CEN/TC 264/TFE N 96

Handling and reporting of concentrations below Limit of Detection or Limit of Quantification regarding European Standards (CEN/TC 264, stationary source emissions)

Version 03 (2019-01-10)

Introduction

CEN/TC 264 "Air Quality" decided at the 25th Plenary Meeting in Rome, Italy in May 2015:

> TC 264 asked the Task Force Emissions (TFE) to convert Document N 2345 (= TFE N 48) into a document containing recommendations of how to handle and report concentrations below the limit of detection or limit of quantification in emission measurements. This document shall be made available as soon as possible to provide guidance in this regard to all CEN/TC 264 working groups (Decision 946).

> It was identified during the discussion that a definition of the detection and quantification limit is needed for manual methods. The handling of data below the detection or quantification limit is not only relevant for manual methods but also for automated methods. The handling of such data should be specified for each method. For heavy metals, this should be considered during the revision of the standard.

1 Scope

This Document prepared by Task Force Emission of CEN/TC 264 provides recommendations and guidance to all CEN/TC 264 working groups on the handling and reporting of concentrations below the limit of detection or limit of quantification ~~when preparing or revising European Standards~~.

2 Terms and Definitions

Limit of detection (LoD) means the smallest measurand concentration which can be detected, but not quantified, in the experiment conditions described for the method (ROM, 2018).

Limit of quantification (LoQ) means the smallest measurand concentration which can be quantified, in the experiment conditions described for the method.(ROM, 2018)

Note: If the LoQ is not known or not reported, it can be estimated as a multiple of the LoD, for example by multiplying the LoD given in the relevant (EN) standard with a factor of three for manual methods.

Note: For manual methods the field blank can influence the smallest measurand concentration.

3 Symbols and abbreviations

LoD   Limit of detection

LoQ   Limit of Quantification

ELV   emission limit value

4 General recommendations regarding LoD and LoQ

An overview of the current state on European requirements regarding LoD and LoQ is given in the following annexes to this document

- Annex A:
  a) AQUILA Recommendation 12 (May 2009) (revised for clarification March 2011)
  b) Integrated Pollution Prevention and Control (IPPC) Reference Document on the General Principles of Monitoring, July 2003, MON REF
  c) JRC Reference Report on Monitoring of Emissions to Air and Water from IED Installations, (ROM 2018)

- Annex B:European Standard EN 1948-3 (2006), EN 1948-4 (2010)

- Annex C: DIRECTIVE 2010/75/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL (IED) (extract)

Keeping in mind all the current documents regarding LoD and LoQ the TFE recommends the following:

Handling of values below the limit of detection or limit of quantification

As a general rule, the LoD should be less than 10 % of the emission limit value (ELV) in order to guarantee that the LoQ is clearly below the emission limit value (ROM 2015 (July))

Any measurement method applied should have an appropriate LoD/LoQ in relation to the emission level to be measured. In many cases, the LoD is required to be less than 10 % of the ELV in order to guarantee that the LoQ is clearly below the ELV. (ROM 2018)

Note: Some Member States have set stricter performance requirements, e.g. in France the LoQ should be less than 10 % of the ELV.

Regarding the handling and reporting of LoD and LoQ, there are two different cases to be considered:

1) Periodic measurements

    a) Calculation of average values

        There are different solutions possible (see Annex A).

        In the case of assessment of attainment of ELV individual measurement results but not average values should be compared with the ELV (in some Member states).

        In some Member States (e.g. Germany, the United Kingdom) each measurement result is assessed individually. This procedure is also used in the IED, Annex VI, Part

In other cases different approaches could be applied which are explained in the ROM 2018. Therefore, it is good practice to always report the approach taken together with the results.

b) Summation i.e. total sums of a given group of chemical measurands

In the European context of emission measurements there are the following cases:

b1) PCDD/F emission measurement (EN 1948-3(2006) – according to the IED
The total I-TEQ concentration is calculated by the addition of the concentrations of the 17 individual 2,3,7,8-chlorine substituted PCDDs/PCDFs when multiplied by the appropriate I-TEF:

b2) coplanar PCB-emissions (EN1948-3(2010))

b3) Metals and Arsenic emission measurement – according to the IED
Summation of: Sb + As + Pb + Cr + Co + Cu + Mn + Ni + V and summation of Cd + Tl;
EN 14385 gives no advice, how to deal with LoD and LoQ (see also annex C)

Here it is possible to take over the regulation from the water sector: Directive 2009/90/EC, cited in the ROM 2018 (see above):
"results below the limit of quantification of the individual substances shall be set to zero."

In a prospective revision process of EN 14385 there could be advice, how to deal with LoD and LoQ.

2) Continuous measurements

a) Calculation of average values

– daily average

– monthly average

– yearly (annual) average

In the case of continuous measurements, the AQUILA procedure (see Annex A) could be implemented. This means:

For all measurements, basic values which are greater than or equal to the negative detection limit (–LoD, i.e. the negative value of the detection limit) shall be accepted as they are and used for further evaluations and in all aggregations and calculations.

Values smaller than the negative detection limit shall be discarded.

b) Summation is only carried out with periodic measurements.

c) Calibration of AMS according EN14181

The calibration procedures according EN 14181 consider LoD and LoQ as follows:

Scattering at zero (life zero) data are used as they are.

This should be applied both for AMS and for automated SRM (recommendation of TFE).

"… the offset Z is the difference between the AMS zero reading and zero" (section 6.4.3 b).

NOTE 2 For several AMS the offset is 4 mA.

For procedure b) in EN 14181 it is essential that, prior to the parallel measurements, it is proven that the AMS gives a reading at or below detection limit (as demonstrated in QAL1) at a zero concentration (as stated in 6.2). (EN 14181, section 6.4.3 b)

d) Performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources; EN 15267-3

The performance criteria and test procedures for AMS consider LoD and LoQ as follows:

Lower limit of ranges (section 5.2.3 of EN 15267-3)

The lower limit of calibration is usually zero.

Note 1   The zero value is usually the detection limit.

(section 6.10) Repeatability standard deviation at zero point

The AMS shall meet the performance criteria for repeatability standard deviation at zero point specified in Clause 8 of EN 15267-3.

Note 1   The detection limit is two times the standard deviation at zero.

Note 2   The quantification limit is four times the standard deviation at zero.

Requirement in Table 4 of EN 15267-3 (clause 8)

Repeatability standard deviation at zero

< 5% of emission limit value ELV (that means: LoD < 10% of ELV or LoQ < 20% of ELV)

The AMS shall have a data output with a living zero point (e.g. 4 mA) so that both negative and positive readings can be displayed (section 6.4).

5   Reporting of LoD and LoQ

The Task Force Emissions recommends:

In most of the air-emission documents LoD rather than LoQ is used.

There are different approaches.

It depends on what purpose it is used. But as it is explained in the ROM: "A consistent approach should be applied either for the sector or for the specific country, so that a fair comparison of the data is possible".

If relevant, it is useful to clearly state in the permit the necessary arrangements for dealing with values below the LoD or LoQ, if it is not stated elsewhere in the national regulation. This is particularly important in the case of emission limit values expressed as calculated averages when the LoQ is not far below the emission limit value, as the approach may have an influence on the final result and the subsequent compliance assessment.

Commission Implementing Decision 2012/119/EU on the collection of data and on the drawing up of BREFs specifies in Section 5.4.7.2 that the LoD and LoQ should be given as reference information accompanying emission data during the data collection.

For periodic measurements, the sampling time can be adapted and/or the analytical method can be chosen to reach an acceptable LoQ. Therefore, it is essential that, together with the measurement results, the LoD, and preferably also the LoQ, is reported. This allows a more proper use of data when assessing measurement results (ROM).

EN 15259 requires reporting the Limit of Detection in case of deviations from the standard or in case of instruments without suitability-test (Annex B).

Bibliography

AQULA Recommendation 12 (May 2009) (revised for clarification March 2011)

https://ec.europa.eu/jrc/sites/default/files/aquila-meeting-minutes-20090506-07-recommendations.pdf

DIRECTIVE 2010/75/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 24 November 2010 on industrial emissions (integrated pollution prevention and control)

EN 14181 Stationary source emissions. Quality assurance of automated measuring systems

EN 15259 Air quality - Measurement of stationary source emissions -Requirements for measurement sections and sites and for the measurement objective, plan and report

EN 15267-3 Air quality. Certification of automated measuring systems. Performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources

Integrated Pollution Prevention and Control (IPPC) Reference Document on the General Principles of Monitoring, July 2003

Implementing Decision 2012/119/EU

JRC Reference Report on Monitoring of Emissions to Air and Water from IED Installations; Thomas Brinkmann, Ralf Both, Bianca Maria Scalet, Serge Roudier, Luis Delgado Sancho; European Union, 2018

Annexes

Annex A

Current state on European requirements regarding LoD, LoQ

a) AQUILA Recommendation 12 (May 2009) (revised for clarification March 2011)

Handling of values below the detection limit
Legal reference: None.
For all measurements, basic values which are greater than or equal to the negative detection limit (–DL, i.e. the negative value of the detection limit) shall be accepted as they are and used for further evaluations and in all aggregations and calculations.
Values smaller than the negative detection limit, shall be discarded. Only in cases where values which are greater than or equal to the negative detection limit but lower than the detection (or quantification, if available) limit are not accessible, these values shall be replaced by half the detection (or quantification, if available) limit and flagged.
Example
The detection limit is 2 µg/m³. A value of -3.1 µg/m³ is below the negative detection limit and has to be discarded. A value of -2µg/m³ is equal to the negative detection limit and therefore has to be considered as a valid measurement value (validity flag = 2, i.e. value below detection limit but measurement value given).

Discussions about two alternatives could arise in the case of -2.1 µg/m³:
1) -2.1 is smaller than -2 , so the value has to be discarded (validity flag = -1);
2) -2.1 is rounded to -2 and therefore equal to the detection limit, so the value has to be considered as a valid measurement value (validity flag = 2);
The correct answer is that the value is rounded with one decimal place (1≤|-2.1|<10, see **(2)**

Number of significant digits and rounding) and has to be discarded (option 1).
These provisions generally apply for all kinds of measurements. The rounding should be done according to the commercial rounding rules.

The only exceptions are some continuous PM monitoring techniques where negative values below the negative DL have a physical reason and shall therefore not be discarded in the course of data validation. Such values are to be considered as valid measurement values with a validity flag = 2.
The above rules were drawn by AQUILA and could be found at:
https://ec.europa.eu/jrc/sites/default/files/aquila-meeting-minutes-20090506-07-recommendations.pdf

See also Annex D.

Recommendation 12 (May 2009) (revised for clarification March 2011)
Data should be delivered to EEA with the same number of digits as they are handled in the network. Basic values must have at least one digit more than the relevant limit ortarget value. The detection limit (DL) for SO2, NO/NO2/NOx, CO and ozone shall be calculated by multiplying the standard deviation of zero signals with a factor of 3.3 as described in the revised versions of EN 14211, 14212, 14625 and 14626. This does not apply for the DL as described in the EN standards for PAH's, metals and benzene.
For all measurements basic values greater or equal –DL shall be accepted as they are and used for further evaluations. Values smaller than –DL must be discarded. Only in cases where values greater or equal -DL but lower than DL are not accessible, these values shall be replaced by 0.5*DL and flagged. These provisions generally apply for all kinds of measurements.
For manual methods and laboratory analyses, e. g. gravimetric measurements of PM and analyses of heavy metals, PAH etc. in PM, the following cases have to be considered:

Generally, any sample cannot have negative amounts of substance (PM, heavy metals, PAH etc.). Therefore, negative values should not occur. In these cases, values lower than DL, but greater or equal zero shall be taken as they are and used for all calculations. Only in cases where these values are not available, they shall be set to 0.5*DL and flagged, because there is no knowledge about the statistical distribution of values in this range. AQUILA N 143 (revised)

In some cases, for example the analysis of heavy metals in PM sampled on filters, it is necessary to subtract laboratory blank values that are directly related to specific types and/or even batches of filters used. In these cases, also small negative values may occur. Then values greater or equal –DL but lower than DL shall be taken as they are and used for all calculations. Only in cases where these values are not

available, they shall be replaced by 0.5*DL and flagged. Values lower than –DL must be discarded. In contrary to laboratory blanks as described above, field blanks should generally not be subtracted from measured values. All these values as defined above have to be used for all aggregations and calculations. When reporting individual values all values lower than DL (but greater or equal –DL) shall be reported as "<DL". Rounding has to be the very last step of any calculation, i.e. immediately before comparing the result with the limit or target value (rounding has to be done only once).

For rounding purposes, only the so-called commercial rounding has to be used.

b) Integrated Pollution Prevention and Control (IPPC) Reference Document on the General Principles of Monitoring, July 2003, MON REF

Five different possibilities for handling values below the **detection limit** are presented in Section 3.3, however, none of them have been singled out as the preferred option.

Values under the limit of detection

[Mon/tm/66]

Measuring methods normally have limitations with regard to the lowest concentration that can be detected. Clarity on the handling and reporting of these situations is essential. In many cases the problem can be minimised by using a more sensitive measuring method. Therefore, a proper monitoring strategy should attempt to avoid results under the limit of detection, so that only for less interesting low concentrations do values under the limit of detection occur. In general, it is good practise to use a measurement method with detection limits of not more than 10 % of the ELV set for the process. Therefore, when setting ELVs, the limits of detection of the available measurement methods need to be taken into account.

It is important to distinguish between the limit of detection (LOD - the lowest detectable amount of a compound) and the limit of quantification (LOQ - the lowest quantifiable amount of a compound). The LOQ is usually significantly larger than the LOD (2 - 4 times). The LOQ is

sometimes used to assign a numeric value when handling values under the limit of detection, **however the use of the LOD as a reference value is widely spread**. Problems with values of concentrations below the LOD are primarily connected to the

calculation of averages. Particularly, when the LOD is close to the emission limit value, the handling of these values has a significant importance. There are only a few written rules in the field, and as a consequence the handling varies between and even within different sectors. There are principally five different possibilities for handling values below the detection limit:

1. The measured value is used in the calculations, even if it is unreliable. This possibility is only available for certain measuring methods.

2. The limit of detection is used in the calculations. In this case the resulting mean value is normally stated as <(less than). This approach tends to overestimate the result.
3. Half of the detection limit is applied in the calculations (or, possibly, another predefined fraction). This approach may over or underestimate the result.
4. The following estimation:

   Estimation = (100 %-A)*LOD,

   where A = percentage of samples below the LOD

   Therefore if, for instance, 6 samples out of 20 are below the LOD the value that would be used for the calculations would be (100 - 30) * LOD, which is 70 % of the LOD.

5. Zero is used in the calculations. This approach tends to underestimate the result.

Sometimes the value is reported to be between two values. The first value is obtained by using zero for all measurements below the LOD, and the second by using the LOD for all measurements below the LOD. It is good practice to always report the approach taken together with the results. It is useful if the permit clearly states the appropriate arrangements to deal with these values under the limit of detection. Where possible, the choice should be consistent with that applied throughout the sector or within the own country so that fair comparisons of the data are possible. Examples are available that show the difference in results when using different approaches.

EXAMPLES OF DIFFERENT APPROACHES TO VALUES UNDER THE LIMIT OF DETECTION (LOD)

The following two examples show the differences in results when using the different approaches listed in Section 3.3.

To recap, these approaches are:

1. the absolute measurement value is used in the calculations

2. the limit of detection is used in the calculations

3. half of the limit of detection is used in the calculations (or, possibly, another predefined

fraction)

4. the percentage method, i.e. the following estimation is used in the calculations:

Estimation = (100 %-A)*LOD,

where A = the percentage of samples below the LOD

5. zero is used in the calculations.

In 'Example 1' there are 2 groups of figures, and in 'Example 2' there are 4 groups of figures,

each group has a different number of samples below the LOD.

In each group of figures:

column 1 is the flow (Q)

column 2 is the concentration (c)

column 3 is the load when using choice 3 (i.e. half of the LOD)

column 4 is the load when using choice 5 (i.e. zero)

column 5 is the load when using choice 4 (i.e. the percentage method).

In Example 1, the LOD is 20.

Example 1

Example 1

Q	C	1/2 det.lim. load	<det.lim=0 load	% meth. load	Q	c	1/2 det.lim. load	<det.lim=0 load	% meth. load
2035	<20	20350	0	16280	2035	26	52910	52910	52910
2304	<20	23040	0	18432	2304	<20	23040	0	32256
1809	21	37989	37989	37989	1809	21	37989	37989	37989
1910	26	49660	49660	49660	1910	26	49960	49960	49960
2102	<20	21020	0	16816	2102	25	52550	52550	52550
1981	22	43582	43582	43582	1981	22	43582	43582	43582
2025	<20	20250	0	16200	2025	22	44550	44550	44550
1958	<20	19580	0	15664	1958	<20	19580	0	27412
1895	21	39795	39795	39795	1895	21	39795	39795	39975
2134	<20	21340	0	17072	2134	<20	21340	0	29876
	SUM	296606	171026	271490		SUM	384996	321036	410580

4 of 10 above det.limit
<20 = 8

7 of 10 above det.limit
<20 = 14

In Example 2, the LOD is 30.

Example 2

Example 2

Q	c	1/2 det.lim. load	<det.lim=0 load	% meth. load	Q	c	1/2 det.lim. load	<det.lim=0 load	% meth. load
10934	<30	164010	0	0	10934	<30	164010	0	218680
12374	<30	185610	0	0	12374	35	433090	433090	433090
10298	<30	154470	0	0	10298	31	319238	319238	319238
	SUM	504090	0	0		SUM	916338	752328	971008

All below det.limit
<30 = 0

2 of 3 above det.limit
<30 =20

Q	C	1/2 det.lim. load	<det.lim=0 load	% meth. load	Q	c	1/2 det.lim. load	<det.lim=0 load	% meth. load
10934	<30	164010	0	109340	10934	32	349888	349888	349888
12374	<30	185610	0	123740	12374	35	433090	433090	433090
10298	31	319238	319238	319238	10298	31	319238	319238	319238
	SUM	668858	319238	552318		SUM	1102216	1102216	1102216

1 of 3 above det.limit
<30 =10

all above det.limit

3.4.4.4 Limit of detection and limit of quantification (excerpt from ROM 2018)

Laboratories adhering to EN ISO/IEC 17025:2017 are required to validate non-standard methods, laboratory-developed methods and standard methods used outside their intended scope or otherwise modified as well as to determine their performance characteristics [1, CEN 2017]. Validation usually includes the determination of the limit of detection (LoD) and of the limit of quantification (LoQ).

In the field of water analysis, there was no generic EN standard or specification in 2017 defining LoD or LoQ. However, a definition is given in Directive 2009/90/EC laying down technical specifications for chemical analysis and monitoring of water status pursuant to the Water Framework Directive [40, EC 2009]:

- **Limit of detection** means the output signal or concentration value above which it can be affirmed with a stated level of confidence that a sample is different from a blank sample containing no determinand of interest. determined with an acceptable level of accuracy and precision. The limit of quantification can be calculated using an appropriate standard or sample, an
- **Limit of quantification** means a stated multiple of the limit of detection at a concentration of the determinand that can reasonably be d may be obtained from the lowest calibration point on the calibration curve, excluding the blank.

Even though the monitoring of industrial waste water is not covered by Directive 2009/90/EC, the aforementioned definitions could be used in this context.

For measurements of emissions to air, a similar but more general definition is given in EN 14793:2017 [27, CEN 2017]:

- **Limit of detection** means the smallest measurand concentration which can be detected, but not quantified, in the experiment conditions described for the method;
- **Limit of quantification** means the smallest measurand concentration which can be quantified, in the experiment conditions described for the method.

Further specifications are given in some individual standards (e.g. in EN 1948-3:2006 for the measurement of PCDD/PCDF emissions to air from stationary sources [41, CEN 2006]).

There are several other terms in use, such as limit of determination, limit of application, practical reporting limit or demonstrability limit, but it appears that they are mostly used in the sense of limit of quantification (LoQ).

Any measurement method applied should have an appropriate LoD/LoQ in relation to the emission level to be measured. In many cases, **the LoD is required to be less than 10 % of the ELV** in order to guarantee that the LoQ is clearly below the ELV. Some Member States have set stricter performance requirements, e.g. in France the LoQ should be less than 10 % of the ELV [133, FR 2013].

Directive 2009/90/EC for chemical analysis and monitoring of water status pursuant to the Water Framework Directive is an example of EU requirements in the field of water analysis, which is, however, not relevant for emissions from IED installations. The Directive requires that the LoQ for all methods of analysis shall be equal to or below a

value of 30 % of the relevant environmental quality standards [40, EC 2009].

The LoD and LoQ strongly depend on the performance of the laboratory and the possible modifications or adaptations to specific circumstances. For instance, for periodic measurements, the **sampling time can be adapted and/or the analytical method** can be chosen to reach an acceptable LoQ. Therefore, it is essential that, together with the measurement results, the LoD, and preferably also the LoQ, is reported. This allows a better use of data when assessing measurement results. In that sense, Commission Implementing Decision 2012/119/EU on the collection of data and on the drawing up of BREFs specifies in Section 5.4.7.2 that the LoD and LoQ should be given as reference information accompanying emission data during the data collection, if available. Moreover, the aforementioned Decision stipulates in Section 3.3 that it is acceptable to use an expression of the type '< X to Y', when the lower end of the range cannot be accurately defined, when the data reported are close to the LoD [39, EU 2012].

> **If the LoQ is not known or not reported, it can be estimated as a multiple of the LoD, for example by multiplying the LoD given in the relevant (EN) standard by a factor of three.** However, the use of laboratory-specific performance characteristics of the method is preferable.
>
> For the averaging of measurement results, the way in which values below the LoD or LoQ are taken into account needs to be defined. This implies also judging if the measured pollutant is relevant for the installation under investigation and therefore whether it may be present in the release. If the best available information indicates that a pollutant is not released, there is no need to measure that pollutant or report any data. If there are indications that the pollutant could be released, even if it is not detectable at present, the data should be reported and the LoD and the LoQ should be expressed.

There are different ways to explicitly handle values below the LoD or LoQ, for example:

- Article 5 of Directive 2009/90/EC for chemical analysis and monitoring of water status pursuant to the Water Framework Directive specifies the following rules for the calculation of average values [40, EC 2009]:
  - Where the amounts of physico-chemical or chemical measurands in a given sample are below the limit of quantification, the measurement results shall be set to half of the value of the limit of quantification concerned for the calculation of mean values.
  - Where a calculated mean value of the measurement results referred to in paragraph 1 is below the limits of quantification, the value shall be referred to as 'less than limit of quantification'.
  - Paragraph 1 shall not apply to measurands that are total sums of a given group of physico-chemical parameters or chemical measurands, including their relevant metabolites, degradation and reaction products. In those cases, results below the limit of quantification of the individual substances shall be set as zero.
- In **Denmark**, the approach of Directive 2009/90/EC is modified for the monitoring of industrial waste water in order not to lose useful information for pollutants with very low concentrations (e.g. organic micro-pollutants) [38, DK EPA 2012]:
  - If less than 10 % of all samples have concentrations above the LoD, no average will be calculated.
  - If more than 10 % but less than 50 % of all samples have concentrations above the LoD, the measurement result for all values below the limit of

detection will be set as zero for the calculation of the average.
- o If 50 % or more of all samples have concentrations above the LoD, the measurement result for all values below the limit of detection will be set to half the value of the limit of detection for the calculation of the average.
- For the reporting to the **Scottish** Pollutant Release Inventory, the measurement results should be set as zero when multiple results for a pollutant are all below the LoD and there is no other reason to believe that the pollutant is present. When there is reason to believe that a pollutant is present, the measurement results should be taken as half the value of the LoD. When some values are above the LoD and some are below, then those above the LoD should be taken as the measured values, unless it can be demonstrated that the measurements are false, and the readings below the LoD should be taken as half the value of the LoD [33, SEPA 2011].

- In **France**, for summing up and averaging measurement results for emissions to air, the individual result is taken as half the value of the LoQ for concentrations below the LoQ and as zero for concentrations below the LoD [133, FR 2013].In other Member States, there might be different approaches for taking the LoD and/or the LoQ into account when measurement results are averaged. Therefore, it is **good practice to always report the approach taken together with the results**. This also applies to average emission data submitted for the drawing up or review of BREFs.

   If relevant, it is useful to clearly state in the permit the necessary arrangements for dealing with values below the LoD or LoQ, if it is not stated elsewhere in the national regulation. This is particularly important in the case of ELVs expressed as calculated averages when the LoQ is not far below the ELV, as the approach may have an influence on the final result and the subsequent compliance assessment.

NOTE: All bibliographic references in this chapter you will find in the ROM 2018.

Annex B

European Standard EN 1948-3 (2006), EN 1948-4(2010)

limit of detection (*LOD*)

minimum value of the measurand for which the measuring system is not in the basic state, with a stated probability

NOTE 1   The limit of detection, also referred to as capability of detection, is defined by reference to the applicable basic state. But it may be different from "zero", for instance for oxygen measurement as well as when gas chromatographs are used.

[prEN ISO 9169:2004, definition 3.2.6 [v]]

NOTE 2   The measurement value can be distinguished from the analytical blank value with a confidence of 99 %. The limit of detection is expressed as the mean analytical blank value (b_{ave}) plus three times the standard deviation of the analytical blank (s_b).

$$LOD = b_{ave} + 3\ s_b \ (1)$$

Where

LOD is the detection limit;
b_{ave} is the mean analytical blank value;
s_b is standard deviation of the analytical blank.

NOTE 3   In this European Standard the limit of detection should preferably be calculated from the analytical blank b_{ave}. If this is not possible, the limit of detection can be calculated from the signal to noise ratio according to 8.1.

limit of quantification (*LOQ*)

limit above which a quantification of the measurand is possible, expressed as the mean analytical blank value plus, either, five to ten times the standard deviation of the analytical blank. The factor *F* depends to the accepted measurement uncertainty.

$$LOQ = b_{ave} + F\ s_b \ (2)$$

Where

LOQ is the quantification limit;
b_{ave} is the mean analytical blank value;
s_b is standard deviation of the analytical blank.

NOTE   In this European Standard the limit of quantification should preferably be calculated from the analytical blank b_{ave}. If this is not possible, the limit of quantification can be calculated from the signal to noise ratio according to 8.1 using the requirement of Clause 8.3e.

Calculation of the measurement results

PCDD/PCDF emissions are expressed as the mass per dry standard cubic meter of waste gas and reference oxygen (or carbon dioxide) content.
The total I-TEQ concentration is calculated by the addition of the concentrations of the 17 individual 2,3,7,8-chlorine substituted PCDDs/PCDFs when multiplied by the appropriate I-TEF (see Annex A of EN1948-1:2006)

$$C_T = \frac{1}{V_{nr}} \sum \left(Q_{i\ 12c} \times \text{I-TEF}_i \right) \qquad (8)$$

where

C_T is the concentration of the emitted PCDDs/PCDFs expressed as I-TEQ under standard conditions dry and reference conditions;

$Q_{i\ 12c}$ is the amount of the emitted congener i;

V_{nr} is the waste gas volume of the sample under standard conditions dry and reference conditions calculated in accordance with Equation (5) of EN 1948-1:2006;

I-TEF_i I-TEF of congener i.

If the mass of a congener or congeners is below the limit of quantification, then two I-TEQ concentrations should be reported per sample

a) with the mass of those congener(s) below the limit of quantification being taken as equal to the limit of quantification;

b) with the mass of those congener(s) taken as zero.

The same regulation in EN 1948-4 (2010) coplanar PCBs

If the mass of a congener or congeners is below the limit of quantification, then two WHO-TEQPCB concentrations should be reported per sample:

a) with the mass of those congener(s) below the limit of quantification being taken as equal to the limit of quantification;

b) with the mass of those congener(s) taken as zero.

Annex C

DIRECTIVE 2010/75/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL (IED) (extract)

Average emission limit values (mg/Nm3) for the following heavy metals over a sampling period of a minimum of

30 minutes and a maximum of **8 hours**

Cadmium and its compounds, expressed as cadmium (Cd) Total: 0,05

Thallium and its compounds, expressed as thallium (Tl)

Mercury and its compounds, expressed as mercury (Hg) 0,05

Antimony and its compounds, expressed as antimony (Sb) Total: 0,5

Arsenic and its compounds, expressed as arsenic (As)

Lead and its compounds, expressed as lead (Pb)

Chromium and its compounds, expressed as chromium (Cr)

Cobalt and its compounds, expressed as cobalt (Co)

Copper and its compounds, expressed as copper (Cu)

Manganese and its compounds, expressed as manganese (Mn)

Nickel and its compounds, expressed as nickel (Ni)

Vanadium and its compounds, expressed as vanadium (V)

These average values cover also the gaseous and the vapour forms of the relevant heavy metal emissions as well as their compounds. 1.4.

C — total emission limit values for heavy metals (mg/Nm^3) expressed as average values over the sampling period of a minimum of 30 minutes and a maximum of 8 hours (O$_2$ content 6 % for solid fuels and 3 % for liquid fuels)

Polluting substances	C
Cd + Tl	0,05
Hg	0,05
Sb + As + Pb + Cr + Co + Cu + Mn + Ni + V	0,5