Publishable JRP Summary Report for JRP ENV01 MACPoll Metrology for Chemical Pollutants in Air

Background

The measurement of gaseous pollutants in air is a sensitive and priority issue that has a large impact on human health and environment. The European Directive on "Ambient air quality and cleaner air for Europe" (2008/50/EC) sets limit values and data quality objectives for the measurement in EU member states of air pollutants in ambient air. However, no legislation is currently present for the monitoring of indoor air and concern over the exposure of humans to indoor chemical pollutants is increasing.

Need for the project

JRP MACPoll addresses the need to improve the metrological traceability and comparability of measurements using current air monitoring techniques and the need to set-up a metrological basis for the new sensor technologies used in air quality applications.

These needs are demonstrated by:

- The poor agreement between reference standards prepared by NMIs for the calibration of air monitoring equipment;
- The lack of reliable "zero gas" for the zeroing of analysers and the preparation of gas standards used in ambient air monitoring;
- The lack of accurate reference methods and traceable reference materials for the measurement of (semi)volatile organic compounds ((S)VOC) in indoor air and constant emitting (S)VOC materials;
- The lack of low-cost indicative measurement methods for Air Quality assessment that permit proper spatial coverage.

Scientific and technical objectives

One of the main objectives of JRP ENV01 is to comply with the challenging requirements of calibration gases, both for span and zero gas, as set out in documentary EN standards related to the air quality directive (2008/50/EC) reference methods. In order to achieve this goal, the JRP will focus on:

- Improvement of dynamic and static dilution, as alternative preparation methods to gravimetry, for the generation of reactive primary gas mixtures, namely nitrogen oxides (NO and NO_2) and sulphur dioxide (SO_2) at the limit values mandated by the air quality directive.
- Development of a new approach for single and simultaneous assessment of impurities in zero gas. Nitrogen and pure air, used for zeroing gas analysers and for dilution purposes, should be free of contaminants that may interfere with measurements.

The second objective of the JRP is the development of references systems for (S)VOCs in indoor air pollution. To address this, the JRP will:

EMRP European Metrology Research Programme Programme of EURAMET
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Improve reference methods and develop reference materials for harmful (S)VOCs originating from emission of building materials.

Develop a constant emitting reference material for the quality control of emission test chamber measurements.

The third objective is the applicability of **micro-sensors in air quality monitoring.** Micro-sensors are emerging measuring devices for the "indicative measurements" specified in the air quality directive (2008/50/EC). The JRP will:

Validate existing sensor systems and investigate the metrological aspects (traceability and uncertainty) of the results.

Develop a new highly sensitive and selective NO_2 sensor using the promising 2 dimensional material graphene.

Expected results and potential impact

The JRP will have a direct impact on NMIs dealing with the measurement of trace levels gases. An outcome of the project is to improve the measurement capabilities of these organisations in terms of better standards and lower uncertainties. A number of JRP-Partners are designated Air Quality National Reference Laboratories (NRLs) and their task of ensuring traceability and comparability to the air monitoring networks will greatly benefit from the JRP results. NRLs and air monitoring networks also benefit from the JRP's work on micro-sensors for air pollution applications, because, if successful, it will help to implement the EU Air Quality Directive (2008/50/EC) and in particular the use of indicative methods for ambient air monitoring. The development of measurement standards and methods for (S)VOC is impacting emission labelling schemes, responsible for the detection of hazardous compounds emitted by building materials. By improving the quality of these schemes, the risk of indoor pollutants in buildings will be reduced.

All JRP-Partners are closely linked to stakeholders and to the end-users of this JRP. JRP-Partners are members of key environmental networks, such as the AQUILA (Air Quality Reference Laboratories) European network, they participate in standardisation activities (CEN/TC264 Air Quality, ISO/TC146 Air Quality and ISO/TC158 Gas Analysis), collaborate with National Environmental Institutes and provide traceable calibrations standards to air quality monitoring networks.

An evaluation of the dynamic and static preparation methods for traceable calibration gases for SO_2 and NO_x ambient air pollutants at concentrations close to the limit value has been completed. The evaluation has shown that the purity of the gases, including the dilution gas, is an important source of uncertainty. Meanwhile, a highly accurate portable system for the preparation of low concentration NO_2 gas mixtures, based on permeation, is fully assembled, leak-proof and operational. This system has been validated for stability and robustness and it is currently being used as a transfer standard to demonstrate the comparability of NMIs' calibration gas standards. Two other travelling standards, to demonstrate the comparability of NO and SO_2 dynamic methods, have been characterised and validated. These standards are dynamic dilutors based on molbloc/molbox systems.

In this JRP particular emphasis is on the reactive contaminants in zero gas. Because zero gas (nitrogen or purified air) is used in analyser calibration, these impurities need to be minimized to (sub)-ppb level and their presence quantified by traceable measurements using optical systems, such as Cavity Ring Down Spectroscopy (CRDS). Once this measurement approach is fully traceable, a protocol for the certification of the impurity composition of zero gas will be made available. Major achievements have been made so far in this field. A comprehensive study of adsorption of ammonia at trace concentration levels (part-per-billion) on different tubing materials (coated and non-coated stainless steel and various polymers) has been completed.

Further to this, for some low adsorbing polymers, differences in adsorption could be determined for the first time.

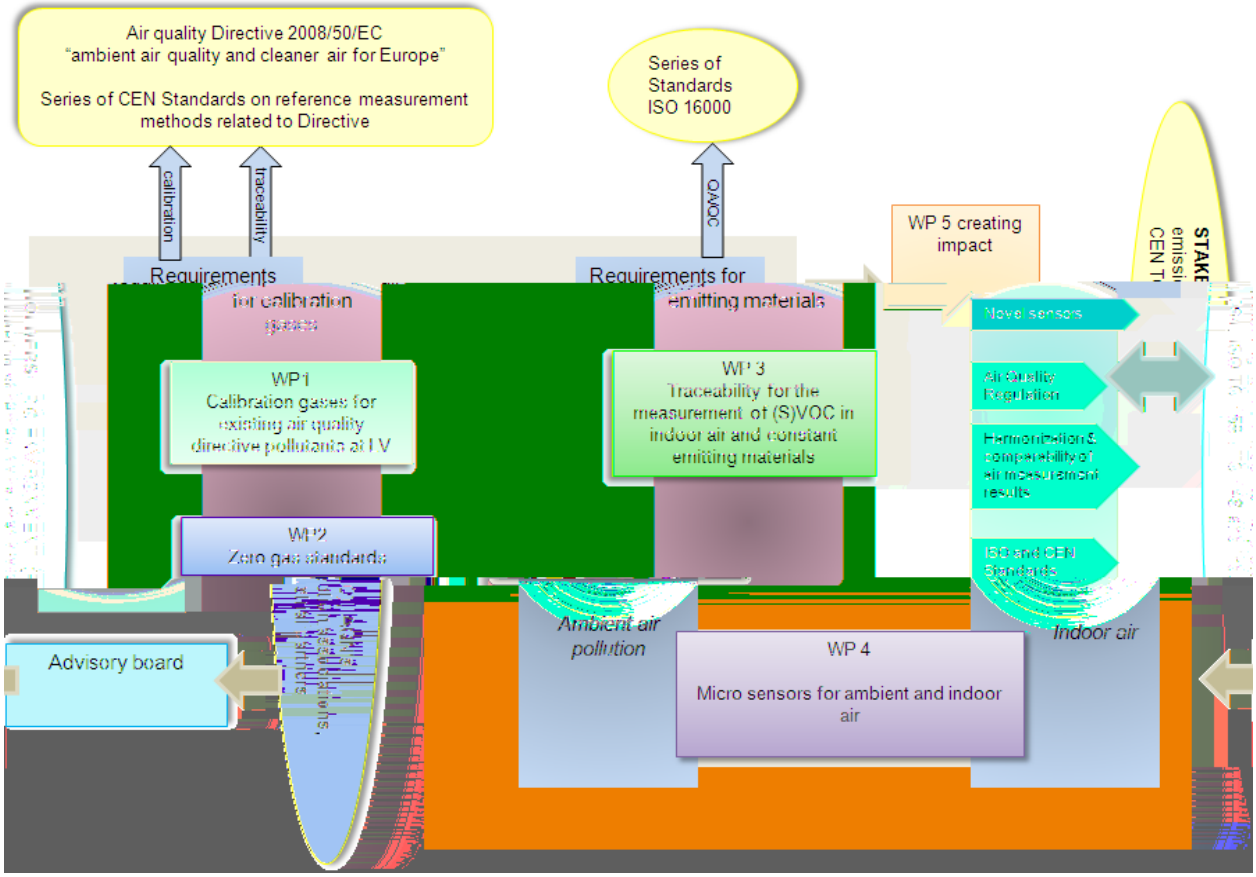
In addition, for the measurement of NO_2 impurities in zero gas, an innovative and sensitive cavity-enhanced detection system has been constructed based on a high-power blue light emitting diode. The performance of this system has been compared to the 'classical' chemiluminescence detector for the trace level measurement range (part-per-billion) with good results.

In order to obtain zero gases, purifiers and zero gas generators are often used. The evaluation of a selection of commercial filter systems and zero gas generators is currently on-going. The aim is to demonstrate their efficiency in removing reactive contaminants that may interfere in the analysis of NO_x and SO_2.

The work related to indoor air pollution is focusing on a range of challenging (S)VOC that are typically emitted from building materials: hexadecane, dibutyl phthalate (DBP), dimethyl phthalate (DMP), 2-ethyl-1-hexanol, 1-methyl-2-pyrrolidone and styrene. The research activities on the generation of reference (S)VOC gas mixtures at low concentration levels have shown that for the very high boiling point compounds, such as dibutyl phthalate (bp 350 C), this is a challenging objective because of condensation issues. Further investigation is needed to improve the methodology and to ensure traceability of these compounds. Meanwhile, the first tests on the production of a VOC constant emitting material have been carried out with promising results.

Research activities aiming at the development and validation of micro-sensors for air monitoring purposes are progressing beyond state of the art. Currently, two types of graphene sensors, based on epitaxial and exfoliated graphene, are available and they are under testing for the measurement of ambient levels of NO_2. A laboratory validation of 11 selected micro sensors for the measurement of ozone has been performed based on a self-designed validation and calibration protocols. At this stage, the same validation procedure is being applied to NO_2 sensors (graphene and commercially available sensors).

To date, ENV01 MACPoll has been presented at international conferences, to specific ISO and CEN standardisation committees involved in air quality and gas analysis, and to stakeholders. The project has over 30 examples of dissemination including papers, oral and poster presentations. An advisory board, made up of policy makers (European Commission), standardisation committees (CEN TC264, ISO TC158), air research institutes (NILU) and indoor air emission experts (AgBB) have been also set up. Further, collaboration agreements have been formalised with industrial collaborators, research organisations and air monitoring networks.



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