

FACILITIES FOR THE TESTING OF GAS MONITORING INSTRUMENTS AT THE NATIONAL PHYSICAL LABORATORY

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1 INTRODUCTION

The National Physical Laboratory (NPL) has developed facilities for the laboratory testing and calibration of gas monitoring instruments as part of the VAM (Valid Analytical Measurement) programme of the Department of Trade and Industry. This paper describes some of these facilities, including gas cells for testing open-path instruments and a laboratory which is dedicated to the testing of CEMs (Continuous Emission Monitors). The latter is UKAS accredited for a range of tests which include the laboratory test requirements of the MCERTS Certification Scheme. A comprehensive facility is also described which is being developed for the performance testing of pumped adsorption tubes and diffusion tubes, which are used increasingly for measurements of ambient air and occupational exposure.

2 CEM PERFORMANCE TEST FACILITY

This facility is used to test the laboratory performance of CEMs. It not only applies gas concentrations to the CEM but also allows the environmental conditions of ambient temperature and humidity, sample gas temperature and sample gas pressure to be varied. The facility will allow instruments to be accurately calibrated but is mainly intended to test manufacturer's equipment for accuracy, linearity, time response, drift, detection level, repeatability and the susceptibility to environmental conditions.

The facility is UKAS accredited and suitable for laboratory testing to the requirements of the MCERTS certification scheme.

2.1 CEM Types

Continuous emission monitors are used to measure pollutant gas concentrations within stacks and ducts. They may employ various measurement techniques but which one is used has little influence on the way in which they are tested. Of more concern is the method by which they access the sample in the stack. There are essentially three types of monitor :-

- extractive, where a sample is drawn from the stack through a heated sample line
- in-stack, where the sample is measured within the stack by a single entry point probe
- cross-stack, where the sample is measured within the stack by a double entry point probe

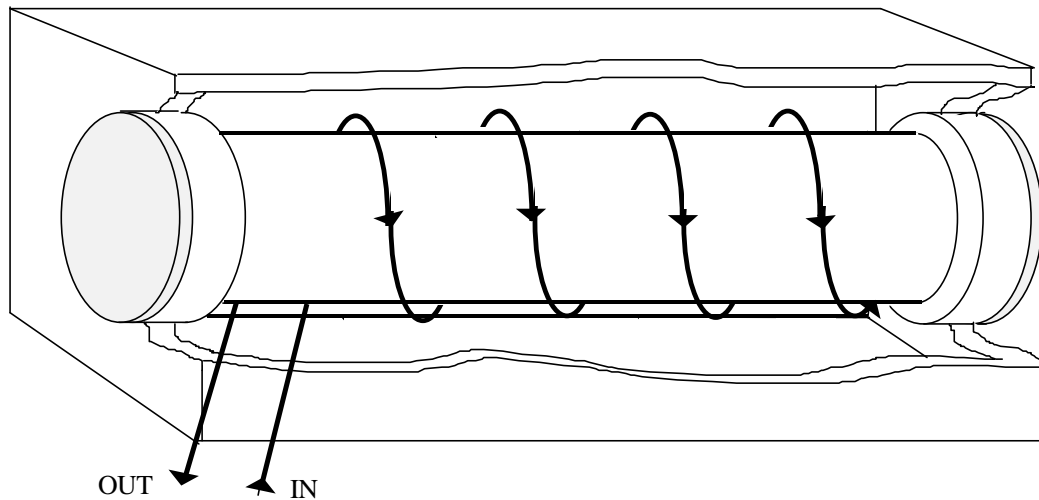
The test facility can accommodate all three types in its Probe Chamber.

2.2 The Probe Chamber

The probe chamber provides the means of placing the CEM's probe in the test environment. Two alternative probe chambers are used, according to the size of the probe. Each is a 1.08 m long cylinder and the internal diameters are 125 mm and 190 mm. Metal plates at the ends of the cylinder represent small areas of the stack wall while the length of the cylinder equates to the diameter of the stack. The probe of the CEM under test is

mounted through one or both of the end plates. The probe chamber is enclosed by an oven, as shown in Figure 1, which can be controlled from ambient to 200°C.

Figure 1



Test gases are pre-heated in the oven before being fed into one end of the probe chamber. They then flow down the length of the chamber and are exhausted into a control valve. The control valve, and a pressure sensor which monitors the inside of the probe chamber, form part of a closed-loop system which allows the chamber pressure to be regulated from close to atmospheric pressure and up to 10% above.

2.3 Test Gas Sources

The test gases must meet different requirements for different tests. The accuracy test requires defined absolute concentrations while the linearity test needs a series of concentrations which are precisely defined with respect to one another. The remaining tests rely upon the applied concentrations remaining stable over many hours. All three requirements are not needed simultaneously and two alternative sources of test gas are used to provide the desired specification.

Where absolute concentrations are necessary NPL gravimetric standards are used. These have an accuracy of generally better than 0.2% relative.

The second source of test gases is a dynamic dilution network. This uses conventional mass flow controllers in a novel way to blend high concentration gas mixtures with diluent air or nitrogen to produce concentrations with dilution ratios and stability of better than 0.5% relative. By comparing the output of the dilution network with a suitable gravimetric standard it may be calibrated to provide absolute concentrations with an uncertainty of better than 1% relative.

Water vapour is required as a test gas in concentrations of 1% to 50% by volume. These concentrations are obviously not available as standards, in cylinders. NPL has therefore developed a dynamic water vapour generator using gravimetrically calibrated liquid and gas mass flow controllers. This can produce water / air mixtures with an uncertainty generally better than $\pm 0.4\%$ water vapour by volume.

2.4 Environmental Room

The probe chamber provides the in-stack environment seen by the probe of the CEM. The environment outside of the stack is controlled by an environmental room into which the probe chamber and its enclosing oven are placed, together with the other CEM components. This room may be controlled over a temperature range of -20°C to +70°C and a relative humidity range of 5% to 95%.

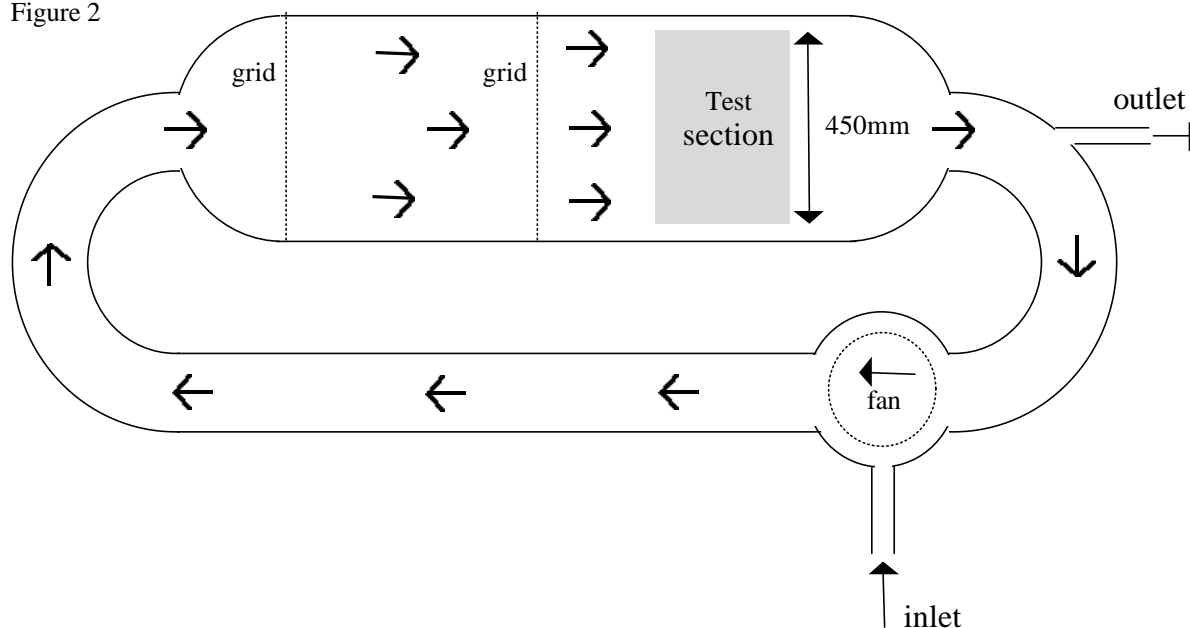
3 AMBIENT ANALYSER PERFORMANCE TEST FACILITY

It is intended that the continuous emission monitor performance test facility will be expanded to allow similar performance testing of ambient monitoring instruments.

4 DIFFUSION TUBE AND PUMPED ADSORPTION TUBE TEST FACILITY

A facility for testing the performance of diffusion tubes and pumped adsorption tubes has been constructed and is currently being commissioned. This facility generates air containing accurate concentrations of gases and vaporised liquids and flows it over the tubes being tested. The air velocity, temperature, humidity and pressure can be varied to evaluate their effect on the trapping efficiency of the tubes.

Figure 2



The test facility comprises means of generating the air/gas/vapour/water mixtures and a wind tunnel in which the mixtures are circulated by a fan. The wind tunnel is shown diagrammatically in Figure 2. The test section in which the tubes under test are placed is 450 mm in diameter. The tubes are located perpendicular to the air flow in one plane over which uniform air velocity is achieved by the use of up-wind grids.

The mixtures are injected into the wind tunnel close to the fan, which ensures good mixing with the recirculated air. A restrictor in the outlet can be used to control the internal pressure. The whole wind tunnel is temperature controlled over the range 5°C to 40°C.

5 ANALYSER CALIBRATION FACILITIES

NPL holds a large stock of traceable gas concentration standards and, as the national standards laboratory of the UK, is able to produce a wide range of calibrated gas mixtures which may be used for the calibration of gas analysers. Some gas mixtures are too reactive to be held in cylinders and must be dynamically generated. If the generation technique does not yield a mixture with adequate concentration uncertainty a calibrated analyser is used to determine its value. An example of this is in the calibration of ozone analysers. NPL has recently established a UKAS accredited ozone analyser calibration facility which uses a standard reference photometer to calibrate the output of an ozone generator.

6 OPEN PATH INSTRUMENT CALIBRATION FACILITIES

Open path instruments use optical techniques to determine gas concentrations in a sample which has no physical boundary but is defined by an optical beam passing through the atmosphere. The fundamental measurement unit is average concentration multiplied by path length and because instruments are in essence “molecule counters” readings must be related to the prevailing ambient temperature and pressure. Instruments employ a variety of measurement techniques, can operate over sample lengths of cm to km and may be small enough to carry or too big to be portable. However, they may all be calibrated by inserting into some part of their optical measurement beam a calibration gas of known concentration occupying a known length of the beam.

Calibration of open path instruments therefore requires :-

- calibrated gas mixtures
- gas cells to accurately define the length of gas in the measurement beam
- means of filling gas cells
- traceable temperature and pressure measurements

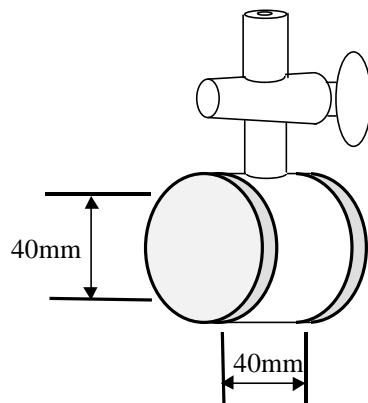
The variety of open path instrument types necessitates a variety of calibration cells and methods of filling them with gas mixtures. It is also important that the means of retaining gas in the cells allows the optical measurement beam to pass through without interfering with the instrument’s measurement technique.

NPL uses three gas cell types and associated filling facilities for testing open path instruments.

6.1 Forty Millimetre Sealed Cell

The smallest type of gas cell is typified by the 40 mm long cell, illustrated in Figure 3. This cell will accommodate an optical beam of up to 40 mm diameter. This is generally smaller than the beam transmitted to the atmosphere by open-path instruments but the cell is suitable for placement within the instrument in a section of the beam which has been reduced in diameter by the instrument’s light collection and focusing optics. This cell type allows an “internal calibration” of the instrument to be performed.

Figure 3



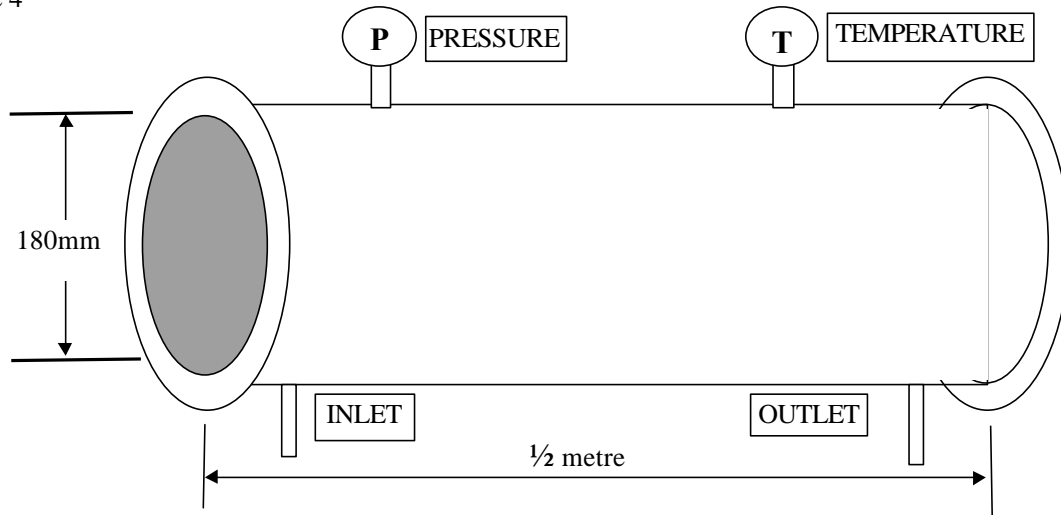
Cells are constructed around a borosilicate glass tube with a filling tap and windows sealed onto each end. For operation in the visible and infra red regions the window material may be calcium fluoride but other materials could be chosen as appropriate. To avoid shifting of the beam axis as it passes through the cell all faces of the windows are parallel.

Cells are filled by evacuating, filling and closing off with the tap. NPL has a filling apparatus in which standard gases may be precisely diluted before being flowed into an evacuated cell. The effective concentration in the cell depends not only on the concentration of the filling gas mixture but also on the filling temperature and pressure, which are both monitored at the moment of sealing the cell. Once the cell has been sealed the effects of internal temperature and pressure changes cancel each other.

6.2 Half Metre Flow-through or Sealed Cell

A large diameter gas cell is required for “external calibration” of instruments by the introduction of the test gas into the transmitted optical beam. The NPL test facility uses the cell illustrated in Figure 4. This has a gas path length of $\frac{1}{2}$ m and a clear aperture of 180 mm diameter.

Figure 4



The cell comprises a stainless steel cylinder with end flanges which allow windows to be attached. Windows of calcium fluoride are used for operation in the visible and infra red spectrum and zinc selenide windows are used in the ultra violet spectrum. The windows are wedged to reduce fringing problems from multiple reflections. Some surface reflection is generally tolerable because the type of instrument being tested receives a relatively strong signal directly from its transmitter. The small displacement of the optical axis which is produced by the wedged windows is not significant compared to the beam diameter.

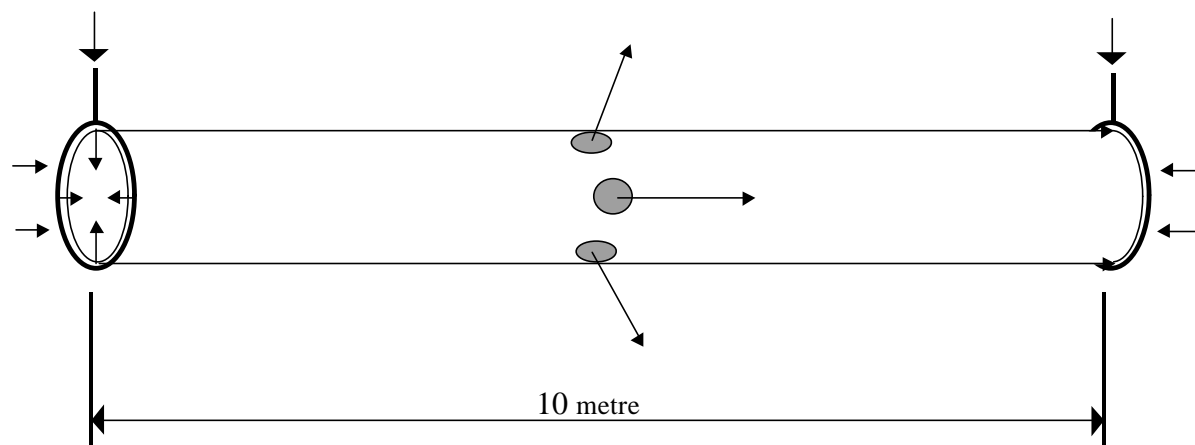
The preferred method of filling this cell is to evacuate, fill and seal it. However, if the gas is reactive and the concentration decay over the duration of the test would be significant, then the facility allows for the gas to be continuously flowed through the cell. Any loss to the walls is thereby replenished and the concentration remains constant. To avoid concentration decay in the cell it is internally coated with PFA to form an inert barrier which reduces surface reactions.

In the sealed mode of operation of the cell the temperature and pressure at the time of sealing are important, but once sealed a change of temperature or pressure will have no effect. In the flowing mode of operation the temperature and pressure have a direct effect and are continuously monitored and used for compensation.

6.3 Ten Metre Windowless Cell

This is the largest gas cell used by NPL, being 10 m long and 1 m diameter. It was designed to allow the external calibration of DIAL type instruments which transmit a powerful light beam into the atmosphere but receive back a very weak return signal resulting only from scatter off particulates and aerosols. Because the return signal is so weak it is essential that the gas cell does not reflect any of the transmitted light beam back to the receiver. The large diameter of the cell ensures that the transmitted beam can pass through the cell without reflection off the walls. Reflections from end windows are avoided by having no windows.

Figure 5



To maintain a concentration of gas within the open ended cell it is operated in a dynamic mode. Figure 5 is a schematic of the cell which is essentially a steel tube with open ends. The method of maintaining a known mixture of gas within the cell is to continually flow the mixture into the two ends and remove it from the centre. A ring of apertures around the central length of the cell are connected to fans which extract from the cell and in so doing draw ambient air into the cell at its two ends. A gas mixture is introduced at each end of the cell by a ring of nozzles which propel the gas radially across the cell ends where it is drawn in and at the same time diluted by the ingoing air. A continuous measurement of the concentration of the gas mixture in the cell is determined from samples extracted along its length.

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Brian Goody is a Senior Research Scientist and Project Manager in the Environmental Standards Section, which he joined in 1993. He currently has particular responsibility for the open-path and continuous emission monitor test facilities and is developing the diffusion tube test facility. Other work at NPL has included a nation-wide survey of landfill sites for methane emission and field measurements of hydrocarbon emissions. Prior to joining NPL his environmental metrology experience began at Siemens where he developed and put into production an open-path infrared measurement instrument. His previous background was the nuclear instrumentation industry where he worked on battlefield dosimetry systems and control and safety instrumentation for mobile nuclear reactors.