunities and uncertainties". A key conclusion that was highlighted is the importance of metrology to underpin nanoscience and nanotechnology. This report specifically recommends the standardisation of measurement at the nano-scale through the National Measurement System portfolio of programmes. The VAM-Physical programme is central to underpinning many aspects of nanoscale metrology development from measurement of nanoparticles to the surface analysis of surfaces, fibres and particles with nanoscale resolution.

Measurement issues

Extensive consultation with industry has been conducted through a series of in-depth, one-on-one discussions with industry experts, BSI and other stakeholders as well as a detailed consultation workshop and survey of measurement needs [NPL Report DQL-AS (RES) 013].

The industry experts identified a growing need for surface analysis work in the UK that has major impact on innovation, global competitiveness, high added-value products, micro- and nano-technology, health and personal care and recommended that growth in this important VAM work is needed to keep up with demands.

Major measurement issues identified by experts include the need for analysis of surfaces with topography, quantitative analysis of compound layers, the development of a measurement infrastructure to support accreditation and quality systems, measurements of micro-scale wettability for microfluidics and ink-jet printing, molecular analysis of functional layers, underpinning measurements for new cluster ion beams, the analysis of delicate materials, quantification of lateral force and optimisation of high-resolution AFM and nanomechanics.

Consultation with industry has highlighted the need for comparable and traceable measurement with strong support for the development of ISO standards as well as commenting on standards proposed outside of the UK, conducting major international comparisons through VAMAS and the development of international comparability in the Consultative Committee on the Amount of Substance.

Another growing need is to provide chemical analysis on surfaces that are not vacuum compatible such as creams for topological application, skin and tissues as well as rapid analyses of pharmaceuticals. In this programme, we thus propose new work on ambient surface mass spectrometry in partnership with LGC. The proposed programme addresses each of these key issues and the strategic direction of the research is shown in the following roadmap.

Theme Roadmap

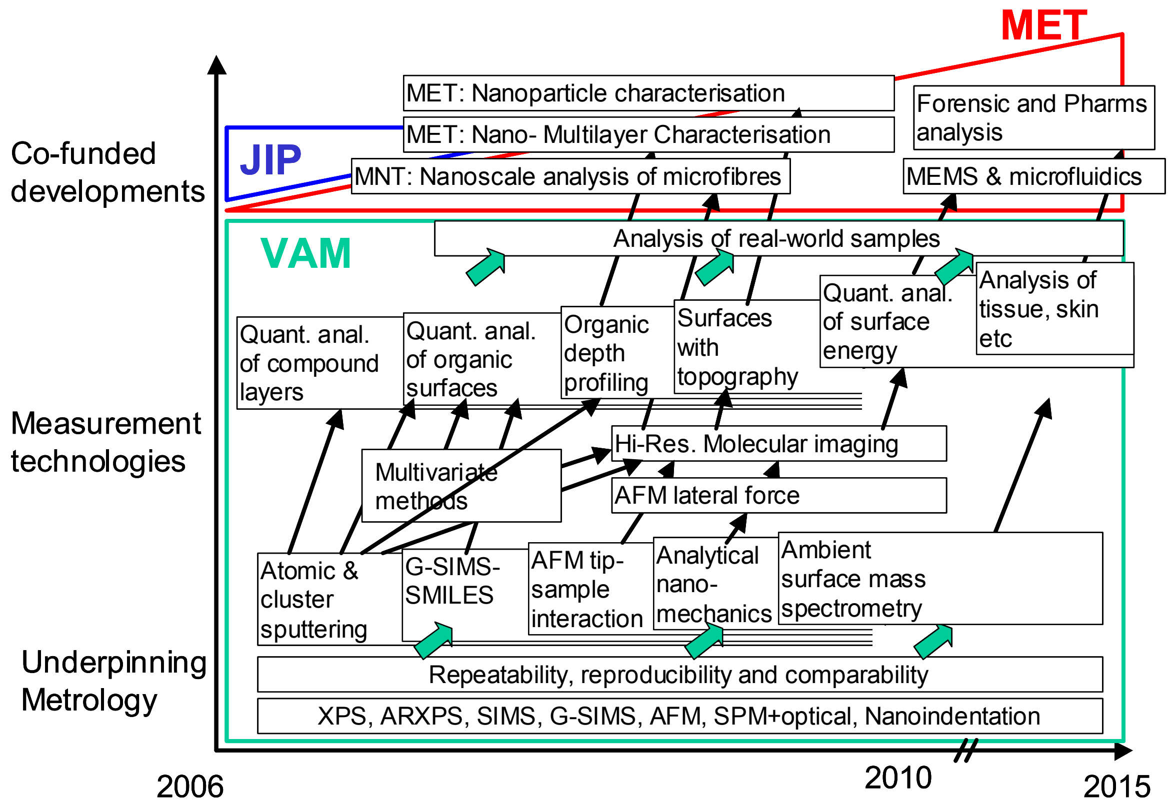

Figure 12: Surface and nano-analysis roadmap showing the strategic development of research within this Theme. It illustrates the feed through of underpinning metrology in VAM through to collaborative industry JIP and MET projects. The timings in the roadmap are indicative and show relative order.

Project S01	Quantitative analysis of surfaces with sub-micron topography – XPS, SIMS, AFM combined study

Project aim and objectives

The aim of this project is to investigate and resolve issues on the effects of surface topography for three principal industry techniques, XPS, SIMS and AFM.

The objectives of the project are:

- To complete a systematic study of the effects of surface topography for XPS and SIMS with AFM to define the effects of topography from flat surfaces to rough surfaces, cylinders and spheres.
- To provide a fundamental method for in-depth quantification of chemical composition, non-destructively by ARXPS and development of the 'thickogram' method for coating thickness measurement from samples with topography;
- The development of procedures and guides to interpret topography effects and reduce these effects for practical analysis.
- To perform quantitative analysis of high information density three-dimensional static SIMS images.
- To develop micro- and nanoscale mechanical evaluation of fibres by scanned probe microscopy (SPM) to allow correlation of mechanical properties with, for example, macroscopic personal care treatments.

Background and rationale

Surface topography is a crucial issue for the analysis of innovative devices such as microfluidic systems, MEMS devices, fibres, composite materials, sensors, organic electronics and biomedical devices. Key recent reports have highlighted the role surface topography plays in the development of innovative new products. A good example is the report, Materials Foresight for Smart Materials, which identifies the performance of coatings, adhesives and composites as highly dependant on surface topography. The associated foresight report, Materials Foresight for Biomaterials, gives as priority topics, surface and interface morphology and interfacial control for drug release and surface design. New technologies which are highly dependent on surface topology such as microfluidics, lab-on-a-chip and MEMS are prominent in almost all current foresight reports. The EPSRC and BBSRC Strategic Plans also highlight the need for the study of the surface chemistries of real surfaces, particles and clusters and the importance of new techniques for the modification and subsequent evaluation of surface chemistry and topography.

Products formulated to control the behaviour of fibres and particles already contribute extensively to the UK's GDP. For example, fabric detergents, hair conditioners and shampoos have global sales of \$77B per year, of which the UK has a >20% market share [Euromonitor]. Healthcare is also a key area (>£9B in UK) with formulated organic coatings for water retention as well as smart fibres for cell growth and wound healing. For particles in both advanced manufacturing and pharmaceuticals, the surface may have anti-agglomeration, anti-static, dispersants and surfactant coatings and for drug delivery systems may have specific controlled release layers and molecular targeting surfaces. In addition, modern automotive and aerospace components are produced from multi-layered carbon fibre structures in an adhesive matrix. The strength and durability of these components is critically dependent on the surface molecular interaction of the fibre and the matrix. Surface chemical analysis of these systems is critical to relate treatments to product performance.

A major issue identified by surface analysts during consultation was that practical samples have important surface topography and are not atomically flat. This surface topography in real life samples causes many unwanted artefacts in surface analysis. These artefacts, introduced into spectra and images, lead to ambiguity, difficulty in interpretation and reduced clarity to process engineers and R&D scientists developing new products and processes. In XPS, topography modifies the equations that would be used to determine a layer thickness. Many innovative products have significant topography including MEMS devices, microfluidic systems, fibres, composite materials, smart materials, sensors, organic electronics and biomedical devices.

Summary of technical work

A systematic study will be made of the effects of surface topography for model systems for the principal techniques XPS, SIMS together with AFM for topography mapping. Model archetypal systems will be developed building from flat surfaces to rough surfaces, cylinders and spheres. This framework will provide the basic understanding to develop procedures and guides to interpret common effects and reduce these effects where possible. The ‘thickogram’ method for XPS for calculating the thickness of ultra-thin layers at surfaces, developed by NPL for flat surfaces, will be extended to cope with the topography of cylindrical fibres, resulting in a method that is just as easy to apply, together with recommendations for fibre orientation, that, if available to the analyst to control, will ensure the most reliable results. Work will be undertaken to improve the quantitative analysis of SSIMS images. A method will be developed for rapid processing of high-resolution raw spectral data in SIMS images using multivariate methods. In addition, methods will be developed for characterising fibres by nanomechanical analysis using AFM. The effect of fibre curvature on nanomechanical measurements will be studied theoretically and experimentally.

Deliverables

S01.1 Development of a model archetypal system building from flat surfaces to rough surfaces, cylinders and spheres.
S01.2 Development of methods for quantitative analysis of surfaces with topography for XPS and SIMS with topography defined by AFM and practical guides for analysts.
S01.3 Development of a reliable depth-profiling protocol and algorithm by Angle Resolved XPS, and thickograms for fibres.
S01.4 Development of a guide to Angle Resolved XPS Depth Profiling as draft for ISO TC201.
S01.5 Development of a protocol for the characterisation of fibres at the nanoscale using nanomechanical analysis.
S01.6 Development of multivariate methods for quantitative analysis of SSIMS images.
S01.7 Knowledge transfer including provision of expert advice to UK industry and academia, key input into BSI C11/60 and ISO TC201, presentations at meetings, website material and the hosting of an NMAET workshop on Fibre Analysis.

Project S02	Quantitative analysis: In-depth composition for surfaces and fibres with radii of curvature >10nm

Project aim and objectives

The aim of this project is to provide surface analysis infrastructure for quantitative in-depth analysis for surfaces and fibres of radii of curvature >10nm.

The objectives of the project are:

- To provide in-depth quantification and compositional analysis of surfaces and fibres with radii > 10 nm using depth profiling XPS (with sputtering).
- To provide in-depth quantification and compositional analysis of surfaces and fibres with radii > 10 nm using SIMS for trace element detection.

Background and rationale

The chemical characterisation of particles and fibres from their surface to their core as well as for surfaces with topography is needed for innovative high added-value coatings, dispersants, anti-statics, medical devices and implants, personal and health care. The requirement for such chemical profiling at the nanoscale is supported by the Chemical Industries R&D Roadmap for Nanomaterials by Design and the recent RS&RAE "Nanoscience and nanotechnologies" report.

This project is key to developing high added-value products, feeding directly into UK innovation and competitiveness. A key example of this is in the personal care and laundry markets where products such as fabric detergents, hair conditioners and shampoos have global sales of $77B per year, of which the UK has a >20% market share [Euromonitor]. In order to maintain and extend the UK's market share of this fiercely competitive global market, high levels of innovation need to be maintained. The leading manufacturers in this area now look at chemical characterisation of fibres on the nanoscale with the intention of understanding the interaction of their products with hair or other fibres. A similar story can be told for other industries in areas where UK industry has a market share that allows it to capitalise on this innovation: chemicals and fibres, biomedical products and materials for improved environmental goods and services. Innovation in this area requires the solving of practical industrial problems such as analysing rough, textured or fibrous surfaces, a great technical challenge on several levels, which needs long-term development of reliable measurement and quantification standards and methods.

Thin coatings are of increasing importance in packaging, printing, organic electronics, and polymer coatings with biomedical applications. The oxidation state of the polymer surface is critical, and the industrial method of choice for determining this is XPS. This work aligns well with the new work item: Guide on Non-Destructive Depth Profiling of Surfaces under ISO TC201 with which this project will be involved.

Summary of technical work

In this project, a method and procedure will be developed to evaluate the sputtering rate for textured surfaces, curved surfaces and nanoparticles. The sputtering process provides the depth profiling for both SIMS (needed for trace elements) and XPS for quantification. For XPS the additional complication of the X-rays, sputter beam and analyser being on different axes and the effect of different sputtering rates for different inclinations will be evaluated to provide recommended operating conditions for practical analysis.

Deliverables

S02.1 Data and method for the effective sputtering rate of textured and curved surfaces.
S02.2 A method to determine the compositional structure of fibres with radii > 10 nm using XPS.
S02.3 A method to compositional structure of fibres with radii > 10 nm using SIMS.
S02.4 Knowledge transfer, expert technical advice and technical inputs provided to UK industry through oral presentations at national and international meetings and peer-reviewed publications. Representation and promotion of the UK view for these topics at relevant international meetings.

Project S03	Quantitative analysis of major constituents in compounds and compound layered structures at surfaces

Project aim and objectives

The aim of this project is to develop methods to allow a general and valid quantification of major constituents in general compounds at surfaces for the development and innovation of materials used in protective coatings, sensors, electronic devices and in nanotechnology.

The objectives of the project are:

- To develop methods, data and an ISO standard for compounds at the outer surfaces of solids.
- To develop methods and data for quantification of contaminated solid surfaces.
- To develop methods and data for quantifying the depths of different layers when sputtering to generate the depth profile.

Background and rationale

Thin and ultra-thin compound layers and their integrity are key across the whole gamut of applications involving protective and optical films, functional and sensor layers and the corrosion and degradation of products. Industrial sectors utilising thin films are diverse and contribute substantially to UK GDP. Key sectors such as Aerospace and Defence (£24B), Food packaging (£9B), Health and personal care (£15B), Pharmaceuticals (£53B) [Figures UK Sales, DTI Value Added Scoreboard 2005] are heavily dependent on thin film coatings. Innovation in thin film coatings has a major impact on product development from increasing shelf life of pre-packed food to regulating the delivery of drugs in the body. The development of improved measurement capability for the composition of ultra-thin and thin films will be an enabler to innovation for the benefit UK high added-value industry. This is borne out by the 2005 Institute of Materials Foresight "Smart Materials for the 21st Century" where the development of material requiring the quantitative analysis of compounds at surfaces and in thin/ultra-thin layers is highlighted. These thin films are also critical for nanoscale technology as emphasised in the metrology challenges in DTI's UK Strategy for Nanotechnology "New Dimensions for Manufacturing" and similarly the Chemical Industry R & D Roadmap for Nanomaterials by Design (2004).

Analyses of these films are conducted mainly by X-ray photoelectron spectroscopy (XPS) using a non-destructive route, for ultra-thin films <10 nm thick, and a sputtering route for thin films between 10 nm and some microns thick. Practical surfaces are contaminated and this affects present quantification procedures for ultra-thin films. Additionally, analysts use a Shirley background in data reduction that current VAM work shows may not be valid for different compounds. Valid rules, algorithms, data and methodology are required to quantify compositions in ultra-thin films. Relevant aspects need incorporation into a new ISO standard. This project will lead to significant improvements across a very wide range of applications and industries.

For layers thicker than the outermost atomic layer, analysis is by sputter removal using energetic ions (Ar^+ or C_{60}^+). Here a lack of known sputtering yields and a general theory of sputtering in compounds leaves analysts with errors in composition and the depth. Definitive data are required to start to put in place these corrections for quantitative analysis in depth profiling compound layers more than 10 nm thick. New methods will be developed based on work developed in previous VAM Programmes for a selected group of compounds.

The objectives in this project are highlighted in the top five of over 50 topics ranked as priority items by ISO in ISO/TC 201/SC 5 and SC 7. They were all very highly rated during the extensive consultation process that has taken place during the formulation of this programme.

Summary of technical work

Compounds are measured at surfaces by XPS across the whole gamut of materials and applications. In practical XPS, these may be clean if analysed *in situ* but are more generally analysed separately and are then covered in a thin carbonaceous contamination that affects the relative intensities of peaks. Furthermore, analysts use a Shirley background to determine the peak area with associated sensitivity factors. New work shows that such methods may not be transferable from compound to compound, and that quantification by this popular method has no validity. In this work, new general methods will be developed and tested for their accuracy on selected groups of compounds for quantification. These methods, correcting for the contamination, and for the relevant sensitivity factors using an improved background will be made available in the usual way as well as on the NPL website. An ISO standard draft will be developed to cover contamination correction.

Compositional structure in compound layers is obtained by XPS with sputtering using argon ions in analysis of materials ranging from sensors to those undergoing corrosion. At present there is no theory, empirical or otherwise to predict, accurately, the yield. Definitive data are needed for model systems to start this process in order to obtain meaningful quantitative analysis by XPS and AES in compositional depth profiling layers more than 10 nm thick involving compounds.

In this work, the sputtering yields of oxides will be measured using both argon and C_{60}. The quantification methods established for AES and XPS in previous work will be extended to include these effects, where possible.

Deliverables

- S03.1 Development of a method and procedure for the compositional quantification of compounds at surfaces.
- S03.2 Development of a method and procedure for the compositional correction for contamination.
- S03.3 Development of a method and procedure for the sputtering yields for oxides for depth scale calibration.
- S03.4 Development of theory for extrapolation and interpolation to other ion energies and other materials.
- S03.5 Participation in national and international standardisation activities, publications, development of a draft ISO standard, presentations at meetings and the production of website material.

Project S04	Development of measurement infrastructure for QC, accreditation and valid measurements by static SIMS

Project aim and objectives

The aim of this project is to develop the essential measurement infrastructure and standards for UK industrial analysts for QC, accreditation and valid analysis by static SIMS.

The objectives of the project are:

- To develop a new draft ISO standard for calibration of the mass scale and progress a draft standard for repeatability and constancy to publication in ISO/TC 201;
- To develop a method and procedure to establish the linearity of the intensity scale;
- To provide a procedure for relative quantification using SSIMS, improved negative ion repeatability and conduct an interlaboratory study.

Background and rationale

High added-value products are key to both economic prosperity as well as improved healthcare. Major growth areas include molecular engineered surfaces for medical devices to improve biocompatibility and anti-fouling properties (for example catheters and stents). The rapidly growing health care and personal care sectors are driving the requirements for laboratories to be accredited and follow quality systems. UK industry experts consulted during formulation and industry and government reports have also identified major growth in pharmaceuticals, functional coatings for personal care applications, diagnostic arrays and point-of-care diagnostics, fragrances, smart packaging, technical textiles, processed foods (barriers to reduce fat penetration and fat content) as well as multilayer laminate systems for aerospace and defence and speciality adhesives. Industry is moving away from costly trial-and-error approaches to more productive analytical solutions.

A number of high profile reports have validated the key message of the importance of surface chemistry to a huge range of new technologies and high added-value products. Amongst these, the DTI report on "New Dimensions for Manufacturing – A UK strategy for Nanotechnology" highlights drug delivery (>$30B global sales), sensors (>$50B potential market) and tissue engineering as three economically leading nanotechnologies where the UK is in a good position to develop market share. The recent Materials Foresight reports provide more detail, highlighting the importance of surface functionalisation and molecular self assembly, smart biomedical materials, interfacial control for controlled drug release and surface design including molecular orientation for adaptive materials and incorporation of anti-microbial, anti-thrombogenic, analgesic capabilities. In addition, the recent RS/RAE "Nanoscience and nanotechnologies" report identifies nanomaterials including thin films and layers at surfaces, nanoparticulates for targeted drug delivery, and dendrimer molecules for coatings, inks, drug delivery and molecular clean-up. These reports identify development of new surface chemical coatings as a key priority, which has to be underpinned by appropriate surface analytical methods and tools. The Chemical Industries R&D Roadmap for Nanomaterials by Design supports this conclusion by explicitly stating the need for high sensitivity tools for chemical characterisation at the nanoscale for essential and enabling R&D.

Static SIMS is a powerful frontline technique for qualitative and quantitative surface chemical analysis by industry. It has excellent spatial resolution of up to 100 nm, sensitivity better than parts-per-million and specificity of the chemical structure at the surface far exceeding other methods. Critical analyses by SIMS can identify reasons for device or production failure saving industry millions of pounds. The gearing of benefit to cost of such analysis is outstanding and so must the reliability and confidence of the data that forms the basis of such important decisions. The VAM Programme has been fundamental to the major improvements in repeatability and confidence in the technique. This project addresses measurement needs, identified by direct consultation through site visits with leading analysts in UK industry. There is an urgent need to develop ISO standards for repeatability and constancy of the intensity scale, for the linearity of that scale and for calibration of the mass scale suitable for use in quality systems and accreditation to ISO 17025. The topics in this project were ranked, by industry, as top in a recent ISO survey of needs.

Summary of technical work

A draft ISO standard will be developed for calibration of the mass scale for TOF-SIMS instruments based on foundation science developed in the 2003-2006 VAM Programme. Additionally, this project will progress a draft standard for repeatability and constancy in ISO/TC 201 to publication as an International Standard. The issues of linearity of the intensity scale and massive sputter yield events will be studied and a method and procedure developed to establish the linearity of the intensity scale using micro-channel plate detectors. The effect of usage of all the ‘stop’ events in a Time-to-Digital-Converter (TDC) by high intensity low mass ions will be studied. The capability to provide relative quantification using SSIMS will be developed and the reasons for poor negative ion repeatability will be studied and a methodology developed to improve repeatability. These methods and procedures will be tested in an interlaboratory study.

Deliverables

S04.1 Development of Standards for ISO TC 201/SC6 Static SIMS (a) calibration of the mass scale for TOF-SIMS instruments and (b) repeatability and constancy of the relative intensity scale.

S04.2 Development of a method to establish linearity of the intensity scale and characterise massive sputter yield events from new cluster ion sources.

S04.3 Development of a procedure for relative quantification using SSIMS, improved negative ion repeatability and testing in an interlaboratory study.

S04.4 Participation in national and international standardisation activities (e,g. key input into BSI C11/60 and ISO TC201), publications, presentations at meetings and the production of website material.

Project S05	Micro- and nano-scale measurement of surface chemistry for surface wettability

Project aim and objectives

The aim of this project is to extend the measurement of wettability, governed by surface composition, into the micro- and nano-spatial regimes in order to facilitate development of microscopic devices and sensors.

The objectives of the project are:

- To relate the traditional methods of the measurement of wettability to the surface analytical methods of X-ray photoelectron spectroscopy (XPS) and Secondary Ion Mass Spectrometry (SIMS).
- To demonstrate that changes in wettability at the micrometre length scale can be reliably inferred from XPS and SIMS analyses.
- To develop new methods using the Atomic Force Microscope (AFM) for high-resolution and in situ measurement where traditional methods cannot be used.

Background and rationale

Microscopic control of wettability is critical within a number of emerging technologies, including microfluidics, microarrays, MEMS and organic electronics. The failure of devices can often be attributed to inappropriate flow of liquid resulting from microscopic flaws in surface properties. These issues and the associated measurement challenges are highlighted in the recent NPL report 'Survey and analysis of measurement requirements for liquid / surface interactions' (in press). The direct measurement of wetting using contact angle methods is generally limited to the mm-scale and requires flat surfaces; wettability in real systems cannot be measured microscopically using such approaches. Microdrop (50 μm) delivery of liquids addresses this problem to some extent, although rapid liquid evaporation can be a problem and sample topography is still an issue. Consequently there is a pressing requirement to develop techniques that determine wettability at the μm-scale on actual devices to facilitate improved device performance and understanding of device failure.

Although direct measurements of wettability with 10 μm or better spatial resolution cannot be made, elucidation of the key chemical components that contribute to surface energy is possible using XPS and SIMS. These surface analytical techniques have the requisite spatial resolution for the measurement need, what is lacking is a reliable protocol for inferring wettability from spectroscopic data. Sample-tip interactions in AFM are governed by similar forces to those that determine wettability and direct measurement of wetting forces with nanometre resolution will be achieved using this approach.

Microfluidics is identified as a key area of development in the DTI Technology Strategy with a projected market of $2B in 2005. This theme is identified in the current Foresight project 'Detection and Identification of Infectious Diseases' and the BBSRC 'Tools and Technology' strategy. The surface characteristics of the microchannels and other components play a major role in the performance of microfluidic devices. Similarly, MEMS devices already command a $11B global market and are identified as another key technology with a high priority in

DTI's technology strategy. The UK is a leader in the OLED field with 'imaging and computer vision' being cited as a key area in the DTI technology strategy. Organic electronics are highlighted in the Materials Foresight Report on 'Functional Materials – Future Directions'.

Summary of technical work

In this work, information from a range of surface techniques that possess microanalytic capability will be related to wettability data. These include: XPS, which has clear quantification and traceability possibilities down to <10 μm resolution; SIMS, which has clear speciation and sub-micrometre resolution; AFM, which has near atomic resolution and clear speciation. Work will progress by equating functional groups identified by XPS and SIMS with contributions to surface energy (e.g. Lifshitz – van der Waals, acid and base contributions from the van Oss – Good – Chaudury formulation) on the mm-scale before progressing to microscopic analysis and comparison with novel micro-drop wettability measurements. AFM analysis with a functionalised tip will also be related to wettability. Ellipsometry will be used for quantification of the layer thickness.

Deliverables

S05.1 Verification of reference surfaces for correlating wettability measurements by traditional methods and the micro-drop method with analysis by XPS, SIMS and AFM.
S05.2 Development of a protocol for measuring microscale wettability by XPS and SIMS.
S05.3 Development of a protocol for measuring nanoscale wettability by AFM.
S05.4 Participation in national and international standardisation activities (e,g. key input into BSI C11/60 and ISO TC201), publications, presentations at meetings and the production of website material. Organisation of a one-day workshop on wettability on the nano- and microscale.

Project S06	Ambient and imaging mass spectrometry

(**Note:** Project S06 is a proposed collaborative project between NPL and LGC. The equivalent project in the VAM-Chemical Programme is Project O3.)

Project aim and objectives

The aim of this project is to begin the establishment of a metrology infrastructure for an important and powerful new ambient mass spectrometry technique Desorption Electrospray Surface Ionisation (DESI) and related methods.

The objectives of the project are:

- To configure a DESI instrument (this will be done at LGC who already have the necessary equipment).
- To develop method and models to evaluate the projectile-surface interaction to develop an understanding of the process.
- To compare the resulting mass spectra with SSIMS, G-SIMS and electron impact mass spectra.
- To evaluate the repeatability of the method for test materials.
- To identify the mass range over which the desorption or 'chemical sputtering' event is effective.

Background and rationale

There is a major requirement for analytical techniques with high chemical specificity and sensitivity that can operate in ambient conditions and in a portable unit from such key UK industries as Pharmaceuticals (£22.9B), Chemicals (£7.6B), Health and Personal care (£5.1B) [DTI Value Added Scoreboard 2005] and other high innovation sectors. In these industries innovation is essential for the UK's competitiveness and prosperity in the global economy. For many of their devices and products the surface chemistry is critical but the samples are not compatible with vacuum based surface chemical analytical techniques. The need for operation in vacuum has inhibited the development of mass spectrometry relating to surfaces for these important products and sectors including direct analysis of pharmaceutical creams, high throughput analysis of tablets as well as the analysis of pharmaceuticals and cosmetics on skin and hair. Many reports, for example Chemical Industry 2020 vision, DTI New Dimensions for Manufacturing, EPSRC Strategic Plan and OST Foresight, identify the need for new techniques and tools for the chemical analysis of surfaces. In addition, the Royal Society report "Making the UK safer" highlights the need for detection of chemical agents by first responders at the scene using mass spectrometry.

In 2004 a powerful new mass spectrometry technique was developed – Desorption Electrospray Surface Ionisation (DESI) [Science 306 (2004) 471]. Whilst a very new technique, its unique capability to provide highly sensitive mass spectrometry of surfaces without the need for vacuum has caused significant interest and rapid development. At least 3 academic groups in the UK have commenced work in this area and two UK instrument manufacturers are actively looking at possibilities for exploitation. In this project, we begin the basic characterisation of the method to evaluate its applicability for industrial analysis and application for forensics, law enforcement and homeland security. Recent publications have illustrated the power of the method to study the elution of ingested pharmaceuticals through the skin, pharmaceuticals as tablets, creams and liquids as well as transdermal patches. Another key application is for the secure environment; excellent sensitivities of better than 1 pg/cm^2 to plastic explosives such as RDX, TNT and PETN, on a variety of substrates have been reported. Clearly, if the technique is to flourish the basic metrology needs to be in place. This work would form part of the VAM forward-looking research.

Summary of technical work

The first stage of this project will be to configure a DESI instrument at LGC using an existing 'nanospray' Electrospray source onto a Q-TOF MS/MS spectrometer. This will be undertaken by LGC with input from NPL so that experiments may be appropriately configured. In order to develop a method and models to evaluate the projectile-surface interaction and develop an understanding of the process; surfaces with molecular assemblies of varying binding energies will be developed to allow the effect of elecrospray source voltage, droplet size and type to be mapped out. The mass spectra from DESI will be compared with cluster-SSIMS and G-SIMS to elucidate the DESI ionisation and desorption process. A similar approach will be used to evaluate the maximum sized intact or large fragment molecules that can be detected using this method.

Deliverables

S06.1 Development of a method and model to evaluate the projectile-surface interaction in order to gain an understanding of the process.
S06.2 Comparison of the resulting mass spectra with SSIMS, G-SIMS and electron impact mass spectra.
S06.3 Identification of the mass range over which the desorption or 'chemical sputtering' event is effective.
S06.4 Knowledge transfer including publications, presentations at meetings and the production of website material.

Project S07	Quantitative depth profiling and cluster sputtering of thin organic layers at surfaces

Project aim and objectives

The aim of this project is to develop organic depth profiling using cluster ion beams.

The objectives of the project are:

- To develop methods for molecular depth profiling of thin organic and biomaterial layers at surfaces.
- To provide essential metrology of sputter yields of organic and biomaterials for cluster beams.
- To define the fluence limit to avoid damage for molecules and delicate materials by electrons, cluster primary ion beams.

Background and rationale

The need for human communication in a global context will drive the requirement for cheaper, flexible, faster, lighter and smaller displays at an unparalled rate. Flexible displays and electronic transistors will have major global impact with the global Flat Panel Display market estimated to be $85 billion by 2006 of which the market for organic LED displays is expected to be worth between $3 billion and $5 billion by 2009 with an annual growth rate of 42% [i-Suppli]. Commensurate with this is the rapid growth in ink-jet printing technology with a market of $14.5 billion. The use of ink-jet systems to cheaply and rapidly create high-resolution molecular assemblies for organic LED displays and organic electronics (including FETs, RFID tags) as well as array diagnostics and sensors is predicted to grow rapidly [Physics World Jan 2006]. In support of this conclusion the Materials Foresight report identifies as top of the list, "organic electronics" as the next revolution and smart labelling and packaging (including RFID) as a key area with major needs for development in surface analysis. For industry, it is essential to characterise the organic multilayer structure so that process technology can be developed and compared with other methods such as spin-casting. Similarly, the characterisation of elution of pharmaceuticals from controlled release layers is essential for drug delivery systems (>$30 billion global market), tissue engineering and medical devices such as coronary stents (>2.5 million stents fitted per year globally). These innovative devices have significant impact on both quality of life and prosperity. The Materials Foresight report on Priority topics for biomaterials, highlights the need for surface and interface morphology and chemistry, the surface design, molecular orientation and functionality for anti-microbial, anti-thrombogenic and analgesic capabilities.

Analogously to the silicon based semi-conductor industry there is a critical need to characterise the chemical composition with depth of complex device structures. In the silicon based semiconductor industry depth profiling by SIMS is the workhorse on-line technique in Fabrication facilities and has been key in the development of the low cost, high performance and high reliability of modern consumer electronics. Hitherto, this depth profiling has not been possible for organics because of primary ion beam damage. Recently, the use of C_{60} to sputter organic materials has opened up these possibilities. This project addresses measurement needs, identified by direct consultation with UK industry through site visits and other formulation activities as well as from input from the NMS Measurements for Emerging Technology Programme.

Summary of technical work

In this project, the metrology and understanding or organic profiling using cluster ion beams will be developed so that industry analysts have confidence and have clear guidance. An understanding of the degradation process for thin organic films is urgently required. The energetics of the collision cascade will be studied using organic (sensor-like) layers for different cluster ion beams for a range of energies and materials. This will be developed into a methodology so that practical guidance for analysts can be provided. In addition, sputter yields for cluster primary ion beams will be investigated. Data and procedures will be developed for measuring sputtering yields of cluster ion beams including metal clusters involving from 1 to 5 atoms as well as C_{60} for organic and biomaterials materials for depths to 1 μm.

There is an urgent need to define a fluence limit for ions and electrons where surface damage limits the optimum resolution obtainable for delicate molecules, organics and bio-molecules. This is especially critical for new cluster ion beams. The traditional static SIMS limit is only valid for atomic ions on robust polymer materials. A method and procedure will be developed for users to minimise the effects, suitable for incorporation into ISO standards.

Deliverables

S07.1 Development of a method and model for cluster primary ion beam sputtering of organic materials.
S07.2 Development of a method and model for cluster primary ion beam sputtering of bio-materials.
S07.3 Sputtering yields for cluster primary ion beams for organic materials – development of theory and experimental data.
S07.4 Development of a method and procedure to define a fluence limit where surface damage limits the optimal resolution for cluster and atomic primary ion beams for delicate materials and biomaterials. Development of a practical guide for analysts for optimal operation – available on the website.
S07.5 Knowledge transfer including publications, participation in national and international standardisation activities, presentations at meetings and the production of website material. Organisation of the 46th IUVSTA Workshop "Sputtering and Ion Emission by Cluster Ion Beams".

Project S08	Quantitative analysis of organic coatings and molecular layers

Project aim and objectives

The aim of this project is to develop key methods for the identification, interpretation and quantitative analysis of organic and molecular surfaces.

The objectives of the project are:

- To provide methods to quantitatively analyse nanometre molecular coatings.
- To develop methods for equivalence and comparability to relate spectra between different cluster and atomic primary ion beams.
- A novel method to determine molecular structure using G-SIMS with SMILES.

Background and rationale

The top ranking high added value UK industries include Pharmaceuticals (£22.9 billion), Chemicals (£7.6 billion), Health and Personal care (£5.1 billion), Processed Food and Beverages (£30.8 billion) and Aerospace and Defence (£10.4 billion) [DTI Value Added Scoreboard 2005]. These high added value and high innovation sectors are essential for the UK's competitiveness and prosperity in the global economy. Surface molecular chemistry is critical to a very wide range of devices including drug delivery systems, medical devices, wound healing and tissue engineering, diagnostics, smart materials, packaging and personal care products. The Materials Foresight report – Priority topics for biomaterials, highlights the need for surface and interface morphology and chemistry as well as the surface design, molecular orientation and functionality for anti-microbial, anti-thrombogenic and analgesic capabilities. Developments in these areas will have a major impact on the economy as well as quality of life with over 2.5 million coronary stents and more than 100,000 heart valves fitted annually and by reducing the current figure of over 1000 deaths annually from bacterial infection of catheters. The NHS alone spends more than £450 million per year surgically replacing urinary catheters. Further requirements for the analysis of organic layers can be found in the OST Foresight report which highlights the need to characterise surface chemistry for molecular binding and recognition for healthcare applications and also in the Chemicals Foresight report – A chemical renaissance that highlights the development of pharmaceuticals (including drug delivery) which depend on chemistry at the molecular level. The EPSRC strategic plan also flags the need for determination of chemical structure.

Static SIMS is a powerful frontline technique for surface chemical analysis by industry. It has excellent spatial resolution of up to 100 nm, sensitivity of better than ppm and specificity of the chemical structure at the surface far exceeding other methods. Consultation with industry through site visits as well as a formulation workshop has clearly shown that the analysis of functional surfaces with sub-monolayer and nanometre molecular films is a growing requirement for many sectors. New cluster primary ion beams have revolutionised the capability for the analysis of molecular and complex organic surfaces by static SIMS. At present, the benefits or otherwise of using cluster beams (Bi_n and C_{60}) are not clear. This project will develop the urgently needed measurement base for the analysis of organic and molecular layers from 0 to 100 nm to provide the essential foundations for interpretation. A wide range of cluster primary ion beams are now in use in industry and academia including $Bi_{3,5,7}$ and $Au_{1,2,3}$ as well as C_{60}. Relative spectral intensities vary significantly between cluster and atomic primary ion beams and between different types of cluster ion beams. This behaviour needs to be characterised so that spectral data can be compared between different instruments and with existing data in libraries. The identification of complex molecules and their molecular structure is critical for many of the high added-value industries discussed above. The molecular structure is needed since the mass alone cannot give unique identification. G-SIMS-FPM has been shown to be a powerful method to evaluate the molecular structure of complex molecules at surfaces. This provides identification where mass alone is insufficient to determine the molecule.

Summary of technical work

A key part of this project is to advance the analysis of nanometre molecular coatings by investigating the effect on the ion intensities of different atomic and cluster primary ions (Bi_n and C_{60}) and different energies will be characterised. A framework will be developed to understand the differences providing guidance for optimal use. For this, there are two key requirements. The first is to improve understanding of the benefits or otherwise of using cluster ion beams. The second is the need to establish the information depth for small atomic secondary ions as well as larger molecules through sub-monolayer and nanometre films.

A wide range of cluster primary ion beams are now in use in industry and academia including $Bi_{3,5,7}$ and $Au_{1,2,3}$ as well as C_{60}. Relative spectral intensities vary significantly between cluster and atomic primary ion beams and between different types of cluster ion beams. In this work, the effect on the ion intensities of different atomic and cluster primary ions (Bi_n and C_{60}) and different energies will be characterised. A framework will be developed to understand the differences providing guidance for optimal use.

A new NPL SMILES (Simplified Molecular Input Line Entry Specification) fragmentation system provides a framework to determine molecular structure at surfaces when matched to G-SIMS data. Throughout mass spectrometry there are no, or inadequate, automated systems for interpreting molecular structure from mass spectra. This may also have applicability in a wider range of mass spectrometry techniques.

Deliverables

S08.1 Quantitative molecular analysis of sub-monolayer and nanometre molecular films (0-100 nm) using cluster ion beams $Bi_{1,3,5}$ and C_{60}.

S08.2 Characterisation of the changes in spectral intensity with different cluster ion beams and the development of a practical guide for analysts to select the optimal primary ion beam configuration for different analytical systems.

S08.3 Development of NPL's SMILES (Simplified Molecular Input Line Entry Specification) fragmentation system to provides a framework for an automated systems for interpreting molecular structure from mass spectra.

S08.4 Validation of the SMILES method on a range of complex molecules.

S08.5 Assembly of a SMILES fragment architecture library for determination of molecular structure.

S08.6 Knowledge transfer activities including publications, participation in standardisation activities, presentations at meetings and the production of website material.

Project S09	Practical analysis of delicate materials

Project aim and objectives

The aim of this project is to develop key methods for analysts for analysis of organic and delicate surfaces with modern industry cluster ion sources.

The objectives of the project are:
- To write practical guide to the use of multivariate methods for static SIMS data.
- To define the fluence limit to avoid damage for molecules and delicate materials by electrons.

Background and rationale

The analysis of organic surfaces is important for modern industry and there is a strongly growing demand for analysis of delicate materials. This project will benefit UK industry in healthcare, personal care and high added value products where the analysis and location of molecules and components at surface is critical for correct device functionality and performance. This includes medical implants, anti-fouling catheters, controlled release coronary stents, drug delivery systems, pharmaceuticals, polymer additives, organic electronics, functional and active coatings, smart materials, packaging and personal care products.

This project addresses measurement needs, identified by direct consultation with UK industry, required for high added value industry essential for wealth creation and health care. The requirement for this work is supported by the DTI Report – 'New Dimensions for Manufacturing: A UK Strategy for Nanotechnology' which identifies the need for chemical analysis at less then 100nm resolution and the development of analytical techniques to give the highest possible resolutions, greatest sensitivities and ultimate detection limits as critical research topics. The types of measurements to be developed are crucial to give chemically rich information about delicate materials such as cellular materials, fibres, biomaterials, organics, value added functional materials and nanoparticles at the nanoscale. These advanced materials need development of underpinning metrology projects, such as micromeasurements for multifunctional materials and systems and rapid, reliable multi-property analysis techniques as suggested by the NMS Measurement for Emerging Technologies Programme. The 'US Chemical Industry Vision2020 R&D Roadmap for nanomaterials by design: From fundamentals to functions' also includes nanoparticle surface structure and chemical composition as a key challenge. This project helps address many of these key issues.

There is strong interest and demand for analytical techniques to condense large datasets and extract information quickly. EPSRC highlight the need for analytical science using chemometrics to push the limits of measurements available. UK industry analysts are beginning to take advantage of multivariate methods but there is still much that remains mysterious and mythical. A practical guide is urgently needed and was ranked highly in industry consultations and the consultation (SAMS) workshop.

Summary of technical work

In this work a practical guide to multivariate methods for the data reduction of spectra will be developed, identifying when the methods are appropriate for use and giving recommendations for the effects of data normalisation. A workshop will be held to improve the uptake and confidence of these methods in UK industry.

Additionally, work will be undertaken to define a fluence limit for electrons where surface damage limits the validity of spectra, repeatability and the optimum spatial resolution obtainable for delicate molecules, organics and bio-molecules. A method and procedure will be developed for users to minimise the effects, suitable for incorporation into ISO standards.

Deliverables

S09.1 Development of a practical guide to the use of multivariate methods for identification and interpretation of SSIMS data.
S09.2 Evaluation of the effect of electron beam damage on delicate materials and polymers and provide analysts with a guide and procedures to minimise this.
S09.3 Knowledge transfer including publications, presentations at meetings and the production of website material. Organisation of a workshop or tutorial on multivariate methods for analysts.

Project S10	Quantification issues in lateral resolution and lateral force in AFM

Project aim and objectives

The aim of this project is to develop measurement methods to resolve quantification issues in analytical measurement of surfaces by atomic force microscopy (AFM).

The objectives of the project are:

- To provide traceable calibration of lateral force in AFM for the first time.
- To develop a method to calibrate AFM tip shape and provide a procedure.
- To evaluate the imaging capabilities, resolution and durability of carbon nanotube AFM tips.
- To investigate the resolution of force-distance mode.

Background and rationale

The recent RS/RAE report on Nanoscience and nanotechnologies highlighted the importance of metrology that underpins these emerging areas of science. It recommends the standardisation of measurement at the nano-scale through the NMS portfolio of programmes. This is supported by the EU commission strategy for research funding “Towards a European Strategy for Nanotechnology” and the DTI's UK Strategy for Nanotechnology "New Dimensions for Manufacturing" where analysis at sub-100 nm resolution is described as a key metrology challenge. The recent EPSRC Research priorities and opportunities report also highlights work in this area as essential. AFM can potentially offer the high-resolution imaging and analysis required with lateral force AFM providing high-resolution chemical composition of the first atomic layer at resolutions approaching 10nm. These measurements are essential for new nanotechnology products in for example, health and personal care, sensors (>$50 billion potential market) and drug-delivery (>$30 billion global sales).

The US “Chemical Industry R&D Roadmap for Nanomaterials by Design: From Fundamentals to Functions” states a key challenge as the development of nanoprobes capable of measuring physical and chemical properties where reproducible inter-probe performance is needed. Essential components to address these issues is the requirement for a method to characterise AFM probe tips, along with the assessment of reliability of new carbon nanotube AFM tips, which offer unsurpassed resolution. Further requirements are expressed in the Materials Foresight report on priority topics for future biomaterials development that identifies the characterisation of the surface and interface morphology and chemistry as important for future technology developments. Force-distance mode AFM can potentially give some of the required analytical and characteristic parameters. Work in this project will aim to determine the resolution of this technique when used to analyse interfaces and coatings.

This project will underpin future metrology in VAM, enabling fundamental improvements in quantitative AFM measurement. Currently, these measurements suffer from poor repeatability and difficult-to-use calibration methods, which will be addressed in this project.

Summary of technical work

This project will address four critical issues in this area.

The first is lateral force measurement by AFM. These measurements are extremely sensitive to information on chemical composition of the first atomic layer at resolutions approaching 10nm. Nevertheless these measurements have been plagued by poor repeatability, absent traceability and difficult-to-use calibration methods. The previous VAM Programme developed a prototype MEMS device capable of quantifying the lateral force of an AFM cantilever quickly and traceably. In this work, the efficacy of this device will to be established via an interlaboratory study.

The second issue to be addressed is the tip shape and size. These are the main sources of uncertainty in nearly all aspects of AFM from high-resolution imaging through to materials properties measurement. However, the tip shape is very difficult to characterise at the required level. In this work, new methods will be developed, analysing samples of known shape at the atomic level using, for example, crystal features or molecular structures. Procedures and guidelines will be developed leading to future draft ISO standards.

The third part of the project will look at using carbon nanotubes as AFM tips. Carbon nanotubes have long been cited as being able to provide a step change in AFM imaging quality by offering high resolution and well defined tip radii, however problems over reliability, life-time issues, sturdiness and fabrication methods have hindered their uptake. These will be investigated here.

The final part of the project will be to investigate the resolution of AFM measurements in force-distance mode. Force-distance curves using AFM give information on the mechanical information of the near surface region at the nanoscale, however, the spatial resolution of this mode is not well defined. Here, model systems consisting of two materials of different moduli will be analysed both experimentally and theoretically to define this. A guide for UK analysts will be produced for optimisation and characterisation of the resolution in force-distance microscopy.

Deliverables

S10.1 Interlaboratory study of lateral force calibration, refereed publication as the basis for a possible ISO draft.
S10.2 Development of structures to calibrate AFM tip shapes and development of a procedure to calibrate AFM tip shape for use as a future draft ISO standard.
S10.3 Investigation of and recommendations for carbon nanotube tips for use as AFM probes.
S10.4 Investigation of the resolution of AFM force-distance mode.
S10.5 Knowledge transfer including publications, key input to ISO TC201 and TC229, presentations at meetings and the production of website material.

Project S11	Optimisation of operating conditions for high-resolution AFM nanomechanics measurements

Project aim and objectives

The aim of this project is to develop measurement methods in order to resolve quantification issues using AFM to optimise operating conditions, improve reproducibility and additionally of the measurements.

The objectives of the project are:

- To improve the reproducibility and consistency of normal force and modulus measurement at the nano-scale via an interlaboratory study.
- To optimise operating conditions for force modulation microscopy and phase imaging.
- To develop methods for depth profiling using AFM.

Background and rationale

Atomic Force Microscopy (AFM) has emerged strongly from industry consultations as of increasing importance in surface and nano-analysis, even relative to the increasing importance of surface analysis as a whole. The reasons are technological (ambient analysis, and very high-nm scale-spatial resolution) and economic (relatively cheap and compact). Growth in applications is inhibited by the present very modest metrology infrastructure that needs development. The growth areas where innovation plays a key role are in high value-added products such as MEMS devices, microfluidic systems, sensors, diagnostic arrays, organic electronics and fibre treatments which rely on surface structure and chemical composition on the micro and nano-scale. Major UK industries such as Pharmaceuticals (£22.9 billion), Chemicals (£7.6 billion), Health and Personal care (£5.1 billion) and Aerospace and Defence (£10.4 billion) [DTI Value Added Scoreboard 2005] are increasingly dependent on product innovation on the nanoscale for competitive advantage in the global economy. However, analysis at sub-100 nm is still a key metrology challenge as identified in the DTI's UK Strategy for Nanotechnology "New Dimensions for Manufacturing". The recent EPSRC "Research Priorities and Opportunities" report also highlights this requirement. AFM is one of the only techniques, which can successfully address these measurement challenges at the high resolution required.

The 2004 Royal Society and Royal Academy of Engineering report – "Nanoscience and Nanotechnologies: Opportunities and Uncertainties" recommends supporting the standardisation of measurement at the nanometre scale required by regulators and for quality control in industry under the National Measurement System portfolio of Programmes. The government response, published in 2005, stated that the Government will continue to support the NMS working on the development of traceable methods related to dimensional, chemical and functional aspects of nanotechnologies. This is essential since the annual value for all nanotechnology-related products is estimated to be $1000 billion by 2011-2015 [US National Science Foundation]. Building on the Royal Society report, the Micro- and nano-technology (MNT) Roadmap in Metrology, published in September 2004 stated that the UK should become world leaders in MNT measurement solutions, with the base at NPL being well established to achieve this goal. The use of advanced metrology such as AFM to support high-value nano-manufacturing is a key recommendation from this roadmap.

Summary of technical work

This project will address the optimisation of operating conditions of several AFM modes for high-resolution nanomechanical measurements. VAM has previously developed effective methods to measure modulus using AFM but issues identified during consultation with industry need resolving using an interlaboratory study. This will include the novel NPL electrical Nanobalance for calibrating the AFM normal spring constant.

Surface sensitivity is greatly increased using dynamic modes of AFM operation. Force modulation and phase imaging can provide quantitative nanomechanical information, uptake of this method has been hampered by a lack of any metrology infrastructure. Here, guidelines will be developed for the analyst to optimise the instrumental set-up for quantitative force modulation mode and phase images and to evaluate the associated uncertainties. In the allied area of nanoindentation, rules have been imported from microindentation that previous VAM work has shown can be grossly in error at the nanoscale. This needs to be analysed in more detail for AFM users. In this work, the depth that the AFM senses into a surface using force modulation mode and phase imaging will be investigated. A model and analytical equations will be developed, tested and refined for the analysis and characterisation of ultra thin organic layers by AFM. The analytical equations developed will then be compared with experimental data to monitor when substrate properties become apparent.

Deliverables

S11.1 Development of a procedure for and carry out an interlaboratory study of normal force / modulus leading to a future draft ISO standard.
S11.2 Development of a method and procedure to determine the optimal parameters for quantitative force modulation and phase imaging.
S11.3 Development of a method and procedure for depth sensing using force modulation mode.
S11.4 Development of a method and procedure for the analysis of thin layers and coatings on substrates by AFM.
S11.5 Knowledge transfer including publications, key input to ISO TC201 and TC229, presentations at meetings and the production of website material.

7. Management and knowledge transfer Theme

Introduction to Theme

Key to the success of any programme of work is the effective management and co-ordination of projects and activities within the programme. The management project in this Theme ensures the cost-effective delivery of all projects along with regular reporting of progress. Associated with the management of the VAM-Physical Programme is the need to coordinate activities with the contractors delivering the VAM-Chemical half of the VAM Programme to ensure seamless delivery. Work in this Theme will coordinate both technical and knowledge transfer activities between the two sides of the VAM Programme. Another key aspect of this Theme is the organising and managing of the transfer of all VAM-Physical supported facilities and services to new, purpose designed, laboratories which will occur during the lifetime of this Programme.

Dissemination of the knowledge developed and other outputs of the Programme to beneficiaries is of major importance to maximise the value added to the UK economy and society by work in the Programme. The majority of knowledge transfer is tightly bound up in the delivery of the outputs of specific technical projects. Most of the knowledge transfer and exploitation in this Programme is therefore associated with each technical project where particular audiences can be targeted. Typical activities in these projects might include: publications, presentations, participation in specific national or international standardisation activities, internet related delivery, best practice guides or measurement protocols, handling and answering enquiries etc. Work in this Theme will ensure the coordination and effectiveness of project level knowledge transfer to ensure best practice is employed and developed in all aspects of knowledge exploitation.

While maximum benefit is often gained through dialogue and knowledge exchange with particular audiences at the project level, there is also a requirement to address a broader community which is the role of knowledge transfer activities within this Theme. Ensuring the impact of technical work within the VAM-Physical Programme reaches policy makers and other key stakeholders is an important feature along with representation and supporting of UK interests on major international bodies that directly affect UK industry, government, regulators and other end users.

In order to determine the future direction of work in the VAM-Physical Programme a dialogue with beneficiaries, technical experts in industry, government, regulators and other policy makers is continually underway. This knowledge exchange may well change the direction and content of the Programme during its lifetime if new, important requirements emerge. These activities will intensify mid-way through the Programme in order to focus on formulation of the next VAM-Physical Programme including the development of proposals of work to address future UK measurement requirements.

Theme aims

The Management and knowledge transfer Theme has the following aims:

- To manage the Programme successfully, thus ensuring cost-effective delivery.
- To ensure a successful decant into NPL's new laboratory facilities.
- To formulate proposals for the 2009-2012 VAM-Physical Programme.
- To maximise the impact of the VAM-Physical Programme through high-level knowledge transfer activities.

Project M01	Programme management and decant

Project aim and objectives

The aim of this project is to ensure effective coordination of the VAM-Physical Programme across all projects and contractors so that the Programme delivers maximum benefit to the UK. In addition, this project has the aim of successfully managing the decant of facilities to new laboratories such that the minimum of disruption occurs.

The objectives of the project are:

- To ensure cost-effective delivery of the Programme.
- To coordinate activities between VAM-Physical and other NMS Programmes.
- To provide reports on progress on a monthly and annual basis.
- To successfully decant all facilities and projects to new laboratories.

Background and rationale

The VAM-Physical Programme consists of a number of projects whose delivery is dependent on efficient use of resources and facilities. The complexity of the technical and knowledge transfer activities will require close monitoring of progress and a flexible approach to resourcing. This project coordinates activities in the Programme to ensure that all milestones are completed in a timely and cost-effective manner and that all reporting requirements to the DTI and its advisors are met.

The VAM-Physical and VAM-Chemical Programmes are delivered by different contractors although they share several key dissemination vehicles such as the VAM Bulletin and the VAM website. An important part of this project is to ensure full collaboration and cooperation such that the Programme appears seamless to user communities. Effectively achieving this goal will give maximum impact from the KT activities in the VAM Programme and ensure that coordination between technical projects on both sides of the VAM Programme deliver optimum benefit.

During the lifetime of this Programme the facilities and projects that comprise the VAM-Physical Programme will move to new laboratories. Such a move is a major undertaking, requiring careful coordination as well as supporting the actual decant and setting up of facilities in new laboratories. A substantial part of this project will support the move to new laboratories.

Summary of work

This project will ensure that regular meetings occur between the major contractors on the VAM-Physical and VAM-Chemical Programmes to ensure seamless and effective delivery to the user communities addressed by VAM. A significant part of the work in this project will be to ensure timely and cost-effective of all milestones in the VAM-Physical Programme. Reports on progress will be provided monthly along with invoices to the DTI. On each anniversary of the start of the Programme an annual report on progress will be provided for the VAM working group as well as the DTI.

The successful move of facilities supported by the VAM Programme to new laboratories will be a major objective of this project in this Programme. Work in this project will support and oversee the dismantling, moving and re-build and testing of facilities.

Deliverables

M01.1 Coordination of regular meetings of the major contractors delivering the VAM-Physical and VAM-Chemical Programmes to ensure seamless and effective Programme delivery.
M01.2 Report progress in the Programme to the DTI through a series of monthly invoices and summary reports.
M01.3 Provide an annual reports of progress in the Programme to the DTI and the VAM Working Group.
M01.4 Ensure the successful and timely decant of all VAM-supported facilities to NPL's new laboratory.

Project M02	Formulation of the next VAM-Physical Programme

Project aim and objectives

The aim of this project is to produce a proposal of work for the next VAM Programme through consultation with industry, regulators and other end users in order to determine future measurement requirements or other issues that need to be addressed.

The objectives of the project are:

- To conduct a wide ranging consultation exercise with industry, regulators, government and other end users to determine current and future measurement requirements.
- To draw together all inputs received during formulation into a new Programme of work that will form the next VAM Programme.

Background and rationale

As the UK economy evolves and technology trends develop, new measurement requirements emerge. To underpin growth in the economy these measurement requirements must be successfully addressed as early as possible. Also new regulatory controls on the environment, health and transport demand new or better measurement techniques. In order to determine the present and future measurement requirements of industry, regulators, government and other end users, it is important that their views are sort through a wide ranging consultation process. This process includes looking at independent evidence of measurement needs as expressed in government reports, foresight activities, industry roadmaps etc. as well as conducting meetings, surveys and interviews focusing on specific topics of interest. Collation and assessment of information from all sources enables a list of activities to address the measurement needs to be formulated. An independent prioritisation of these activities enables the optimum programme of work to be developed which will maximise the impact of available funding on UK measurement requirements.

Summary of work

This project will ensure the successful formulation of the 2009-2012 VAM Physical Programme through staging a wide-ranging consultation exercise with key end-users in UK industry, government and other bodies.

The direction and strategy of the Programme will be determined by a ‘trend spotting forward look’ review of measurement requirements and a subsequent Orientation meeting. Technical projects will then be developed and presented to the VAM Working Group at its Annual Review. After the scope of the Programme has been focussed further, the remaining portfolio of projects will be presented for public comment before being prioritised by the Working Group. The projects selected by the prioritisation process will comprise the final Programme, which will developed into a form suitable for ministerial approval before commencement of the technical work of the Programme in October 2009.

Deliverable

M02.1 Production of a proposal of work for the 2009-2012 VAM-Physical Programme.

Project M03	Dissemination and representation

Project aim and objectives

The aim of this project is to maximise the impact of the VAM-Physical Programme through high level knowledge transfer activities and to represent UK interests through participation in high level metrology and standardisation committees for the benefit of UK industry and regulators.

The objectives of the project are:

- To support organisation of and participation in key science conferences that have a broad scope, covering the interests of a technical area or the whole Programme.
- To manage active and relevant contributions to high level national and international standardisation bodies and aid in formulating UK views to the benefit of UK industry.
- To represent UK interests on international measurement committees.
- To achieve high impact knowledge transfer through support for secondments.

Background and rationale

Knowledge transfer (KT) is the vehicle through which the knowledge and benefits developed in the VAM Programme are disseminated to industry, government, regulators and other end users. As such it is an essential component of the Programme. KT activities are embedded within each technical project for maximum effectiveness in transferring knowledge generated in projects to the relevant user communities. However, there are several, cross-cutting, KT activities that are essential in order to gain the maximum benefit from the VAM Programme. A key role for the VAM Programme is the support for representation of UK interests on many international measurement and standardisation committees such that UK industry and government benefits from: the prevention and reduction of trade barriers (support for the MRA); better informed regulation; and the harmonisation of industry standards. The benefit of these activities is demonstrated in a recent DTI report 'The Empirical Economics of Standards' which showed that standardisation activity had contributed around 13% of post-war productivity growth in the UK. This project will cover relevant participation in the work of the high level and influential CCQM and EUROMET metrological committees as well as supporting work in top level technical committees under the auspices of ISO, CEN and VAMAS.

Much of the work of the VAM Programme is disseminated through presentations and papers at both national and international meetings and this work is largely included within technical projects. However, there are a couple of events that cut across many different projects and as such they sit outside any individual project. The two events are firstly the ISO Gas Symposium which is a high profile, high impact, international meeting in the area of gas standards. The second event is the highly successful and well regarded, Nano-Molecular Analysis for Emerging Technologies meeting. Both of these events are highly effective dissemination vehicles for knowledge generated in the VAM-Physical Programme.

One of the key mechanisms for developing strong links and maximising knowledge transfer is the use of secondments to and from projects in the VAM Programme. A secondment to another organisation, such as another research institute or a commercial company creates a conduit where measurement best practice can be transferred along with new technological innovations, both of which benefit the participating organisations. Of longer-term benefit are the contacts made and the potential for collaborative projects into the future.

Summary of work

The majority of KT in the VAM Programme is embedded directly in technical projects as it is directly related to the work within a project. However, there are a few KT events, such as some conferences, that cover a broad range of activities in the Programme. One of these is the ISO Gas Symposium and another in the Nano-Molecular Analysis for Emerging Technologies (NMAET) conference. Both of these meetings have previously been extremely successful and have demonstrated substantial impact. This project will support participation in one ISO Gas Symposium and organisation of two NMAET conferences.

High level international committees such as: CCQM plenary and its working groups; ISO and CEN Technical Committees (TCs); and BSI EH2 and PTI/015 committees are core metrology activities for the VAM Programme. This project will support UK representation on the high level CCQM committees and TC level work under ISO, CEN and BSI. Work on individual working groups of CEN and ISO is covered under each technical project. One of the most beneficial mechanisms for achieving high impact KT both into and out of NMS Programmes is the secondment of key staff to other institutions and companies. This project will support a small number of secondments where support is not available from other sources. Impact will be demonstrated through a minimum of three case studies per year demonstrating how VAM-Physical outputs have been implemented in industry or the wider user community.

Deliverables

M03.1 Support the organisation of, and participation in, key conferences that have a broad scope (covering the interests of a technical area or the whole Programme).
M03.2 Ensure that active and relevant contributions are made to high-level national and international standardisation bodies. Aid in formulating UK views which will benefit the nation’s industry.
M03.3 Representation of the UK’s interests on international measurement committees
M03.4 Achievement of high impact knowledge transfer of the VAM-Physical Programme through support for secondments.
M03.5 Annual reporting of KT activities in the VAM-Physical Programme and three case studies per annum.

REFERENCES

[1] Nanoscience and nanotechnologies: opportunities and uncertainties, Royal Society and Royal Academy of Engineering, July 2004

[2] Characterising the potential risks posed by engineered nanoparticles: A first UK Government research report, HM Government / Defra, November 2005.

[3] Measurement needs analysis, PA Consulting / DTI NMSD, April 2005.

[4] Review of the rationale and economic benefit of the National Measurement System, DTI, November 1999.

[5] Innovation report – 'Competing in the global economy: the innovation challenge', DTI, December 2003.

[6] www.bipm.org/en/cipm-mra/

[7] http://kcdb.bipm.org/

[8] www.dti.gov.uk/technologystrategy/

[9] Technology Strategy Board – A call to action, November 2005.

[10] www.rsc.org/images/February AD News_tcm18-44091.pdf

[11] N Kunzli et al, The Lancet, September 2000 (356), 795-801.

ANNEX: LIST OF ABBREVIATIONS AND ACRONYMS

AES	Auger Electron Spectroscopy
AFM	Atomic Force Microscopy
AQEG	Air Quality Expert Group
AQUILA	Ambient Air Quality Reference Laboratories
ARXPS	Angle Resolved X-ray Photoelectron Spectroscopy
BAM	Federal Institute for Materials Testing and Research, Germany
BBSRC	Biotechnology and Biological Sciences Research Council
BIPM	International Bureau of Weights and Measures
BSI	British Standards Institute
BTC	British Technical Council for the Motor and Petroleum Industries
CAFE	Clean Air For Europe
CATFAC	Controlled Atmosphere Facility
CCQM	Consultative Committee for Amount of Substance
CEHAPE	Children's Environment and Health Action Plan for Europe
CEN	European Committee for Standardization
CIPM	International Committee for Weights and Measures
Defra	Department for the Environment, Food and Rural Affairs
DESI	Desorption Electrospray Surface Ionisation
DMA	Differential Mobility Analyser
DTI	Department of Trade and Industry
EC	European Commission
EPSRC	Engineering and Physical Sciences Research Council
EU	European Union
EUROMET	European Collaboration in Measurement Standards
FCO	Foreign and Commonwealth Office
FET	Field Effect Transistor
FID	Flame Ionisation Detector
FPM	Fragmentation Pathway Mapping
FTIR	Fourier Transform Infrared
GAW	Global Atmospheric Watch
GC	Gas Chromatography
G-SIMS	Gentle Secondary Ion Mass Spectroscopy
HSE	Health and Safety Executive
IDMS	Isotope Dilution Mass Spectrometry
INTEGAIRE	Integrated Urban Governance and Air Quality Management in Europe
IPR	Intellectual Property Rights
IR	Ionising Radiation
IRMM	Institute for Reference Materials and Measurements
ISE	Ion Selective Electrode
ISO	International Organisation for Standardization
IUVSTA	International Union for Vacuum Science, Technique and Applications
JIP	Joint Industry Project
JRC	Joint Research Centre
KT	Knowledge Transfer
LC	Liquid Chromatography
LED	Light Emitting Diode
LGC	Laboratory of the Government Chemist
LNG	Liquefied Natural Gas
LPG	Liquid Petroleum Gas
MEMS	Micro Electro Mechanical Systems
MET	Measurements for Emerging Technology

METAS	Metrology and Accreditation Switzerland
MNT	Micro- and Nanotechnology
MRA	Mutual Recognition Arrangement
MS	Mass Spectrometry
NEL	National Engineering Laboratory
NERC	Natural Environment Research Council
NMAET	Nano-molecular Analysis for Emerging Technologies
NMI	National Metrology Institute
NMS	National Measurement System
NMSD	National Measurement System Directorate
NPL	National Physical Laboratory
OLED	Organic light-emitting diode
OST	Office of Science and Technology
PM	Particulate Matter
PT	Proficiency Testing
PTB	Physikalisch-Technische Bundesanstalt, Germany
QC	Quality Control
Q-TOF	Quadrupole Time-of-Flight
RAE	Royal Academy of Engineering
RCUK	Research Councils United Kingdom
REACH	Registration, Evaluation and Authorisation of Chemicals
RFID	Radio Frequency Identification
RS	Royal Society
SAMS	Surface and nano-analysis measurement steering
SC	Sub-committee
SEMI	Semiconductor Equipment and Materials International
SERS	Surfaced Enhanced Raman Spectroscopy
SI	International System of Units
SIMS	Secondary Ion Mass Spectroscopy
SME	Small and Medium Enterprise
SMILES	Simplified Molecular Input Line Entry Specification
SSIMS	Static Secondary Ion Mass Spectroscopy
STA	Source Testing Association
STAG	Standard Test Atmosphere Generator
TC	Technical Committee
TCD	Thermal Conductivity Detector
TD	Thermal Desorption
TDD	Time Division Dilution
UKAS	United Kingdom Accreditation Service
UNECE	United Nations Economic Commission for Europe
VAM	Valid Analytical Measurement
VAMAS	Versailles Project on Advanced Materials and Standards
VOC	Volatile Organic Compound
WG	Working Group
WHO	World Health Organisation
WMO	World Meteorological Organisation
XPS	X-Ray Photoelectron Spectroscopy