

v2

**EXPLANATORY & GUIDANCE  
document (E&G-d)  
on IED-based (draft)  
Waste Incineration BREF  
and BAT conclusions**

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**ANNEX 3  
Monitoring & Uncertainties**

**Annex 3.c  
QAL2 calibration examples**

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## Annex 3.c: QAL2 calibration examples

### Abbreviations and explanations

See **Annex 1** to this Explanatory and Guidance document (E&G-d) for:

- a summary of abbreviations
- and, in **its section 2**, explanations on measurements wording.

### 1. Standards requirements on calibration

#### 1.1 WI BREF BAT conclusions requirement

WI BREF BAT conclusion n° 4 requires to monitor channelled emissions to air in accordance with EN standards, including “Generic EN Standards”: “EN 15267-1, EN 15267-2, EN 15267-3 and EN 14181”.

#### 1.2 Standards requirements on calibration

As stated in its scope, Standard EN 14181 “specifies procedures for establishing quality assurance levels (QAL) for automated measuring systems (AMS) installed on industrial plants for the determination of the flue gas components and other flue gas parameters.

*This European Standard specifies:*

- a procedure (QAL2) to calibrate the AMS and determine the variability of the measured values obtained by it, so as to demonstrate the suitability of the AMS for its application, following its installation;
- a procedure (QAL3) to maintain and demonstrate the required quality of the measurement results during the normal operation of an AMS, by checking that the zero and span characteristics are consistent with those determined during QAL1;
- a procedure for the annual surveillance tests (AST) of the AMS in order to evaluate (i) that it functions correctly and its performance remains valid and (ii) that its calibration function and variability remain as previously determined.

*This European Standard is designed to be used after the AMS has been certified in accordance with the series of European Standards EN 15267.”*

As detailed in the General principles (§ 5.1) of EN 14181 Standard:

*“QAL2 is a procedure for the determination of the calibration function and its variability, and a test of the variability of the measured values of the AMS compared with the maximum permissible uncertainty given by legislation. The QAL2 tests are performed on suitable AMS that have been correctly installed and commissioned. A calibration function is established from the results of a number of parallel measurements performed with the standard reference method (SRM). The variability of the measured values obtained with the AMS is then evaluated against the maximum permissible uncertainty.*

*The QAL2 procedures are repeated periodically, after a major change of plant operation, after a failure of the AMS or as required by legislation.”*

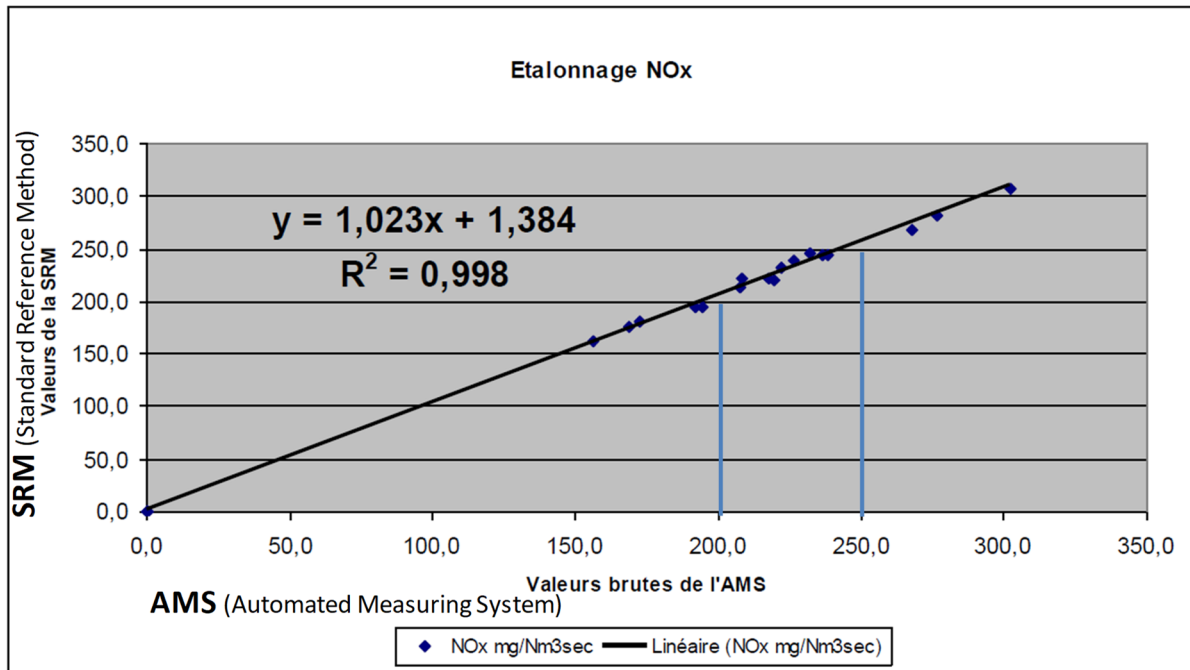
QAL2 procedure must be repeated every 3 years for incineration plants, every 5 years for Large Combustion plants. As its name says, AST (Annual Surveillance Test) is to be repeated every year.

See also [section 2 of Annex 1](#) to this E&G-d.

### 1.3 Example of QAL2 calibration graph

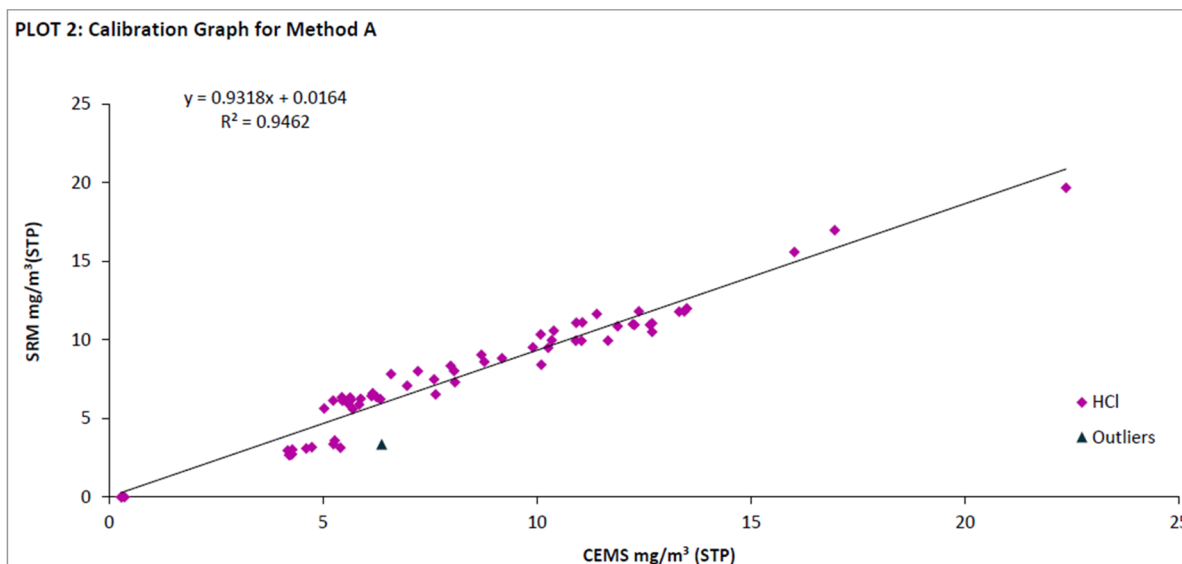
Examples of QAL2 calibration curves are given in the INERIS report (see annex C, pp. 101-116), from WtE plants from several EU Member States.

The QAL2 tests are usually presented on graphs showing on the X-axis the reading of the AMS and, on the Y-axis, the reading of the SRM, which is assumed to give the true value. Ideally, the 2 instruments should read the same and therefore the calibration should be  $y = x$ .



**Figure 3.c-1:** Example of QAL2 calibration function obtained for NOx.

The calibration function for NOx on [Figure 3.c-1](#) is very good with a slope of nearly 1, a small y-intercept (1,384) in respect of the ELV (200 mg/Nm³) and, above all, the coefficient of determination nearly equal to 1 ( $R^2 = 0,998$ ). This excellent result is due to the fact that the concentrations are significant and varying during the test (from 150 to 300 mg/Nm³).



**Figure 3.c-2:** Example of QAL2 calibration function obtained for HCl.

The concentration range covered during the test (see [Figure 3c.2](#)) is large and at a high concentration level: 5 to 22 mg/Nm<sup>3</sup>, giving a satisfactory calibration function  $y = 0.9318 + 0.0164x$  with  $R^2 = 0.9462$ . The variation in concentration was possible on this line using a dry Flue Gas Cleaning process where the set-point can be adjusted.

## 2. INERIS study findings on onsite QAL2 and AST calibration

The INERIS study includes the examination of about 40 QAL2 reports carried out in Europe. The investigation on these QAL2 reports corroborates the above conclusions (see [Annex 2.b](#) to this E&G-d) on the real uncertainties being higher than requested by Standards at the very low concentration levels achieved by incinerators (see INERIS report, pp. 19-22):

*“(..) AMS calibration by SRM implies having SRMs with much lower uncertainties than the ones required by the IED for the values given by the AMS. However, given the collected data, this prerequisite for calibration is not fulfilled (except for NO<sub>x</sub>), thus impacting the quality of current QAL2 results. The non-respect of this rule causes a calibration of poor quality and therefore a higher uncertainty of the measurements given by the AMS. (...). For some substances, CO, TOC, Dust, HCl, HF or NH<sub>3</sub>, the actual uncertainty of the SRM (estimated from ILCs) at the current Daily ELVs is higher than the uncertainty required in the standard describing the reference method. (...)”*

*This shows that the GUM approach, which assesses the suitability of the measuring system according to the SRM, does not reflect the actual uncertainty in the field. This real uncertainty affects the quality of the QAL 2 performed.”*

In the following sections, examples of calibration function graphs have been selected to illustrate the high relative uncertainties obtained during QAL2 or AST tests when emissions are low. Most of them come from Annex C of the INERIS report, which includes a few more.

### 2.1 Very low and not varying concentrations

Another finding of the INERIS study is that when the concentrations are very low and not varying during the calibration test, the test provided by the standard to check the quality of the calibration is

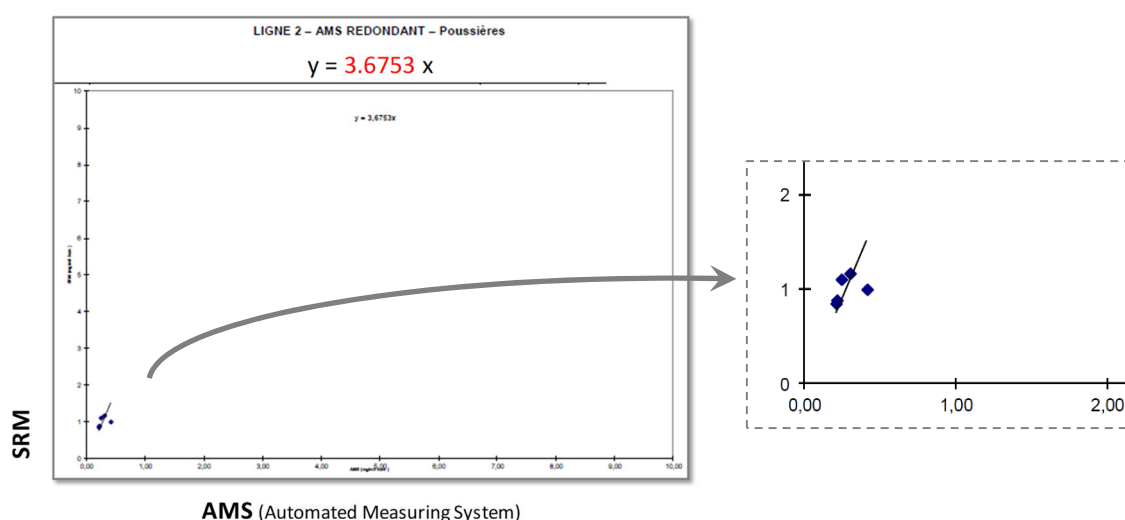
always positive, whatever the calculated calibration function, and therefore in such case is inadequate (see INERIS report, summary p. 21):

*“It should be noted that EN 14181 provides a variability test to ensure that the uncertainty at the daily ELV is lower than the maximum uncertainty allowed by IED. However, because of the nature of the variability test formula, when the measured concentrations are much lower than ELV, the variability test always passes, even if values are very scattered. In other words, the fact that the variability test is successful gives no information on the actual uncertainty when emission concentrations are much lower than the ELV.”*

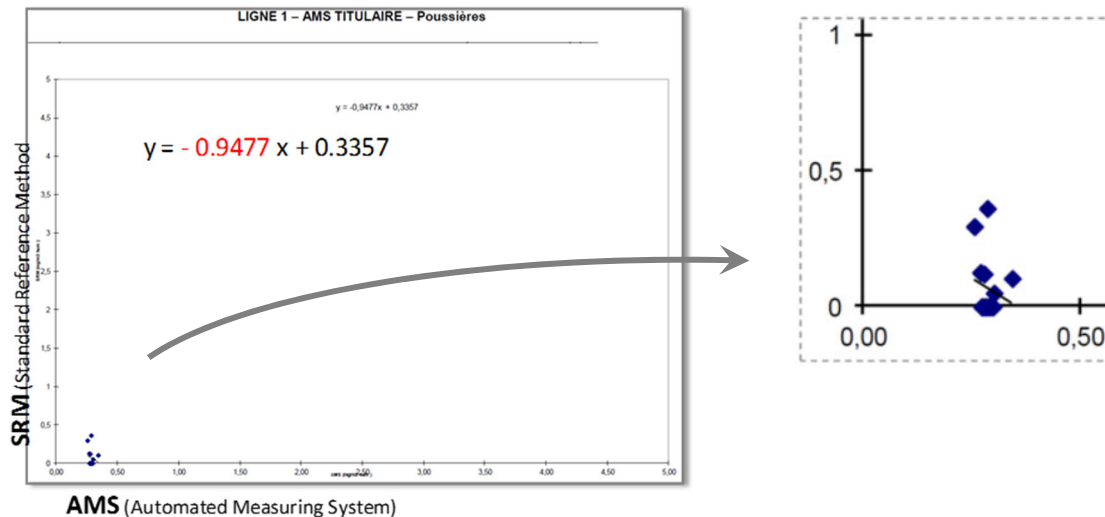
*“As it can be seen in Annex A of the report this is what happened for many calibration tests of the QAL2 reports which were investigated in the study for which the measured concentrations were very low: although the calculated calibration functions do not show good correlations sometimes, the variability tests were always satisfied: for example if a daily ELV equal to 10 mg/Nm<sup>3</sup> with related maximum uncertainty of 20%, then the variability test will accept to have a difference of up to 2 mg/Nm<sup>3</sup> between the reading of AMS and the value measured by the SRM which, for instance, could be 3 mg/Nm<sup>3</sup>. In this case:*

- *the uncertainty level at 3 mg/Nm<sup>3</sup> is much higher than 20% ( $2/3 = 67\%$ )*
- *and since it provides no information on the differences between AMS and SRM readings at 10 mg/Nm<sup>3</sup> the test does not give any idea on the level of uncertainty actually performed at 10 mg/Nm<sup>3</sup>.”*

This can be seen for instance on the following graphs.



**Figure 3.c-3:** Example of QAL2 calibration function obtained for dust. For easier reading, the low concentrations area of the graph is enlarged on the right. The reading of the SRM on the Y-axis is supposed to give the true value. The dots shown are very close to each other and at very low concentration. Although the variability test is OK, the test does not allow to raise satisfactory conclusions. Here, the slope of the calculated correction function is most probably too high (3.6753) whilst the y-intercept, which is equal to 0, when it should most probably be around 0.7. (See INERIS study Annex C, Dust, Example 1).

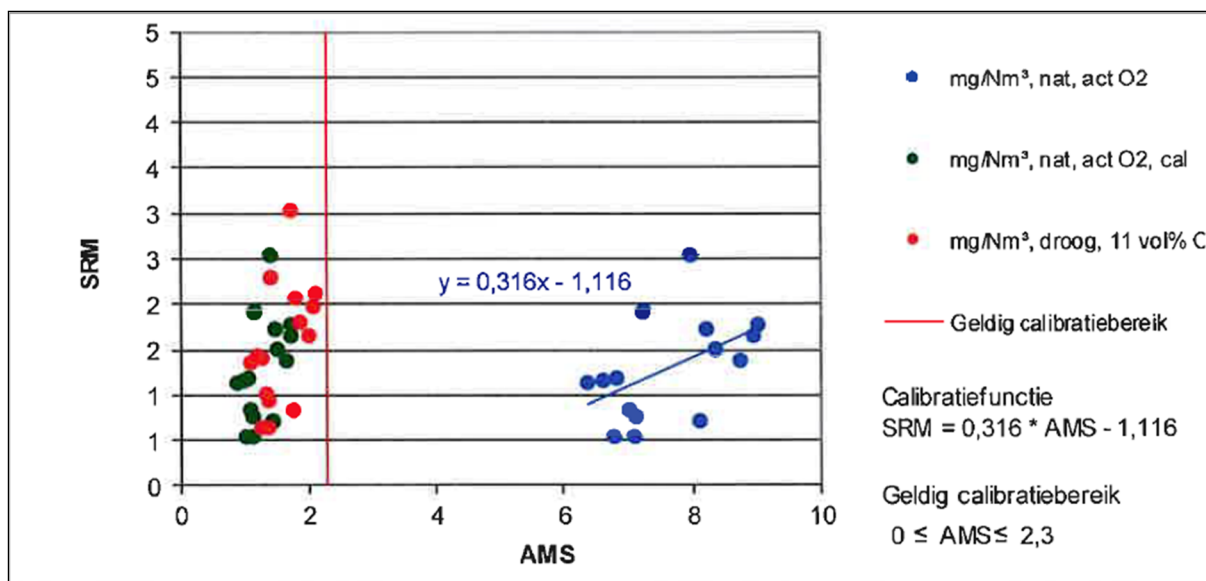


**Figure 3.c-4:** Example of QAL2 calibration function obtained for dust on line 1 of the same plant as in **Figure 3.c-3**. Quite similar to the one above. However here, the obtained correction function keeps a negative slope, meaning that, if it were to be used, the higher the value read by the online instrument, the lower the corrected value. (See INERIS study Annex C, Dust, Example 2).

When looking at the outcome of the QAL2 tests shown on **graphs 3.c-3 and 3.c-4:**

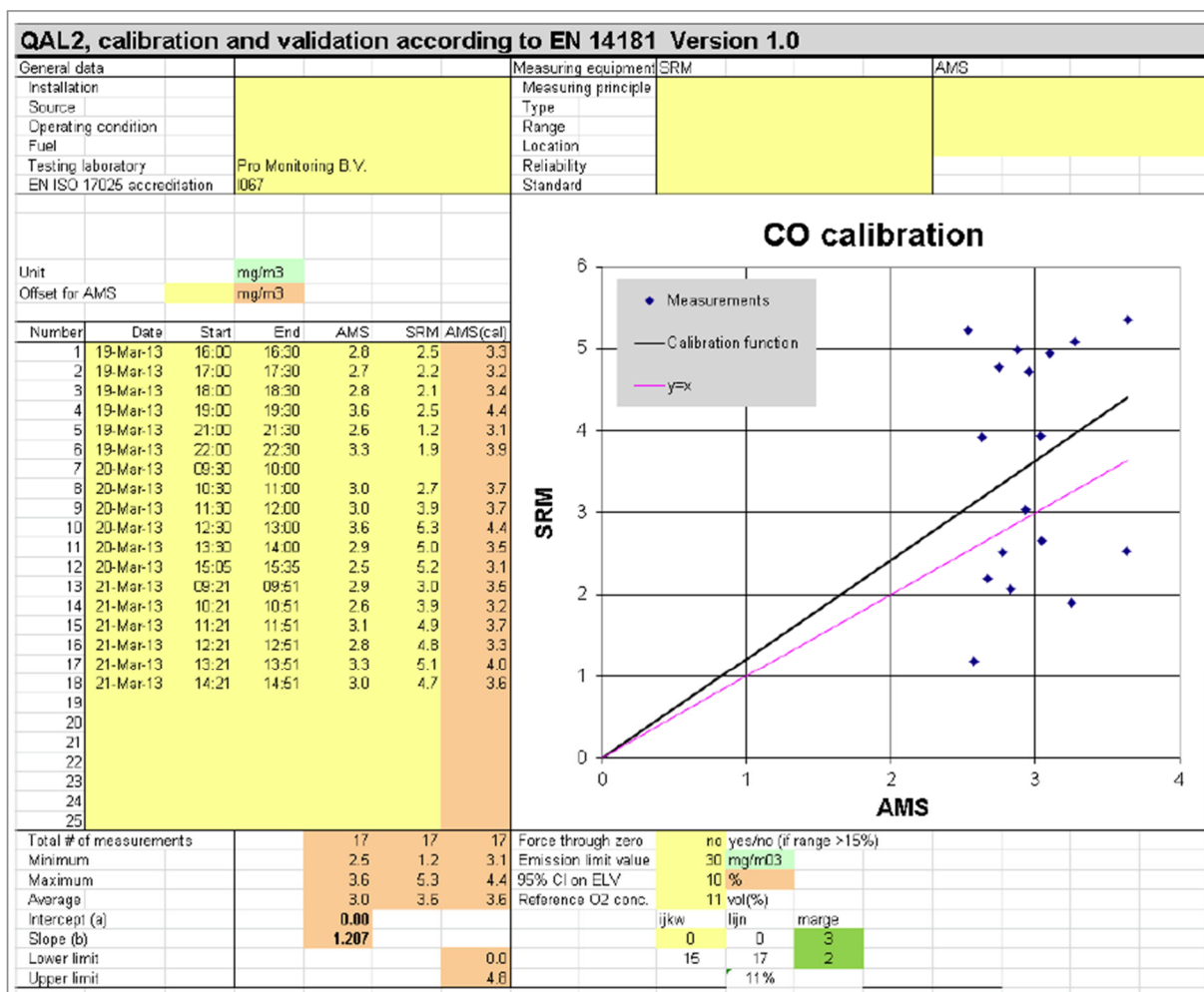
- although statistically validated (variability test fulfilled because the differences between SRM and AMS readings are small in respect of the ELV),
- the calibration function is not satisfactory;
- however, although the relative uncertainties are high,
- the readings of the two instruments are not so different in absolute values.
- Lowering the ELV would just worsen the situation in terms of QAL2 and uncertainty without reducing the emissions of that plant.

The following graphs show similar situations where the concentrations are too low and not varying for a real comparison between AMS and SRM readings, which leads to high relative uncertainties at low concentration levels.



**Figure 3.c-5:** Example of QAL2 calibration function obtained for HCl. The concentration range covered during the test is limited: the SRM measurements are between 0.5 and 2.5 mg/Nm<sup>3</sup>. The AMS measures the highest values, from 6 to 9 mg/Nm<sup>3</sup>. This yields a calibration function whose slope is very different from 1: 0.316, and whose intercept is high given the concentration. Also, the dispersion of the points shows a weak correlation between SRM and AMS. (See INERIS study Annex C, HCl, Example 4).

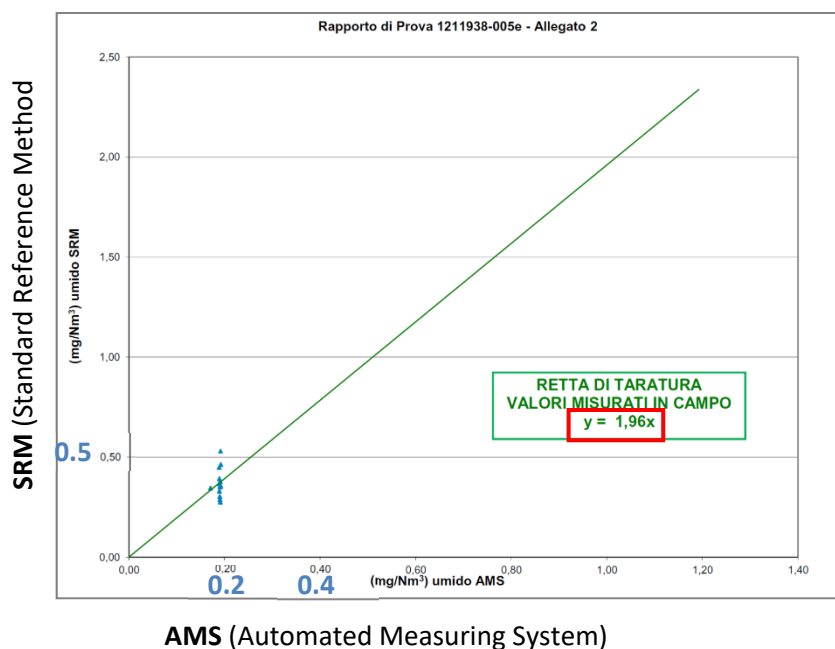




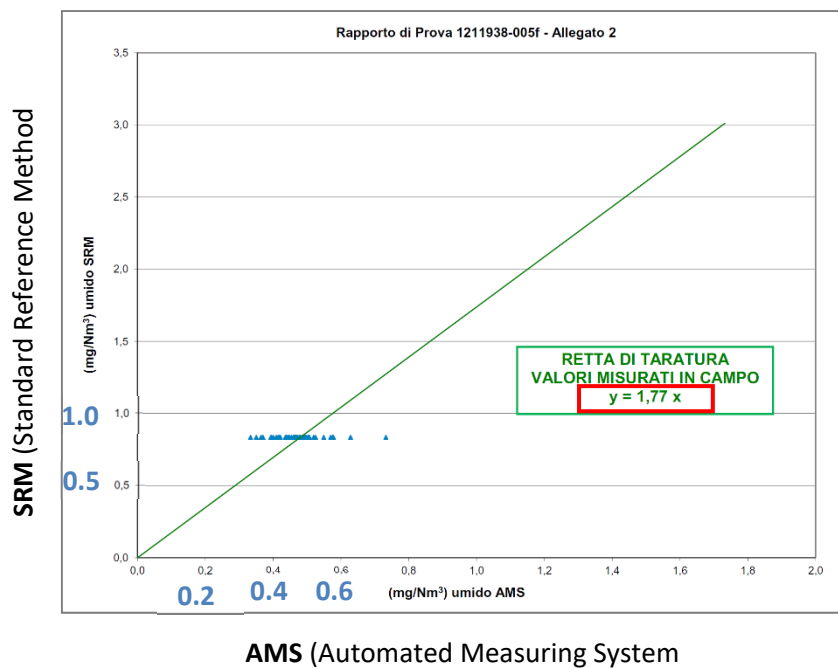
**Figure 3.c-6:** Example of QAL2 calibration function obtained for CO. The narrow concentration range covered during the measurements, from 1 to 5 mg/Nm<sup>3</sup>, is at a relatively low concentration level, leading to a cloud of points. It is thanks to a zero measurement that the slope is at 1.2. The spread of the values obtained at the concentration level measured during the QAL2 control raises doubts on the values that would be given by the AMS by applying this function at that level. Lowering the Daily ELV to these concentration levels would raise serious doubts on the compliance/incompliance declaration. (See INERIS study Annex C, CO, Example 1).

## 2.2 Near the Limit of Quantification

The emission levels are sometimes so low that one or the two instruments are at their Limit of Quantification or below. See the two graphs below. In such case, even if the variability test is satisfied the calibration curve is meaningless. Here even if the relative uncertainty is high, the reading of the two instruments are not so different in absolute values.



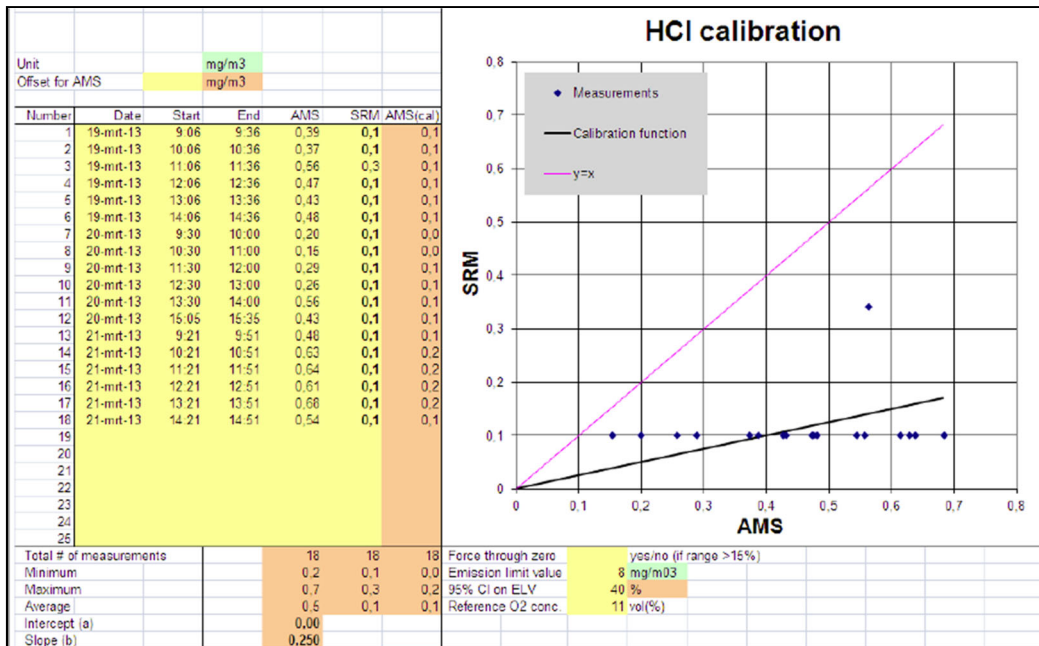
**Figure 3.c-7:** Example of QAL2 calibration function obtained for dust. During the test, the readings of the AMS were constant. For the AMS, as for the SRM, the concentrations likely are close to, or even below the Limit of Quantification. It is thanks to a zero measurement that a calibration function has been calculated but this function,  $y = 1.96$ , is not really funded. (See INERIS study Annex C, Dust, Example 3).



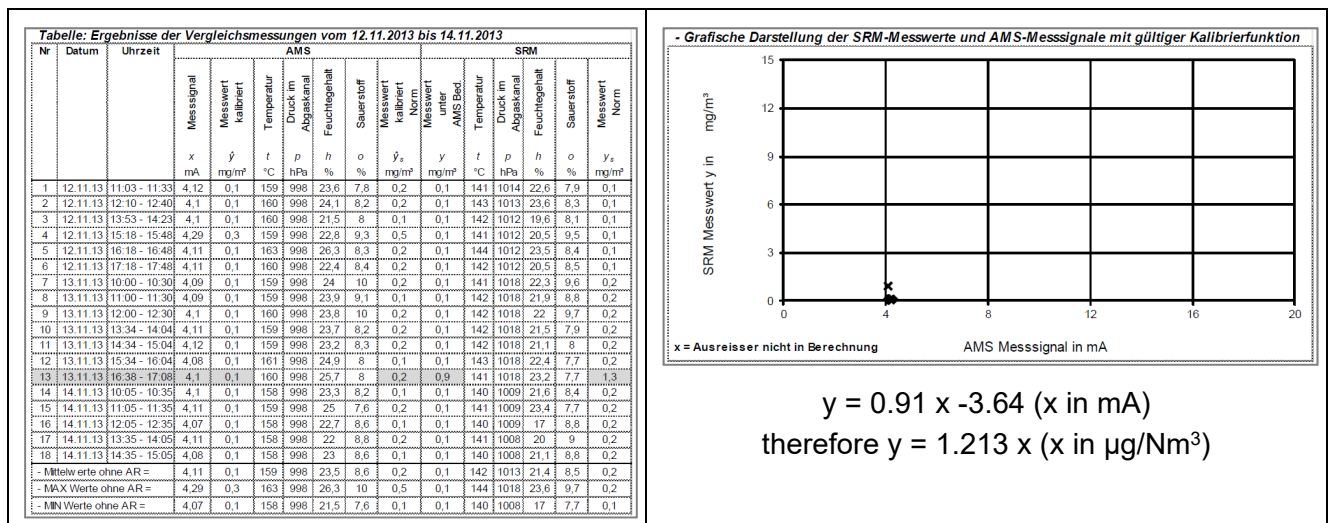
**Figure 3.c-8:** Example of QAL2 calibration function obtained for TOC. During the test, the readings of the SRM were constant. For the SRM, as for the AMS, the concentrations likely are close to, or even below the Limit of Quantification. The calculated correction function,  $y = 1.776x$ , is not really funded. (See INERIS study Annex C, TOC, Example 1).

For the two above tests, it is thanks to a zero measurement that the slope is not infinite ( $x=0.2$  for Figure 3.c-7) or zero ( $y=0.8$  for Figure 3.c-8). However, with a slope of 1.96 or 1.77, there can be doubts on the measurements that would be given at the current Daily ELVs of  $10 \text{ mg/Nm}^3$  for both dust and TOC, applying these functions, if a malfunction of the installation or of the Flue Gas Cleaning led to an emissions peak. Doubts can also be expressed on the possibility to check the AMSs compliance if the ELVs were lowered around the levels of the measured values.

Similar problems are shown on the following graphs.



**Figure 3.c-9:** Example of QAL2 calibration function obtained for HCl. The measured concentrations are low: < 1 mg/Nm<sup>3</sup>. The SRM's readings are constant. Both for the AMS and the SRM, the concentrations likely are close to, or below, their LoQs. It is thanks to a zero measurement that a non-nil slope can be obtained. This slope nevertheless is 0.25, hence very far from 1. (See INERIS study Annex C, HCl, Example 1).



**Figure 3.c-10:** Example of QAL2 calibration function obtained for dust. The AMS reading are shown in a range from 4 to 20 mA instead of in mg/Nm<sup>3</sup>. All the SRM's values are equal to 0.1 mg/Nm<sup>3</sup>, likely because they are near, or below, the LoQ. The function was determined by a zero measurement. (See INERIS study Annex C, TOC, Example 5).

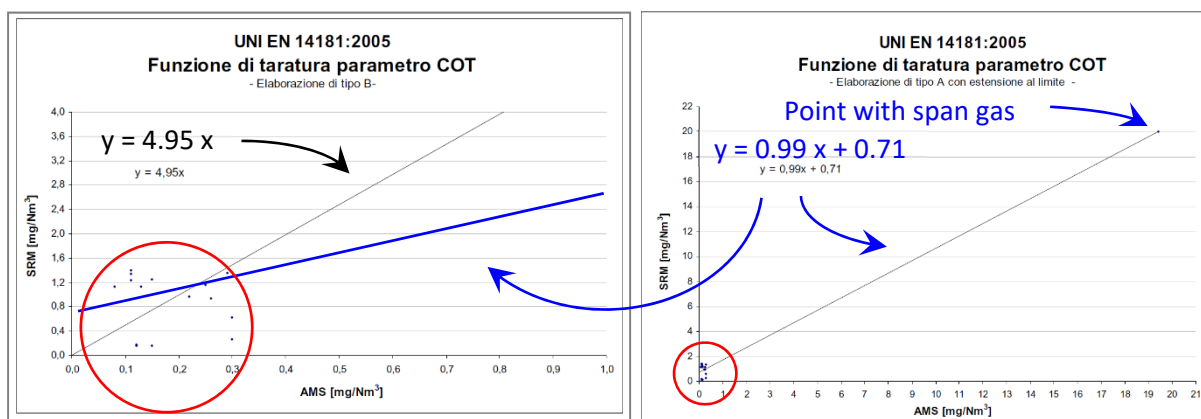
### 2.3 Use of a span gas

As said in the INERIS report p. 22, span gas (also 'spike gas') can be used when the concentrations are low and not varying but this does not improve the accuracy at low levels:

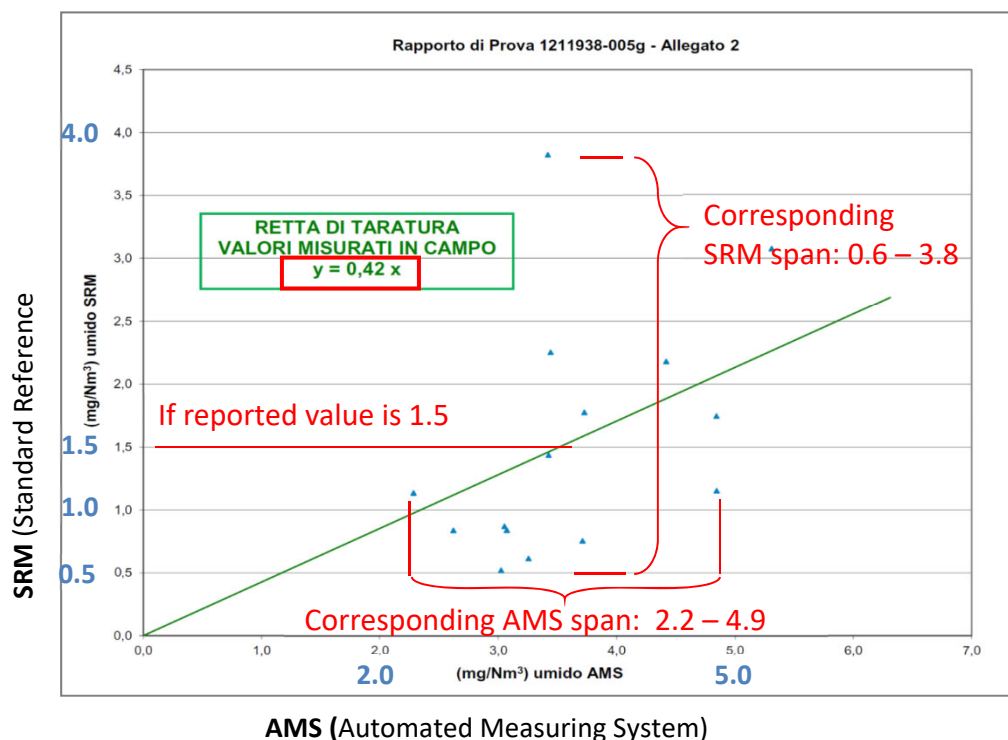
"In the case where the cloud of points is at low concentration levels, the test is irrelevant because one cannot conclude on the behaviour of the equipment with measurements near the Daily ELV. For

this reason, where concentrations are under 30% of the Daily ELV, the French Guide FD X 43-132 proposes for the determination of the calibration function, to combine with AMS/SRM data those obtained when a span gas is injected to the AMS at zero and another one at the concentration of the Daily ELV. Thus, required uncertainty at the Daily ELV will be better respected and the AMS will more likely show a correct value around the daily ELV, if the installation was to emit at this level of concentration. A similar procedure (known as method C) is provided in the revised version of Standard EN 14181 (December 2014). It is important to note that using span gas does not help improving the uncertainty at very low concentrations where the effect of interfering substances can be significant. A selection of graphs presenting the measurements [AMS;SRM] during QAL2 controls and calibration lines can be found in Annex C. They highlight the important increase and difference in measurement results between AMS and SRM in relative value when concentrations are much lower than IED daily ELVs.

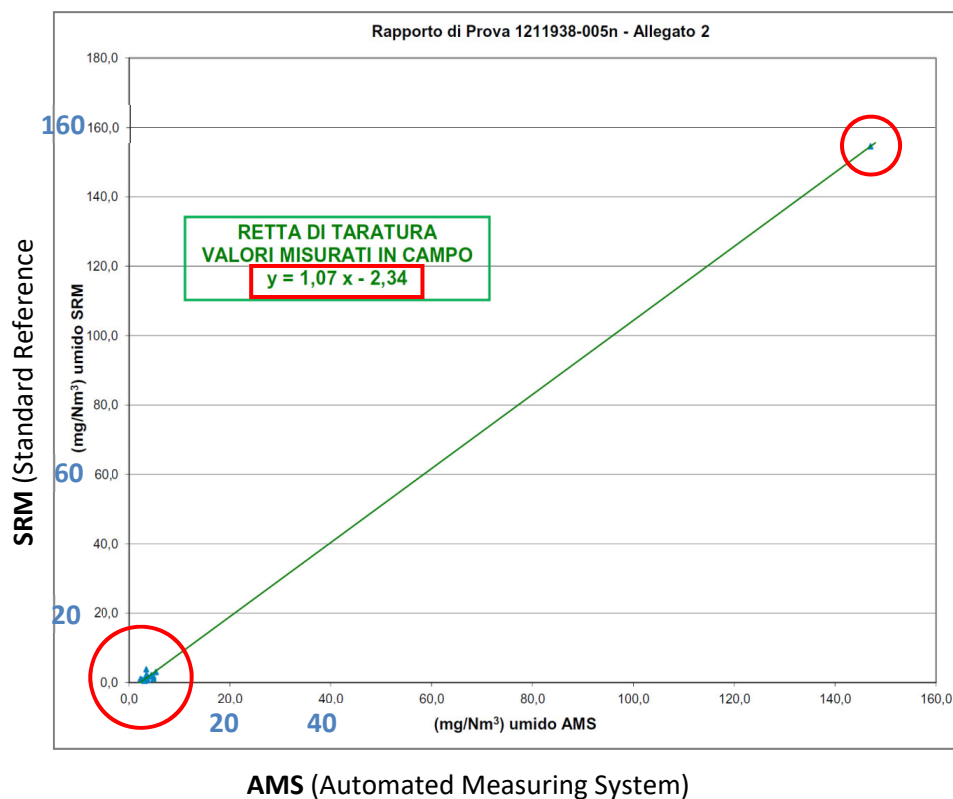
Laboratories already often use calibration gases in combination with results of comparison (AMS/SRM), and sometimes more extensively than what is allowed by EN 14181, e.g. at concentrations much higher than the daily ELV. There are some limitations to the use of calibration gases, mainly because they may not exist at very low concentrations and that diluting a calibration gas with a level of concentration would introduce an additional source of uncertainty. Moreover, using reference materials is not always possible, e.g. for dust calibrations. When reference materials exist and when the level of emission concentration is very low compared to ELV, a combination of the results of a limited number of measurement results (AMS/SRM) and of the results of the adjustment of the AMS by the reference material is an acceptable procedure to obtain a realistic calibration function (see EN 14181 § 6.4.3 c) but the use of reference material only which was the common practice before EN 14181 was launched, is no more accepted by this standard.”



**Figure 3.c-11:** Example of QAL2 calibration function obtained for TOC. First time without span gas, the slope was 4.95 (see black straight line). On the second graph with span gas, injected at 20 mg/Nm<sup>3</sup> the slope was nearly 1, which is much better. However, the accuracy at low concentrations (see the red circle) was not improved. NB: The calibration line obtained with the span gas ( $y = 0.99x + 0.71$ ) has been displayed as well on the left graph, in blue. Note that the proportions between the Y-axis and the X-axis scales are not the same in the two graphs. (See INERIS study Annex C, TOC, Example 2)



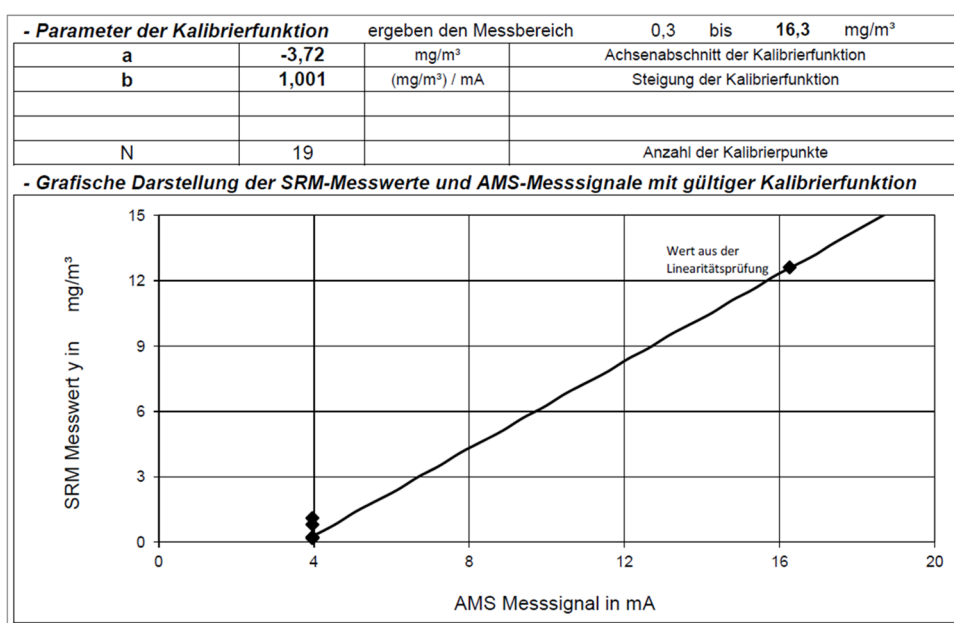
**Figure 3.c-12:** Example of QAL2 calibration function obtained for SO<sub>2</sub>. Both AMS and SRM show low readings in respect of the daily ELV (50 mg/Nm<sup>3</sup>). However if the reported value is 1.5 mg/Nm<sup>3</sup>, the AMS may have read a value between 2.2 and 4.9 mg/Nm<sup>3</sup> whilst the SRM was reading a value between 0.6 and 3.8 mg/Nm<sup>3</sup>. Once again the relative uncertainty is very high even if the absolute uncertainty remains reasonable in respect of the daily ELV. (See INERIS study Annex C, SO<sub>2</sub>, Example 1).



**Figure 3.c-13:** Same set of test points as in previous graph, plus injection of span gas. The QAL2 calibration function obtained for SO<sub>2</sub> looks better ( $y = 1.07 x - 2.34$ ) and calibration value is probably better for high

concentrations. However the span gas concentration (150 mg/Nm<sup>3</sup>) was very high in respect of the daily ELV (40 mg/Nm<sup>3</sup> on this line) and the y-intercept of -2.3 shows that for the site concentration level, the SRM and AMS measurements are offset: the uncertainty at the low site concentration level is not improved by the use of span gas. (See INERIS study Annex C, SO<sub>2</sub>, Example 2.)

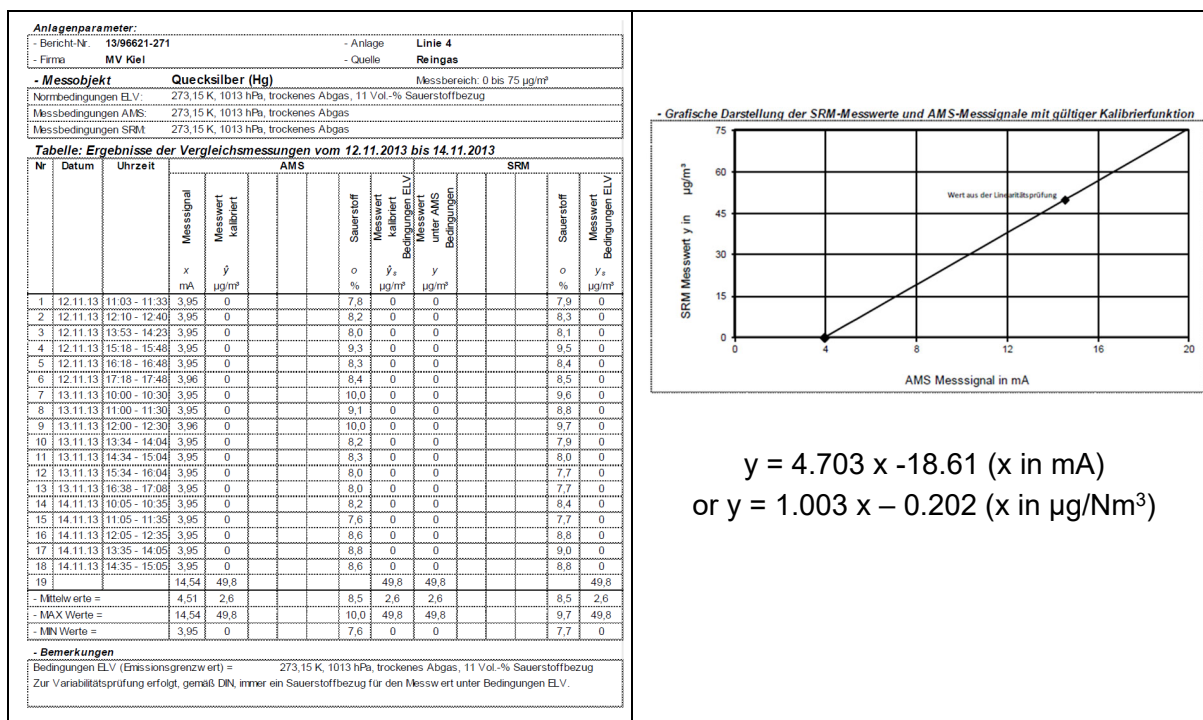
These examples (see **graphs 3.c-11 to 3.c-13**) confirm the interest to use a span gas for calibration. The slope is nearly 1 in both cases and most probably more realistic than the slopes of 4.95 and 0.42 calculated without span gas (see **graphs 3.c-11 to 3.c-12**). However, if the injection of span gas at 20 mg/Nm<sup>3</sup> for TOC is still acceptable since it is twice the current daily ELV, the injection at 150 mg/Nm<sup>3</sup> for SO<sub>2</sub> is too high since the daily ELV is at 40 mg/Nm<sup>3</sup> for this plant (50 mg/Nm<sup>3</sup> required by IED Annex VI). At lower concentration levels, these calibration functions will lead to a discrepancy between the SRM and the AMS and to the impossibility of checking compliance with the Daily ELV if it is lowered to the level of the values measured during the QAL2 control.



$$y = 1.001 x - 3.72 \text{ (x in mA)} - \text{ or } y = 1.068 x - 0.284 \text{ (x in } \mu\text{g/Nm}^3\text{)}$$

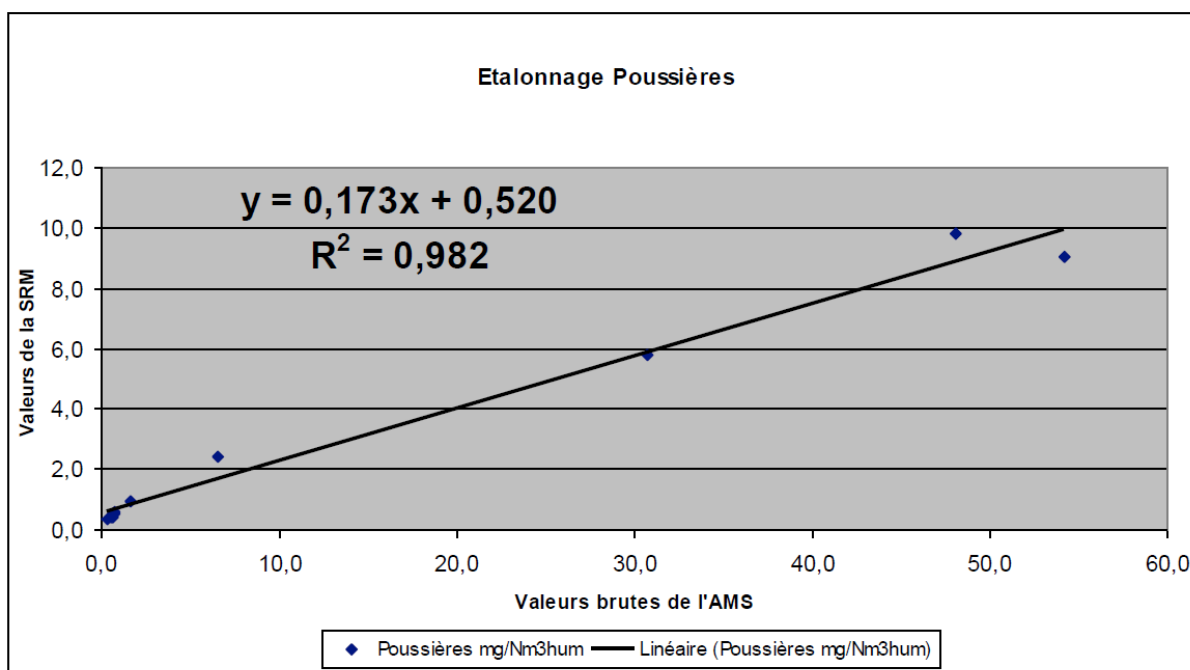
**Figure 3.c-14:** Example of QAL2 calibration function obtained for HCl. The AMS reading are shown in a range from 4 to 20 mA instead of in mg/Nm<sup>3</sup>. The concentrations are low (SRM measurements from 0.2 to 1.1 mg/Nm<sup>3</sup>), the variation range is limited and the AMS gave constant measures of 3.96 mA, or -0.04 mg/Nm<sup>3</sup>, which is a negative value. Adding a span gas measurement value for calibration at 13 mg/Nm<sup>3</sup> gives a calibration function with a correct slope, but the AMS and SRM correlation is difficult to assess because the concentrations likely are close to, or below, the LoQ of both measurement instruments. (See INERIS study Annex C, HCl, Example 2)





**Figure 3.c-15** Example of QAL2 calibration function obtained for Hg (mercury). The AMS readings are from 4 to 20 mA instead of in mg/Nm³. All the AMS and SRM measurements are zero, likely because they are under their respective LoQs. It is through span gas injection that the calibration function is calculated. However, this raises the question on what calibrated values will be given for concentrations above the LoQ of the AMS. (See INERIS study Annex C, Hg, Example 2)

## 2.4 Impact of the change of conditions during the calibration test

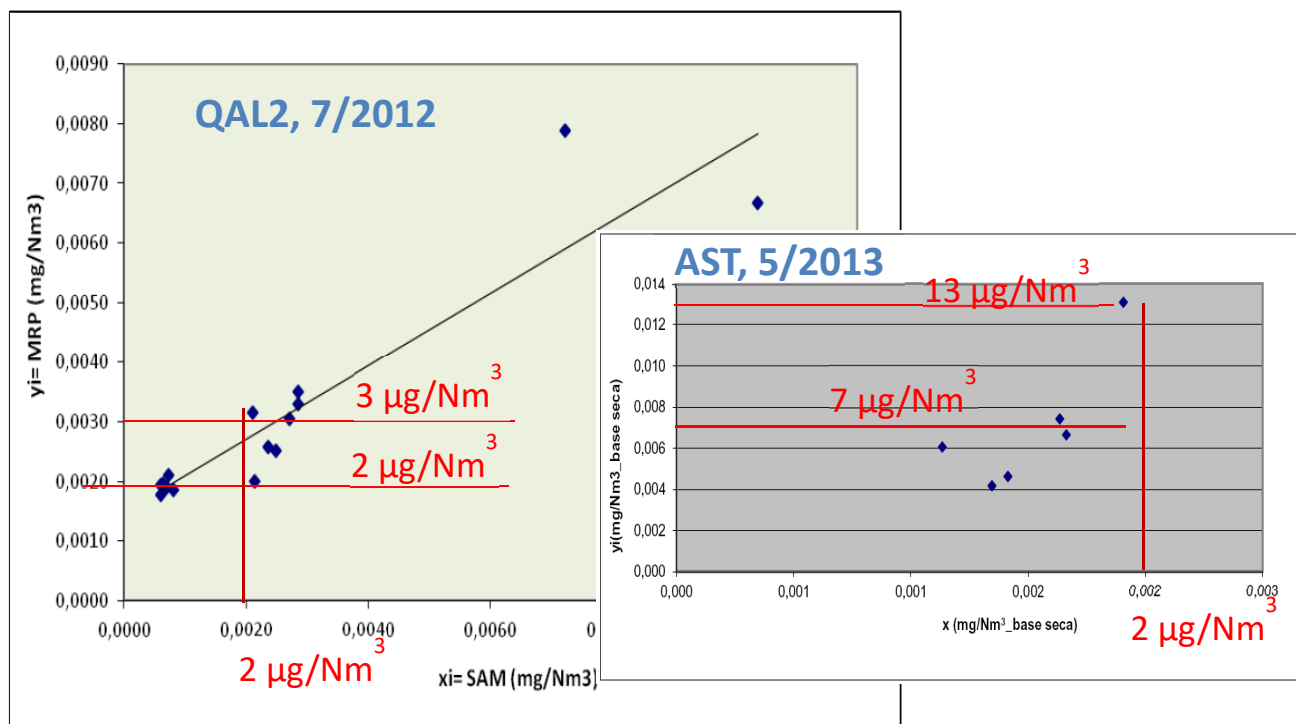


**Figure 3.c-16:** Example of QAL2 calibration function obtained for dust. The concentration range covered during measurement is wide, giving a calibration function that could seem satisfactory:  $R^2 = 0.9825$ , but the slope is very different from 1: 0.173, and the intercept point is 0.5 mg/Nm³, which is high for levels under 2 mg/Nm³. (See INERIS study Annex C, Dust, Example 6).



In the calibration test shown on [Figure 3.c-16 above](#), the 3 highest measurements likely were obtained by enriching (spiking) or by changing the installation's operating conditions. The differences between SRM and AMS can be caused by the used particulates, resulting from these changed conditions, and that would have different physical characteristics than those in the duct (the optical AMSs are very sensitive to particulates' physical characteristics), or because a lack of a reference material, the AMS could not be tuned, explaining the measurement bias.

## 2.5 Drift



**Figure 3.c-17:** QAL2 and AST on mercury on the same line. During QAL2, when the AMS reads  $2 \mu\text{g}/\text{Nm}^3$ , the SRM reads between 2 and  $3 \mu\text{g}/\text{Nm}^3$ . 10 months later, when the AMS reads  $2 \mu\text{g}/\text{Nm}^3$ , the SRM reads between 7 and  $13 \mu\text{g}/\text{Nm}^3$ .

The two graphs on the above [Figure3.c-17](#) show that there can be a significant drift on some instruments. When the AMS reads  $2 \mu\text{g}/\text{Nm}^3$ , the SRM reads first between 2 and  $3 \mu\text{g}/\text{Nm}^3$  but 10 months later, the SRM reads between 7 and  $13 \mu\text{g}/\text{Nm}^3$ , i.e. 200 to 400% more. Lowering the Daily ELV to these concentration levels would raise serious doubts on the compliance/incompliance declaration.

## 3. Learnings from QAL2 calibration tests

The validation of the quality of the QAL2 (Quality Assurance Level 2) tests and of the AST (Annual Surveillance Test), which depends on daily ELVs, are already doubtful with current IED Annex VI daily ELVs and can *a fortiori* not be ensured if the daily ELVs are significantly lowered. Indeed the correction curves obtained during QAL2 tests are already often unrealistic for the daily ELVs indicated in IED Annex VI for certain pollutants. Most of the time, this is due to the impossibility to vary emission concentrations as well as to the high uncertainty of the existing reference methods (SRMs) used for QAL2 at these emission levels. The use of reference materials can help. However, they often are limited to concentrations above the current IED ELVs, which makes it difficult to use them for ELVs at lower levels.