

Report on test protocol for sensor testing

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Report on test protocol for sensor testing

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III

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SUMMARY

Sensors (or Low-Cost Sensors: LCS) are generally considered as key emerging measuring devices for the monitoring of air quality. LCS can supplement existing reference grade monitor networks by extending their spatial coverage and increasing monitoring density to provide more localized insights. LCS can support policy-relevant air quality analysis, including assessing levels of pollution and identifying sources. However, the use of sensors and acceptance of their data is somewhat hampered due to unknown quality of the data.

To ensure reliable air pollution data, it's essential to evaluate and compare these sensors against well-established measurement methods using harmonised testing protocols and performance metrics. This can help guiding sensor selection, identify major issues, and also help guide sensor improvement and provide clarity for manufacturers, developers, and consumers.

Within this reference task we developed a laboratory test protocol for the evaluation of gas and particle sensors. This document reports on activities and outcomes of the reference tasks. First, we give a short overview of existing test protocols (Chapter 1). In Chapter 2 we describe the laboratory infrastructure and test protocol for gas sensors and in Chapter 3 for PM sensors. Each chapter includes the description of the infrastructure (including gas- or particle generation, exposure chamber and reference monitors), the test protocol, the evaluation parameters and lessons learned from the tests performed. Chapter 4 gives some information on infrastructure for outdoor/mobile testing. Chapter 5 summarizes the conclusions and next steps. More background information on the validation of the gas test chamber is given in Annex A and background information on the final set-up of PM tests is given in Annex B.

The test infrastructure for **gas sensors** is an exposure chamber where the environmental conditions (e.g., temperature, RH, air exchange rate) can be controlled and monitored. The total internal volume of the chamber is 237 dm³ and it can host multiple sensor systems. The homogeneity inside the test chamber is realised by a mixer and a multi-point air inlet distribution system. The exposure test chamber is placed inside a thermostatic room to control the temperature (between -10°C ÷ +60°C). The gas mixture is generated by diluting certified gas mixture with carrier gas using mass flow controllers and the RH is controlled (between 5% – 90%) using a Bronkhorst controlled evaporator mixer.

The gas test protocol comprises different steps:

A first step (at average T and RH) where sensors are exposed to different concentrations to evaluate the accuracy, the linearity, difference between sensors. A step to evaluate impact of T and RH and a step to evaluate impact of interferences;

The test infrastructure for **PM sensors** includes an exposure chamber and two particle generation systems; One is a Particle Dispenser System (PALAS) coupled to a dilution and injection system to supply particle loaded air to the exposure chamber. Another is an aerosolizer installed directly inside the exposure chamber for generating different particle sizes.

The test protocol for PM sensors includes a laboratory test to evaluate linearity and ability to measure coarse PM fractions. This dedicated test is introduced because some sensors can only measure PM_{2.5} and calculate PM₁₀ concentrations based on PM_{2.5} measurements.

The developed test protocol was set up for gas sensors (focus on NO/NO₂ and O₃ but can be extended to other regulated pollutants) and PM sensors (PM_{2.5}, PM₁₀).

IV

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SAMENVATTING

Sensoren (of Low-Cost Sensors: LCS) worden algemeen beschouwd als nieuwe meetinstrumenten voor het monitoren van de luchtkwaliteit. LCS kunnen referentiemetingen aanvullen door een grotere ruimtelijke dekking om meer gelokaliseerde inzichten te bieden. LCS kunnen beleidsrelevante analyses van de luchtkwaliteit ondersteunen, inclusief het beoordelen van de concentratieniveaus en het identificeren van bronnen. Het gebruik van sensoren en de acceptatie van hun data wordt echter belemmerd vanwege de onbekende (kennis van) kwaliteit van de data.

Om betrouwbare luchtkwaliteitsmetingen te garanderen, is het essentieel om deze sensoren te evalueren en te vergelijken met gevestigde meetmethoden met behulp van geharmoniseerde testprotocollen en prestatie-karaktersitatieken. Dit kan helpen bij het selecteren van sensoren, het identificeren van issues, het verbeteren van sensoren en het bieden van duidelijkheid voor fabrikanten, ontwikkelaars en consumenten.

Binnen deze referentietoek hebben we een laboratorium testprotocol ontwikkeld voor de evaluatie van gas- en deeltjessensoren. In dit document wordt gerapporteerd over de activiteiten en uitkomsten van de referentietoek. Eerst geven we een kort overzicht van bestaande testprotocollen (Hoofdstuk 1). In Hoofdstuk 2 beschrijven we de laboratoriuminfrastructuur en het testprotocol voor gassensoren en in Hoofdstuk 3 voor PM-sensoren. Elk hoofdstuk bevat de beschrijving van de infrastructuur (inclusief gas- of deeltjesgeneratie, blootstellingskamer en referentiemonitoren), het testprotocol, de evaluatieparameters en de lessen die zijn getrokken uit de uitgevoerde tests. Hoofdstuk 4 geeft enige informatie over de infrastructuur voor buiten/mobiel testen. Hoofdstuk 5 vat de conclusies en volgende stappen samen. Meer achtergrondinformatie over de validatie van de gastestkamer wordt gegeven in Bijlage A en achtergrondinformatie over de uiteindelijke opzet van PM-testen wordt gegeven in Bijlage B.

De testinfrastructuur voor **gassensoren** omvat een blootstellingskamer waar de omgevingscondities (bijvoorbeeld temperatuur, RV, luchtuitwisseling) kunnen worden gecontroleerd. Het totale interne volume van de kamer is 237 dm³ en er kunnen meerdere sensorsystemen in worden ondergebracht. De homogeniteit in de testkamer wordt gerealiseerd door een mixer en een meerpunts-luchtinlaatdistributiesysteem. De blootstellingstestkamer wordt in een thermostatische kamer geplaatst om de temperatuur te regelen (tussen -10°C ÷ +60°C). Het gasmengsel wordt gegenereerd door het gecertificeerde gasmengsel te verdunnen met draaggas met behulp van massadebietsregelaars en de RV wordt geregeld (tussen 5% – 90%) met behulp van een Bronkhorst-gestuurde verdampmixer.

Het gastestprotocol bestaat uit verschillende stappen:

Een eerste stap (bij gemiddelde T en RH) waarbij sensoren worden blootgesteld aan verschillende concentraties om de nauwkeurigheid, de lineariteit en het verschil tussen sensoren te evalueren. Een stap om de impact van T en RH te evalueren en een stap om de impact van interferenties te evalueren;

V

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De testinfrastructuur voor **PM-sensoren** omvat een blootstellingskamer en twee deeltjesgeneratiesystemen; Eén daarvan is een Particle Dispenser System (PALAS) gekoppeld aan een verdunnings- en injectiesysteem om met deeltjes beladen lucht naar de blootstellingskamer te voeren. Een andere is een aerosoliser die direct in de blootstellingskamer is geplaatst voor het genereren van verschillende deeltjesgroottes.

Het testprotocol voor PM-sensoren omvat een laboratoriumtest om de lineariteit en de mogelijkheid om grove PM-fracties te meten te evalueren. Deze speciale test wordt geïntroduceerd omdat sommige sensoren alleen PM_{2,5} kunnen meten en PM₁₀-concentraties kunnen berekenen op basis van PM_{2,5}-metingen.

Het ontwikkelde testprotocol is opgezet voor gassensoren (focus op NO/NO₂ en O₃ maar kan worden uitgebreid naar andere gereguleerde verontreinigende stoffen) en PM-sensoren (PM_{2,5}, PM₁₀).

VI

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TABLE OF CONTENTS

AUTHORS	III
SUMMARY	IV
SAMENVATTING	V
TABLE OF CONTENTS	VII
LIST OF FIGURES	IX
LIST OF TABLES	X
LIST OF ACRONYMS	XI
1 INTRODUCTION	1
1.1 Aim	1
1.2 CEN standardisation work.....	2
1.3 Other initiatives on testing protocols	2
1.3.1 AQ-SPEC	2
1.3.2 AirLab	3
1.3.3 US-EPA	3
1.3.4 ASTM standards	4
2 TEST PROTOCOL AND INFRASTRUCTURE FOR GAS SENSORS	5
2.1 Introduction	5
2.2 Test chamber or exposure chamber	5
2.2.1 Introduction.....	5
2.2.2 Description of the test infrastructure.....	6
2.3 Gas mixture generation.....	9
2.4 Measurement reference instruments.....	10
2.5 Test protocol	11
2.5.1 Step 1: Pre-test of sensor system under controlled conditions	11
2.5.2 Step 2: Temperature effect	12
2.5.3 Step 3: Humidity influence	12
2.5.4 Step 4: Cross-sensitivities of gaseous interfering compounds.....	13
2.6 Sensor evaluation parameters	14
2.6.1 Response time.....	14
2.6.2 Lack of Fit	14
2.6.3 Repeatability and Limit of Detection (LOD)	16
2.6.4 Accuracy (AQ-SPEC).....	17
2.6.5 Intra-Model Variability (IMV) (AQ-SPEC)	17
2.6.6 Between instrument uncertainty of the sensor systems (BIU)	17
2.6.7 Mean Absolute Error (MAE).....	18
2.6.8 Mean Bias Error (MBE).....	18

2.6.9	Root Mean Square Error (RMSE) and Normalized Root Mean Square Error (NRMSE).....	19
2.6.10	Impact of Temperature.....	19
2.6.11	Impact of RH.....	20
2.6.12	Impact of interferences	20
2.7	Lessons learned from first tests	22
2.7.1	Preliminary test before starting extended test program	22
2.7.2	Impact of changing ambient conditions on stabilization of sensor signal	23
2.7.3	Zero values.....	25
2.8	Evaluation of results.....	26
3	TEST PROTOCOLS FOR PM SENSORS	27
3.1	Aim and specific needs for PM sensors	27
3.2	Particle generation and exposure chamber	28
3.2.1	PM generation system	28
3.2.2	PM exposure chamber and reference instrument.....	29
3.3	Coarse laboratory test.....	30
3.3.1	PM generation for PM _{coarse} test.....	30
3.3.2	Evaluation.....	31
3.4	Linearity test (LoF).....	32
3.5	Examples of tested PM sensors.....	34
3.5.1	Projects and sensors tested.....	34
3.5.2	Examples PM: Linearity and accuracy	36
3.5.3	Example PM: PM _{coarse} test.....	36
4	ADDITIONAL INFRASTRUCTURE	39
5	CONCLUSIONS AND OUTLOOK	40
ANNEX A: Validation of the gas test chamber		41
5.1	Introduction	41
5.2	Temperature and relative humidity stability	41
5.3	Efficiency of the internal exposure test chamber air mixing.....	42
ANNEX B: Evaluation of the PM test chamber and settings.....		44
5.4	Introduction	44
5.5	Homogeneity if the test chamber.....	44
5.6	Stability and levels of PM concentrations	44
5.7	Coarse tests.....	45
5.7.1	Particle composition for generation	45
5.7.2	Timing of particle introduction for stable concentration.....	47

LIST OF FIGURES

Figure 1: Schematic overview of test protocol of US-EPA including base and enhanced testing	4
Figure 2: Exposure test chamber	6
Figure 3: Mixing system of the exposure chamber	7
Figure 4: Gas generation system and humidification of inlet air to the test chamber	7
Figure 5: 14 dm ³ exposure test chamber.....	9
Figure 6: Schematic overview of gas mixing and humidification system.....	10
Figure 7: An overview of the test sequence and conditions performed in Step 1.....	11
Figure 8: An overview of the test sequence and conditions performed in Step 2.....	12
Figure 9: An overview of the test sequence and conditions performed in Step 3.....	13
Figure 10: An overview of the test sequence and conditions performed in Step 4.....	13
Figure 11: Example of bad calibrated sensor output (top) and improved signal using simple calibration model (bottom).....	23
Figure 12: NO ₂ sensor signal with changing RH conditions at zero concentration.....	24
Figure 13: NO ₂ sensor signal with changing RH conditions at span concentration.....	24
Figure 14: Example of sensor signal at zero concentration plotted, together with signal of reference monitor for NO ₂	25
Figure 15: PM ₁₀ and PM _{coarse} of sensor in function of reference for sensor tested in VAQUUMS project.....	27
Figure 16: Schematic of particle generation and distribution system.....	28
Figure 17: Exposure chamber with generation system on top (left) and reference instrument	29
Figure 18: Set-up for PM coarse test (left) and co-spheric dust used for the generation of fine and coarse dust (right).....	31
Figure 19: AirsensEUR sensor box showing (A) entire unit with PM sensor inlet (B) details of OPC and PMS inlet and (C) sensor units (OPC left and PMS right).....	34
Figure 20: PM sensors tested as part of the benchmark study (from left to right): PAM (2BTech), GeoAir, SODAQ NO ₂ (SODAQ), SODAQ Air (SODAQ), PMscan (TERA), Open Seneca (Open Seneca) and ATMOTube Pro (ATMO).....	35
Figure 21: Time trends for fine (left) and coarse (right) tests measured by reference monitor	36
Figure 22: Coarse PM testing results with consecutive 5-min generation periods of coarse (7.75 μm) and fine (1.18 μm) PM peaks (upper panel; measured by Grimm monitor) and resulting sensor response (μg/m ³) in the lower panels (adopted from Hofman et al., 2024).....	38
Figure 23: Exposure shelter for sensors (left: outside of the shelter and right: inside with sensors).....	39
Figure 24: Prototype of mobile evaluation platform	39
Figure 25: A schematic overview of the tracer gas system.....	42
Figure 26: Homogeneity test with Dusttraks: intercomparison of instruments after correction (left) and concentrations measured at different locations (right).....	44
Figure 27: Stability test of PM concentrations for different PM injection rates.....	45
Figure 28: TSP concentrations in exposure chamber with injection frequency of 8 sec (left peak) and 2 sec (right peak).....	45
Figure 29: Example of coarse test (aiming for coarse test conditions) with PSL particles showing sensor and monitor (Grimm) values for PM _{2.5} and PM ₁₀	46
Figure 30: Size distribution measured during coarse test (aiming for coarse test conditions) using PSL particles	46
Figure 31: PM concentrations for different settings of the generation.....	47

LIST OF TABLES

Table 1: Target gas test concentration levels defined in CEN/TS 17660-1.....	11
Table 2: Summary of sensor evaluation parameters	14
Table 3: Selected limit values (LV) of compounds.....	21
Table 4: Max concentrations of interfering compounds for calculation of uncertainty.....	21
Table 5: Results of tests performed on PM portable sensors	33
Table 6: Overview of performance (Accuracy, MAE, R ²).....	36
Table 7: Ratio sensor/reference for different fine (1A-1D) and coarse (2A-2B) test conditions	37
Table 8: Factor of sensor response change from fine to coarse test conditions F(fine/coarse)	37
Table 9: An overview of the temperature and relative humidity measurements during the performed stability test.....	41

LIST OF ACRONYMS

CO	Carbon monoxide
COV	Coefficient of variance
DQO	Data Quality Objectives
EPA	Environment Protection Agency
LCS	Low Cost Sensors
MAE	Mean Absolute Error
NO	Nitrogen oxide
NO ₂	Nitrogen dioxide
O ₃	Ozone
PM	Particulate matter
PSL	Polystyrene latex
R ²	Square of correlation
RMSE	Routh Mean Square Error
SD	Standard deviation
SO ₂	Sulphur dioxide
VOC	Volatile Organic Compounds

1 INTRODUCTION

1.1 Aim

Sensors (or Low-Cost Sensors: LCS) are generally considered as key emerging measuring devices for the monitoring of air quality.¹ Sensor systems provide a fast and low-cost alternative to the reference methods as defined in Directive 2008/50/EC. LCS can supplement existing reference grade monitor networks by extending their spatial coverage and increasing monitoring density to provide more localized insights. Therefore, sensors are increasingly used to assess air quality by (local) authorities, citizens, researchers, ... Low-cost air quality sensor systems (LCS) are a key emerging class of technologies for expanding policy-relevant air quality analysis, including assessing levels of pollution, identifying sources, and producing forecasts.

However, the use of sensors and acceptance of their data is somewhat hampered due to unknown quality of the data. In general, LCS provide lower accuracy (i.e., including both trueness and precision), sensitivity, and specificity in their data and typically have a shorter operational lifetime compared with a reference monitors. Whereas sensors have been extensively used and tested under specific conditions or at specific locations, data are difficult to compare.

To ensure reliable air pollution data, it's essential to evaluate and compare these sensors against well-established measurement methods using harmonised testing protocols and performance metrics. This can help guiding sensor selection, identify major issues and also help guide sensor improvement and provide clarity for manufacturers, developers, and consumers.

In fact, there are a few things important when evaluating sensor systems to be used in a sensor network:

- How good do values obtained from the sensor systems correspond with the 'true value'. What is the systematic error to the reference?
- How good do repeated measurements correspond? This includes a) how close are repeated measurements of the same situation and instrument; b) how good is the correspondence of different instruments when exposed to the same concentration?
- How are sensor system's values affected by ambient conditions? In fact, this relates also to the first bullet point because if a sensor system's values are strongly impacted by ambient conditions, resulting in deviating values, this will have a negative effect on the outcome of the first bullet point.
- For gaseous pollutants: How are sensor system's values affected by other pollutants (interference)
- For particulates: How are sensor system's values affected by size and composition of particulates

¹ GAW Report No.293. Integrating Low-cost sensors Systems and Networks to Enhance air Quality Applications

1.2 CEN standardisation work

TC264 WG42 (Working Group 42 of Technical Committee 264 on Air Quality of the European Committee for Standardization) has been working on a protocol for the evaluation of sensor systems for air quality monitoring. The protocol applies to sensor systems as individual measurement devices for outdoor measurements at fixed sites. The procedure evaluates if the measurement uncertainty defined in Directive 2008/50/EC as Data Quality Objective (DQO) for indicative measurements and for objective estimation is met. The Technical Specification (CEN/TS 17660-1) for the evaluation of regulated gaseous pollutants (O₃, NO, NO₂, CO, SO₂ and benzene) is published and the protocol for PM sensors (PM₁₀ and PM_{2.5}) is being developed (CEN/TS 17660-2).

1.3 Other initiatives on testing protocols

Other initiatives to evaluate sensors are shortly discussed below.

1.3.1 AQ-SPEC

The Air Quality Sensor Performance Evaluation Center (AQ-SPEC) program was set up by South Coast AQMD (Air Quality Management District). Within this program, a test chamber was built, and a test protocol was set up (including laboratory and field tests). A wide range of testing is performed. The aim is to do an extensive characterization of commercially available sensors and sensor systems. They also want to give guidelines on sensor technologies and data-interpretation, in addition to support the development of sensor technologies.

<http://www.aqmd.gov/aq-spec>

AQ-SPEC started with criteria pollutants² (NO, NO₂, O₃, SO₂, CO, PM₁, PM_{2.5}, PM₁₀). But also, H₂S and CH₄ can be considered.

It has now extended its test program with a specific protocol for:

- For emerging pollutants (VOCs)³
- Mobile applications⁴

Both laboratory and field evaluation are included. The evaluation parameters (stationary applications) included are coefficient of determination (R²), intra-model variability, accuracy, precision, co-pollutant interference, temperature, and RH influences.

² Papapostolou V, Zhang H, Feenstra B, and Polidori A. (2017). Development of an environmental chamber for evaluating the performance of low-cost air quality sensors under controlled conditions. *Atmospheric Environment*, 171: 82-90.

³ Air Quality Sensor Performance Evaluation Center (AQ-SPEC) of the US South Coast Air Quality Management Division; Papapostolou et al. Laboratory Evaluation of VOC sensors – June 2023. <https://www.aqmd.gov/aq-spec/evaluations/voc/laboratory> last accessed 26 June 2024.

⁴ <https://www.aqmd.gov/aq-spec/special-projects/mobile-sensors> and Mui W., et al. 2021. Development of a Performance Evaluation Protocol for Air Sensors Deployed on a Google Street View Car. *Environmental Science and Technology*, Vol. 55, 3 <https://pubs.acs.org/doi/abs/10.1021/acs.est.0c05955>

The main difference with the EU procedure is that in AQ-SPEC testing protocol for most evaluation parameters, the average of the three sensors under evaluation is used instead of the individual sensor data.

1.3.2 AirLab

AirLab is an open innovation laboratory of AIRPARIF and its partners. The aim is to use an open innovation model to stimulate cooperation between different entities that focus on innovation in air quality. <https://airlab.solutions/en/notre-equipe-76>

One of the initiatives is the “microsensor challenge”. The AIRLAB Microsensors Challenge seeks to meet the growing demand from potential users for an independent and objective evaluation of the performance of microsensor based devices for air quality measurement applications. The AIRLAB Challenge goes beyond data accuracy criteria to also consider the utility, usability, portability, and cost of the considered devices. It is designed as a periodic event in which all candidate sensing platforms are evaluated in parallel, therefore providing a snapshot of the state of the art of commercially available microsensor platforms at the time of the evaluation.

The tests are performed following a fixed protocol. They consider eight usage categories characterized by intended application, a combination of the micro-environment (outdoor air, indoor air and citizen air) and aim of data collection (e.g. monitoring, awareness,...).

Most recent report including the protocol can be found here:

https://airlab.solutions/sites/default/files/2023-07/Protocole%20-%20Challenge_Microcapteurs%202023%20EN.pdf

1.3.3 US-EPA

The US-EPA (Environment Protection Agency) has developed performance testing protocols, metrics and target values for PM_{2.5}⁵ and ozone sensors⁶. These are targeted for non-regulatory supplemental and informational monitoring (NSIM) applications in ambient, outdoor, fixed site environments.

Test include base testing (field evaluation) and enhanced testing (laboratory evaluation). The base testing consists of two field deployments (co-located with reference instruments) for at least 30 days each. The two field deployments can include two different seasons or two different locations. The enhanced testing includes laboratory testing..., effect of interferences, effect of T and RH, evaluation of drift (after 60 days), accuracy at high concentration.

Performance metrics include precision (SD and COV), bias (slope and intercept), linearity (R²), error (RMSE) and meteorological effects (based on graphical evaluation for field test and change in sensor response between two meteorological conditions for enhanced tests). Drift and accuracy at high concentration is only evaluated based on enhanced testing.

⁵ EPA (2021). Performance Testing Protocols, Metrics, and Target Values for Fine Particulate Matter Air Sensors EPA/600/R-20/280

⁶ EPA (2021). Performance Testing Protocols, Metrics, and Target Values for Ozone Air Sensors EPA/600/R-20/279.

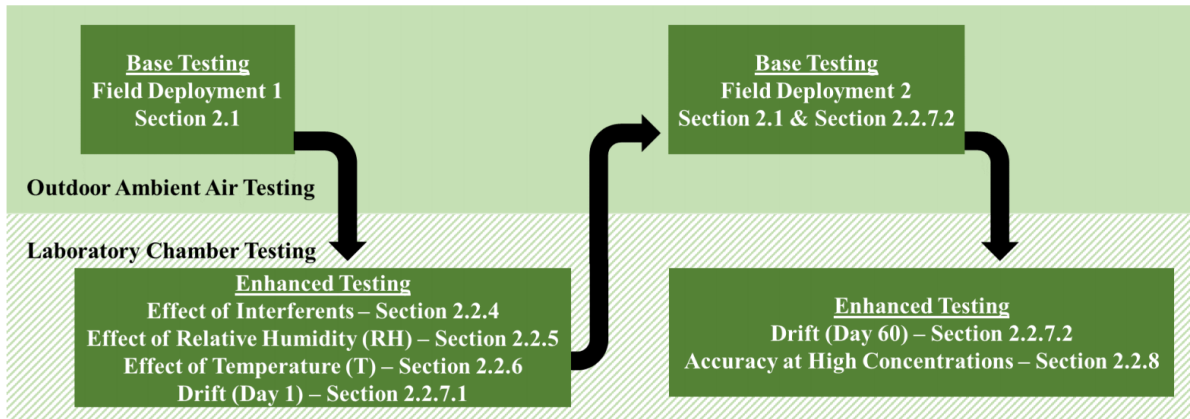


Figure 1: Schematic overview of test protocol of US-EPA including base and enhanced testing

They have recently added other pollutants:

- PM₁₀⁷
- NO₂, CO, SO₂⁸

See also:

<https://www.epa.gov/air-sensor-toolbox/air-sensor-performance-targets-and-testing-protocols>

1.3.4 ASTM standards

On an international level the American Society for Testing and Materials (ASTM) prepared standards for sensor testing for ambient measurements.

The “Standard Practice for Performance Evaluation of Ambient Outdoor Air Quality Sensors and Sensor-based Instruments for Portable and Fixed-point Measurement”, ASTM International’s air quality committee (D22) has developed standards to evaluate the performance of ambient outdoor air quality sensors. Two standards have been developed:

- D8406-22⁹: describing the performance test
- D8559-24¹⁰: describing the criteria for ambient outdoor air quality sensors.

Both are focused on PM_{2.5} and PM₁₀, sulfur dioxide (SO₂), ozone (O₃), carbon monoxide (CO), or nitrogen dioxide (NO₂).

⁷ Duvall, R., A. Clements, K. Barkjohn, M. Kumar, D. Greene, T. Dye, V. Papapostolou, W. Mui, AND M. Kuang. PM10 Supplement to the 2021 Report on Performance Testing Protocols, Metrics, and Target Values for Fine Particulate Matter Air Sensors. U.S. Environmental Protection Agency, Washington, DC, EPA/600/R-23/145, 2024.

⁸ Duvall, R., A. Clements, K. Barkjohn, M. Kumar, D. Greene, T. Dye, V. Papapostolou, W. Mui, AND M. Kuang. NO₂, CO, and SO₂ Supplement to the 2021 Report on Performance Testing Protocols, Metrics, and Target Values for Ozone Air Sensors. U.S. Environmental Protection Agency, Washington, DC, EPA/600/R-23/146, 2024.

⁹ ASTM D8406-22. Standard Practice for Performance Evaluation of Ambient Outdoor Air Quality Sensors and Sensor-based Instruments for Portable and Fixed-point Measurement

¹⁰ ASTM D8559-24. Standard Specification for Ambient Outdoor Air Quality Sensors and Sensor-Based Instruments for Portable and Fixed-Point Measurement.

2 TEST PROTOCOL AND INFRASTRUCTURE FOR GAS SENSORS

2.1 Introduction

This chapter describes the test protocol for gas sensors. Test protocol for PM sensors is described in Chapter 3.

Note that different test protocols use different metrics for evaluation. Our test protocol is based on CEN/TS 17660-1 and we also calculate additional evaluation parameters (to be comparable with other test protocols, see also paragraph 1.3).

A review study performed by JRC¹¹ concluded that a majority of the studies only evaluate R^2 and slope and intercept of the regression line, when studying performance of AQ sensors, especially for field tests. For laboratory test, commonly reported variables are: R^2 , slope and intercept of the regression line, RMSE, Measurement uncertainty (U), MAE, bias.

Main issues related to gas sensors are:

- impact of RH
- impact of T
- cross sensitivity

The following paragraphs describe the exposure test chamber, the gas generation systems and the reference instruments used.

2.2 Test chamber or exposure chamber

2.2.1 Introduction

To assess the performance of sensor systems, the units are placed in an exposure chamber. In contrast to analyzers, most sensor units do not have active sampling. Therefore, they need to be placed in a test chamber which has a uniform concentration inside. Reference monitors need to sample from the test chamber.

A guidance for design of the exposure chamber is described in Annex C of CEN/TS 17660-1.

The guidance describes that:

- The exposure chamber should be made of inert materials;
- the exposure chamber should be large enough to test multiple sensor systems (the TS requires 3 systems to be tested);
- the exposure chamber should allow for generation of multi gas mixtures and be able to control and change T, RH, and possibly wind velocity and pressure;
- the exposure chamber should be equipped with a dynamic multi gas mixture composition system for generating, mixing and delivering known concentrations to the chamber;
- the gas concentration and other parameters should be recorded using calibrated measuring instruments during the tests;
- the homogeneity should be checked and satisfy requirements set in TS (par 7.2);

¹¹ Karagulian F.; Barbieri, M.; Kotsev, A.; Spinelle, L.; Gerboles, M.; Lagler, F.; Redon, N.; Crunaire, S.; Borowiak, 2019. A. Review of the Performance of Low-Cost Sensors for Air Quality Monitoring. Atmosphere, 10, 506.

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

- a zero air generator should be able to deliver high air flows with low impurity levels (lower than 1ppb for NO, NO₂, SO₂ and O₃; and lower than 15 ppb for CO);
- temperature and relative humidity should be controlled and stable to: RH \pm 5% and T \pm 1.0°C.

2.2.2 Description of the test infrastructure

The exposure chamber for sensor system evaluations is a stainless-steel test chamber (Figure 2), where the environmental conditions (e.g., temperature, RH, air exchange rate) are constantly controlled and monitored. The total internal volume of the chamber used for the tests is 237 dm³ (905(W) x 535(H) x 490(D) mm). The homogeneity of the chamber's inner atmosphere is provided by the active mixer installed at the middle of the back wall (below the air inlet) of the chamber. The air is introduced in the chamber via an inlet air distribution system (a multi-point diffuser installed over the entire length of the chamber) to assure equal dispersal of incoming air inside the chamber. To provide controlled temperature conditions during the tests, the exposure test chamber is placed inside a thermostatic room. The thermostatic room is capable to continuously regulate the temperature between -10°C ÷ +60°C with setting temperature accuracy of \pm 0.1 °C. The temperature and relative humidity inside the exposure test chamber are continuously monitored and recorded (as a minute average) using calibrated T/RH monitor (Testo 175H1, with accuracy of 0.4°C and 2% RH).

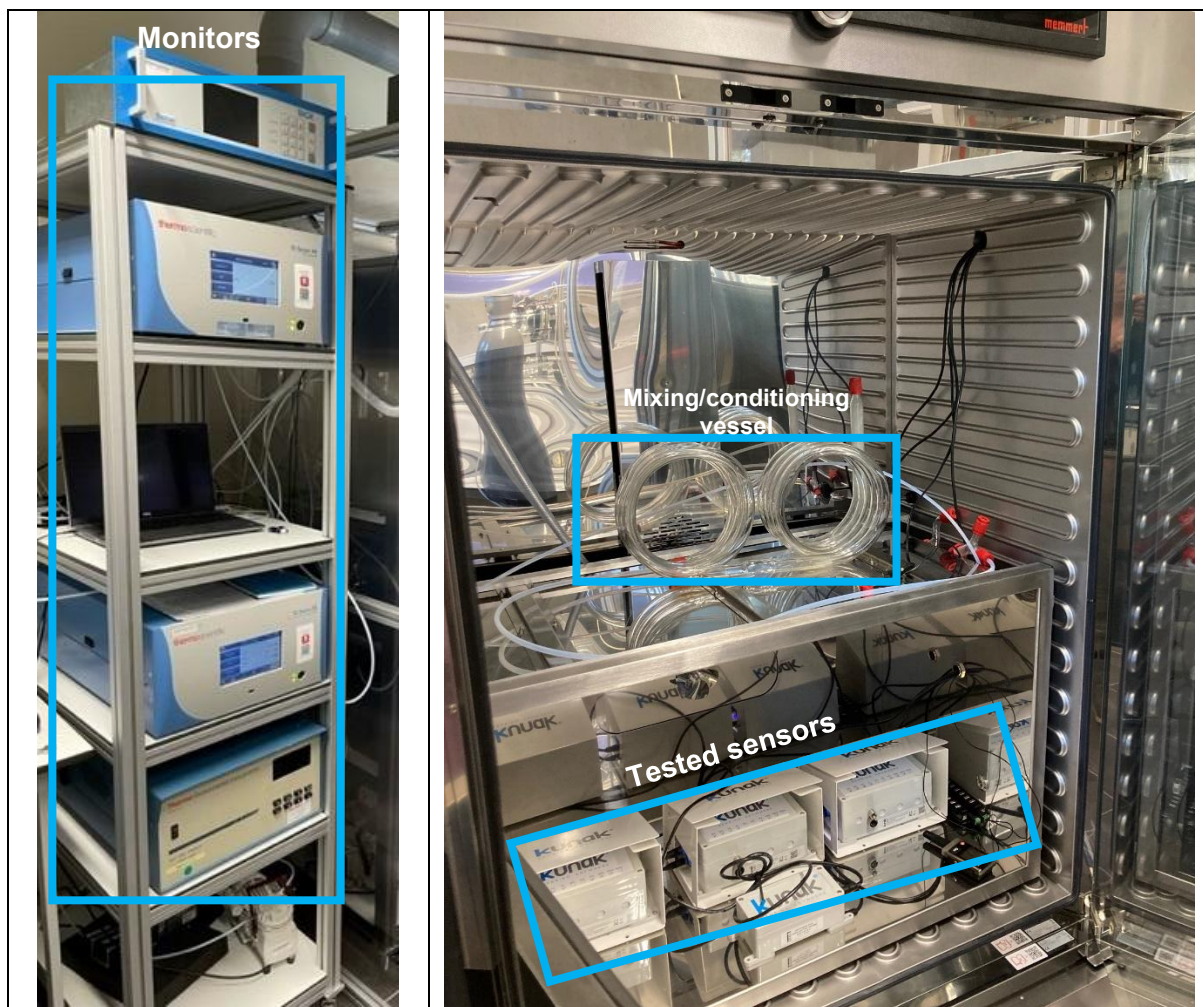


Figure 2: Exposure test chamber

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

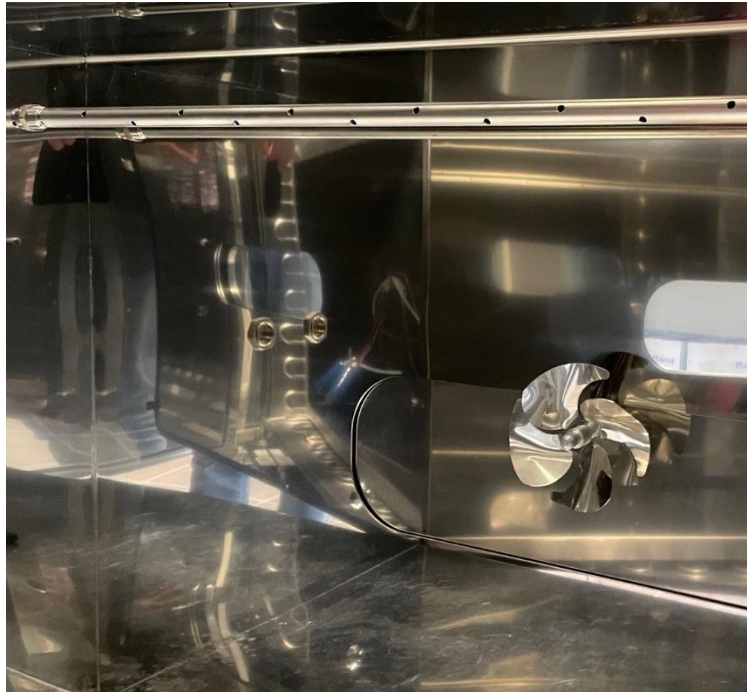


Figure 3: Mixing system of the exposure chamber



Figure 4: Gas generation system and humidification of inlet air to the test chamber

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

During the performed tests the exposure test chamber is supplied with a constant and controlled flow of carrier gas at rate of 50 L/min. The residence time t of the gas molecules inside the chamber for the used carrier gas flow rate (50 L/min) and total volume of 237 dm³ is estimated at 4.7 min, calculated by Eq. 1

$$t = \frac{V}{Q} = \frac{237 \text{ dm}^3}{50 \frac{\text{dm}^3}{\text{min}}} = 4.7 \text{ min} \quad 1$$

where:

- t is the residence time of the gas molecules inside the exposure test chamber, min;
- V is the volume of the exposure chamber, dm³;
- Q is the total carrier gas flow through the exposure test chamber, dm³/min.

To reach a steady-state condition (i.e., stable target gas concentration) inside the chamber, 2 – 3 residence times are needed, resulting to a total of about 9.4 – 14.1 minutes.

The validation of the test chamber is described in Annex A.

In addition to the described above 237 dm³ test chamber, a smaller 14 dm³ exposure chamber is also available, where much faster changes in the concentrations of the exposure target gases could be performed. These fast concentration changes are required for assessment the response time of the sensor systems under test.

The 14 dm³ exposure test chamber is made from glass with passive mixer (made from PTFE) to ensure homogeneity of the chamber's inner atmosphere (Figure 5). The temperature and relative humidity inside the exposure test chamber is continuously monitored and recorded using calibrated T/RH monitor (Testo 175H1, with accuracy of 0.4°C and 2% RH). The T/RH measurements are typically recorded as a minute average.

During the sensor systems testing the exposure test chamber is typically supplied with a constant and controlled flow of carrier and target gases at a rate of 37 L/min, which results to a residence time t of the gas molecules inside of 0.38 min (calculated by Eq. 1). The typical steady-state condition of the target gas concentration inside the chamber at those flows is typically reached in about 0.76 – 1.14 minutes.



Figure 5: 14 dm³ exposure test chamber

2.3 Gas mixture generation

Pure (pollutant free) air is used as carrier gases during the tests. The carrier gas is humidified to the desired humidity level prior entering the exposure test chamber using a Bronkhorst controlled evaporator mixer (Bronkhorst CEM EVAPORATOR W-202A). This instrument is capable to provide a constant and controlled humidification of the carrier gas flow ranging between 5% and 90% RH.

The various concentrations inside the exposure test chamber are produced by mixing a controlled flow of a target gas of known (certified) concentration (e.g., 932 ppm for NO₂) into the stream of the already humidified carrier gas (clean air). Both gas flows (i.e., target and carrier gas) are mixed before entering the exposure chamber in the mixing/conditioning vessel (Figure 2). For gases with higher adsorption potential to the walls of the mixing/conditioning vessel or short stability time, the target gas flow is added to the carrier gas stream at the inlet (after the mixing/conditioning vessel) of the air distribution system of the chamber. Precise mass flow controllers are used to control the flows of the target and carrier gas (clean air) during testing.

The target gas concentrations inside the exposure test chamber are calculated from the mass flow rate measurements of the target (e.g., NO₂) and carrier (clean air) gases following Eq. 2

$$C_{mix} = C_{ref} \times \frac{Q_{mt}}{Q_{mt} + Q_{mc}} \quad 2$$

Where,

- C_{mix} is the concentration of the mixture, ppb
- C_{ref} is the concentration of the target gas, ppb
- Q_{mt} is the target gas flow rate, cm³/min
- Q_{mc} is the carrier gas flow rate, cm³/min

A schematic overview of the gas mixing system is given in Figure 6.

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

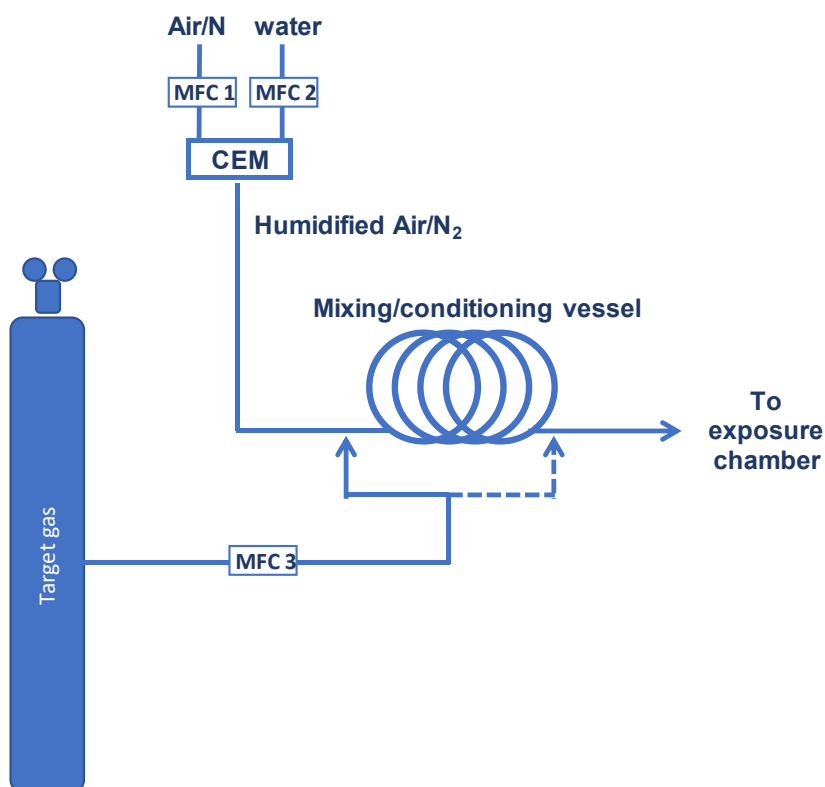


Figure 6: Schematic overview of gas mixing and humidification system

2.4 Measurement reference instruments

All gas flows during the experiments were controlled using high accuracy mass flow controllers (EL-FLOW SELECT, Bronhorst). All gas flows are measured using calibrated flow meters (Brooks Vol-U-Meter (standard measurement uncertainty $\pm 0.3\%$), Brooks Instruments and DryCal 1020 Primary Flow Calibration Standard (standard measurement uncertainty $\pm 0.5\%$), Mesa Labs).

Calibrated NO-NO₂-NO_x gas analyser 42iQ from Thermo Fisher Scientific (Chemiluminescence measurement principle, dynamic range 0.4 ppb – 1000 ppb, LOD: 0.050 ppb, zero drift negligible, linearity: $\pm 1\%$ of full-scale) is used to continuously monitor and record the NO₂ concentrations inside the exposure test chamber during the performed experiments.

Calibrated O₃ gas analyser 49iQ from Thermo Fisher Scientific (UV Photometric measurement principle, dynamic range 0 ppb – 200 ppm, LOD: 0.5 ppb, zero drift < 2.0 ppb, linearity: $\pm 1\%$ full-scale) is used to continuously monitor and record the O₃ concentrations inside the exposure test chamber during the performed experiments.

Calibrated T/RH monitor/logger (Testo 175H1, accuracy of 0.4°C and 2% RH) is used during the performed tests to log the temperature and relative humidity inside the exposure test chamber.

2.5 Test protocol

In this section the different test conditions for the performed evaluation tests on air quality measuring sensor system according to *CEN/TS 17660-1 “Air quality – Performance evaluation of air quality sensor systems – Part 1; Gaseous pollutants in ambient air”* are described using the target gas concentration levels for NO₂. For other target gases the concentration levels need to be adjusted as described in “Table 2 – Test levels for laboratory experiments” of *CEN/TS 17660-1 “Air quality – Performance evaluation of air quality sensor systems – Part 1; Gaseous pollutants in ambient air”* (see Table 1).

Table 1: Target gas test concentration levels defined in *CEN/TS 17660-1*.

Target gas	Concentration test levels. µg/m ³
SO ₂	0; 75; 125; 200
NO ₂	0; 40; 100; 140; 200
NO	0; 40; 100; 250; 400
O ₃	0; 60; 120; 180; 240
Benzene	0; 5; 25; 45
CO	0; 2 000; 7 000; 10 000

The durations of each individual test of the described below evaluation steps is set to at least 2 hours¹² after the moment when the conditions inside the exposure test chamber reach the conditions pre-defined for the selected test.

2.5.1 Step 1: Pre-test of sensor system under controlled conditions

During this step, the sensor system output is evaluated at different concentration levels of the target gas (NO₂) generated inside the exposure test chamber following the sequence shown in Figure 7.

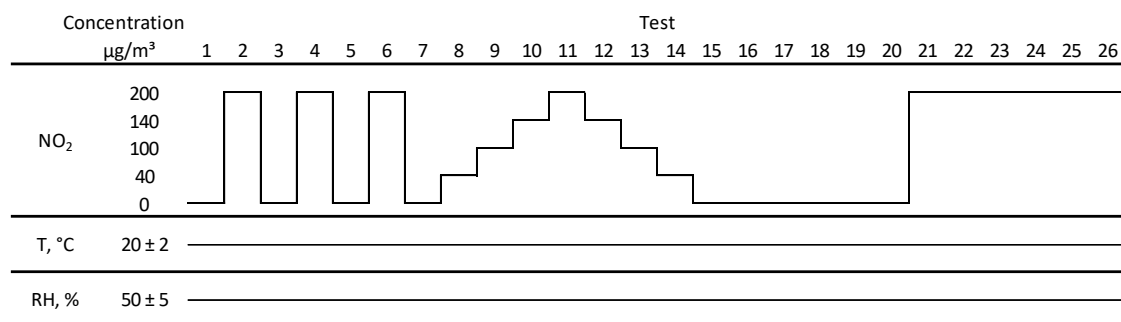


Figure 7: An overview of the test sequence and conditions performed in Step 1

During this step the environmental conditions inside the exposure test chamber are kept at constant temperature (with allowed deviation of $\pm 1^\circ\text{C}$) and relative humidity (with allowed deviation of $\pm 5\%$).

¹² 2 hours in addition to stable ambient conditions is set to stabilize the sensor signal. Test showed that fast changing ambient conditions could influence the stabilization time of sensors. We recommend taking this into account and if needed increase stabilization time (See also lessons learned).

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

2.5.2 Step 2: Temperature effect

In this step, the influence of different temperature levels on the sensor system output is evaluated. During this step, the output of the sensor system at $0 \mu\text{g}/\text{m}^3$ and $200 \mu\text{g}/\text{m}^3$ of target gas (NO_2) is assessed at different levels of the temperature inside the test chamber, following the sequence shown in Figure 8.

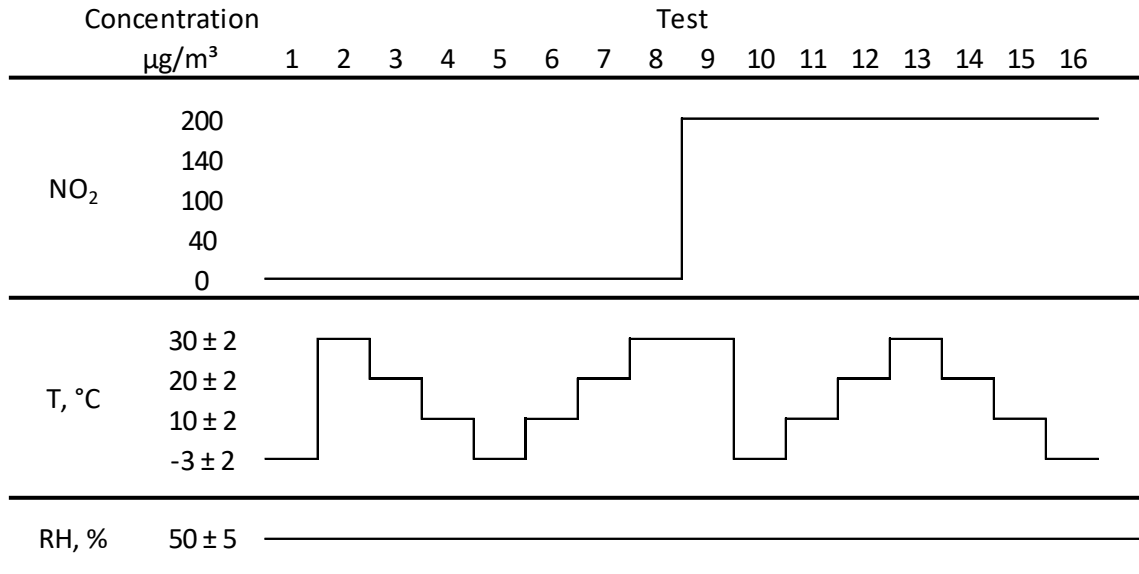


Figure 8: An overview of the test sequence and conditions performed in Step 2

During this step the relative humidity inside the test chamber is kept constant at desired level with allowed deviation of $\pm 5\%$.

2.5.3 Step 3: Humidity influence

The influence of different relative humidity levels on the sensor system output is evaluated during this step. The output of the sensor system at $0 \mu\text{g}/\text{m}^3$ and $200 \mu\text{g}/\text{m}^3$ of target gas (NO_2) is assessed at different relative humidity levels inside the test chamber. The test is conducted following the sequence shown in Figure 9.

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

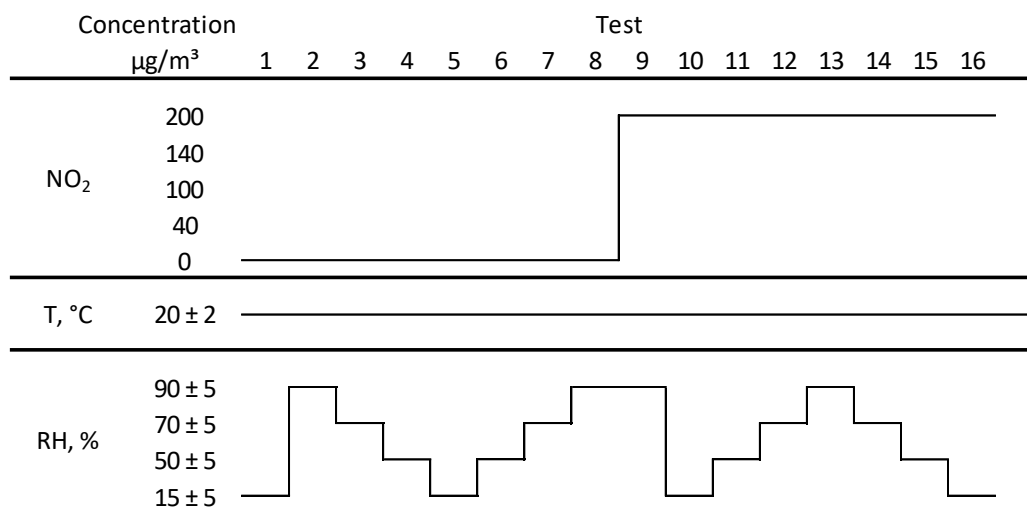


Figure 9: An overview of the test sequence and conditions performed in Step 3

During this step the temperature inside the test chamber is kept constant at desired value with allowed deviation of $\pm 1^\circ\text{C}$.

2.5.4 Step 4: Cross-sensitivities of gaseous interfering compounds

The influence of other gaseous compounds onto the sensor system output is evaluated during this step. The output of the sensor system at $0 \mu\text{g}/\text{m}^3$ and $100 \mu\text{g}/\text{m}^3$ of target gas (NO₂) is evaluated at different concentrations of the selected interfering compound (O₃)¹³. The test is conducted following the sequence shown in Figure 10.

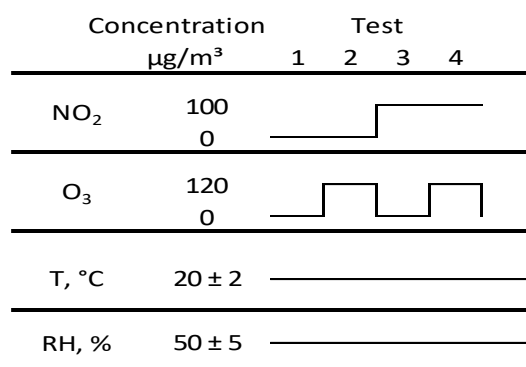


Figure 10: An overview of the test sequence and conditions performed in Step 4

During this step the environmental conditions inside the exposure test chamber are kept at constant temperature (with allowed deviation of $\pm 1^\circ\text{C}$) and relative humidity (with allowed deviation of $\pm 5\%$).

¹³ A list of interfering components and the corresponding concentration levels for other target gases can be found in "Table 5 – Interfering gases and VOC mixtures for cross-sensitivity tests" of CEN/TS 17660-1 "Air quality – Performance evaluation of air quality sensor systems – Part 1; Gaseous pollutants in ambient air".

2.6 Sensor evaluation parameters

The sensor evaluation parameters are summarized below.

Table 2: Summary of sensor evaluation parameters

CEN/TS/17660-1	Test
Response time (s)	Step 1
Lack of fit (LoF)	Step 1
Repeatability r	Step 1
Limit of detection (LOD)	Step 1
Between sensor uncertainty or between-instrument uncertainty	Step 1
Slope, intercept regression lin (lab)	Step 1
Impact of T	Step 2
Impact of RH	Step 3
interferences	Step 4
Other	
RMSE; MAE, MBE	Step 1
AQ-SPEC	
Accuracy	Step 1
Intra-model variability (IMV)	Step 1

2.6.1 Response time

No infrastructure for larger sensors is available at this moment. We have adapted infrastructure for small sensor units.

2.6.2 Lack of Fit

The uncertainties due to lack-of-fit of the regression function of the tested sensor systems were calculated using the data obtained during the concentration ramping tests. The regression function of the individual sensor system is determined by plotting the obtained sensor response versus the measured reference values during the tests in Step 1 (see 2.5.1). A linear regression function in the form of Eq. 3 is used.

$$Res_i = a + bRef_i \quad 3$$

where:

Res_i is the concentration of the target parameter obtained from the tested device.

Ref_i is the reference concentration level of the target parameter.

a is the intercept of the regression model.

b is the slope of the regression model

The linear regression function is made by calculation of the function according to Eq. 4

$$Res_i = A + b(Ref_i - Ref_a) \quad 4$$

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

For the regression calculation all measuring points (including zero) are considered. The total number n of measuring points is equal to the number of concentration levels multiplied by the number of repetitions at each concentration level.

The coefficient A is obtained according to Eq. 5

$$A = \sum_{i=1}^n \frac{Res_i}{n} \quad 5$$

where:

A is the average value of Res_i

Res_i is the concentration of the target parameter obtained from the sensor system.

n is the number of measuring points.

The coefficient b is obtained from Eq. 6

$$b = \frac{\sum Res_i \times (Reff_i - Reff_a)}{\sum (Reff_i - Reff_a)^2} \quad 6$$

where:

b is the slope of the regression model.

$Reff_a$ is the average of the $Reff_i$ calculated according to Eq. 7

$$Reff_a = \sum \frac{Reff_i}{n} \quad 7$$

The Eq. 4 is converted to Eq. 3 through calculation of a by Eq. 8

$$a = A - b \times Reff_a \quad 8$$

The average of Res_i is calculated according to Eq. 9

$$Res_a = \sum_{i=1}^m \frac{Res_i}{m} \quad 9$$

where:

Res_a is the average value of Res_i at the same concentration level.

Res_i is the concentration of the target parameter obtained from the sensor system.

m is the number of repetitions at one and the same concentration level.

The residual of each average at each concentration level is calculated following Eq. 10

$$residue_{c,i} = Res_a - (a + b \times Reff_i) \quad 10$$

where:

$residue_{c,i}$ is the residual of each average at each concentration level.

Res_a is the average concentration of the target parameter obtained from the sensor system at each concentration level.

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

The standard uncertainty of the lack of fit of the regression function of each parameter is calculated according to Eq. 11

$$u(lof) = \frac{residue_{c,i,max}}{\sqrt{3}} \quad 11$$

where:

$u(lof)$ is the standard uncertainty associated with lack of fit.
 $residue_{c,i,max}$ is the maximum residual of individual $residue_{c,i}$

The expanded uncertainty associated with lack of fit $U(lof)$ is calculated according to Eq. 12

$$U(lof) = k \times u(lof) \quad 12$$

where:

$U(lof)$ is the extended uncertainty associated with lack of fit.
 k is the coverage factor ($k = 2$).

2.6.3 Repeatability and Limit of Detection (LOD)

The repeatability of a tested sensor system's response is evaluated by calculating the standard deviation of the output of the sensor system for at least six consecutive individual measurements at constant conditions (concentration, T, and RH) during Step 1 (see 2.5.1). To calculate the standard deviation of the repeatability the Eq. 13 is used.

$$s_r = \sqrt{\frac{\sum_{i=1}^n (C_i - C_a)^2}{n-1} - \frac{\sum_{i=1}^n (x_i - x_a)^2}{n-1}} \quad 13$$

where:

s_r is the standard deviation of repeatability.
 C_i is the i^{th} value of the sensor system response.
 C_a is the average of the values C_i .
 x_i is the i^{th} x-value (reference measurement).
 x_a is the average of x_i .
 n is the number of measurements.

The repeatability of the sensor system is calculated using the Eq. **Error! Reference source not found.**

$$r = t_{n-1,\alpha} \times s_r \quad 14$$

where:

r is the repeatability of the sensor system
 s_r is the standard deviation of repeatability.
 $t_{n-1,\alpha}$ is the two-sided Students t-factor at the confidence level of $1-\alpha$, with $n-1$ degrees of freedom.

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

To calculate the limit of detection (LOD), the standard deviation of the repeatability of the sensor signal while the sensor system is exposed to zero air is used. The LOD of the sensor system is calculated using Eq. 15.

$$LOD = k \times s_r \quad 15$$

where:

LOD is the Limit of Detection of the sensor system for the target component.

s_r is the standard deviation of repeatability.

k is coverage factor ($k = 3$).

2.6.4 Accuracy (AQ-SPEC)

The accuracy assessed for the tested sensor systems is defined as the degree of closeness between the obtained sensor response and the measured reference values during the performed tests. The accuracy of the sensor systems is calculated considering the outputs obtained during the steady-state period for each individual exposure concentration level, using Eq. 16.

$$A(\%) = 100 - \frac{|\bar{X} - \bar{R}|}{R} * 100 \quad 16$$

where:

$A(\%)$ is the accuracy of the sensor system.

\bar{X} is the average concentration measured from the sensor system at the steady-state period at selected concentration level.

\bar{R} is the average concentration from the reference measurement obtained during the same steady-state period.

2.6.5 Intra-Model Variability (IMV) (AQ-SPEC)

The intra-model variability (IMV) is a parameter used for assessment of how close the measurements between the tested sensor systems are to each other. The outcomes from all tested sensor systems obtained under same test conditions are used to calculate this parameter. The IMV is calculated according to Eq. 17.

$$IMV(\%) = \frac{Mean_{highest} - Mean_{lowest}}{Mean_{average}} * 100 \quad 17$$

where:

$IMV(\%)$ is intra-model variability.

$Mean_{highest}$ is the highest of the sensor system.

$Mean_{lowest}$ is the lowest of the sensor system.

$Mean_{average}$ is the average of the sensor system.

2.6.6 Between instrument uncertainty of the sensor systems (BIU)

Between instrument uncertainty (BIU) of the tested sensor systems is assessed during the exposure of at least three sensor systems simultaneously to the same test conditions. The values of the BIU are calculated from the measurements obtained from the sensor systems during the steady state period for different concentration level performed in Step 1 (see 2.5.1).

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

The BIU values of the sensor systems tested during this study are calculated using Eq. 18

$$u(bs, s) = \sqrt{\left(\frac{\sum_{i=1}^n \sum_{j=1}^{n_s} (y_{i,j} - \bar{y}_i)^2}{n(n_s - 1)} \right)} \quad 18$$

where:

$u(bs, s)$ is the between instrument uncertainty of the tested sensor systems.

$y_{i,j}$ is the value of the sensor system output for period i of sensor system j .

\bar{y}_i is the average for period i of the n_s replicate sensor systems.

\bar{R} is the average concentration from the reference measurement during the same steady-state period.

n is the number of the valid data points from the measurements.

n_s is the number of replicate sensor systems.

2.6.7 Mean Absolute Error (MAE)

The mean absolute difference between the measurements obtained by the tested sensor systems and the reference instrument measurement are expressed with the MAE. The MAE values are calculated from the measurements obtained during the steady state period for each concentration level.

The MAE values of the sensor systems tested during this study are calculated using Eq. 19.

$$MAE = \frac{1}{n} \sum_{i=1}^n |(\bar{X} - \bar{R})| \quad 19$$

where:

MAE is the mean absolute error.

\bar{X} is the average concentration measured from the sensor system during the steady-state period at selected concentration level.

\bar{R} is the average concentration from the reference measurement during the same steady-state period.

n is the number of the valid data points from the measurements

2.6.8 Mean Bias Error (MBE)

The MBE is defined as the difference between the measurements from the tested sensor systems and the reference instrument during the same steady-state period for each concentration level. The MBE is used to assess the tendency of the tested sensor systems to underestimate (negative MBE values) or overestimate (positive MBE values) the reference instrument measurements. The MBE in this study is calculated using Eq. 20.

$$MBE = \frac{1}{n} \sum_{i=1}^n (\bar{X} - \bar{R}) \quad 20$$

where:

MBE is the mean absolute error.

\bar{X} is the average concentration measured from the sensor system during the steady-state period at selected concentration level.

\bar{R} is the average concentration from the reference measurement during the same steady-state period.

n is the number of the valid data points from the measurements.

2.6.9 Root Mean Square Error (RMSE) and Normalized Root Mean Square Error (NRMSE)

RMSE is used as a metric to assess the error associated with the measurements of the tested sensor systems. RMSE is defined as the error between the sensor system and the reference instrument. This parameter is also calculated considering the data obtained during the steady-state period for each of the tested concentration levels. The RMSE and NRMSE are calculated according to Eq. 21 and Eq. 22.

$$RMSE = \sqrt{\frac{\sum_{i=1}^n (\bar{X} - \bar{R})^2}{n}} \quad 21$$

where:

RMSE is the root mean square error.

\bar{X} is the average concentration measured from the sensor system during the steady-state period at selected concentration level.

\bar{R} is the average concentration from the reference measurement at the same steady-state period.

n is the number of the valid data points from the measurements

$$NRMSE(\%) = \frac{RMSE}{\bar{R}} * 100 \quad 22$$

where:

NRMSE is the normalized root mean square error.

\bar{R} is the average concentration from the reference measurement during the steady-state period at selected concentration level.

2.6.10 Impact of Temperature

The influence of the temperature onto the measurements of the target component from the sensor system is evaluated using the system's output obtained during the tests performed at step 2 (see 2.5.2).

To assess the temperature effect, two regression models of the sensor system's output are plotted against the temperature at zero target gas (tests 2 to 8), and at high level of target gas (tests 10 to 16). The slope and intercept of each of the models are calculated.

The standard uncertainty used to estimate the effect of the temperature on the sensor system measurements is calculated using Eq.23

$$u(T) = b_a \frac{T_{max} - T_{min}}{\sqrt{12}} \quad 23$$

where,

$u(T)$ is the standard uncertainty of the estimated temperature effect.

b_a is the average of the slopes of the regression models at zero and high level of target gas.

T_{max} is the maximum value of the tested temperature.

T_{min} is the minimum value of the tested temperature.

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

The $T_{\max} - T_{\min}$ parameter for the calculations is set at 30°C. This can be adapted if sensor systems need to be evaluated for other conditions (use)

It is also recommended to plot the sensor values (for zero and high level) in function of T, together with the reference values. If reference values show a trend this should be considered.

2.6.11 Impact of RH

The influence of the humidity onto the measurements of the target component from the sensor system is evaluated using the system's output obtained during the tests performed at step 3 (see 2.5.3).

To assess the humidity effect, two regression models of the sensor system's output are plotted against the relative humidity measured inside the exposure test chamber at zero target gas (tests 2 to 8), and at high level of target gas (tests 10 to 16). The slope and intercept of each of the models are calculated.

The standard uncertainty used to estimate the effect of the humidity on the sensor system measurements is calculated using Eq.24

$$u(T) = b_a \frac{RH_{\max} - RH_{\min}}{\sqrt{12}} \quad 24$$

Where,

$u(\text{RH})$ is the standard uncertainty of the estimated humidity effect.

b_a is the average of the slopes of the regression models at zero and high level of target gas.

RH_{\max} is the maximum value of the tested relative humidity.

RH_{\min} is the minimum value of the tested relative humidity.

The $RH_{\max} - RH_{\min}$ parameter for the calculations is set at 50%. This can be adapted if sensor systems need to be evaluated for other/specific conditions (use).

It is also recommended to plot the sensor values (for zero and high level) in function of T, together with the reference values. If reference values show a trend this should be considered.

2.6.12 Impact of interferences

The standard uncertainty due to selected interferent compound is calculated from the data obtained during the performed test in Step 4 (see 2.5.4).

The influence quantity of the selected interferent is calculated at zero (Tests 1 and 2 (Figure 10)) and at test level (Tests 3 and 4 of the target gas). The overall quantity of the interferent Y_{int} at the tested limit values is estimated using Eq. 25

$$Y_{\text{int}} = (Y_{\text{int},c} - Y_{\text{int},z}) \times \frac{L}{X_c} + Y_{\text{int},z} \quad 25$$

where,

Y_{int} is the influence quantity of the interferent at limit value L^*

$Y_{\text{int},c}$ is estimated influence quantity of the interferent at presence of target gas

$Y_{\text{int},z}$ is estimated influence quantity of the interferent at zero target gas level

L is the level for which the data quality objective is set.

X_c the high level of the test gas during interference testing

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

* the values are displayed in Table 3.

- Note that the values correspond to the limit values of the current AQ Directive and could be changed in future.
- Note that for some compounds there are different limit values at different averaging time; Table 3 summarises the most appropriate limit values that are used for the evaluation (not taking into account the averaging time for the respective values).

Table 3: Selected limit values (LV) of compounds

Compound	Limit value ($\mu\text{g}/\text{m}^3$)*
Benzene	5 (y)
O ₃	120 (8h)
NO ₂	200 (h)
NO	-
SO ₂	10 (24h)
Compound	Limit value (mg/m^3)*
CO	10 (8h)

* at 20°C and 101.3 kPa

The standard uncertainty due to interference at maximum concentration of the interferent is calculated according to Eq.26

$$u(int) = \left| \frac{Y_{int}}{X_{int}} \right| \times \frac{C_{int,max} - C_{int,min}}{\sqrt{3}} \quad 26$$

where,

$u(int)$ is the standard uncertainty associated with the influence of a gaseous interferent.

$C_{int,max}$ is the maximum concentration of the interferent compound (see Table 4)

$C_{int,min}$ is the minimum concentration of the interferent compound

X_{int} the high level of gaseous interfering compound

According to the requirements stated in *CEN/TS 17660-1* the $C_{int,min}$ values are set to zero and $C_{int,max}$ are defined according to the interference compound and listed in Table 4.

Table 4: Max concentrations of interfering compounds for calculation of uncertainty

Interference compound	Target max concentration ($C_{int,max}$) ($\mu\text{g}/\text{m}^3$)*
Benzene	3
O ₃	100
NO ₂	100
NO	200
CO	800
SO ₂	10

* at 20°C and 101.3 kPa

2.7 Lessons learned from first tests

Sensors have some peculiarities that need specific attention when testing them. When testing sensors which performance is unknown, it is recommended to start with some preliminary tests. Also, the response time to varying test conditions needs to be evaluated. E.g., it was observed that a fast change in RH might result in long period before stabilization of sensor signal, which needs to be considered when assessing impact of RH. Also, some sensors round negative values to zero which might influence their performance. Some examples are given in paragraphs below.

2.7.1 Preliminary test before starting extended test program

It is recommended to evaluate the data of the first step prior to proceeding with interference test (Step 2 - 4); in this way, extensive testing with very bad performing sensors can be prevented.

If sensors have a good data platform, sensor output can be followed up easily. However, some sensors only have local data stored and this means that the test chamber needs to be opened for retrieving the datasets.

The example below shows a sensor that was tested as part of the benchmark study¹⁴ for VMM. One of the sensors showed an almost constant zero value for different steps. However, detailed comparison with reference data and showed that this was due to a bad calibration since we observed that a factor could be applied resulting in better response to the applied concentrations.

¹⁴ Hofman, J.; Lazarov, B.; Stroobants, C.; Elst, E.; Smets, I.; Van Poppel, M. Portable Sensors for Dynamic Exposure Assessments in Urban Environments. State of the Sci. 2024, <https://doi.org/10.3390/s24175653>

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

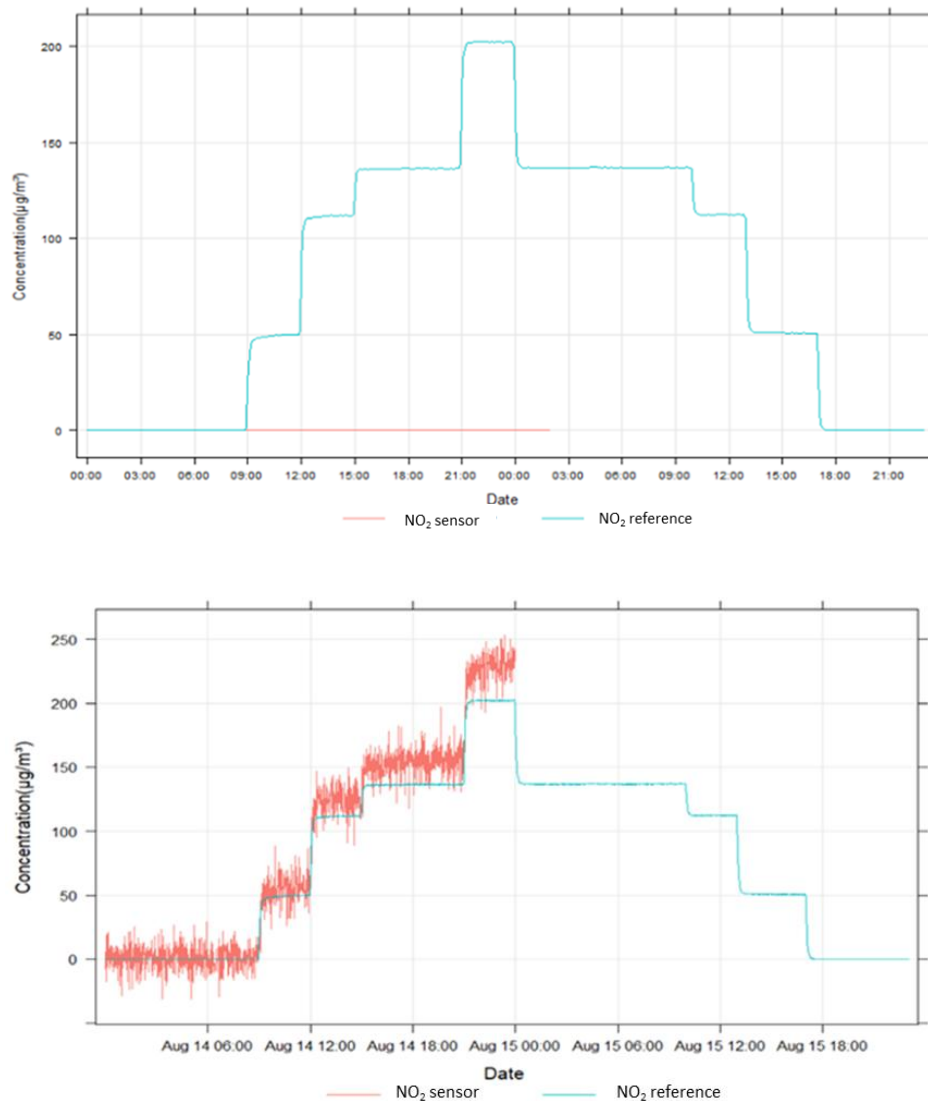


Figure 11: Example of bad calibrated sensor output (top) and improved signal using simple calibration model (bottom)

2.7.2 Impact of changing ambient conditions on stabilization of sensor signal

The figure below shows test of RH of NO₂ sensors. The tests were performed at zero concentrations and shows some memory effect of the sensors; the last blue peak appeared after a strong RH change, and it took about 6h before the sensor signal was again at zero.

The figure below shows sensor and reference values at span concentration for different RH. We observed that on some occasions changing RH (e.g. from 50% to 75%) requires a longer stabilization time of the sensor of more than 6 hours. The transient effect is observed in both directions (increasing and decreasing RH).

Note also that when settings for NO₂ are not changed, concentrations decrease with increasing RH due to some reaction of NO₂ with water vapor. We correct for this by increasing the NO₂ flow with increasing RH.

2. TEST PROTOCOLS AND INFRASTRUCTURE FOR GAS SENSORS

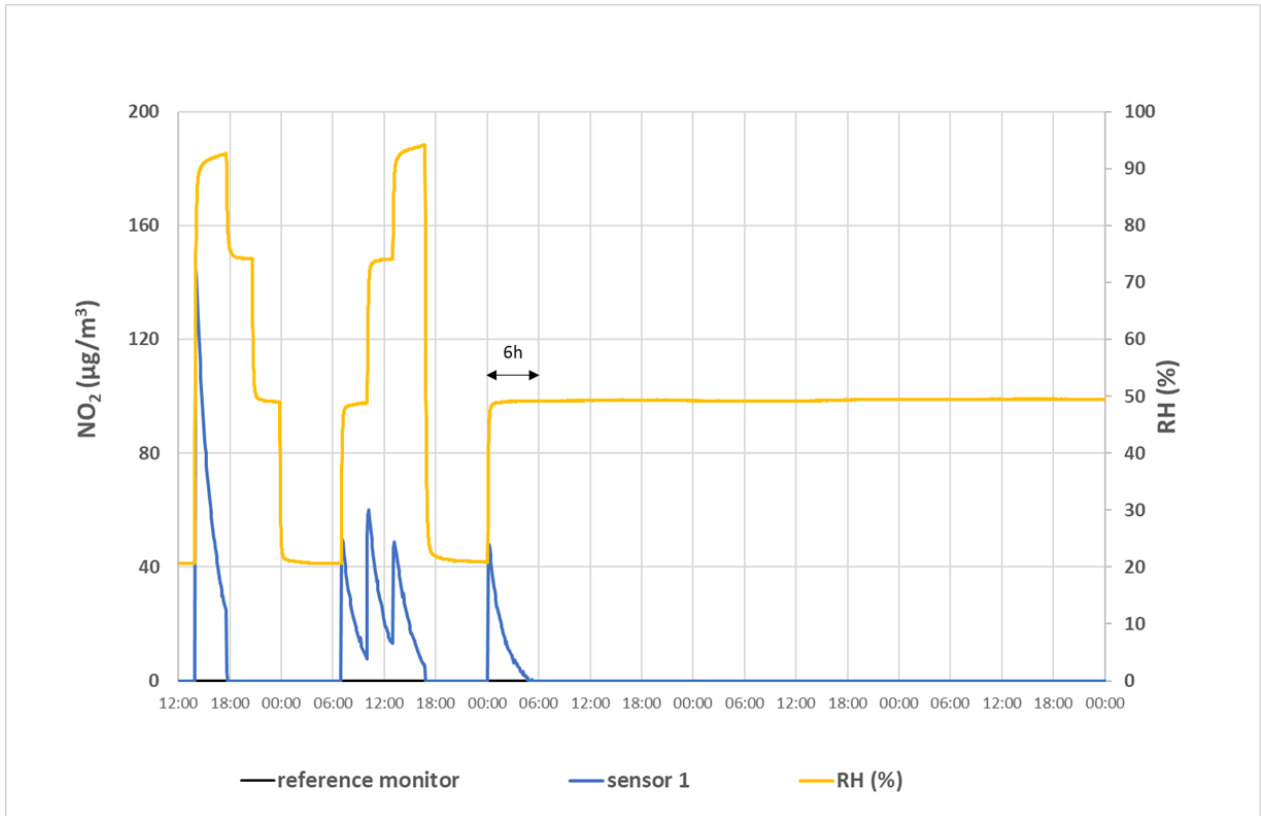


Figure 12: NO₂ sensor signal with changing RH conditions at zero concentration

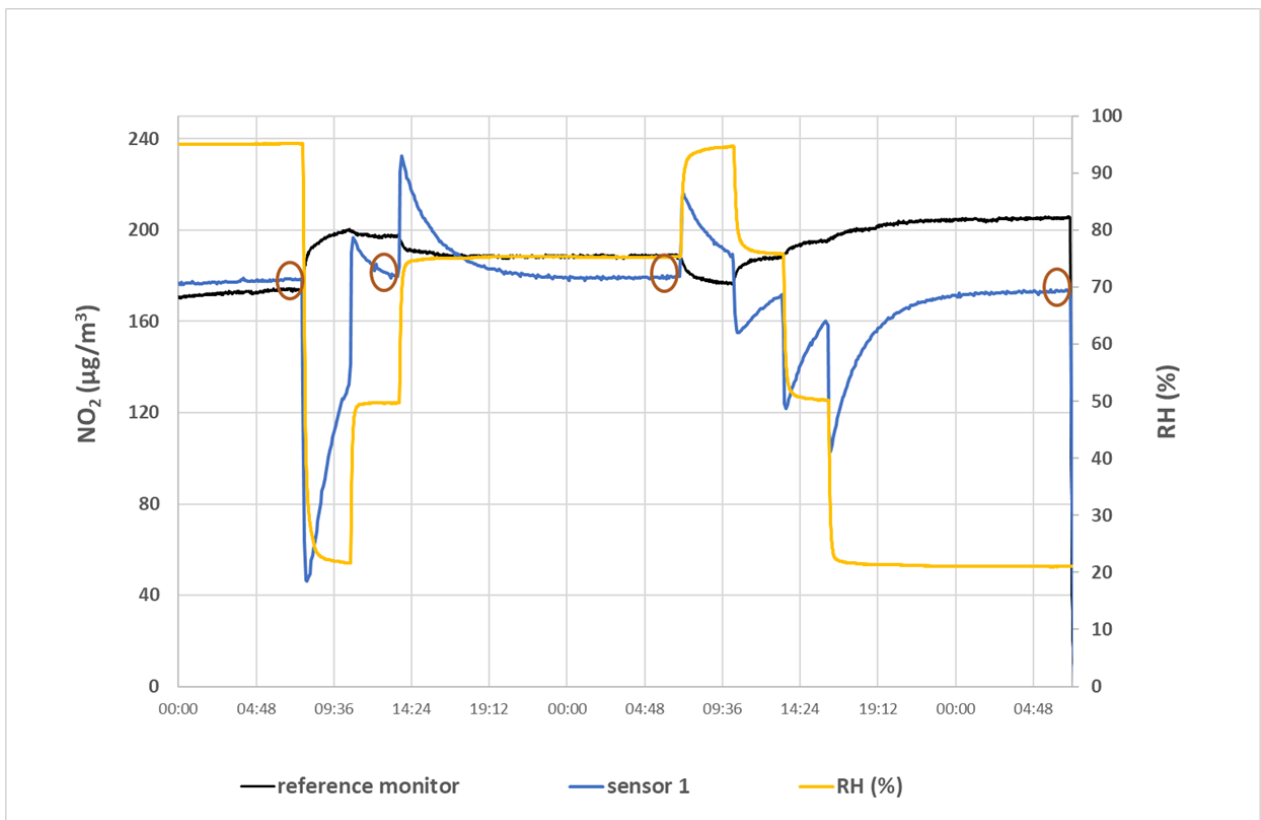


Figure 13: NO₂ sensor signal with changing RH conditions at span concentration

2.7.3 Zero values

Figure 14 below shows the sensor signal and reference signal for test 3 in Step 1. The aim is to calculate LOD as explained in 2.6.3 (following CEN/TS17660-1). The problem is that some sensors (as is the case for the example given below) round value at zero if sensors value tend to go below zero. As a result, this method (using a standard deviation of repeatability at zero) is not suitable for sensors that round negative values to zero because the zero values do not show a variability around zero and as a result, LOD would be zero ($LOD = 0$). A way to overcome this is to use the second lowest concentration to calculate LOD.

The AQ-SPEC protocol has a way to overcome this (for VOC sensors)¹⁵ by exposing the sensors to serially decreasing concentrations to until the lowest-possible concentration is reached that elicits a response that is statistically different than zero or the signal noise. As also indicated by AQ-SPEC, this approach is extremely resource- and time-intensive. The detection limit is then calculated $3.3 \times \sigma / S$ (with σ the residual standard deviation of the regression line and S the slope of the regression line). This is not included in this protocol.

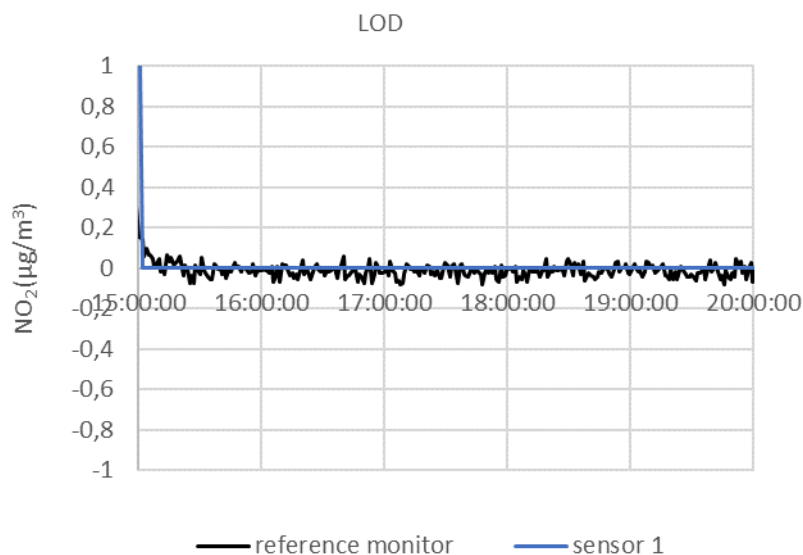


Figure 14: Example of sensor signal at zero concentration plotted, together with signal of reference monitor for NO_2

¹⁵ Papapostoulou et al., 2023. AQ-SPEC Laboratory Evaluation of VOC Sensors. <https://www.aqmd.gov/aq-spec/evaluations/voc/laboratory>

2.8 Evaluation of results

This protocol does not describe an evaluation of results. How performance of sensors is evaluated depends on requirements and application of use of sensors.

For the use of sensors in regulated monitoring as individual sensor systems as indicative measurements or objective estimations, we refer to TS-17660 which describes three classes, corresponding to indicative measurements, objective estimations, and other use with more relaxed requirements. Note that the evaluation of the TS also considers field tests (which are not described in this test protocol).

Note that we did not explicitly describe the long-term drift test as was described in the TS-17660.

The AQ-SPEC does not give a classification but an evaluation for reporting the results.

3 TEST PROTOCOLS FOR PM SENSORS

3.1 Aim and specific needs for PM sensors

PM sensors are different from gas sensors and have specific needs in terms of testing. Moreover, PM reference instruments measure at 24h resolution and reference mixtures are not available like for gaseous compounds.

The outcome of the VAQUUMS project - which tested PM sensors on a larger scale for the first time in Flanders - showed that PM sensors can show a dependency on RH and temperature, which can result of overestimation of sensor readings at high RH. In addition, when sensors are intended to measure PM₁₀, it should be checked if PM₁₀ is not calculated based on PM_{2.5} and if the coarse PM is estimated correctly.¹⁶ An example is given in Figure 15.

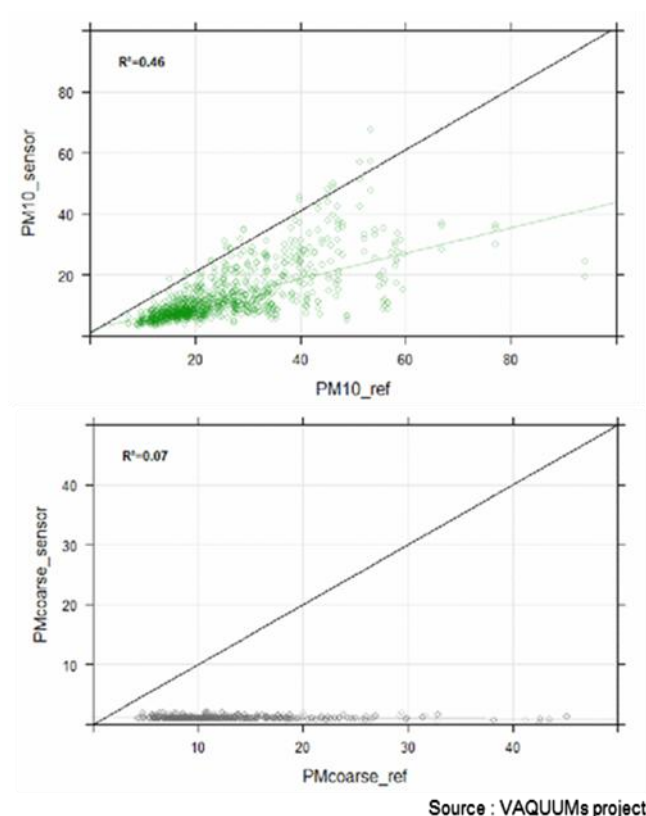


Figure 15: PM₁₀ and PM_{coarse} of sensor in function of reference for sensor tested in VAQUUMS project

The evaluation of PM sensors following the TS-17660 – Part 2 (which is currently in preparation) includes mainly field tests and has a laboratory test included to evaluate the ability of the sensor to measure PM₁₀. In addition, it suggests some calculations to evaluate the RH dependency and ability to measure PM₁₀ using field test data.

¹⁶ Vercauteren, J. Performance Evaluation of Six Low-Cost Particulate Matter Sensors in the Field; VAQUUMS: Antwerp, Belgium, 2021; Available online: https://www.vaquums.eu/sensor-db/tests/life-vaquums_pmfieldtest.pdf/view (accessed on 30 August 2024)

This document focuses mainly on laboratory tests for evaluating the LoF and PM coarse test of PM sensors. In future, the laboratory infrastructure would be further developed to be able to perform the evaluation of the T and RH effects on the PM sensors but given the direction of the TS (WG42) we focus here on these tests.

3.2 Particle generation and exposure chamber

3.2.1 PM generation system

The various PM concentrations inside the test chamber are generated using a Particle Dispenser System (PALAS) with Dolomite dust. The particle loaded airflow from the dispenser is inject into a mixing duct, where the initial high particle loaded air flow is diluted by mixing it with additional particles free air (Figure 16). A portion of this diluted mixed particle loaded air is then sampled through a Venturi feed and injected to the inner atmosphere of the exposure chamber. The rest of the mixed particle loaded air in the mixing duct is vented and removed from the system. The concentration of the particles injected to the exposure test chamber are controlled using either the feed rate of the particles in the particle loaded airflow from the PALAS dispenser system or by the rate of particle dilution (by controlling the inflow rate of the particle free air) into the mixing duct supplying the particles through the Venturi feeder into the exposure test chamber.

The actual concentration and particle size distribution inside the exposure test chamber is continuously monitored using a calibrated optical particle counter (Grimm 11-D with heated sampling inlet line (EDM 264, Grimm), particle size range: 0.253 – 35.15 μm , 0 $\mu\text{g}/\text{m}^3$ - 100 mg/m^3 , reproducibility: $\pm 3\%$ for total measuring range).

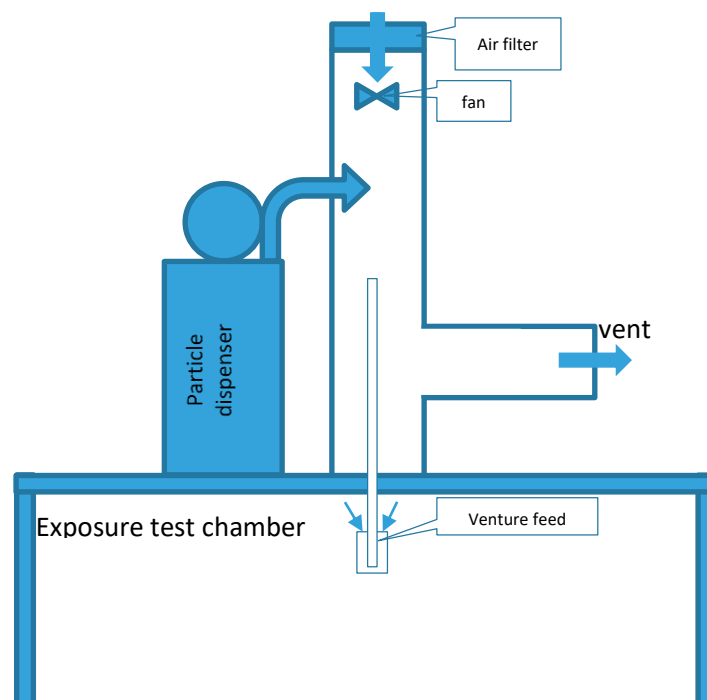


Figure 16: Schematic of particle generation and distribution system

3.2.2 PM exposure chamber and reference instrument

The PM exposure chamber for testing of PM sensors is shown in Figure 17. The chamber is made of aluminum and has ventilators inside to ensure a good homogeneity. The ventilators will also improve the stability of the concentrations since dust has the tendency to settle resulting in fast decreasing concentrations.

The test chamber is equipped with a large (around 50 cm in diameter) sealed door to easily place sensors inside and at the same time quickly ventilate the chamber to reach background concentrations.

The homogeneity of the PM concentrations inside the chamber was evaluated and the settings of the particle generation system to obtain the most optimal conditions for representative and stable PM concentrations were established (see Annex B).



Figure 17: Exposure chamber with generation system on top (left) and reference instrument

3.3 Coarse laboratory test

Coarse particles are defined here as $PM_{10-2.5}$, difference between the reported PM_{10} and reported $PM_{2.5}$ values.

For PM a laboratory test was developed, to evaluate the potential of the sensor to measure the coarse fraction ($PM_{2.5-10} = PM_{10} - PM_{2.5}$) because it is known that some low-cost sensors calculate PM_{10} concentrations based on the measured concentrations of $PM_{2.5}$ and sensors can have various response characteristics regarding size selectivity (Molina et al. 2023¹⁷; Kuula et al. 2019¹⁸).

Sensitivity of PM sensor to the coarse (2.5-10 μ m) particle fraction. The test consists of sequentially dosing of larger particles and smaller particles to the exposure chamber where sensors are installed.

As part of the reference task, VITO contributed to the development of a laboratory test for the TS in CEN/TC264/WG42.

3.3.1 PM generation for PM_{coarse} test

During this test, PM sensor systems are exposed to coarse and fine dust inside the exposure test chamber to evaluate their response to $PM_{10-2.5}$. The size fractions generated inside the test chamber must fulfil the following requirements:

- coarse test: > 80 % $PM_{10-2.5}$ mass concentration in PM_{10}
- fine test: < 20 % $PM_{10-2.5}$ mass concentration in PM_{10}

The dust for the two tests should have the same composition and it is recommended that the concentrations are similar.

An equivalent method (Grimm 11-D EDM264) is used to measure $PM_{2.5}$ and PM_{10} and calculate $PM_{10-2.5}$.

The particle generation during the coarse test utilized an aerosolizer (from the Grimm 7.851 aerosol generator system), installed directly inside the exposure chamber (Figure 18). The aerosolizer uses compressed air to aerosolize the dust placed in a glass container on the device. The pressure of the applied compressed air as well as the duration and the frequency of the 'puffs' were used to generate different concentration and size distributions of the particles inside the chamber during the test.

We extensively tested the setup by varying the frequency of 'puffs', the filling of the vial and by cleaning the vial between experiments.

We also observed that the Cospheric dust tend to 'stick' after some time and needs to be stored in dry conditions.

¹⁷ Molina Rueda E, Carter E, L'Orange C, Quinn C, Volckens J., 2023. Size-Resolved Field Performance of Low-Cost Sensors for Particulate Matter Air Pollution. *Environmental Science & Technology Letters*, 10(3):247-253.27.

(20) (PDF) Portable Sensors for Dynamic Exposure Assessments in Urban Environments: State of the Science. Available from: https://www.researchgate.net/publication/381627545_Portable_Sensors_for_Dynamic_Exposure_Assessments_in_Urban_Environments_State_of_the_Science#fullTextFileContent [accessed Aug 30 2024].

¹⁸ Kuula J, Mäkelä T, Aurela M, Teinilä K, Varjonen S, Gonzales O, Timonen H: Laboratory evaluation of particle size-selectivity of optical low-cost particulate matter sensors; 2019.28.

3. TEST PROTOCOLS FOR PM SENSORS

For these tests we used:

Monodisperse Silica Nanospheres (SiO₂NSP 2.0, Cospheric) of 508 nm, 690 nm, 1180 nm and 7750 nm; with a density of 2.0 g/cc.

Finally test are performed with 508 or 690 and 7750 nm spheres.

Previous test performed to develop this method are summarized in Annex B



Figure 18: Set-up for PM coarse test (left) and co-spheric dust used for the generation of fine and coarse dust (right)

3.3.2 Evaluation

The calculations for the evaluation of this test are given in TS and are summarized here.

- 1) Calculate ratio (R) of output from sensor vs equivalent method
 - a. for PM_{2,5} (measured during fine test)
 - b. for PM_{10-2,5} (measured during coarse test)
- 2) Calculate the factor F(FINE/COARSE) to estimate the response change of the sensor from fine to coarse test conditions

In the TS it is defined that the factor F(FINE/COARCE) cannot be larger than 2.

$$R_{PM_{10-2,5}(COARSE)} = \frac{PM_{10-2,5}(sen,COARSE)}{PM_{10-2,5}(eq,COARSE)}$$

$$R_{PM_{2,5}(FINE)} = \frac{PM_{2,5}(sen,FINE)}{PM_{2,5}(eq,FINE)}$$

$$F\left(\frac{FINE}{COARSE}\right) = \frac{R_{PM_{2,5}(FINE)}}{R_{PM_{10-2,5}(COARSE)}}$$

3.4 Linearity test (LoF)

A LoF test is performed to test the linearity of the sensors and the comparability between the sensors. This test is not included in the current TS but can give some insight in linearity of PM sensors.

The PM concentrations used during the test were selected to be representative for ambient concentrations and at the same time representing high concentration due to peaks that can occur. Currently, the following concentration steps are recommended:

0, 30, 40, 60, 130, 200 and 350 $\mu\text{g}/\text{m}^3$.

These concentrations can be adapted if sensors will be used for specific applications and concentrations.

Note that also the composition of particles can influence the sensor response. Currently we didn't include tests to evaluate the response to different compositions. However, these kinds of tests can be very useful when e.g; wood burning is envisaged.

Based on the lack of fit results, the comparability against the reference¹⁹ is evaluated using the following parameters: R^2 , accuracy (%), Root Mean Squared Error (RMSE), Mean Absolute Error (MAE), Mean Bias Error (MBE). An example of resulting tests of the benchmark study is given in Table 5 below.

The comparability between instruments is evaluated using IMV and BIU as described in 2.6.

¹⁹ We use a Grimm EDS264 here as reference.

3. TEST PROTOCOLS FOR PM SENSORS

Table 5: Results of tests performed on PM portable sensors

	SENSOR SYSTEM	Accuracy (%)			MAE	R ²	Uexp
		PM ₁	PM _{2.5}	PM ₁₀	µg/m ³	-	%
PM	SENSOR 1 (3)	84	65	29	10.0	0.98	47
	SENSOR 2 (3)	83	54	22	12.6	0.99	55
	SENSOR 3 (3)	18	79	47	5.2	1.00	25
	SENSOR 4 (3)	64	70	31	8.9	0.99	40
	SENSOR 5 (3)	68	52	21	10.9	0.99	45
	SENSOR 6 (3)	NA	NA	NA	NA	NA	NA
	SENSOR 7 (1)	63	29	13	17.3	0.96	79

3.5 Examples of tested PM sensors

Different projects could benefit from the infrastructure developed and more specific also from the coarse particle test that has been developed. Some tests used different settings when the final test protocol was under development. On the other hand, we could benefit from the sensor projects to apply the developed methods to sensors of interest.

3.5.1 Projects and sensors tested

3.5.1.1 sensEURcity project

The sensEURcity project aimed to deploy sensors in cities and evaluate calibration methods. A short summary of the project is given in Van Poppel et al., 2023²⁰. A large set of field data is available and therefore it was decided to test also the PM_{coarse} test on these boxes. Moreover, the sensor units had two different PM sensors which made it even more interesting (see Figure 19).

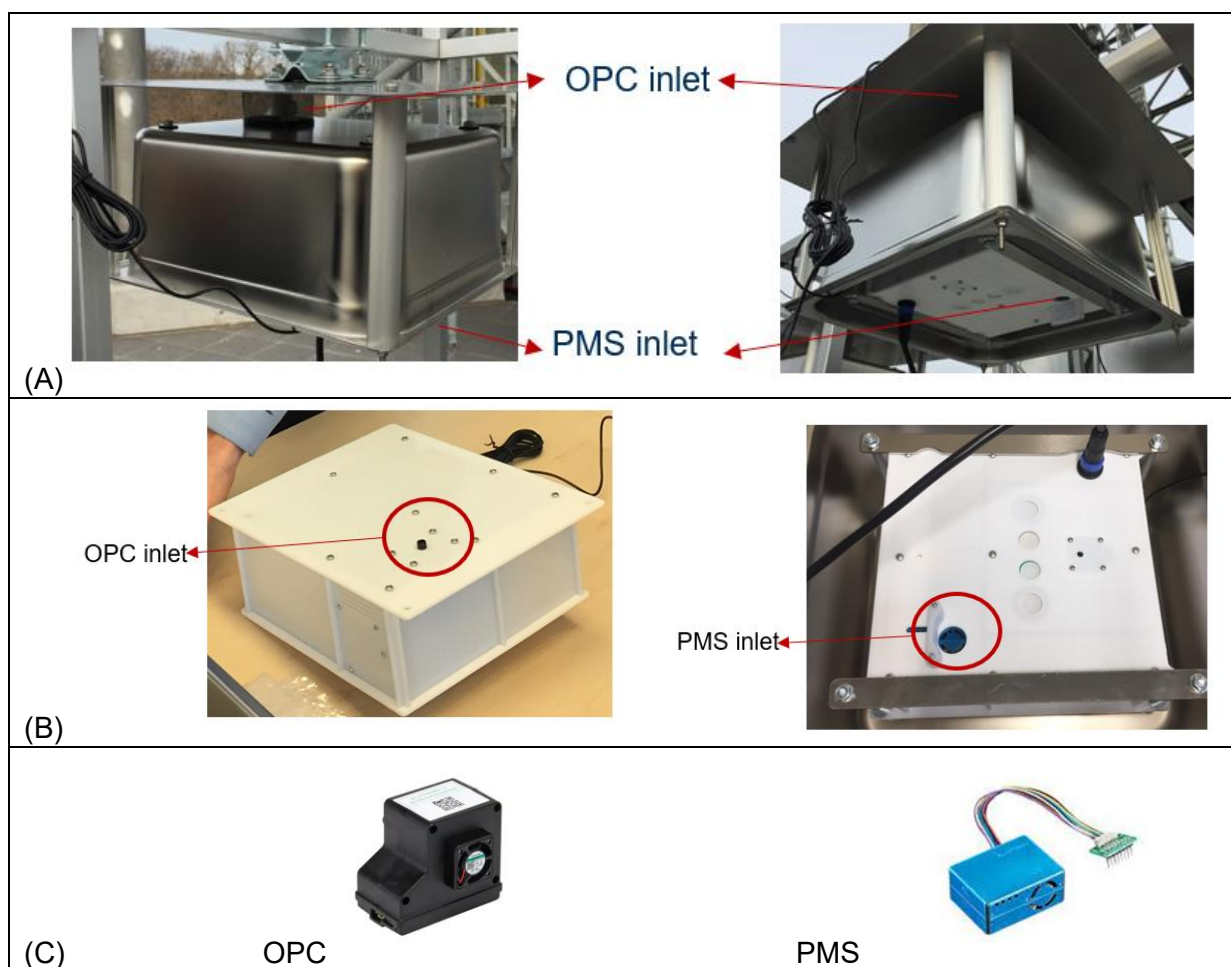


Figure 19: AirsensEUR sensor box showing (A) entire unit with PM sensor inlet (B) details of OPC and PMS inlet and (C) sensor units (OPC left and PMS right)

²⁰ Van Poppel, M., Schneider, P., Peters, J. et al. SensEURCity: A multi-city air quality dataset collected for 2020/2021 using open low-cost sensor systems. Sci Data 10, 322 (2023). <https://doi.org/10.1038/s41597-023-02135-w>

AirSenseEUR is an Open Platform project developed by the JRC, aimed at measuring air quality using low-cost sensors. AirSenseEUR includes a PTFE enclosure (26x22x10cm), inserted in a stainless-steel protecting cover (35x32x30cm). It includes two PM sensors:

- OPC-N3 with 24 size bins (0.3/0.35 – 40 μm)
with a counting efficiency* of 50% @ 0.3 μm and 100% @ 0.35 μm
- PMS5003 with 6 size bins (> 0.3 μm)
with a counting efficiency* of 50% @ 0.3 μm and 100% @ 0.5 μm

*counting efficiencies claimed by the manufacturers

3.5.1.2 Benchmark study

This study included a benchmark of portable sensors to be used in dynamic exposure experiments. The evaluation was part of a study (funded by Innovative Public Procurement Program (PIO), and results were recently published in Hofman et al., 2024²¹. The study included seven PM sensors that were evaluated in the laboratory Figure 20.



Figure 20: PM sensors tested as part of the benchmark study (from left to right): PAM (2BTech), GeoAir, SODAQ NO2 (SODAQ), SODAQ Air (SODAQ), PMscan (TERA), Open Seneca (Open Seneca) and ATMOTube Pro (ATMO).

The study showed that different sensors can have different monitoring resolutions (from 1 sec to 5 min in this study). This has a large impact on the PM test since it requires stable conditions over a number of individual measurements. In this case it was solved by using the average of the last 15 minutes of stable conditions at each setpoint.

²¹ Hofman, J.; Lazarov, B.; Stroobants, C.; Elst, E.; Smets, I.; Van Poppel, M. Portable Sensors for Dynamic Exposure Assessments in Urban Environments: State of the Science. *Sensors* 2024, 24, 5653. <https://doi.org/10.3390/s2417565>

3.5.2 Examples PM: Linearity and accuracy

The results of the benchmark study are shown in Table 6 below (adapted from Hofman et al., 2024).

Table 6: Overview of performance (Accuracy, MAE, R^2)

	SENSOR SYSTEM	Accuracy (%)			MAE	R^2
		PM ₁	PM _{2.5}	PM ₁₀	$\mu\text{g}/\text{m}^3$	-
PM	ATMOTUBE (3)	84	65	29	10.0	0.98
	OPEN SENECA (3)	83	54	22	12.6	0.99
	TERA (3)	18	79	47	5.2	1.00
	SODAQ AIR (3)	64	70	31	8.9	0.99
	SODAQ NO2 (3)	68	52	21	10.9	0.99
	GeoAir (3)	NA	NA	NA	NA	NA
	PAM (1)	63	29	13	17.3	0.96

3.5.3 Example PM: PM_{coarse} test

3.5.3.1 sensEURcity results

The figure below shows the time trends during the tests. Slight differences are observed which is partly related to the level of filling of the cuvette. The aim of this experiment is to see if slight differences in conditions will influence the final conclusions and result of the test.

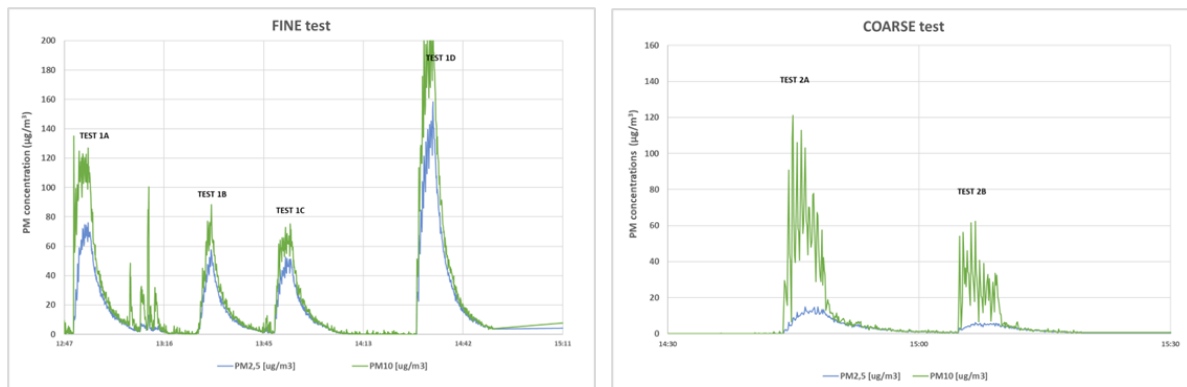


Figure 21: Time trends for fine (left) and coarse (right) tests measured by reference monitor

Table 7 below shows the resulting ratio R for different fine and coarse tests for PMS and OPC. Based on this, the factor $F(\text{fine/coarse})$ is calculated for different combinations of tests, which are presented in Table 8. This shows that PMS fails for coarse test, which means that this sensor does not measure the coarse particles and calculates PM_{10} from measured $\text{PM}_{2.5}$.

3. TEST PROTOCOLS FOR PM SENSORS

Table 7: Ratio sensor/reference for different fine (1A-1D) and coarse (2A-2B) test conditions

TEST	PMS		OPC	
	R _{PM2,5} (FINE)	R _{PM10-2,5} (COARSE)	R _{PM2,5} (FINE)	R _{PM10-2,5} (COARSE)
FINE 1A	0,31	0,30	0,95	0,49
FINE 1B	0,25	0,76	1,00	0,47
FINE 1C	0,27	0,76	0,92	0,44
FINE 1D	0,30	0,57	1,14	0,67
COARSE 2A	0,35	0,08	0,97	1,73
COARSE 2B	0,32	0,06	0,89	1,42

Table 8: Factor of sensor response change from fine to coarse test conditions $F(\text{fine/coarse})$

	PMS		OPC		
	COARSE 2A	COARSE 2B		COARSE 2A	COARSE 2B
FINE 1A	3,6	4,8	FINE 1A	0,55	0,67
FINE 1B	3,0	4,0	FINE 1B	0,58	0,70
FINE 1C	3,2	4,3	FINE 1C	0,53	0,65
FINE 1D	3,6	4,7	FINE 1D	0,66	0,80

From the field data, it was observed that:

- PMS sensors showed a clear relationship with reference instruments for PM_{2,5}, whereas no relationship was observed for PM_{10-2,5}
- A clear relationship was observed between PM_{10-2,5} and PM_{2,5}, from PMS sensor data (e.g. R² around 0.6) whereas no relationship was observed from the reference data (R² < 0.1)

These field observations confirm that PMS do not measure PM₁₀, which was also concluded from the laboratory tests.

3.5.3.2 Benchmark study results

The Coarse PM test size of the monospheric dust in the benchmark study was respectively 7.750 and 1.180 µm. In later experiments, other sizes were used based on discussions in WG42 and to be able to reach the requirements for the % in different size fractions for the experiments as set by the TS.

The visual inspection of the time trends show that sensor systems tend to pick up fine particle spikes but appeared far less responsive to the coarse fraction spikes (Figure 22). Note that in both fine and coarse generation spikes, PM_{2.5} is present. Similar responses are observed between the different sensor systems, which is not surprising, as all sensors are ultimately based on three original equipment manufacturer (OEM) sensors, namely Sensirion SPS30, Plantower PMS, and TERA next-PM. From the calculated change ratios (not shown here) the

3. TEST PROTOCOLS FOR PM SENSORS

sensor/REF ratio changed significantly between the considered particle size conditions (resulting in failing the coarse laboratory test).

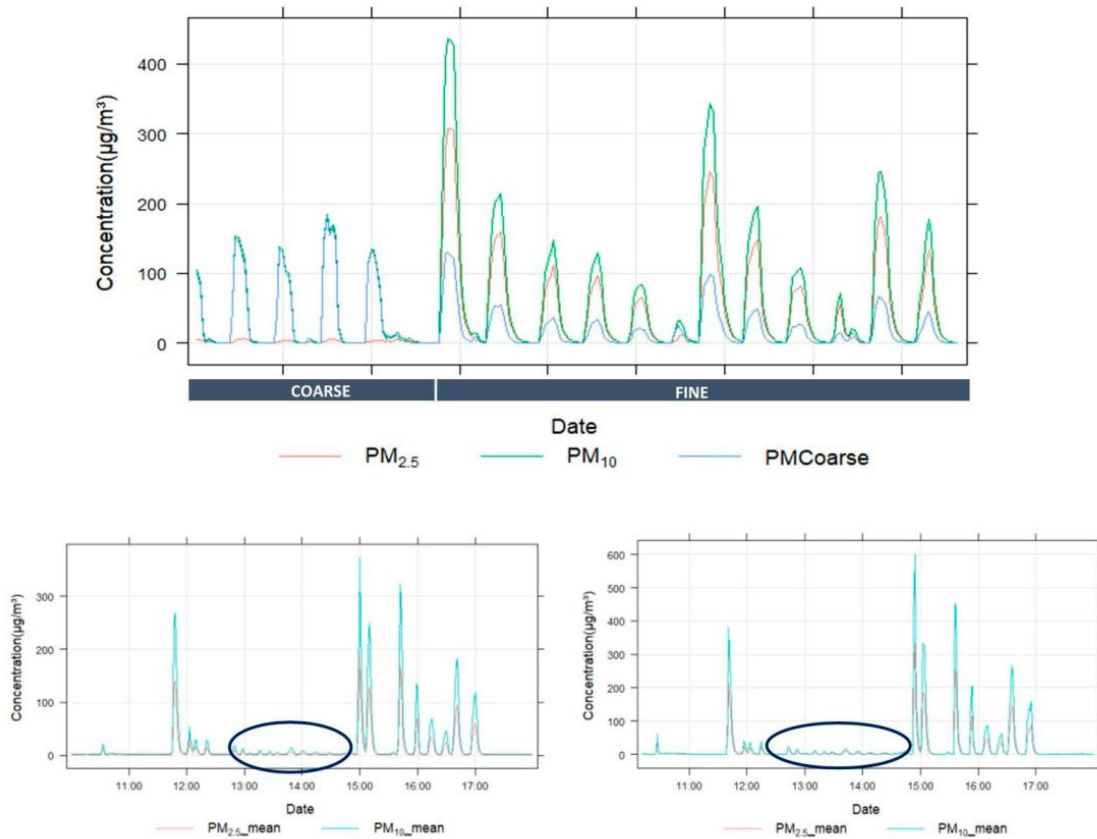


Figure 22: Coarse PM testing results with consecutive 5-min generation periods of coarse (7.75 μm) and fine (1.18 μm) PM peaks (upper panel; measured by Grimm monitor) and resulting sensor response ($\mu\text{g}/\text{m}^3$) in the lower panels (adopted from Hofman et al., 2024)

4 ADDITIONAL INFRASTRUCTURE

Complementary to laboratory testing, field testing can give further insights in sensor performance. Most sensor have weather-proof housing so can be easily deployed next a reference monitoring station. For sensors that are used for specific applications like personal exposure and have no protective housing, a shelter was build. Also, for mobile applications, VITO developed a method to evaluate these sensors (not a final infrastructure yet). These developments were made for the Benchmark study.



Figure 23: Exposure shelter for sensors (left: outside of the shelter and right: inside with sensors)



Figure 24: Prototype of mobile evaluation platform

5 CONCLUSIONS AND OUTLOOK

This protocol does not address **unregulated pollutants**. However, there is a large interest in non-regulated pollutants like VOC's, NH₃, CH₄ and new sensor technologies to measure these pollutants at a high spatial resolution.

The current protocol needs some revisions to address these pollutants. VOC sensors measure a combination of compounds and this needs to be taken into account in the evaluation procedure. Ammonia has other challenges due to its stickiness and absence of a reference method. Another issue is that none of these pollutants have target or reference values and as a result, defining a representative concentration for evaluation is more challenging. Moreover, because these pollutants are not regulated, application of sensors will go beyond the field of regulatory monitoring and representative concentrations need to be defined. Finally, the compounds do not have a 'reference method' like regulated pollutants.

We also do not address requirements of sensors for specific applications/use like **mobile applications**.

Next to the evaluation of sensors prior to their use, also the calibration and AQ/QC during use needs to be taken into account. An example on how sensors can be used is described in [Hofman et al. \(2022\)](#).

Field evaluations were not discussed in this document. The CEN/TS 17660-01 and CEN/TS 17660-02 include strategies to set-up field evaluations and to evaluate sensors during field evaluation. We refer to these documents.

ANNEX A: VALIDATION OF THE GAS TEST CHAMBER

5.1 Introduction

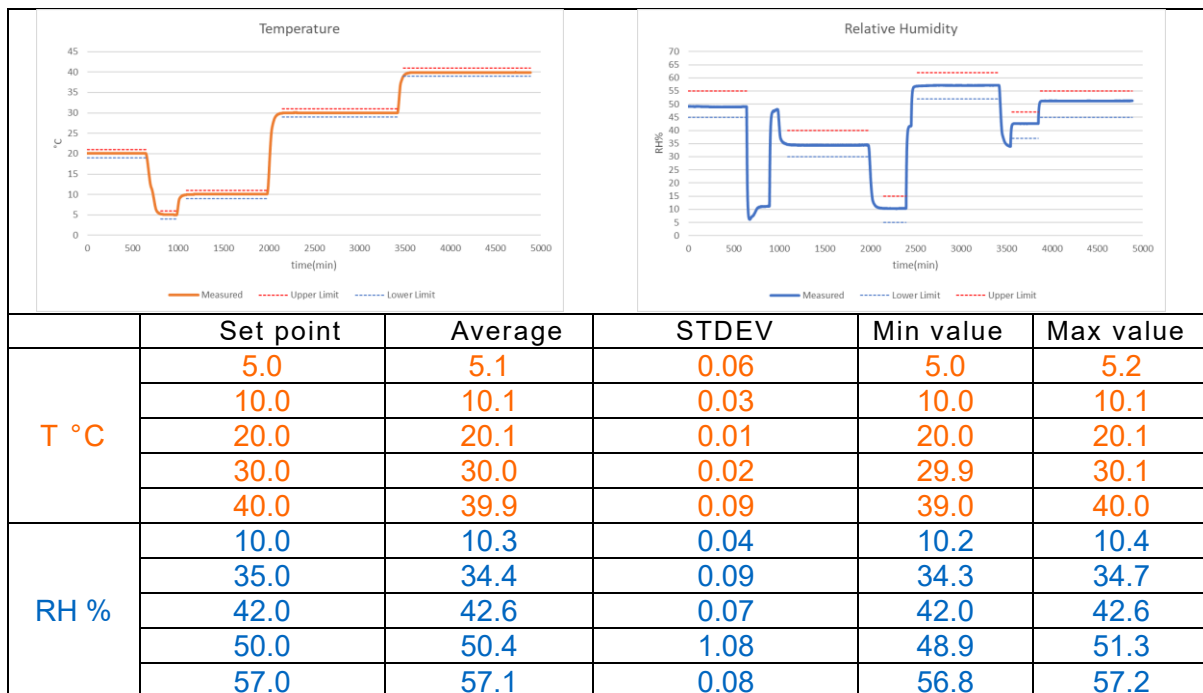
This annex describes the validation of the stainless-steel exposure test chamber currently present at VITO, and its capability to cover the requirements for gas sensor testing described in CEN/TS 17660-1.

5.2 Temperature and relative humidity stability

For testing the capability of the system to continuously control the temperature and relative humidity inside the test chamber, an exposure experiment has been simulated. During the experiment the temperature and relative humidity inside the test chamber were measured every 1 min during a validation period of 72 h using calibrated T/RH monitor/logger (Testo 175H1, accuracy of 0.4°C and 2% RH).

An overview of the temperature and relative humidity measured inside the exposure test chamber is shown in Table 9.

Table 9: An overview of the temperature and relative humidity measurements during the performed stability test



5.3 Efficiency of the internal exposure test chamber air mixing

The efficiency of the internal exposure test chamber air mixing has been tested by using a tracer gas, which is introduced in the chamber (inject into the inlet air stream) at a constant concentration and flow. CO₂ was used as a tracer gas, at a concentration of 1741 ppm. The concentration at the outlet flow of the chamber was continuously measured during the experiment using a calibrated NDIR CO₂ monitor ((SIDOR, 0 – 10000 ppm). Figure 25 shows a schematic overview of the system providing tracer gas to the chamber.

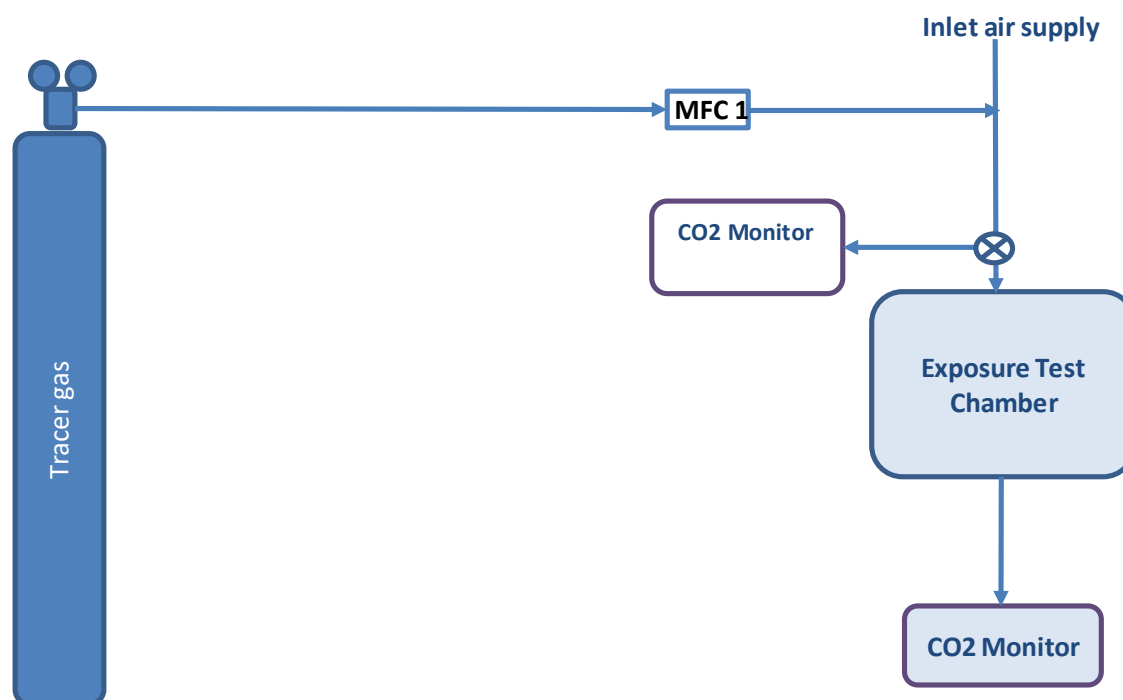


Figure 25: A schematic overview of the tracer gas system

The experimental curve (concentration vs. time) is then compared to the theoretical curve of the well mixed system (calculated according to Eq.27) for the same time settings.

$$C = C_0(1 - e^{-Nt}) \quad 27$$

where:

C is the concentration of the tracer gas measured in the chamber

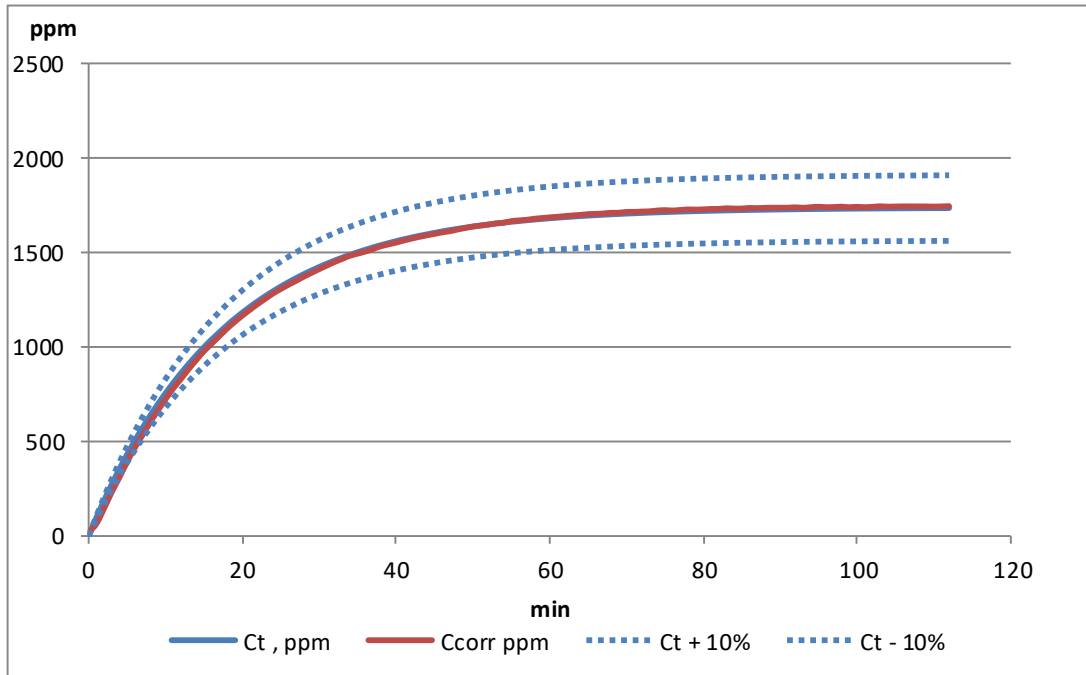
C_0 is the concentration of the tracer gas at the inlet of the chamber.

t is time.

N is the air exchange rate ($N = Q/V$, where Q – flow rate through chamber; V – chamber volume)

The internal exposure test chamber air is considered as well mixed when the difference between the observed and theoretical air concentrations at selected time points are within $\pm 10\%$.

ANNEX A: VALIDATION OF THE GAS TEST CHAMBER



- C_t Concentration for a perfectly mixed system, calculated by $C(t) = C_o (1 - e^{-Nt})$, ppm
- N Chamber air exchange rate, h⁻¹
- t Time, h
- C_o Tracer gas concentration in chamber exhaust, ppm
- C_{ocorr} Tracer gas concentration in chamber exhaust corrected by blank value, ppm
- C_t + 10% Upper acceptable limit for concentration for perfectly mixed system, ppm
- C_t - 10% Lower acceptable limit for concentration for perfectly mixed system, ppm

ANNEX B: EVALUATION OF THE PM TEST CHAMBER AND SETTINGS

5.4 Introduction

This Annex describes some of the experiments that were performed to develop the PM sensor test infrastructure (generation, settings and exposure chamber). The development was also partly trial and error and not all steps are described in detail. This annex is to illustrate the preparative work that has been done to realize the set-up of the PM sensor testing infrastructure.

5.5 Homogeneity of the test chamber

The **homogeneity of the test chamber** was evaluated by placing 3 DustTraks at different locations (left, middle, right) in the chamber, and recording the PM concentrations. First an intercomparison of the three DustTraks was performed and the inter-instrument correction factor was applied to take into account inter-instrumental differences. The comparison of the instrument readings after correction is shown in Figure 26 (left) and the readings of the DustTraks (after correction) at the three locations is shown in Figure 26 (right). The concentrations at different locations in the test chamber correspond quite well; the slightly lower concentration of one of the instruments was due to lower instruments readings even after correction. The tests were performed for total PM concentrations.

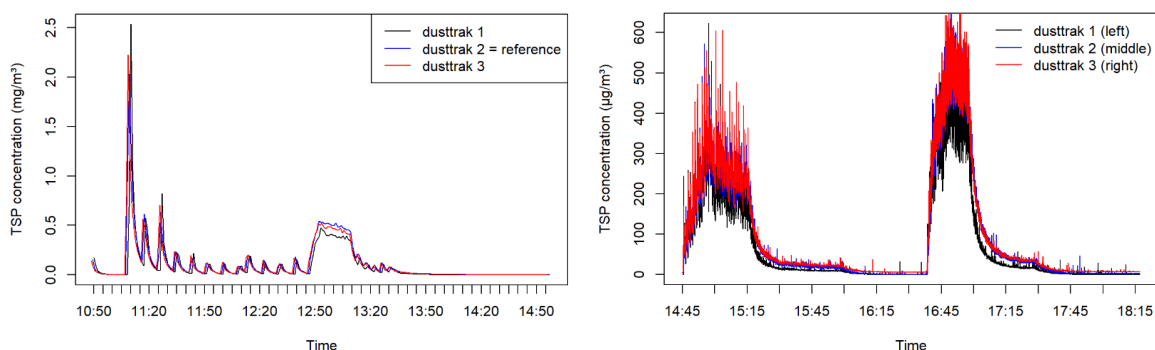


Figure 26: Homogeneity test with Dusttraks: intercomparison of instruments after correction (left) and concentrations measured at different locations (right)

5.6 Stability and levels of PM concentrations

In addition, tests were performed to optimize the settings of the PM generation. The aim was to get a relative **stable PM concentration**. The dust injection rate was varied for fixed ventilator settings (20V) and venturi pressure (± 1.5 bar); an example of different injection rates (every 32 seconds and every 256 seconds is given in Figure 27, showing a stable concentration with more frequent injections as compared to lower frequency injection. Further tests showed that an optimal injection rate was reached around 8 seconds, and when further decreasing the time between two injections higher concentrations were reached and these decreased faster but with less stable conditions.

ANNEX B: EVALUATION OF THE PM TEST CHAMBER AND SETTINGS

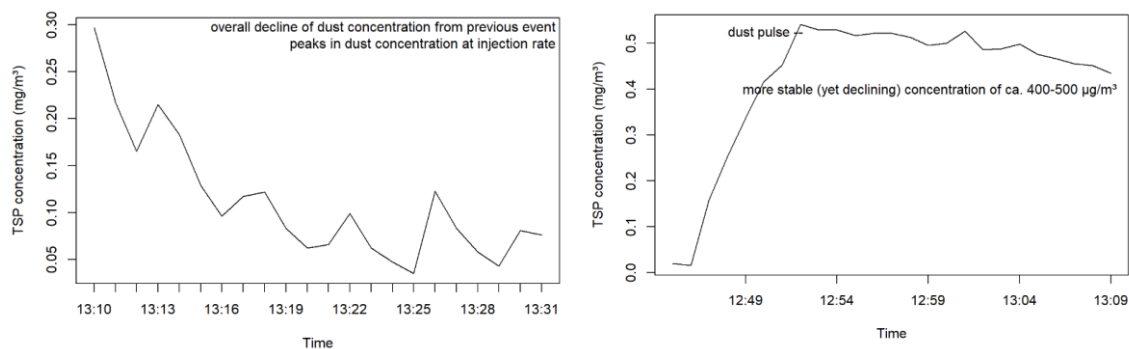


Figure 27: Stability test of PM concentrations for different PM injection rates

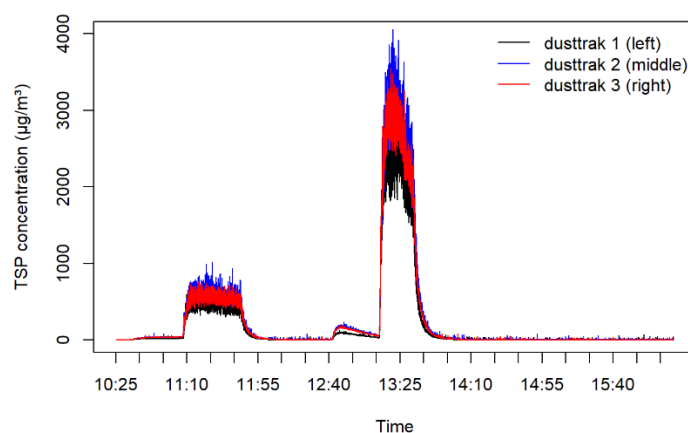


Figure 28: TSP concentrations in exposure chamber with injection frequency of 8 sec (left peak) and 2 sec (right peak).

5.7 Coarse tests

For coarse test, different set-ups were tested and are reported below.

5.7.1 Particle composition for generation

First experiments were performed using PSL particles. These particles are commonly used to evaluate size measurements. We used a commercial-type atomizer (TSI type constant output atomizer 3076) combined with a drying column to remove the dispersion liquid. The idea was to generate 600 nm and 6 µm particles. Size and mass concentration were measured using Grimm and APS (TSI). Whereas this test showed that sensors did not detect larger particles (see figure), the amount of small particles was too large to be a good candidate for further tests.

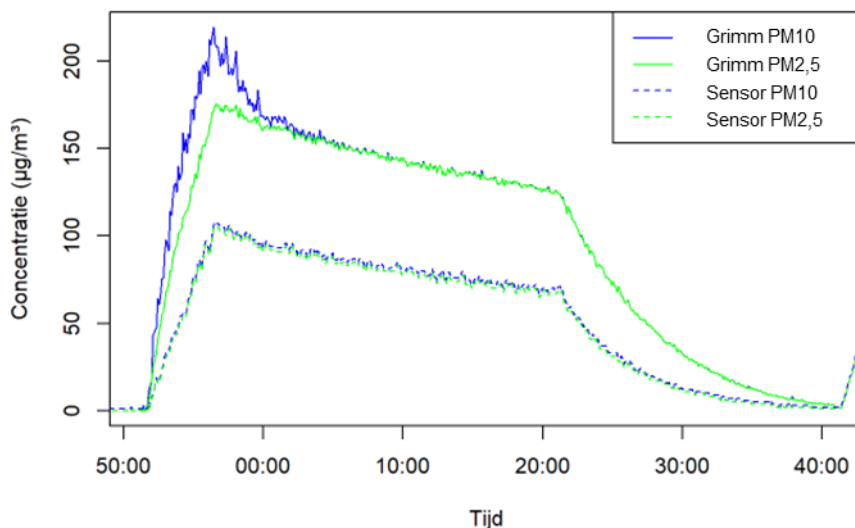


Figure 29: Example of coarse test (aiming for coarse test conditions) with PSL particles showing sensor and monitor (Grimm) values for $\text{PM}_{2.5}$ and PM_{10}

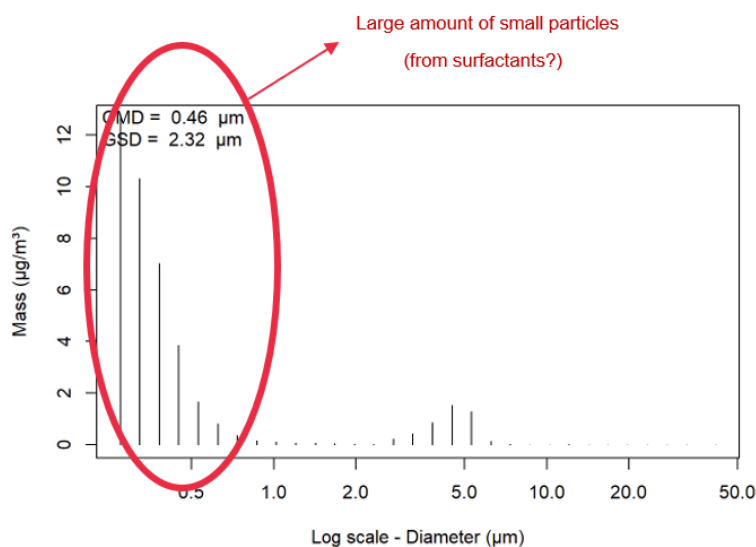


Figure 30: Size distribution measured during coarse test (aiming for coarse test conditions) using PSL particles

5.7.1.1 Note: Experiences from the international community

We contacted AQ-SPEC and they confirmed the issue with a lot of small particles when using larger PSL particles, probably due to surfactants present in the PSL sphere stock solution. Also, colleagues from NILU confirmed that it is challenging to generate a large amount of larger particles without small ones (around 200-300 nm).

In the ASTM standard (ASTM 8402-21), sensors are exposed to coarse particles but aiming to assess interference of coarse particles to the $\text{PM}_{2.5}$ sensor which is a different aim. Also, they use a different type of dust (using NaCl for fine and Arizona Road dust (ATD-A4) for a

mixture with fine and coarse) which is not advisable since composition can have an effect on sensor response.

Another option could be to use fractions of Arizona road dust (Fiatec, Germany) but this was not further explored.

5.7.2 Timing of particle introduction for stable concentration

Different settings for particle generation were tested and the homogeneity and levels of the PM concentrations inside the chamber were evaluated in function of the settings. Some examples are given below for different size fractions.

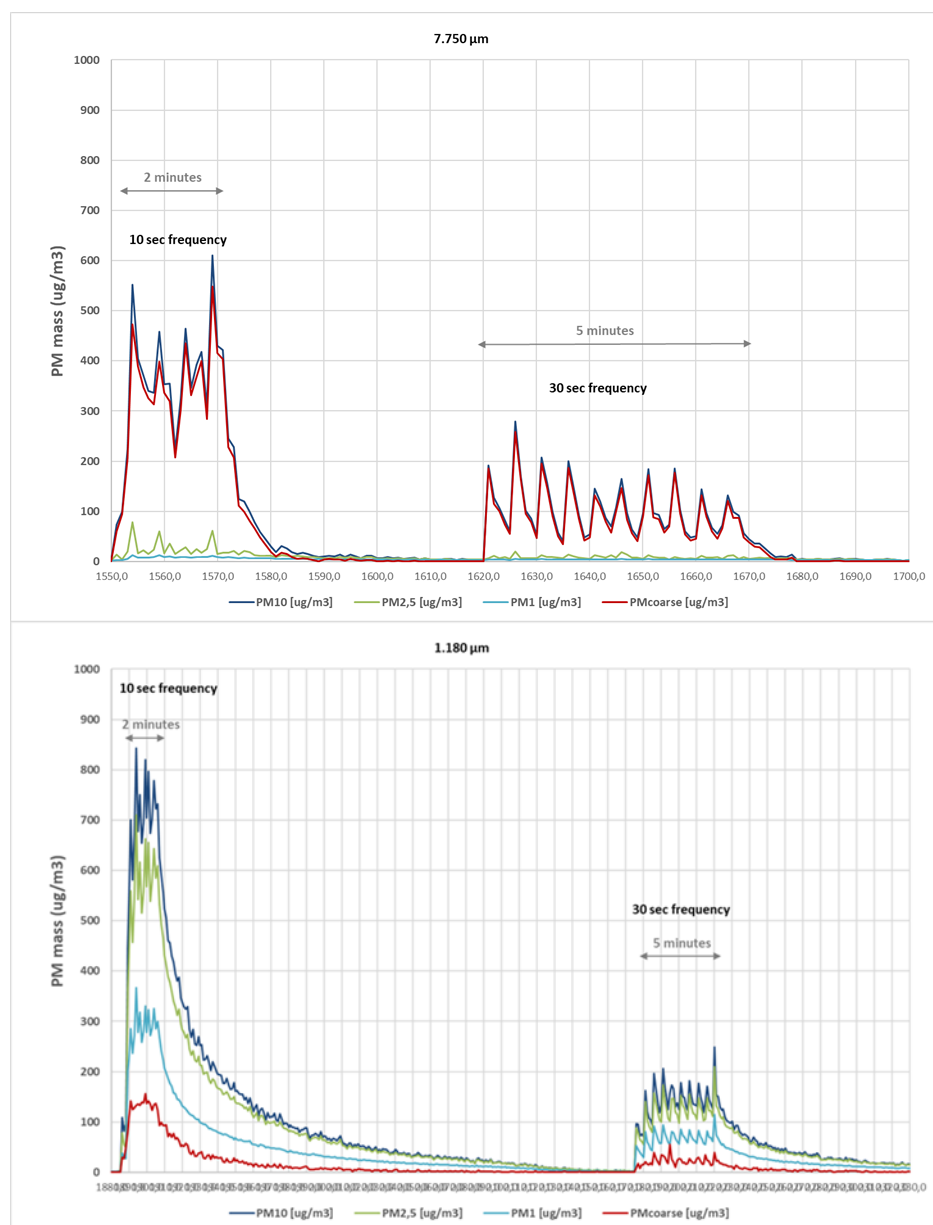


Figure 31: PM concentrations for different settings of the generation

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