Publishable Summary for 15SIP01 Zero Gas
Standard to certify the purity of zero gas in ambient air measurements

Overview

Zero gases, such as nitrogen or synthetic air, are ultra-high purity gases used as reference points in the calibration of equipment in ambient air pollution measurements. This project aimed to maximise the uptake of the Certification Protocol for Zero Gas, developed by EMRP ENV01 MACPoll, by converting it into a draft documentary standard under ISO Technical Committee 158 "Gas Analysis.

Besides the incorporation of the Certification Protocol for Zero Gas into the documentary standard dealing with purity determinations in gas analysis (ISO 19229), this project developed and provided training courses to facilitate the uptake of the Certification Protocol for Zero Gas by National Reference Laboratories (NRLs) and the gas industry. The endorsement and implementation of the Certification Protocol and later of the ISO standard by European Network of Air Quality Reference Laboratories (AQUILA) and by the gas industry will improve compliance of the air monitoring networks with EU regulation and allow better reliability of the measurement data generated.

Need

The air monitoring networks of EU countries are mandated by the EU Directive on 'Ambient Air Quality and Cleaner Air for Europe' (2008/50/EC) and Commission Directive 2015/1480/EU to perform and guarantee SI traceable measurements of gas pollutants at extremely low (parts per billion) levels. To measure at low levels, the zero gas should be the "anchor point" for the analytical equipment from which minimum changes in instrument response are detected.

The European documentary standards (EN standards), that describe the reference methods for measuring air gas pollutants, specify the criteria for the purity of the zero gas but do not give explicit instruction on how to achieve the requirements. For example, when measuring NO_2 at the EU Annual Limit Value of 21 nmol/mol with a maximum allowed uncertainty on the measurement result of 15 %, the presence of 1 nmol/mol in the zero gas counts already for one-third of the allowed uncertainty. The standards specify that the content of NO_2 in zero gas shall be less than 1 nmol/mol. However, a zero gas with such level of metrological accuracy does not exist on the market. Hence, **there is an urgent need for a standardised purity assessment protocol and Zero Gas Calibration Standards to 'zero' the gas analysis equipment with greater accuracy**.

EMRP JRP ENV01 MACPoll delivered a Certification Protocol for Zero Gas that enabled the metrological assessment of the purity of zero gases, and generated SI traceable and ultra-accurate Zero Gas standards down to the nmol/mol.

However, the **Certification Protocol for Zero Gas** had **no formal standing**. International acceptance would be achieved by incorporating it in an ISO standard under TC158, the Technical Committee involved in the preparation of documentary standards for gas analysis. The Chair of **ISO/TC158, the primary supporter of this project,** supported the preparation of a draft New Work Item Proposal (NWIP) **to amend ISO 19229** standard "Gas analysis – Purity analysis and the treatment of purity data" so that it would include the relevant parts of the Certification Protocol for Zero Gas.

A second fundamental output of EMRP JRP ENV01 MACPoll was the successful implementation of the Certification Protocol for Zero Gas by the project partners to test gas purification systems, namely gas filters, zero gas generators and best grade compressed gases in cylinders.

Articles and presentations at symposia given by industrial parties demonstrated the challenge and the need for developing Zero Gas Calibration Standards. Industrial producers of zero gases, of gas purification systems and of zero gas generators usually provide conservative and therefore inaccurate specifications which do not meet the metrological requirements (accuracy and SI traceability) prescribed by the EN standards and the EU Air Quality Directives as above. This situation resulted in a non-compliance of the EU air monitoring networks

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with existing legislation and in a lack of confidence in the measurement data provided for policy makers to improve EU Air Quality. Therefore, at the start of this project, there was a need for training events for the EU air monitoring networks and industrial zero gas producers in the correct implementation of the Certification Protocol for Zero Gas in order that they may comply with legislation.

Objectives

The objectives of the project were:

1. To incorporate the "Certification Protocol for Zero Gas" and practical case studies developed in EMRP ENV01, into ISO 19229 (to Committee Draft status).
2. To facilitate the uptake of the Certification Protocol by direct users of Zero Gas Calibration Standards, namely the AQUILA Network of Air Quality National Reference Laboratories (NRLs) and by industrial producers of zero gases, namely gas producers and manufacturers of gas purification systems and zero gas generators. This objective would complement objective 1, by focusing on dedicated training for the correct implementation of the Protocol and of the future ISO standard.

Results

Objective 1

Within ISO/TC158 "Gas Analysis" a New Work Item Proposal covering the incorporation of the "Certification Protocol for Zero Gas" into ISO 19229 was submitted in 2016, after start of this project, and accepted.

The project partners worked closely with WG3 of ISO/TC158, responsible for the revision of the documentary standard, to ensure the acceptance of the Working Draft Document and to further proceed to achieve the status of Committee Draft (CD). The CD ISO 19229 was accepted in 2017 and the comments provided at CD ballot stage were discussed during ISO/TC158 meetings and the document amended. The project partners went beyond the project objective and prepared the Draft International Standard (DIS) version for enquiry. The DIS version of the standard has been submitted by ISO/TC158 secretariat (NEN) to ISO and it has been circulated for voting among the member states in February 2018. The results of the voting were published in July 2018, after the project lifetime. The DIS version was accepted with comments. The comments are currently addressed so that the DIS version can be amended and the final version published promptly.

The objective of a revised ISO 19229 standard in CD stage has been successfully achieved, being the standard even at a further stage (DIS) towards publication.

Objective 2

To achieve the second objective, dedicated (hands-on) training courses were scheduled and organised for industrial producers of zero gases and members of the AQUILA Network. These events focussed on the practical use of the protocol and on the data handling described in the Certification Protocol for Zero Gas.

The training material included an introduction part to illustrate the content of the revised ISO 19229, two theoretical presentations on the procedures for the assessment of the impurities in zero gases and relative data calculations, and an informational section on the impact of zero gas standards in air quality. The practical part, carried out in the laboratory, consisted of two sessions demonstrating the use of the protocol with measurement instruments having a defined and not-defined zero. Two demo videos demonstrating the two practical procedures were prepared and they were used when the training location would not guarantee access to the laboratories.

Three successful hands-on training sessions were held in the second half of 2017. They were given by the project partners to zero gas stakeholders: industrial parties, to NMIs and to AQUILA. Specifically, one hands-on training for gas producers and zero gas instruments' manufacturers and NMIs was given at VSL- Dutch Metrology Institute, Delft, The Netherlands in June 2017. Twelve participants took part in the training. Two training courses on zero gas were organised for AQUILA members and Nordic European Air Quality National Reference Laboratories and they were held respectively at the European Commission/Joint Research Centre (EC/JRC), Ispra, Italy in September 2017 (35 participants) and at the Finnish Meteorological Institute (IL), Helsinki, Finland in October 2017 (9 participants).

About three months after the trainings, the trainees were surveyed by the partners with an online questionnaire to ascertain the uptake of the Protocol and an assessment of the benefits of the course was done.

The assessment indicated that both zero gas industrial producers and AQUILA members were interested in the theoretical and practical modules of the training. Notably, industrial producers appreciated the practical module where advanced spectroscopy is used to determine the quality of zero gas, while AQUILA members thought the application to determine impurities using a purifier coupled to a not-defined zero instrument was

the most useful. While several zero gas industrial producers already applied the developed certification protocol for zero gas, showing the direct uptake of the training material, the NRLs have not done it yet. Some are planning to do that in the future.
One of the raised issues was that the application of the protocol was costly and time consuming. Once the revision of the ISO 19229, including the details of the certification protocol for zero gas is published, the expectation is that laboratories will gain capabilities and experience to carry out time and cost efficient certification protocol for their own zero gas.

Impact

The primary beneficiaries of the project activities were the AQUILA members, namely the EU NRLs, and producers of zero gases and of zero gas equipment, namely producers of compressed zero gas, gas purification system and zero gas generator manufacturers. These are the main players in ensuring accuracy and SI traceability to the measurement data provided by the air monitoring networks in Europe.

With the help of ISO/TC158, the Primary Supporter, the Certification Protocol and examples of its use have been successfully incorporated in the ISO standard dealing with purity determinations in gas analysis (ISO19229), so that the protocol has now formal status and, once published as final version, it will be adopted at both European and international level.

The development of an ISO standard describing the metrological assessment of the purity of zero gas and the dedicated (hands-on) training courses for stakeholders have therefore helped:

- ISO/TC158 Primary Supporter. The successful outcome of this project has provided ISO/TC 158 with a draft DIS standard that, following the formal ISO voting procedures, can be accepted and published as an ISO standard. The revision of the ISO 19229 standard and the project have been part of the presentation given by the ISO/TC 158 chair at the opening of the international conference “Gas Analysis 2017”, held in Rotterdam in 2017.
- The zero gas industry. Producers of zero gases and of zero gas equipment are enabled to deliver SI traceable, accurate and unambiguous products (Zero Gas Calibration Standards) to the market that meet the needs of the users, and in particular the air monitoring networks.
- The European NRLs. They can now apply the protocol and provide verified zero gas standards to the air monitoring networks.
- The presence on the market of Zero Gas Calibration Standards and the capability of direct users, the NRLs, in applying the ISO standard enable compliance of the air monitoring networks with the EU Air Quality legislation requirements. These networks can now calibrate their analytical equipment more accurately and produce reliable data.

Ultimately, the confidence in the accuracy of these data will help the European Commission and EU Governments in applying more appropriate strategies for air pollution abatement and contribute to a cleaner air for European citizens.

Thanks to the adoption of the ISO standard, gas producers and manufacturers of purification and zero gas generator systems are able to provide evidence of the quality of their products: SI traceable, accurate and unambiguous Zero Gas Calibration Standards. The availability on the market of these products will, in the long term, have a positive economic impact, as these products are used daily for the calibration of gas analysers of the ca. 8000 environmental monitoring stations located in Europe.

<table>
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