THE MULTIPLE ATMOSPHERE CONTROLLED ENVIRONMENT (MACE): ADVANCED TYPE TESTING FACILITY FOR GAS SENSORS

INTRODUCTION

In recent years, there has been a surge in emergent technologies, particularly concerning gas sensors, which can complement traditional reference grade instrumentation. Realizing this potential depends on effective performance evaluations of such technologies, building confidence in their adoption for air quality monitoring strategies. In response to this trend, the European Committee for Standardization (CEN) has recently published a Technical Specification (CEN/TS 17660-1:2021) addressing the requirements for such performance appraisals in laboratory tests and field co-location studies. Furthermore, this specification has now been incorporated as a UK MCERTS standard. In response, NPL has recently developed the Multiple Atmosphere Controlled Environment (MACE), a type testing facility conceived to assist industry in developing new products to meet the monitoring requirements of Air Quality legislation. A wide range of gas pollutant concentrations may be introduced and monitored under controlled environmental conditions in advanced purpose-built exposure chambers designed to evaluate the performance of low cost gas sensors.

MACE capabilities

The MACE facility consists of up to six state-of-the-art exposure chambers and a gas delivery system to deliver advanced testing and performance evaluation under different experimental regimes. Traceable gas compositions are generated and controlled via a gas mixing unit, which dynamically combines multiple gas sources to deliver the desired concentrations. A vaporizer unit generates humidified gas streams covering the entire range of relative humidity that is in representative conditions for ambient air monitoring applications. Specialised exposure chambers with low internal volume can be used simultaneously to expose the instrumentation undergoing assessment to the generated atmospheres. These chambers are located in a temperature-controlled environmental room, which is capable of reproducing real world temperature conditions. A suite of reference grade instrumentation enables continuous measurements of the conditions being generated, such as gas composition, temperature, relative humidity and pressure. Figure 1 shows three of six exposure chambers of the MACE facility accommodating air quality sensor systems for testing.

Figure 1: Photograph showing three of the exposure chambers of the MACE facility.

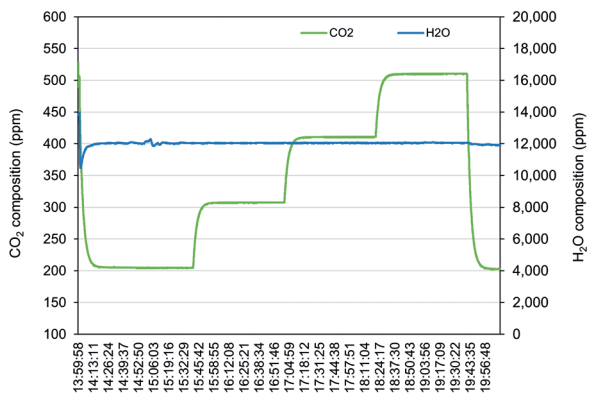

Figure 2: Results of the CO_2 multipoint calibration. Time series of CO_2 and H_2O compositions (ppm) measured by a reference grade gas analyser sampling from one of the MACE exposure chambers.

The facility has been designed to house sensors measuring the regulated compounds NO_2, NO_x, O_3, CO, SO_2, together with other molecules including CO_2, NH_3 and volatile organic compounds (VOCs). A number of research and development projects, as well as bespoke measurement services, have been supported by the MACE facility in the recent past. One of such projects, entailed the validation of low-cost carbon dioxide (CO_2) sensors with respect to temperature and response to water vapour. The following section focuses on the diagnostic results measured by calibrated reference quality instrumentation employed to showcase the performance of the MACE facility. The proprietary results obtained from the low cost sensors under evaluation have not been included.

Case study: validation of carbon dioxide (CO_2) sensors

Water vapour is likely to cause cross-interference in carbon dioxide measurements by optical (NDIR) sensors. To support industry in improving the performance of CO_2 sensor systems, NPL has deployed the MACE facility to generate empirical evidence for the effects of absolute humidity, relative humidity and temperature on the performance of these sensors. The whole programme of work entailed a number of exposure regimes, three of which are highlighted below. The full set of results is being used to enable improvements in the proprietary compensation algorithms used for CO_2 low cost sensors.

The initial example is a multipoint calibration of CO_2, which had the objective of establishing traceable calibration factors for the sensors' response this gas. The target conditions were four

stages of CO_2 composition (200 ± 10 ppm to 500 ± 10 ppm ppm) under a constant temperature of 20 ± 1 °C, constant water vapour concentration of 12,000 ± 500 ppm and constant pressure of 101.3 ± 1 kPa. To demonstrate the multipoint calibration results, Figure 2 shows the time series of CO_2 and H_2O compositions (ppm) measured by a LI-COR 7815 reference grade gas analyser sampling from one of the MACE exposure chambers. It can be observed that there is a high degree of stability of the generated conditions inside the chamber, as well as a good adherence to the target gas concentrations.

The second example shows a multipoint calibration of H_2O under constant CO_2 composition of 405 ± 10 ppm and environmental conditions similar to the previously described example. This assessment had the objective of establishing traceable calibration factors for the humidity effect on the sensors' response to CO_2. To demonstrate these multipoint H_2O calibration results, Figure 3 shows the time series of CO_2 and H_2O compositions (ppm) measured by a reference grade gas analyser sampling from one of the MACE exposure chambers. Once again, good stability and adherence to the target levels can be observed.

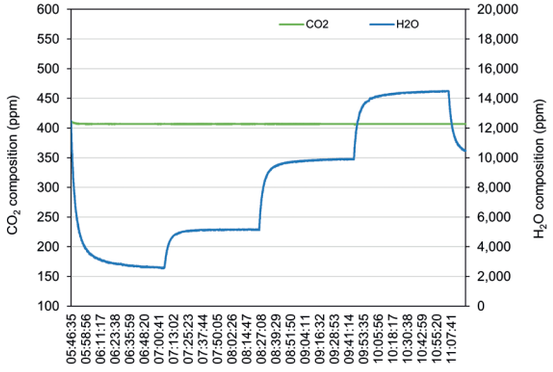

Figure 3: Results of the H_2O multipoint calibration. Time series of CO_2 and H_2O compositions (ppm) measured by a reference grade gas analyser sampling from one of the MACE exposure chambers.

Finally, the third example is a more complex experimental design, which had the objective of discriminating the cross-interference effects of absolute humidity and relative humidity on the sensors' response to CO_2. To achieve this goal, the exposure regime aimed at generating changes in relative humidity by alternating means: changing temperature and changing absolute humidity. The CO_2 composition and pressure were kept constant at 405 ± 10 ppm and 101.3 ± 1 kPa, respectively. Figure 4 shows the time series of temperature (°C) and relative humidity (%) measured by a calibrated Michell Instruments WR293 reference hygrometer inside one of the MACE exposure chambers. Blue bars on the top of the graph indicate the periods in which the absolute humidity was kept constant. The vertical black lines indicate the moment where the absolute humidity was changed to a different value.

Authors:

Nick Martin is a Principal Research Scientist and the Science Area Leader of the Air Quality and Aerosol Metrology Group at NPL. He is a Member of the Royal Society of Chemistry, a Chartered Chemist, Chartered Scientist (MRSC CChem CSci) and an Associate of King's College London (AKC). He joined NPL in 1990, having completed his BSc in Chemistry, AKC, and PhD in kinetic spectroscopy at King's College London. This was followed by postdoctoral research at the Department of Physical Chemistry, University of Cambridge employing infrared lasers with molecular beams. At NPL he was first involved in developing a ground-based laser heterodyne spectrometer to detect stratospheric molecules. More recently, Nick developed pumped and diffusive sampling methods for measurements of nitrogen dioxide, VOCs, and ammonia using a Controlled Atmosphere Test Facility (CATFAC). He was involved in the "Breathe London" pilot consortium, which implemented a new network of low-cost sensors for hyperlocal monitoring of air quality in London and projects concerned with measuring in-cabin air quality and bleed air on aircraft, and QA/QC for NO_2 diffusion tube networks. Nick is a member of DEFRA's Air Quality Expert Group (AQEG) and is involved in CEN standardization committees including CEN TC264 WG42 for low-cost sensors.

Gabriel Garcia graduated in chemistry (BSc) and conducted academic research in atmospheric chemistry to conclude his master's and PhD degrees at the Sao Paulo State University (Brazil). During his studies he delivered projects for developing passive samplers for tropospheric ozone measurements and investigating the relevance of atmospheric deposition as sources of nitrogen and phosphorus for aquatic ecosystems. Gabriel joined NPL in 2019 as a Higher Research Scientist and is a Member of the Royal Society of Chemistry. More recently, he has been involved in laboratory and field-based research projects and bespoke customer measurement services regarding gas sensors' testing, ambient and indoor air quality assessments, and quality assurance for atmospheric measurements.

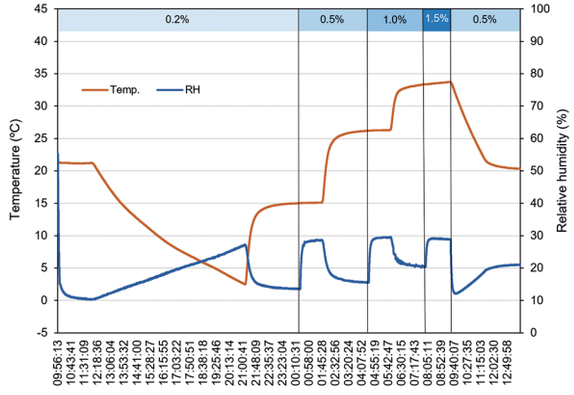

Figure 4: Time series of temperature (°C) and relative humidity (%) during the water vapour cross-interference test.

During the periods of constant H_2O concentration, the changes in relative humidity were caused by the applied changes to the temperature. The events marked by vertical lines indicate the changes to relative humidity caused by increased absolute humidity. Additionally the test was set up to deliver three episodes of 30 ± 1 % relative humidity at different combinations of temperature and absolute humidity.

Correlating the sensors' response to CO_2 with these environmental parameters should identify which factor(s) are most influential on the sensors performance (absolute or relative humidity) and calculating adjustments to the compensation algorithms.

Conclusions

The Multiple Atmosphere Controlled Environment (MACE) is a recently commissioned type testing facility for evaluating the performance of low cost gas sensors. It has been successfully deployed in metrological research projects that underpin product development for air quality monitoring purposes. The presented case study highlighted how the MACE capabilities can be deployed to validated novel carbon dioxide (CO_2) low cost sensor systems, which involved the generation of traceable gas compositions alongside the stringent control of environmental conditions such as humidity, temperature and pressure. These capabilities are directly applicable to the performance evaluations required by the CEN/TS 17660-1:2021 and NPL will soon obtain UKAS accreditation for providing this service.

Further details of other exciting projects and initiatives can be found at: www.npl.co.uk/commercial-services

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