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ABSTRACT

To carry out metrological tests on the automatic devices used in air quality monitoring networks, INERIS follows a standard text. In order to apply it, the instruments have been fed with reference mixtures of gases in several reproducible concentrations. Compliance with this requirement is quite easy to obtain, in the case of conventional techniques, but the problems remain with optical remote sensing instruments. The difficulties can be solved by using the Beer-Lambert law indicating that the product of the concentration by the distance is a constant value. To implement this optical law, INERIS uses a system with a two meters glass cell with quartz windows inserted in the instrument beam and fed dynamically with known concentrations of reference mixtures of gases. The test-bench facility is set up in a tunnel 90 meters long in INERIS. Tests on \( \text{SO}_2 \), \( \text{NO}_2 \) and \( \text{O}_3 \) were carried out in 1997 on two types of DOAS systems set up in parallel: an OPSIS system and an Environment SA SANOA system. The following features were tested: the detection limit, the linearity and the drift. The tests results on the response of the two types of instruments are in agreement with the results obtained by the ERLAP at the Joint Research Center in Ispra.

key words: optical remote sensing, evaluation, quality assurance, air quality

1. INTRODUCTION

Since the end of eighties optical remote sensing instruments have been used by French air quality monitoring networks. This type of instrument offers new perspectives to characterise the atmospheric pollution. Indeed, it is possible to measure simultaneously several pollutants like \( \text{SO}_2 \), \( \text{NO} \), \( \text{NO}_2 \), \( \text{O}_3 \), hydrocarbons, and specific pollutants such as \( \text{NH}_3 \), naphtalene, ...

Before putting such an instrument into operation on a monitoring place, it is necessary to evaluate its capacities and also to determine its metrological performances.

INERIS has set up an experimental procedure to validate Differential Optical Absorption Spectroscopy (DOAS) devices. The procedure is based on that used for conventional point monitors which is described in the French standard NF X 20-300. This text drives the evaluations in terms of the following metrological features: response time, linearity, limits, drift, influence of physical parameters, of chemical compounds. In order to apply this text, the instruments have been fed with reference mixtures of gases in several reproducible concentrations. Compliance with this requirement is quite easy to achieve, in the case of conventional techniques (e.g. fluorescence detection in UV, chemiluminescence, photometry) but problems remain with optical remote sensing instruments. Indeed, it is quite impossible to manufacture a measurement cell as long as the optical path (greater than 100 meters).

But this difficulty can be solved by using the Beer-Lambert law indicating that the product of the concentration by the distance is a constant value.

To implement this optical law, INERIS uses a system with short glass cell and quartz windows inserted in the instrument beam and fed dynamically with known concentrations of reference mixtures of gases, in alternance with zero gas (nitrogen).
A standard sequence is defined as the measurement of the zero gas during a period and the measurement of the studied gas concentration during a following period of same duration.

The figure 1 shows the standard sequence.

![Standard sequence diagram](image)

Figure 1: standard sequence representation

On one hand the duration of the sequence integrates the necessary time of cell sweeping to obtain a stable and repeatable measurement values, and on other hand it permits to collect enough data for further calculations.

This procedure is adopted with slight differences by US EPA, and by VDI/DIN in Germany.

2. EVALUATION TESTS ON DOAS INSTRUMENTS ON A SHORT PATH IN AN CLIMATIC ENCLOSURE

At the beginning of the nineties two types of DOAS instrument, an OPSIS system and an Environnement SA SANOA system, were tested in a 40 m² climatic enclosure on a short optical path (about two meters) and by using a two meters glass calibrating cell. As the SANOA system uses O₂ to autolock the spectral band, it was necessary to add a cell containing pure O₂ pressure at high pressure to simulate real environmental conditions on the test path (21 % O₂ equivalence). Unfortunately, the diameter of this cell was smaller than the diameter of the beam and thus only a part of the light emitted by the xenon lamp was taken into account. For the two instruments it was also necessary to modify the light intensity which was too strong for the distance between the projector and the receiver. For that, the operator had to defocus the light beam, by an action on the projector mirrors.

The features were studied with six pollutant gases: sulfur dioxide (SO₂), nitrogen dioxide (NO₂), ozone (O₃), formaldehyde (HCHO), benzene (C₆H₆) and toluene (C₇H₈).

Tests (Poulleau et al, 1993 and 1995) gave the following results:

- the detection limits were identical or even better than those obtained with conventional point monitors for SO₂, NO₂ and O₃; but for organic compounds they were quite similar to the background ambient air concentrations.
- the response coefficient varies from an instrument to the other for the same compounds; in one case they are under estimated, in the other case they are over estimated.
- the study of the temperature influence only with SO₂ showed a significant effect for a 20 degrees variation only for one of the instruments
- the study of chemical interferences carried out by crossed procedure showed significant influence of ozone during the organic compounds -benzene and toluene- measurements
- satisfying results were obtained for linearity, zero drift deviation, signal drift deviation for one concentration value on the scale, and for parasite light influence.

The practice of these devices is easy and the soft is user-friendly.

Although the results were acceptable, using this test-bench is not a reflect of the reality, so it was necessary to find tricks to compensate for the differences between the laboratory conditions and the field conditions. These devices are usually carried out for measurements on distances as few hundred meters in open areas. Therefore, INERIS imagined a test-bench set up in a tunnel present on its premises.

3. TESTS ON DOAS INSTRUMENTS ON A LONG PATH IN A TUNNEL

In order to match more closely the real environmental conditions, in 1997 the test-bench facility has been set up in a tunnel that is 100 meters long and 2.5 meters high in INERIS.

This tunnel presents major advantages such as:
- although the calibrating cell is still 2 meters long, the distance between the projector and the receiver is closer to reality (approximatively 90 meters)
- it is not influenced by the day-light or the wind
- the gas composition, the temperature (measured by Pt sensors in three points in the tunnel) and the humidity were quite constant during the tests (each test lasted few days)

In 1997, the two types of bistatic DOAS, an OPSIS 300 system and a SANOA 3C system set up side by side, were tested in parallel and simultaneously in the tunnel.

The following pollutants were studied successively: SO₂, NO₂ and O₃. The same test procedure as the previous tests was used.

An analyser for each compound was put in the exit of the cells to monitor continuously the gas mixture concentration. The SO₂ monitor was a Siemens IR absorption instrument, the NO₂ monitor a Cosma chemiluminescence instrument and the O₃ monitor an Environnement SA UV photometry instrument. The test-bench is schematized on the figure 2.

![Test-bench for SO₂ set up in tunnel](image)

**Figure 2** : test-bench for SO₂ set up in tunnel
For each gas all the hosepipes were changed.

Besides the zero gas, four concentrations $C_i$ were generated in the cell (from 2 ppm to 30 ppm for $SO_2$ and $NO_2$ and from 0.7 ppm to 16 ppm for $O_3$) and six cycles (zero-$C_i$) per concentration were carried out. For $SO_2$ and $NO_2$ certified gas cylinders associated with a sonic nozzle diluter were used, and for $O_3$ a generator associated with a massflow system was used.

The following characteristics were tested: the detection limit, the linearity and the drift.

Based on Beer-Lambert law, we formulate the following assumption: the product between the concentration and the optical path length is constant, then it is possible to present the results for an equivalent optical path. We choose an equivalent optical path of 300 meters, which is a mean value in real conditions.

Therefore the detection limits for the three gases are:
- between 1.5 and 2 $\mu g/m^3$ for $SO_2$
- between 2.1 and 3.7 $\mu g/m^3$ for $O_3$
- and around 4.7 $\mu g/m^3$ for $NO_2$

The results are acceptable for the drift test (0.04% per day) and for the linearity.

Furthermore our tests results on the response of the two types of instruments are in good agreement with the results obtained by the ERLAP at the Joint Research Center in Ispra (1996 and 1997). The following table 1 presents the results of this comparison.

<table>
<thead>
<tr>
<th></th>
<th>OPSIS instrument</th>
<th></th>
<th>SANOA instrument</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>INERIS</td>
<td>ERLAP(1)</td>
<td>INERIS</td>
<td>ERLAP(1)</td>
</tr>
<tr>
<td>$SO_2$</td>
<td>0.98</td>
<td>1.00</td>
<td>0.97</td>
<td>1.03</td>
</tr>
<tr>
<td>$NO_2$</td>
<td>1.01</td>
<td>0.99</td>
<td>1.04</td>
<td>1.04</td>
</tr>
<tr>
<td>$O_3$</td>
<td>0.90(2)</td>
<td>0.98</td>
<td>0.95</td>
<td>1.06</td>
</tr>
</tbody>
</table>

(1) the optical path was 178 meters long, the test cell 1 meter long and placed on the optical path was fed by gas from cylinders for $SO_2$ and $NO_2$, and by a generator for $O_3$.

(2) this result is influenced by a discrepancy between the measure and the theory for a concentration value during the linearity test.

Table 1: response coefficients of OPSIS and SANOA DOAS obtained for $SO_2$, $NO_2$ and $O_3$ by INERIS and ERLAP

The optical facility developed in INERIS tunnel and validated with different DOAS instrument, is now used systematically to test DOAS systems before they are installed in a air quality monitoring network. It is interesting:

- to have a data bank on metrological features on optical remote sensing monitors
- to test the calibration procedure proposed by the manufacturers
- and to test these instruments for the measurement of pollutants such as hydrocarbons

In 1998, other tests were carried out on the calibration systems used for these devices.

When DOAS instruments are used in fieldworks, it is possible to control their response by using a very small calibration cell (few centimetres). Each instrument is fitted out with a calibration cell integrated on the optical
path of the instrument. For the SANOA this cell is placed in the receiver and is 0.034-meter long, and for the
OPSIS the cell is placed in front of the receiver and is about 0.050 meter long (the exact length is indicated on
the cell).

To use these cells it is necessary to feed them with very high gas concentrations such as few hundred ppms. And
some recommendations are given by the manufacturer, especially on the level of the gas flow and the pressure in
the cell for the SANOA system: 0.5 l/min and 300 ± 50 mbar.

Furthermore, it is recommended to operate preferably in conditions of atmospheric stability, and of constant
pollution during this calibration.

A test on the influence of the gas flow on the response coefficient was carried out on the two calibration systems
and compared with results obtained on our test-bench fitted with two meters long cell.

The devices were set up on the test-bench in the tunnel, and the test-cells (2 meters long) were put out of the
optical path during these tests.

The results are given in the table 2.

<table>
<thead>
<tr>
<th>Gas flow in l/min</th>
<th>OPIS instrument</th>
<th>SANOA instrument</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SO2</td>
<td>NO2</td>
</tr>
<tr>
<td></td>
<td>776 ppm (±2%)</td>
<td>718 ppm (±2%)</td>
</tr>
<tr>
<td>0.3</td>
<td>0.979</td>
<td>1.031</td>
</tr>
<tr>
<td>0.5</td>
<td>0.996</td>
<td>1.002</td>
</tr>
<tr>
<td>0.7</td>
<td>0.999</td>
<td>1.001</td>
</tr>
<tr>
<td>0.9</td>
<td>1.002</td>
<td>1.008</td>
</tr>
<tr>
<td>1.3</td>
<td>1.002</td>
<td>1.006</td>
</tr>
<tr>
<td>2</td>
<td>1.003</td>
<td>1.011</td>
</tr>
</tbody>
</table>

Table 2: response coefficients of OPIS and SANOA DOAS obtained for SO2 and NO2 by using the
calibration cells fed at different values of gas flow

The flow variation between 0.3 to 2 l/min does not influence the response of the instrument.

We did not observe a significant difference between the results obtained with the small calibration cell and the
test-bench cell if the uncertainties from gas generation are taken into account.

On the base of these results, it is possible to accept the procedure proposed by the manufacturers.

ACKNOWLEDGES

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