



Environmental Protection Agency

Office of Environmental Enforcement (OEE) Air Emissions Monitoring Guidance Note

(AG2)

INDEX OF PREFERRED METHODS

Version 4 - published November 2020

This publication is designed to be read in conjunction with [*AG2 Air Emissions Monitoring Guidance Note*](#)

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Wexford, Ireland.

AG2 Index of Preferred Methods

Version 4, June 2020

The index below is designed to guide all test houses/licensees on which methods to select when carrying out emissions testing for compliance purposes. Where a determinand is not listed, or a test house/licensee would like to use an alternative method, permission in writing must be sought from the EPA before any testing proceeds. This guide also contains some further information relating to the standards listed. It should be recognised that the requirements stated in the standards always take precedence when applying and implementing standard methods.

This index also includes a series of appendices. These have been included to provide clarification on a number of the standard methods. This is because standards may contain various options and approaches, as well as potential ambiguities. The appendices have been produced for several standards to ensure that they are applied consistently. They may be used by contractors who are EN ISO/IEC 17025 accredited for the required method(s), subject to compliance with Emissions Monitoring Guidance Note, AG2.

Acknowledgment: The information which appears in the appendices is taken largely from Method Implementation Documents (MIDS) which are produced by the Environment Agency (England). Emission testing reports should list the standard reference method and the implementation notes contained herein as the basis of their testing methodology.

Certified analysers: References in the index to “certified analyser” means any analyser that has been certified in accordance with EN 15267-3 (certification must be valid at the time the equipment is newly purchased) or EN 15267-4 as appropriate. It is noted that EN 15267-3 covers the certification of Automated Measuring Systems (AMS, also known as CEMs) and EN 15267-4 covers certification of Portable Automated Measuring Systems (P-AMS) for Periodic Measurements.

Lists of certified analysers can be found at the following websites:

<https://www.csagroupuk.org/services/mcerts/mcerts-product-certification/mcerts-certified-products/>

<https://www.qal1.de/en/index.htm>

European Standards are numbered with the prefix “EN”. These standards are adopted by the National Standards Authority of Ireland and are published with the prefix “IS EN”. Standards having the same number are deemed equivalent, regardless of the publishing authority, (for example, EN 13284 is interchangeable with IS EN 13284, published in Ireland and BS EN 13284, published in the UK, etc).

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1. ALDEHYDES [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|---|
| CEN/TS 13649 (Non- Isokinetic sampling) | DNPH coated silica gel tube / Analysis by solvent desorption followed by GC-MS or GC-FID | ± 25% | <p>CEN/TS 13649 is not specific to Aldehydes but is a general method for the determination of the mass concentration of individual gaseous organic compounds by adsorption onto charcoal, DNPH coated silica gel is a better trapping agent and must therefore be used instead of charcoal. Ensure trapping efficiency is assessed (analyse front and back of tube) and meets the requirement in the Standard. The stack gas must have a water vapour content of no more than 2% v/v when it passes through the tube to maintain the trapping ability. Dynamic sample dilution will be required for hot / wet stacks with a water vapour content of > 2% v/v.</p> <p>Storage and transportation: Keep below 5°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded. (as per NIOSH 2016)</p> <p>Analysis timescales: Analyse within 34 days of the sample being taken. (as per NIOSH 2016)</p> |
| Based on US EPA M0011 (available here) (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | DNPH solution / Analysis by HPLC | ± 25% | <p>The DNPH solution is made using 4g DNPH crystals, 20ml Orthophosphoric Acid & made up to 1l with Acetonitrile. This is an adaptation of US EPA M316 which is specific to formaldehyde which requires the use of Reagent Grade Water as the sampling medium as opposed to DNPH solution.</p> <p>Storage and transportation: Standard requires them to be kept cool (Section 7.2.4), this will be taken as keeping below 8°C from sample birth to analysis.</p> <p>Analysis timescales: Analyse within 14 days from sample being taken. (as per US EPA M316, not stipulated in US EPA M0011)</p> |
| TGN M22 (available here) (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 25% | Analysis must be performed using instrument specific or transportable references. |

2. AMINES & AMIDES [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|--|
| CEN/TS 13649 (Non- Isokinetic sampling) | Silica gel tube / Analysis by solvent desorption followed by GC-MS or GC-FID | ± 25% | <p>CEN/TS 13649 is not specific to Amines & Amides but is a general method for the determination of the mass concentration of individual gaseous organic compounds by adsorption onto charcoal. Silica gel is a better trapping agent and must therefore be used instead of charcoal. Ensure trapping efficiency is assessed (analyse front and back of tube) and meets the requirement in the Standard. The stack gas must have a water vapour content of no more than 2% v/v when it passes through the tube to maintain the trapping ability. Dynamic sample dilution will be required for hot / wet stacks with a water vapour content of > 2% v/v.</p> <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| TGN M22 (available here) (Instrumental sampling) | FTIR analyser | ± 25% | Analysis must be performed using instrument specific or transportable references. |

3. AMMONIA ([GOTO CONTENTS](#))

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|---|---------------------------|---|
| <p>Procedural requirements of EN 14791 (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling)</p> <p>EN ISO 21877 to be used from 30th June 2021</p> | <p>0.1N H₂SO₄ / Analysis by IC</p> <p>0.05M H₂SO₄ Analysis by IC, spectrophotometry or flow analysis and spectrometric detection.</p> | <p>± 30%</p> <p>± 20%</p> | <p>Method designed for sulphur oxides measurement but can be used for ammonia. Measures total NH₃ and NH₄⁺, therefore beware of interferences from NH₄⁺ salts. Stainless steel is not a suitable probe or filter housing material. Compliant materials include titanium, borosilicate glass, quartz glass and PTFE.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> <p>Measures total NH₃ and NH₄⁺, therefore beware of interferences from NH₄⁺ salts. Stainless steel is not a suitable probe or filter housing material. Compliant materials include titanium, borosilicate glass, quartz glass and PTFE.</p> <p>Storage and transportation: Samples should be shipped in an insulated container and accompanied by ice packs. Storage conditions must be maintained as close as possible to the <5°C requirement of the standard. Analyse as soon as possible.</p> |
| <p><u>Ammonia testing at Poultry farms only</u>, may use:</p> <p>EU Commission Implementing Decision CID 2017/302</p> | <p>Ammonia (or dust) samples are taken on six days, as a minimum, distributed over one year</p> | | <p>Calculation by measuring the ammonia (or dust) concentration and the ventilation rate using ISO, national or international standard methods or other methods ensuring data of an equivalent scientific quality.</p> <p>For further information refer to:</p> <ul style="list-style-type: none"> COMMISSION IMPLEMENTING DECISION (EU) 2017/302 of 15 February 2017 establishing best available techniques (BAT) conclusions, under Directive 2010/75/EU of the European Parliament and of the Council, for the intensive rearing of poultry or pigs here JRC Science for Policy Report Best Available Techniques (BAT) Reference Document for the Intensive Rearing of Poultry or Pigs, 2017 here |
| <p>TGN M22 (available here) (Instrumental sampling)</p> <p>CEN/TS 17337 from 30th June 2021.</p> | <p>FTIR analyser</p> | <p>± 25%</p> | <p>Analysis must be performed using instrument specific or transportable references.</p> <p>If used as an alternative method for measurements in place of the applicable SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate.</p> |

4. BIOAEROSOLS [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|-------------------------|---|
| VDI 4257 (Blatt 2) (Isokinetic sampling) pr EN 17359 (new EN standard in preparation at date of publishing). | Saline / Analysis by serial dilution and cultivation onto agar plates followed by colony counting Liquid impingement | See Further Information | <p>The Standard stipulates the use of a single impinger. For wet processes an extra impinger may be added. It is permitted for the analysis laboratory to filter the rinse solution to obtain a lower limit of detection. The standard defines a number of different bank pass criteria. The field blank is acceptable if the blank agar plate value does not exceed 3 CFU/plate count.</p> <p>Storage and transportation: Maintain a temperature of 5°C ±3 °C immediately after sampling until analysis. Monitor temperature to ensure this temperature range not exceeded.</p> <p>Analysis timescales: Analyse immediately or at least within 24 hours of the sample being taken.</p> <p>The following uncertainties may be applied:</p> <ul style="list-style-type: none"> - 30% for total bacteria cultured onto half strength nutrient agar - 30% for gram negative bacteria cultured onto MacConkey agar - 23% for Aspergillus fumigates cultured onto malt extract agar |

5. CARBON DIOXIDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--------------------------------------|----------------------|--|
| ISO 12039 (Instrumental Sampling) | NDIR analyser | ± 25% | <p>Analyser must be calibrated using calibration gases traceable to Primary Standards at a concentration of approximately 70 – 90% of the range to be used.</p> |
| CEN/TS 17405 to be used from 1 st Jan. 2022 | IR Analyser | | <p>The method is based on an extractive gas and sample conditioning system in combination with an Infra Red analyser.</p> <p>The standard defines performance characteristics for the whole method including the analyser and the gas conditioning system. Conformance with these characteristics must be determined in a general performance test by an independent laboratory in accordance with EN 15267-4: 2017.</p> |
| TGN M22 (available here) (Instrumental sampling) | FTIR analyser | ± 25% | <p>Analysis must be performed using instrument specific or transportable references.</p> |
| CEN/TS 17337 to be used from 30 th June 2021. | | | <p>If used as an alternative method for measurements in place of the applicable SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate.</p> |

6. CARBON MONOXIDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--------------------------------------|----------------------|--|
| IS EN 14181 Work | Any certified analyser | - | A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. |
| EN 15058 Compliance Work (Instrumental Sampling) | NDIR analyser | ± 6% | <p>This is the Standard Reference Method for determination of CO. The calculated uncertainty must be < ± 6% of the Daily ELV.</p> <p>Analyser must be calibrated using calibration gases traceable to Primary Standards at a concentration at either the ELV or 50 – 90% of the range to be used. The range selected must encompass at least 2 x the ELV, but may be set higher if expected emissions are likely to be higher than this.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser [see] at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Any NDIR analyser may be used so long all of the performance checks in the Standard Reference Method have been performed. |
| AM for EN 15058 Compliance Work (Instrumental Sampling) | Not NDIR or FTIR | ± 6% | <p>This is an Alternative Method for determination of CO. The calculated uncertainty must be < ± 6% of the Daily ELV.</p> <p>Analyser must be calibrated using calibration gases traceable to Primary Standards at a concentration at either the ELV or 50 – 90% of the range to be used. The range selected must encompass at least 2 x the ELV, but may be set higher if expected emissions are likely to be higher than this.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser] at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. |
| TGN M22 (available here) Compliance Work (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 6% | <p>This is an Alternative Method for determination of CO. The calculated uncertainty must be < ± 6% of the Daily ELV. Analysis must be performed using instrument specific or transportable references.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser [at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. <p>If used as an alternative method for measurements in place of the applicable SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate.</p> |

7. DIOXINS & FURANS [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|--|
| EN 1948 Parts 1, 2 & 3 (Isokinetic sampling) | Filter, Rinse & XAD / Analysis by GC-HRMS | ± 30% | Refer to Appendix 1 for additional information/requirements. Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded. |

8. FORMALDEHYDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|--|
| US EPA M316 (available here) (Isokinetic sampling where water droplets/particulates are present) | Reagent Grade water / Analysis by spectrophotometry | ± 25% | Storage and transportation: Samples should be shipped in an insulated container and accompanied by ice packs. Storage conditions must be maintained as close as possible to the <8°C requirement of the standard. Analysis timescales: Analyse within 14 days of sample being taken. |
| NCASI Method ISS-FP-A105.01 (available here) (Isokinetic sampling where water droplets/particulates are present) | BHA Solution / Analysis by GC/NPD | ± 25% | This method requires the use of at least 3 impingers (midget / Greenburg Smith or other), each containing chilled BHA Solution (o-benzylhydroxylamine). In addition to the Formaldehyde - Methanol, Phenol, Acetaldehyde, Acrolein & Propionaldehyde are also absorbed. Methanol and Ketones can be analysed using GC-FID. Formaldehyde, Acetaldehyde, Acrolein & Propionaldehyde can be analysed using GC-FID. The NCASI Method has not gone through the US EPA Method 301 validation criteria, unlike the previous version of this Method. It is considered to be a self-validating method. Storage and transportation: Samples should be shipped in an insulated container and accompanied by ice packs. Storage conditions must be maintained as close as possible to the <8°C requirement of the standard. Analysis timescales: Analyse within 14 days of sample being taken. |
| TGN M22 (available here) (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 25% | Analysis must be performed using instrument specific or transportable references. |

9. GAS VELOCITY & VOLUMETRIC FLOW RATE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|---|----------------------|---|
| EN 16911-1 and PD CEN/TR 17078 (Manual Method - preferred) | Pitot tube with pressure measurement device or anemometer, and temperature measurement device | ± 10% | EN 16911-1 is a Standard specific to the measurement, and associated calculations, of velocity and volumetric flow rate in ducts. PD CEN/TR 17078:2017 was published as guidance on the application of EN ISO 16911-1. Refer to Appendix 2 for additional information / requirements. |
| EN 16911-1 (Alternative methods as per Annexes B to E) | Complex sampling, analysis equipment and calculations required | | Vane anemometer – Annex B / Tracer gas dilution method – Annex C / Transit time tracer gas method – Annex D / Calculation from energy consumption – Annex E, (Refer to Agency guidance note AG10 for further information). |

10. HALOGENS & HALIDES [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|---|----------------------|--|
| US EPA Method 26 / 26A (available here) (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | Halides only: 0.05M H ₂ SO ₄ Halides & Halogens: 0.05M H ₂ SO ₄ & 0.1N NaOH / Analysis by IC | ± 25% | <p>Not to be used for HCl, HF & HCN as these have specific CEN / ISO / US EPA Methods applicable to them.</p> <p>Suitable for other hydrogen halides, HX (specifically HBr) and halogens, X₂ (specifically Cl₂, Br₂).</p> <p>Method 26 is for gas-phase halides only whereas Method 26A is for both aerosol and gas-phase halides. If used to measure halogens, the 0.05M H₂SO₄ impinger must be inserted before the NaOH impingers to knock out the halides that may be present.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| TGN M22 (available here) (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 25% | Analysis must be performed using instrument specific or transportable references. Suitable for HBr only as FTIR is not able to detect diatomic molecules which is how the halogens exist (Cl ₂ , Br ₂). |

11. HEXAVALENT CHROMIUM (AKA Cr^{VI}) [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|---|----------------------|---|
| US EPA Method 0061 (available here) (Isokinetic sampling). Alternative methods may be used following agreement with the Agency. | 0.1 or 0.5M KOH solution / Analysis by IC/PCR or any other validated technique (e.g. Colorimetry) | ± 25% | Re-circulating probe method which requires the impinger solution to be pumped up to the nozzle and down the probe to prevent Cr ⁺⁶ from turning into Cr ⁺³ in the sampling train. pH test required at the end of the test to ensure the pH in the impinger solution has not exceeded 8.5. Post-test nitrogen purge of the impingers required for 30 minutes at 10l/min. Final post-test requirement is to filter the samples to remove any particulate using a 0.45-micron acetate paper filter. Storage and transportation: Keep below 5°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded. Analysis timescales: Analyse within 14 days of the sample being taken (as per US EPA Method 0061). |

12. HOMOGENEITY TESTING [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|----------------------------------|--|----------------------|--|
| EN 15259 (Instrumental sampling) | Any suitable analyser taking a grid measurement approach | N/A | Used to determine if a stack gas pollutant concentration is sufficiently homogeneous at the sample plane location to enable it to be sampled from a single point. It usually requires the use of two instrumental analysers, which record gas concentration changes across the sample plane and over the time of the homogeneity test. Statistical analysis is used to show if the gas can be considered homogeneous or not. If the gas is not homogenous an alternative location or a grid measurement approach should be used. The Standard can also be used to determine the location of CEMs. The Standard does not stipulate where a homogeneity test is required but it is recommended for combustion processes and stacks with an area ≥1.0m ² (equivalent to ≥1.13m diameter for circular ducts). Refer to Appendix 3 for additional information / requirements. |

13. HYDROGEN BROMIDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|---|
| US EPA Method 26 (available here) | Sulphuric acid and sodium hydroxide. Analysis by ion chromatography (IC) | - | Sources, such as those controlled by wet scrubbers, that emit acid particulate matter must be sampled isokinetically using Method 26A . |

14. HYDROGEN CHLORIDE ([GOTO CONTENTS](#))

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--------------------------------------|----------------------|--|
| EN 14181 Work | Any certified analyser or EN 1911 | - | A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants. |
| EN 1911 (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | Reagent grade water / Analysis by IC | ± 30% | <p>This is the Standard Reference Method for the measurement of Gaseous Inorganic Chlorides (expressed as HCl). It does not measure HCl, but measures total Cl⁻ reaching the impingers. Subject to interference from other chloride ions (e.g. ammonium chloride) as impossible to differentiate between them. Result valid so long as absorption efficiency requirements in the standard are met.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature. Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| TGN M22 (available here) Compliance Work (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 30% | <p>This is an Alternative Method for determination of HCl. It measures gas-phase HCl directly as opposed to EN 1911 which measures Total Gaseous Inorganic Chlorides (expressed as HCl). Analysis must be performed using instrument specific or transportable references. In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser [at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. <p>If used as an alternative method for measurements in place of the applicable SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate.</p> |

15. HYDROGEN CYANIDE ([GOTO CONTENTS](#))

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--------------------------------------|----------------------|---|
| US EPA OTM29 (available here) (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | ≥ 1.0N NaOH / Analysis by IC | ± 25% | <p>Does not measure HCN, but measures total CN⁻ reaching the impingers. i.e. Total Gaseous Inorganic Cyanide (expressed as HCN). Requires pH of impingers to be checked at no more than 15-minute intervals during the sampling exercise. The pH must not drop below 12 in the final impinger. For highly acidic stack gases, a stronger NaOH solution may be used, alternatively a greater volume of impinger liquid may be used. The Method specifies the use of 6.0N NaOH impinger solution, however it is a dangerous substance to handle and its viscosity can lead to sampling issues, so a weaker concentration may be used so long as the pH of the final impinger remains above 12. As with the CEN methods, it is useful to perform an absorption efficiency test on the impingers to ensure that <5% of the recovered HCN appears in the final impinger.</p> <p>Acceptable changes to the Method which are not required in European or ISO Standards are:</p> <ol style="list-style-type: none"> 1. It is not necessary to carry out a field blank spike. 2. It is not necessary to measure the Carbon Dioxide concentration in the stack for non-combustion sources. 3. A titanium probe and filter holder may be used instead of glass. |

| | | | |
|--|---------------|-------|--|
| | | | <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| <p>TGN M22 (available here) (Instrumental sampling)</p> <p>CEN/TS 17337 to be used from 30th June 2021.</p> | FTIR analyser | ± 25% | It measures gas-phase HCN directly as opposed to US EPA OTM29 which measures Total Gaseous Inorganic Cyanide (expressed as HCN). Analysis must be performed using instrument specific or transportable references. |

16. HYDROGEN FLUORIDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|---|---|
| <p>ISO 15713 (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling)</p> | 0.1N NaOH / Analysis by IC or ISE | ± 25% | <p>Does not measure HF, but measures total F⁻ reaching the impingers. i.e. Total Gaseous Inorganic Fluoride (expressed as HF). This method does not measure fluorocarbons. Where IC analysis is utilised, a caustic eluent must be used on a suitable OH⁻ tolerant IC column. ISO 15713 requires the use of Monel / PTFE / PE / PP / Quartz Glass for sampling train materials. Titanium and Borosilicate glass are not permitted. Refer to Appendix 4 for additional information / requirements.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| PD CEN/TS 17340 | <p>Filter</p> <p>Absorbing soln. (alkali, or DIW - depending on analysis).</p> | <30% at the daily ELV or 0.6 mg/m ³ for daily ELV <2 mg/m ³ | <p>Scope of method covers both gaseous and bound to particulate fluorides (expressed as HF). Sample gas is extracted representatively, particulate bound fluorides are collected on a filter and gaseous fluorides are collected in an absorbing solution that is chosen to match the method of analysis (e.g. DI Water, 0.1N NaOH, or Na₂CO₃/NaHCO₃). Analysis is permitted by one of 3 methods (IC, Spectrophotometry or ISE).</p> <p>Method range is from 0.1 – 10 mg HF/m³ at standard conditions. Performance criteria are specified for sampling and analysis when used as an SRM.</p> <p>There are detailed annexes that describe recovery of the filter fraction and the three analytical techniques.</p> |
| <p>TGN M22 (available here) (Instrumental sampling)</p> <p>CEN/TS 17337 to be used from 30th June 2021.</p> | FTIR analyser | ± 25% | It measures gas-phase HF directly as opposed to ISO 15713 which measures Total Gaseous Inorganic Fluoride (expressed as HF). Analysis must be performed using instrument specific or transportable references. |

17. HYDROGEN SULPHIDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|---|----------------------|---|
| US EPA M11 (available here) (Non-Isokinetic sampling) | Cadmium sulphate solution or zinc acetate solution / Analysis by iodometric titration or DIST-VAS | ± 25% | <p>No filter is required for this test. The US EPA Method states the use of cadmium sulphate solution, it is a highly harmful substance and is often replaced with zinc acetate in the UK. Midget impingers are specified in the method, however larger impingers may be used so long as an absorption efficiency tests proves that less than 5% is present in the final impinger.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 4 weeks of the sample being taken.</p> |
| CEN/TS 13649 (Non- Isokinetic sampling) | Charcoal tube / Analysis by solvent desorption followed by GC-MS or GC-FID | ± 25% | <p>CEN/TS 13649 is not specific to hydrogen sulphide but is a general method for the determination of the mass concentration of individual gaseous organic compounds by adsorption onto charcoal. Ensure trapping efficiency is assessed (analyse front and back of tube) and meets the requirement in the Standard.</p> <p>The stack gas must have a water vapour content of no more than 2% v/v when it passes through the tube to maintain the trapping ability. Dynamic sample dilution will be required for hot / wet stacks with a water vapour content of > 2% v/v. Due to the nature of interferences on the tube, this method is suitable for non-combustion processes where no sulphur dioxide is present.</p> <p>CEN/TS 13649 will most likely give a better Limit of Detection (LOD) than the US EPA M11 test, however it is recommended to check with the analysis lab to confirm this.</p> <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 4 weeks of the sample being taken.</p> |

18. ISOCYANATES [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|---|----------------------|--|
| US EPA CTM 36 (available here) (Isokinetic sampling) | 1-(2-pyridyl) piperazine coated filter / Analysis by HPLC | ± 25% | <p>The Standard refers specifically to TDI, MDI, HDI and IPDI, but may also be used for other isocyanates. The US EPA Method stipulates the use of glass; however, a titanium probe and filter holder may be used as an alternative.</p> <p>The filter will normally be placed inside the stack during sampling. Where the stack temperature exceeds 105°C, out-stack filtration will be employed with the out-stack oven set to 105°C. This will also be the case if water droplets are present in the stack which prevent the use of in stack sampling.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 90 days of the sample being taken.</p> |

19. MERCAPTANS and ORGANO-SULPHUR COMPOUNDS [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|---|
| CEN/TS 13649 (Non- Isokinetic sampling) | Atomic thermal desorption (ATD) tube OR other sorbent tube prescribed by lab. Analysis method to be agreed with accredited laboratory | ± 25% | <p>CEN/TS 13649 is a general method for the determination of the mass concentration of individual gaseous organic compounds by adsorption onto a suitable sampling tube, (refer to section on speciated VOC's for further details).</p> <p>Mercaptans and other organo-sulphur compounds are sources of odour nuisance at low concentrations. The choice of sampling medium and analytical technique should be chosen in consultation with the laboratory, (seek ISO 17025 accredited analysis where possible). Techniques include:</p> <ul style="list-style-type: none"> • <u>Mercaptans</u> - Collection onto a mercuric acetate treated filter, analysis by GC-FPD or GC-MS, (NIOSH 2542 is an analytical method that is commonly employed). • <u>Organo-Sulphur Compounds</u> - Collection on molecular sieve or a dual bed sorbent thermal desorption tube, the tubes are treated with an inert coating, (e.g. sulfinert or sulficarb). Analysis by GC with suitable detector for low level sulphur compounds. These compounds are highly reactive and may damage normal GCMS systems, they will typically be analysed at a laboratory that is equipped with a dedicated analytical system. <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analysis is typically recommended within 3 weeks of the sample being taken.</p> |

20. MERCURY [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|-----------------------------------|---|----------------------|---|
| EN 13211 (Isokinetic sampling) | Potassium dichromate or potassium permanganate solution / Analysis by CV-AAS, ICP-MS or CV-AFS | ± 15% | <p>Refer to Appendix 5 for additional information/requirements. . The appendix allows mercury to be measured off the back of a standard EN 14385 sampling train, so long as the filter and all of the impingers are analysed for particulate and gaseous phase mercury, respectively.</p> <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 2 weeks of the sample being taken.</p> |

21.METALS [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|-----------------------------------|---|----------------------|---|
| EN 14385 (Isokinetic sampling) | Nitric peroxide solution / Analysis by ICP-MS, ICP-OES or ICP-AAS | ± 15% | EN 14385 specifically relates to the determination of total emissions of As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, Tl and V. Refer to Appendix 5 for additional information/requirements. The appendix allows EN 14385 to be extended to cover other metals, subject to the analysis lab confirming their abilities to perform the analysis on these other metals. Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded. Analysis timescales: Analyse within 2 weeks of the sample being taken. |

22.NITRIC ACID VAPOUR [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|--|
| US EPA Method 7D (available here) (Non-Isokinetic sampling) | Potassium permanganate solution / Analysis by IC | ± 25% | Measures NO, NO ₂ & nitric acid vapour. Used on surface treatment of metals processes. Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature. Analysis timescales: Analyse within 4 weeks of the sample being taken. |
| TGN M22 (available here) (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 25% | It measures gas-phase HNO ₃ directly as opposed to US EPA Method 7D which measures NO, NO ₂ and nitric acid vapour. Analysis must be performed using instrument specific or transportable references. |

23.NITROGEN OXIDES (NO & NO₂) [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|---|
| EN 14181 Work | Any certified analyser | - | A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants . |
| EN 14792 Compliance Work (Instrumental sampling) | Chemiluminescence analyser | ± 10% | <p>This is the Standard Reference Method for determination of NO_x. Measures NO (and NO₂ via a NO_x converter). The calculated uncertainty must be < ± 10% of the Daily ELV. Where a NO_x converter is utilised, the converter efficiency must be >95%. Water vapour must be removed before the analyser.</p> <p>Analyser must be calibrated using calibration gases traceable to Primary Standards at a concentration at either the ELV or 50 – 90% of the range to be used. The range selected must encompass at least 2 x the ELV, but may be set higher if expected emissions are likely to be higher than this.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Any Chemiluminescence analyser may be used so long all of the performance checks in the Standard Reference Method have been performed. |
| AM for EN 14792 Compliance Work (Instrumental Sampling) | Not Chemiluminescence or FTIR e.g. NDIR, NDUV | ± 10% | <p>This is an Alternative Method for determination of NO and NO₂. The calculated uncertainty must be < ± 10% of the Daily ELV.</p> <p>Analyser must be calibrated using calibration gases traceable to Primary Standards at a concentration at either the ELV or 50 – 90% of the range to be used. The range selected must encompass at least 2 x the ELV, but may be set higher if expected emissions are likely to be higher than this.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. |
| TGN M22 (available here) Compliance Work (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 10% | <p>This is an Alternative Method for determination of NO and NO₂. The calculated uncertainty must be < ± 10% of the Daily ELV. Analysis must be performed using instrument specific or transportable references.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. <p>If used as an alternative method for measurements in place of the applicable SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate.</p> |

24. NITROUS OXIDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--------------------------------------|----------------------|--|
| EN ISO 21258 (Instrumental sampling) | NDIR analyser | ± 25% | Interferences from CO and CO ₂ . (measure CO ₂ to compensate). Water vapour must be removed before the analyser. |
| TGN M22 (available here) (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | ± 25% | This is an Alternative Method for determination of N ₂ O. Analysis must be performed using instrument specific or transportable references. Use a certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. |

25. ODOUR [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|-------------------------------|---|--|--|
| EN 13725 (Manual sampling) | Odour bag / Analysis by olfactometry and odour panel | Dependant on number of sample runs performed | <p>The unit of measurement is European odour unit per cubic metre (ou_E/m³). Important to prevent the formation of condensation in the bag. Where the stack is hot and wet, static dilution may be performed up to a ratio of 3:1 (3 parts dilution gas to 1 part stack gas). Where greater dilution is required, dynamic dilution must be employed. Dilution ratios can be measured using a direct reading analyser (CO₂ or O₂ and comparing the stack gas concentrations to the concentrations as measured in the odour bag.</p> <p>Where odour testing is performed as part of an odour dispersion modelling exercise, accurate stack gas velocity and volumetric flow rate will be required in order to calculate the odour emission rates. This should be done following the Gas Velocity & Volumetric Flow Rate section in this document.</p> <p>Refer to Appendix 6 for additional information / requirements.</p> <p>Storage and transportation: Keep at ambient temperature (ensure this is above the dew point of the gas in the bag) and in the dark. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 24 – 30 hours of the sample being taken.</p> |

26.OIL MIST, TAR & BITUMEN FUME [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|---|
| EN 13284-1 for sampling (Isokinetic sampling) | Cyclohexane washed GF filter / Analysis by cyclohexane extraction followed by gravimetric analysis | ± 25% | <p>In-stack filtration is employed to reduce the chance of sticky particulates fouling the sampling equipment and thus leading to low recoveries. If out-stack is employed, the train must be heated to the stack temperature. The nozzle, gooseneck (and heated probe if used) must be rinsed with acetone and then analysed using a GC-MS screen to pick out oil mist / tar / bitumen fume components or gravimetrically after filtering out any particulate matter.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 4 weeks of the sample being taken.</p> |

27.OXYGEN [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|--|
| EN 14181 Work | Any certified analyser | - | The analyser must be certified at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants. |
| EN 14789 Compliance Work (Instrumental sampling) | Paramagnetic analyser | ± 6% | <p>This is the Standard Reference Method for determination of O₂. The calculated uncertainty must be < ± 6% of the measured concentration.</p> <p>Where stack Oxygen is > 15% v/v, analyser must be calibrated using dried air (21%) or calibration gas at ~21% traceable to Primary Standards. Where stack Oxygen is < 15% v/v, the analyser must be checked at the expected stack gas oxygen concentration using calibration gases traceable to Primary Standards.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Any Paramagnetic analyser may be used so long all of the performance checks in the Standard Reference Method have been performed. |
| AM for EN 14789 Compliance Work (Instrumental sampling) | Not Paramagnetic e.g. Zirconia Cell (wet and dry) | ± 6% | <p>This is an Alternative Method for determination of O₂. The calculated uncertainty must be < ± 6% of the measured concentration.</p> <p>Where stack Oxygen is > 15% v/v, analyser must be calibrated using dried air (21%) or calibration gas at ~21% traceable to Primary Standards. Where stack Oxygen is < 15% v/v, the analyser must be checked at the expected stack gas oxygen concentration using calibration gases traceable to Primary Standards.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. |

28.PAHs [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|---|
| ISO 11338 Parts 1 & 2 (Isokinetic sampling) | Filter, Rinse & XAD / Analysis by GC-HRMS | ± 25% | <p>The regulatory authority will specify the set of PAH compounds that should be measured. The most common is known as the WID / IED list of PAHs, which comprises of 16 compounds: Anthanthrene; Benzo[a]anthracene; Benzo[b]fluoranthene; Benzo[k]fluoranthene; Benzo(b)naph(2,1-d)thiophene; Benzo(c)phenanthrene; Benzo[ghi]perylene; Benzo[a]pyrene; Cholanthrene; Chrysene; Cyclopenta(c,d)pyrene; Dibenzo[ah]anthracene; Dibenzo[a,i]pyrene; Fluoranthene; Indo[1,2,3-cd]pyrene; Napthalene. It is up to the monitoring organisation to ensure the laboratory analyses for the correct list of PAHs.</p> <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 21 days of the sample being taken.</p> |

29.PARTICULATE MATTER [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|---|
| EN 13284-1 (Isokinetic sampling) This method should be considered as the first option for high or low concentration particulate matter monitoring. | Filter & rinse / Gravimetric analysis | ± 20% | <p>Applicable to both low and high range concentrations. Sampling times from 30 to 120 minutes depending on expected particulate loading. In or out-stack sampling may be utilised, however in-stack should always be selected if possible (unless, for example, the stack temperature is too high to permit this configuration (i.e. seals would be melted) or water droplets are present). In this instance the probe and filter oven must be heated (e.g. a conventional temperature of 160°C is generally convenient, see EN 13284-1).</p> <p>A 5-figure balance (resolution 0.01mg) must be used for analysis. Laboratories wishing to weigh particulate samples must hold EN ISO/IEC 17025 accreditation for the weighing and regularly take part, successfully, in proficiency testing schemes to show its ongoing ability to obtain accurate results.</p> <p>Refer to Appendix 7 for additional information/requirements.</p> |
| ISO 9096 (Isokinetic sampling) | Filter & rinse / Gravimetric analysis | | <p>Applicable to concentrations from 20 mg/m³ to 1 000 mg/m³ under standard conditions. Minimum sampling period of 30 minutes. In or out-stack sampling may be utilised, however in-stack should always be selected if possible (unless, for example, the stack temperature is too high to permit this configuration (i.e. seals would be melted) or water droplets are present). In this instance the probe and filter oven must be heated (e.g. a conventional temperature of 160°C is generally convenient, see ISO 9096).</p> <p>A 5 figure balance (resolution 0.01mg) must be used for analysis. Laboratories wishing to weigh particulate samples must hold ISO 17025 accreditation for the weighing and regularly take part, successfully, in proficiency testing schemes to show its ongoing ability to obtain accurate results.</p> |

30.PARTICULATE MATTER SIZE FRACTIONATION [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|--|
| EN ISO 23210 (Isokinetic sampling) | Impaction on a 2-stage cascade impactor / Gravimetric analysis | ± 25% | Applicable for emissions of Total Particulate Matter (at actual conditions, not referenced) with a concentration of 1 – 50 mg/m ³ . Allows measurement of PM10 and PM2.5. Not suitable to sum all parts to report total particulate matter result. Not suitable for stacks where water droplets are present. Single point sampling is acceptable at a “representative” sample point. Refer to Environment Agency TGN M15 for additional information, (the Standard takes precedence). |
| ISO 25597 (Isokinetic sampling) | 1 or 2-stage cyclone / Gravimetric analysis | ± 25% | Applicable for emissions of Total Particulate Matter (at actual conditions, not referenced) with a concentration of >50 mg/m ³ . Allows measurement of PM10 and PM2.5. Not suitable to sum all parts to report total particulate matter result. Not suitable for stacks where water droplets are present. Multiple point sampling as per the sample points in ISO 9096.Can be used to measure condensable particulates. |

31.PCBs (DIOXIN LIKE) [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|---|----------------------|---|
| EN 1948 Part 4 (Isokinetic sampling) | Filter, Rinse & XAD / Analysis by GC-HRMS | ± 30% | Permissible to sample for PCBs on the same trap as for Dioxins & Furans. All site sampling and sample recovery procedures are identical. Refer to Environment Agency Appendix 1 for additional information / requirements. Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded. |

32.PHENOLS & CRESOLS [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--|----------------------|---|
| CEN/TS 13649 (sampling) (Non- Isokinetic sampling, no water droplets present) OSHA 32 or NIOSH 2546 (analysis) | XAD7 tube / Analysis by solvent desorption followed by GC-MS or GC-FID | ± 25% | CEN/TS 13649 is not specific to phenols and cresols but is a general method for the determination of the mass concentration of individual gaseous organic compounds by adsorption onto charcoal, XAD7 is a better trapping agent and must therefore be used instead of charcoal. Ensure trapping efficiency is assessed (analyse front and back of tube) and meets the requirement in the Standard. The stack gas must have a water vapour content of no more than 2% v/v when it passes through the tube to maintain the trapping ability. Dynamic sample dilution will be required for hot / wet stacks with a water vapour content of > 2% v/v. |

| | | | |
|---|---|-------|--|
| | | | <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| Based on EN 1911 (sampling) (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | Reagent grade water / Analysis by Colorimetry, HPLC or GC | ± 25% | <p>As phenols and cresols exhibit high hydrophilic behaviour, it means they can be trapped in impinger solutions. Suitable for hot / very wet stacks where dynamic dilution for tubes may prove to be unsuitable due to the elevated water vapour content in the stack gas.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |

33.SULPHUR DIOXIDE [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|---|----------------------|---|
| EN 14181 Work | Any certified analyser or EN 14791 | - | A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants . |
| EN 14791 (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | 0.3 or 3% H ₂ O ₂ / Analysis by IC | ± 20% | <p>This is the Standard Reference Method for the measurement of SO₂. The Standard recommends the use of 0.3% H₂O₂ where concentrations are expected to be <1000 mg/m³ . For those expected to be >1000 mg/m³ it is recommended to use 3% H₂O₂. Result valid so long as absorption efficiency requirements in the standard are met. The calculated uncertainty must be < ± 20% of the Daily ELV.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| PD CEN/TS 17021 Determination of the mass concentration of SO ₂ by instrumental techniques | Not specific technique prescribed. Instrumental techniques available include infrared (IR) absorption, ultraviolet (UV) absorption, UV fluorescence and electrochemical cells | ± 15% | <p>This is an Alternative Method for determination of SO₂. The calculated uncertainty must be < ± 15% of the Daily ELV. If there are droplets present in the stack gas it should be discussed with the local competent authority if this method is appropriate.</p> <p>Analyser must be calibrated using calibration gases traceable to Primary Standards at a concentration at either the half hourly ELV or 50 – 90% of the range to be used. The measurement range shall be adapted to the measuring objective. Generally, this means that the measurement range is high enough to cover the peak emission and at least 150 % of the half hourly ELV.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. <p>If used as an alternative method for measurements in place of the SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate.</p> |

| | | | |
|---|---------------|-------|--|
| | | | <p><i>Important note: Some NDIR analysers exhibit a large interference to Methane, and therefore may not be suitable for use when measuring emissions from landfill gas engines and biowaste plants. In these cases, an alternative method (or the Standard Reference Method) must be used.</i></p> |
| <p>TGN M22 (available here) Compliance Work (Instrumental sampling)</p> <p>CEN/TS 17337 from 30th June 2021.</p> | FTIR analyser | ± 20% | <p>This is an Alternative Method for determination of SO₂. Analysis must be performed using instrument specific or transportable references.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. <p>If used as an alternative method for measurements in place of the applicable SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate.</p> |

34. SULPHURIC ACID MIST ([GOTO CONTENTS](#))

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--------------------------------------|----------------------|--|
| US EPA Method 8 (available here) (Isokinetic sampling) | Isopropanol / Analysis by IC | ± 25% | Measures H ₂ SO ₄ and SO ₃ . A combined result is obtained because it is not possible to analyse H ₂ SO ₄ and SO ₃ separately. |

35. TOTAL ACIDS ([GOTO CONTENTS](#))

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|---|----------------------|--|
| Based on EN 1911 (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | Reagent grade water / Analysis by potentiometric titration or IC | ± 25% | <p>Sampling is as per EN 1911. Analysis can be performed in one of 2 ways:</p> <ol style="list-style-type: none"> 1. Potentiometric titration This is the preferred method of analysis. The impinger solution is titrated with dilute sodium hydroxide to neutrality. The molarity of the impinger is calculated and then expressed as the equivalent mass of HCl. To maximise accuracy, the titrations must be carried out using an automated potentiometric titrator. 2. IC This is an alternative method of analysis. This involves the summation of the eluted ions as HCl equivalents based on molecular weight. <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |

36.VOCS (CONDENSABLE) [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|--|
| Based on IS EN 14791 (Isokinetic sampling where water droplets are present, otherwise Non-Isokinetic sampling) | Reagent grade water / Analysis by OX / IR | ± 25% | <p>This method is a wet chemistry method whereby the analysis looks for Total Organic Carbon. Using 4 impingers in series, an efficiency of >75% should be achievable for Condensable VOC concentrations of >10 mg/m³. Permitted sampling train materials are Glass / PTFE / Titanium & Viton. Where water droplets are present, out-stack filtration shall be employed, with the probe and oven set to 120°C or 20K above the stack (acid) dew point.</p> <p>Storage and transportation: Keep at ambient temperature. No requirement to monitor temperature.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |

37.VOCS (NON-METHANE) [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|---|----------------------|---|
| CEN/TS 13649 (Non- Isokinetic sampling) | Charcoal tube / Analysis by solvent desorption followed by GC-MS or GC-FID | ± 25% | <p>Ensure trapping efficiency is assessed (analyse front and back of tube) and meets the requirement in the Standard. The stack gas must have a water vapour content of no more than 2% v/v when it passes through the tube to maintain the trapping ability. Dynamic sample dilution will be required for hot / wet stacks with a water vapour content of > 2% v/v. Analysis is performed using a general semi-quantitative screen which is then summed to give a total for Non-Methane VOCs.</p> <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |

38.VOCS (SPECIATED) [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--|----------------------|---|
| CEN/TS 13649 (Non- Isokinetic sampling) | Charcoal tube / Analysis by solvent desorption followed by GC-MS or GC-FID | $\pm 25\%$ | <p>CEN/TS 13649 is a general method for the determination of the mass concentration of individual gaseous organic compounds by adsorption onto charcoal. Consult the analysis laboratory to ensure that the best type of absorption medium is being used as charcoal may not always be the best material available. Ensure trapping efficiency is assessed (analyse front and back of tube) and meets the requirement in the Standard. The stack gas must have a water vapour content of no more than 2% v/v when it passes through the tube to maintain the trapping ability. Dynamic sample dilution will be required for hot / wet stacks with a water vapour content of > 2% v/v, (gases entering the tube must be < 40°C).</p> <p>Analysis may be performed using a general semi-quantitative screen, or specific VOCs can be targeted using a fully qualitative analysis.</p> <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| CEN/TS 13649 (Non- Isokinetic sampling) | Atomic thermal desorption (ATD) tube / Analysis by solvent desorption followed by ATD-GC-MS | $\pm 25\%$ | <p>CEN/TS 13649 is a general method for the determination of the mass concentration of individual gaseous organic compounds by adsorption onto charcoal. For improved Limits of Detection (and therefore for expected low speciated VOC concentrations), the use of ATD tubes is recommended over other materials such as charcoal. The ATD tube can be packed with a number of different absorption media (e.g. molecular sieve, sulfonert, mixed bed for example Tenax/Carbopack) to trap different pollutants. Consult the analysis laboratory to help decide which is the best type of packing media for the particular application.</p> <p>ATD tubes are highly sensitive to water, therefore the tube must be kept dry. If the tube gets wet, the analysis will fail and cannot be repeated. The analysis lab will normally perform a nitrogen purge before performing analysis, however to be on the safe side, the stack gas must have a water vapour content of no more than 2% v/v when it passes through the tube. Dynamic sample dilution will be required for hot / wet stacks with a water vapour content of > 2% v/v.</p> <p>Analysis may be performed using a general semi-quantitative screen, or specific VOCs can be targeted using a fully qualitative analysis.</p> <p>Storage and transportation: Keep below 25°C from sample birth to analysis. Monitor temperature to ensure this temperature is not exceeded.</p> <p>Analysis timescales: Analyse within 6 weeks of the sample being taken.</p> |
| TGN M22 (available here) (Instrumental sampling) CEN/TS 17337 to be used from 30 th June 2021. | FTIR analyser | $\pm 25\%$ | <p>Analysis must be performed using instrument specific or transportable references. This method has the ability to measure many individual organic compounds simultaneously. It is critical the stack gas matrix is well characterised to enable the FTIR to identify the concentrations of the known pollutants in the stack. Where the stack gas matrix is unknown, it is recommended to perform a charcoal screening run to help identify the components to enable the FTIR software to resolve as many pollutants in the stack gas matrix as possible, Where unknowns are present, and are not resolved, it will more than likely mean that other known components will not be quantified due to the interference of these unknowns.</p> <p>FTIR is a hugely powerful tool, but only in the hands of experienced operatives and where the stack gas matrix has been well characterised, or can be characterised by CEN/TS 13649 testing.</p> |

39.VOCS (TOTAL) [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|---|--------------------------------------|----------------------|---|
| EN 14181 Work | Any certified analyser | - | The analyser must be certified at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants. |
| EN 12619 Compliance Work (Instrumental) | FID analyser | ± 15% | <p>This Standard Reference Method specifies the use of Flame Ionisation Detection (FID) for determination of Total Volatile Organic Carbon in the concentration range up to 1000 mg/m³ expressed as carbon.</p> <p>Analyser must be calibrated using calibration gases traceable to Primary Standards at a concentration at either the ELV or 50 – 90% of the range to be used. Zero gas must be carbon free air (from a cylinder or scrubbed) so long as the oxygen synergy effects are tested and are found to meet the requirements of the Standard. If not, a mixture of air and nitrogen blended to the expected concentration of Oxygen in the stack will need to be used. Instrument calibrated on propane in air. Fuel is a hydrogen or a hydrogen / helium mix.</p> |

40.WATER VAPOUR [\(GOTO CONTENTS\)](#)

| Allowable Standard/s | Sampling Medium / Analysis Technique | Standard Uncertainty | Further Information |
|--|--------------------------------------|----------------------|---|
| EN 14181 Work | Any certified analyser or EN 14790 | - | The analyser must be certified at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants. |
| EN 14790 Compliance Work (Non-Isokinetic sampling) | Water / Gravimetric analysis | ± 20% | This method is applicable to the measurement of water vapour (i.e. not including water droplets). For saturated stack gases, default to the theoretical water vapour tables in Annex A of EN 14790, trying to apply the result from an IS EN 14790 test to a saturated stack gas will most likely end up with an overestimate of the water vapour concentration (as water droplets may have been pulled into the sampling train and then weighed). Analysis must be made gravimetrically (not volumetrically) using a balance with a resolution of at least 0.01g. Minimum sampling time is 30 minutes and a minimum sample gas volume is 50l, higher sample rates are recommended to ensure a good Limit of Detection. |
| TGN M22 (available here) Compliance Work (Instrumental sampling) CEN/TS 17337 from 30 th June 2021. | FTIR analyser | ± 10% | <p>This is an Alternative Method for determination of H₂O. Analysis must be performed using instrument specific or transportable references.</p> <p>In order to use this, either use:</p> <ol style="list-style-type: none"> 1. A certified analyser at a range of a maximum of 1.5 x ELV for Incinerators and a maximum of 2.5 x ELV for Large Combustion Plants and any other process. 2. Performance checks in the Standard Reference Method must have been performed. Additionally, demonstration of equivalence to CEN/TS 14793 – “Intralaboratory procedure for an alternative method compared to a reference method” to the satisfaction of INAB / the regulator will be required. This will include process and range specific field test comparisons with a reference method. <p>If used as an alternative method for measurements in place of the applicable SRM, the P-AMS shall meet the performance criteria of EN 15267 part 3 or 4 as appropriate .</p> |

Note on Appendices

The Environment Agency (England) has produced a series of Method Implementation Documents (MIDs) which have been generated to provide clarification on a number of standards.

The information contained in the following appendices is taken largely from the corresponding Method Implementation Documents.

The Agency recognises that the European and International standards listed in these appendices benefit from the supplemental information contained therein. The appendices provide details on how the preferred standards shall be used for regulatory monitoring and to ensure they are being implemented consistently.

Any comments or suggested improvements to these appendices should be e-mailed to airthematicunit@epa.ie.

Appendix 1 [\(GOTO CONTENTS\)](#)

Supplementary Information to EN 1948-1 (Stationary source emissions – Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs)

This appendix supplements EN 1948 parts 1 - 3. However, it does not re-state all the provisions of EN 1948. The clause numbers in this document follow those of EN 1948, although the text from the standard is not repeated in this appendix.

Part 1: Sampling of PCDDs/PCDFs

1 Scope

This appendix is applicable to the filter/condenser method, using solid absorbent, without flow division. It specifies that the requirements of this method (See Annex B of the standard) shall be met.

A test carried out to measure PCDDs/PCDFs cannot also be used to measure PAHs.

2 Normative references

EN 13284-1:2001 has been replaced by EN 13284-1:2017.

3 Terms and definitions

No additional information

4 Symbols and abbreviations

No additional information

5 Principle of the complete PCDD/PCDF measurement procedure

No additional information

6 Sampling device and materials

6.1 General sampling device and sub sections

No additional information

6.2 Materials

6.2.1 General

No additional information.

6.2.2 Sampling system

Commercial titanium is made to different specifications based on its purity. An appropriate grade of titanium shall be used. Titanium will degrade in gas streams with a fluoride content of greater than 20ppm. Titanium may degrade at temperatures greater than 350°C (refer to supplier's information).

The sample system shall be designed to allow a measurement that is representative of the gas temperature within the condenser or resin trap to be made.

Note: different approaches can be used to enable the gas temperature within the condenser or resin trap to be measured. The following approaches are commonly used:

- a) a thermocouple is fitted directly in the gas stream. This will give a very accurate reading of the gas temperature. However, there is potential for leaks around the fitting. Also, the thermocouples and seals will require cleaning to ensure they do not cause contamination. The requirements on materials of construction in section 6.2 of EN 1948 apply because the thermocouple is inserted into the gas stream.
- b) the thermocouple is placed in an indentation in the sidewall of the condenser or resin trap (this may have to be added by a glass blower).
- c) a surface temperature measurement sensor is used to provide a very good contact between the thermocouple and the surface of the condenser or resin trap.

6.2.3 Sealings (in contact with flue gas)

PTFE must not be used in temperatures above 200°C (refer to supplier's information).

6.2.4 Filter

Quartz filters are recommended.

6.2.5 Quartz wool (if used)

The use of quartz wool is not recommended.

6.2.6 Solid adsorbent

An XAD-2 resin filled container (resin trap) is used for the method described by this appendix.

6.2.7 Cooling fluid

Filter/condenser method uses an ice bath, with a re-circulating pump submerged in iced water for cooling the flue gas and resin trap.

6.2.8 Ice bath

No additional information

6.2.9 Silica gel

Indicating silica gel shall be used to ensure moisture does not break through to the control box and dry gas meter.

6.2.10 Reagents

No additional information

6.3 Cleaning the sampling device

The standard (EN 1948) gives 3 examples of cleaning procedures. Cleaning procedure (1) shall be used, unless the analytical laboratory provides evidence to their accreditation body that an alternative procedure is equivalent.

Note 1: consistently low field blank results help to demonstrate that an alternative cleaning procedure is effective.

When using cleaning procedure number (1), the sampling equipment shall be kept in a muffle oven for at least three hours. Due the length of probe linings, it is not practical to treat them in a muffle oven. The probe liners shall be heated within their outer sheaves to the maximum temperature practical for the probe.

The first impinger (condensate flask) shall also be cleaned.

7 Minimum requirements for sampling

7.1 Method validation criteria

- a) No additional information
- b) The absorbent material (XAD-2) shall be sufficiently well packed and evenly distributed in the resin trap to prevent the formation of channels, which stack gas may pass through without coming into close contact with the absorbent.
- c) In order to validate the adsorption stage efficiency for gaseous PCDDs/PCDFs, it is necessary to put two resin traps in series.

7.2 Minimum requirements for sampling

No additional information

Note: as measurements can be required to be taken over several hours, it is good practice to have a system in place that will detect leaks during the sample run. This can be done on combustion processes by using an analyser to measure the oxygen concentration of the gas exiting the isokinetic sampling equipment and comparing it to the dry stack gas oxygen concentration. An increase in the oxygen concentration from the isokinetic sampling equipment relative to the dry stack gas oxygen may indicate a leak. If this occurs the sample run can be interrupted, and the leak rectified before continuing with the measurement.

7.3 Field blank requirements

The field blank shall be performed before a measurement series (i.e. consecutive measurements on one stack). However, it is acceptable to apply a field blank to more than one stack provided the stacks are in the same location (i.e. sampling platform) and the process conditions are the same. In order to apply a blank to more than one stack in the

same location, the glassware used for sampling shall:

- be the same as used on the previous stack; or
- shall have been prepared/cleaned in the permanent laboratory at the same time and conditions as the glassware used on the previous stack.

7.4 Additional sampling minimum requirements for the filter/condenser method

In order to maintain the required resin trap temperature, the sampling team shall have planned in advance where they will obtain ice and how much ice is required for the measurement.

Note: the amount of ice required is based on the stack gas temperature, the duration of the test and the moisture content of the stack gas.

7.5 Additional sampling minimum requirements for the dilution method

This approach is not applicable to this appendix.

7.6 Additional minimum requirements for the cooled probe method

This approach is not applicable to this appendix.

8 Safety measures and transport

No additional information.

9 Preparations and sampling

9.1 Advance preparations at the sampling site

No additional information

9.2 Advance preparations at the laboratory

No additional information

9.3 Sampling at the plant

9.3.1 Initial checks

No additional information

9.3.2 Preliminary survey

No additional information

9.3.3 Assembly of apparatus

The following components of the sample train shall be prepared/assembled and

disassembled/recovered in a clean area:

- filter and filter holder
- condenser, resin trap and impinger train glassware

When transferring the sample train or train components from and back to the clean area, all openings shall be sealed.

Note: due to safety issues it may not be practical to assemble the entire sample train in a clean area before carrying the equipment to the sample location.

9.3.4 Sampling

No additional information

9.3.5 After sampling

9.3.5.1 Sample recovery from the sampling train

When working on a platform only the probe and filter housing should be disconnected at a location other than the designated clean area. All other parts shall be disassembled at a designated clean area. An exposed working platform shall not be used as a designated clean area.

9.3.5.2 Sample storage

Samples shall be stored during transport in a cool dark place, such as a cool box packed with ice or plastic freezer packs. An appropriate temperature measurement / indication device shall be used to confirm that the temperature is maintained below 25°C. Samples shall be extracted within 30 days of collection and shall be analysed within 45 days of extraction.

Note: EN 1948 does not specify time limits on extraction and analysis, so the requirement specified in US EPA M23 has been specified in this appendix.

9.4 Calculation of flue gas sample volume

No additional information

9.5 Sampling report

No additional information

Annex A (informative) Toxicity and toxic equivalency

No additional information

Annex B Examples of operation

B4 Filter/condenser method - Variant with solid adsorbent upstream of the condensate flask

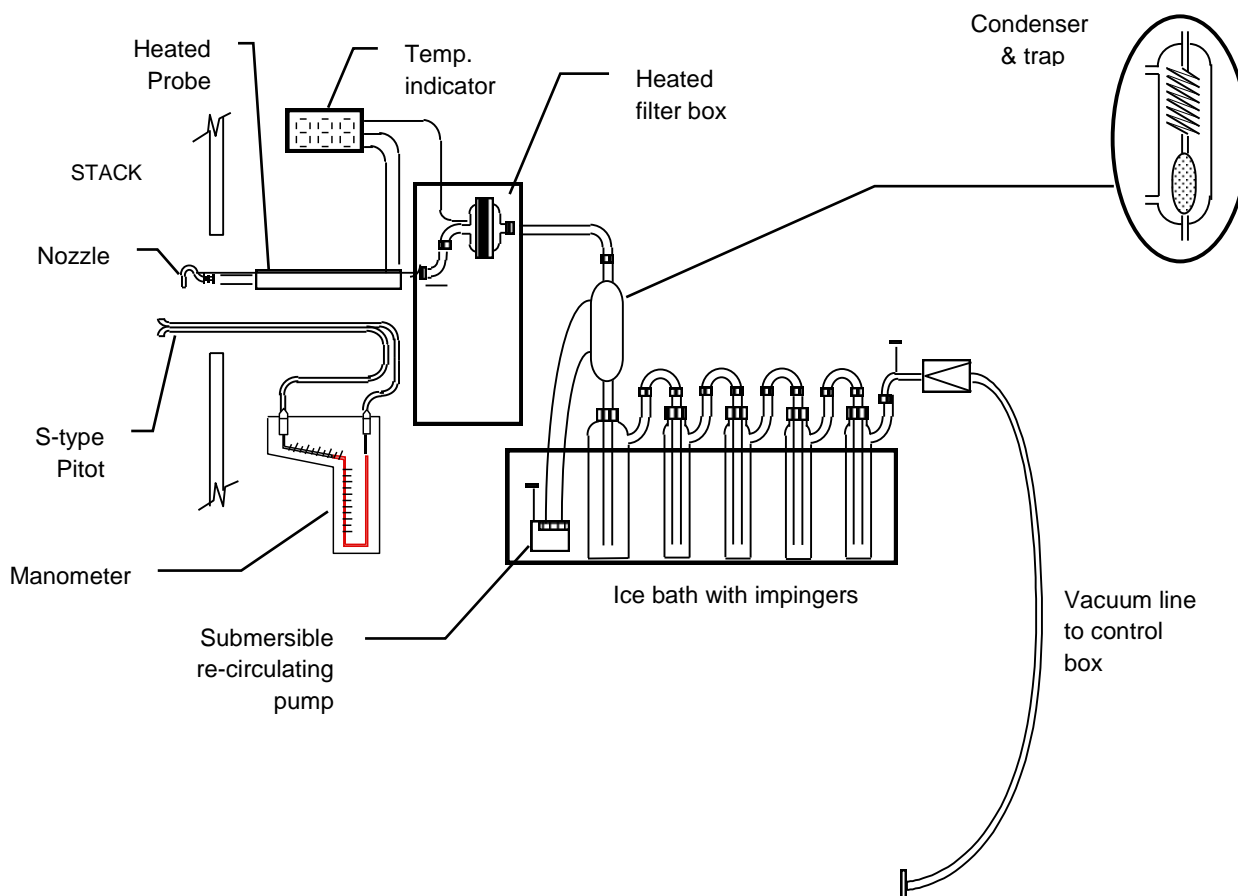
B4. 1. Summary of apparatus design

Equipment used in Ireland to sample PCDDs/PCDFs differs from the example shown in Figure B5 of the standard. Figure 1 below shows the configuration of equipment commonly used in the Ireland¹. This equipment has a solid adsorbent upstream of the condensate flask. A sample train with a solid adsorbent downstream of the condensate flask may also be used.

Section B.4.1 of the standard specifies the use of a glass-lined probe. It is also acceptable to use titanium (see Section 6.2.2).

¹ Typically equipment used in Ireland follows the specifications outlined in US EPA method 23.

Figure 1 Typical Dioxin sample train



B4. 2. Adsorbent and cleaning procedure

No additional information

B4. 3. Sampling train cleaning procedure before sampling

No additional information

B4. 4. Spiking position

B4 specifies that the filter is spiked. It is acceptable to spike the resin trap only or to spike both the resin trap and the filter.

B4. 5. Equipment assembly procedure

The condensate flask may also be located before the solid absorbent.

B4. 6. Leak check procedure

No additional information

B4. 7. Sample gas flow rate control

No additional information

B4. 8. Sampling

No additional information

B4. 9. Equipment disassembly procedure

The condensate collected in the first impinger (condensate flask) shall be stored for analysis.

Annex C Examples of adsorbents and their preparations and cleaning

This appendix specifies that the requirements of Section C.2 XAD-2 shall be followed.

Annex D (informative) Sampling measurement record

No additional information

Annex Za (informative) Clauses of this European Standard addressing essential requirements or other provisions of EU Directives.

No additional information

Part 2: Extraction and clean-up of PCDDs/PCDFs

1 Scope

No additional information

2 Normative references

No additional information

3 Terms and definitions

No additional information

4 Symbols and abbreviations

No additional information

5 Principle of the extraction and clean-up procedure

No additional information

6 Device, materials and ¹³C₁₂-labelled standards

No additional information

7 Method validation and quality control requirements

No additional information

8 Sample pre-treatment and extraction

8.1 Addition of extraction standards

Standards cannot be reliably split into different sampling train components based upon presence of native PCDDs/PCDFs, as it is unlikely that there will be any knowledge of how they are distributed. It is acceptable to use the masses provided in Table 1.

8.2 Sample storage

Brown glass bottles with screw-caps and PTFE-lined seals shall be used. Traps and filters may be wrapped in foil to exclude light.

8.3 Extraction

a) Some particulates on filters are extremely hydrophobic, which may mean that the pre-treatment with hydrochloric acid does not wet the entire surface but forms droplets on the surface of the filter.

8.4 Clean-up

Column chromatography on alumina may be used to separate PCBs from PCDDs/PCDFs.

8.5 Final concentration of the sample extract

No additional information

8.6 Addition of recovery standards

The text in the standard stating that recovery standards are used to measure the recovery of the sampling standards should state that recovery standards are used to

measure recovery of the final quantification step.

9 Report

No additional information

Annexes A – Z

No additional information

Part 3: Identification and quantification of PCDDs/PCDFs

1 Scope

No additional information

.

2 Normative references

No additional information

3 Terms and definitions

No additional information

4 Symbols and abbreviations

No additional information

5 Principles of identification and quantification

No additional information

6 Reagents, materials and equipment

No additional information

7 Safety measures

No additional information

8 Quality control requirements for identification and quantification

8.1 Minimum requirements for identification of PCDF/PCDD congeners

No additional information

8.2 Isomer sums of PCDD/PCDF congeners

No additional information

8.3 Minimum requirements for quantification

- a) no additional information
- b) no additional information
- c) the quantification requirement in this section applies to an initial system suitability check only. It is not necessary to carry out this step on each sample containing 2,3,7,8- TCDD.
- d) no additional information
- e) no additional information
- f) this applies to native PCDDs/PCDFs only, since labelled compound are present at the same concentration in the standards.
- g) no additional information
- h) the equation shown in the standard relates the minimum sensitivity of the measurement to the LOQ. In the original version of the standard this same relationship was presented with the LOD defined in the same manner. Meeting the terms of the original expression is acceptable when stating conformity with the standard.
- i) the standard states that one of the criteria for identifying a congener is if the isotope ratio between the ions matches the theoretical value. If interference results in only one ion being quantified the ion ratio limits would not have been met. However, it does allow quantification of a single ion if interference is unavoidable on the second trace. The use of one ion only must be reported.

9 Quality assurance criteria for extraction/clean-up/quantification procedure blanks

No additional information

10 Calibration of the HRGC/HRMS

If the calibration range is exceeded and the results are over range this shall be stated in the report.

11 Quantification of HRGC/HRMS results

No additional information

12 Calculation of the measurement results

Results should be reported to the toxic equivalency system specified in the Licence. If the Licence does not specify the system used, then the factors in Table A1 of the standard should be applied.

13 Analytical report

No additional information

14 Performance characteristics

No additional information

15 Interferences

No additional information

Annexes A - Z (informative) No

additional information

Appendix 2 [\(GOTO CONTENTS\)](#)

Supplementary Information to EN 16911-1 (Stationary source emissions – Manual and automatic determination of velocity and volume flow rate in ducts)

Introduction

This appendix supplements EN 16911-1:2013 (referred to as EN 16911-1 in this appendix). This appendix does not re-state all the provisions of EN 16911-1, so it is necessary for organisations to refer to both documents. For sections of the standard, where this appendix does not provide additional information, organisations are required to comply with the standard.

1 Scope

This appendix focuses on the measurement of average velocity across a sample plane. This applies to measurements using differential pressure-based techniques, vane anemometers and hot wire anemometers.

This appendix does not apply to the determination of volume flow and average velocity using tracer gas dilution, transit time tracer gas or calculation methods.

When carrying out isokinetic stack-emissions monitoring, it is necessary to assess that the sample plane flow profile meets the requirements of EN 15259. It is also necessary to select an appropriate extraction nozzle size based on the measured velocity at the sample points in the sample plane. EN 16911-1 is used to make these velocity measurements. If the results of isokinetic sampling are to be reported as mass emissions, then the stack gas volumetric flow rate measured during isokinetic sampling shall be used.

This appendix also applies to flow measurements made to calculate mass emissions from stack emissions monitoring measurements.

This appendix does not apply to the calibration of CEMs or for flow measurements made for emissions trading. Information on the application of EN 16911-1 to the calibration of CEMs and emissions trading is contained in PD CEN TR 17078.

2 Normative references

The following referenced sources are essential for the interpretation and application of this appendix:

AG2 Index of Preferred Methods (Appendix 7)
US EPA Method 2G

3 Terms, definitions

No additional requirements.

4 Symbols and abbreviated terms

No additional requirements.

5 Principle

No additional requirements.

6 Selection of monitoring approach

6.1 Monitoring objective

Table 1 – Selection of measurement techniques

- An L-type Pitot may be used for the determination of swirl at the measurement plane.

6.2 Choice of technique to determine point flow velocity

No additional requirements.

6.3 Choice of technique for volume flow rate and average flow determination

No additional requirements.

7 Measuring equipment

No additional requirements.

8 Performance characteristics and requirements

Table 3 – Performance requirements

- Table 3 applies to flow measurement and differential pressure reading devices.
- It is not necessary to carry out the performance tests for the whole measurement system provided the performance requirements of the flow measurement and differential pressure reading devices are demonstrated. The performance requirements of the flow measurement and differential pressure reading devices can be determined using a measurement system that is representative of a typical system.

Note 1: It is preferable for the whole measurement system to be calibrated. However, often this may not be practical because organisations have several differential pressure measurement and reading devices, which are interchangeable between different systems. They may also use different lengths of probes and lines to connect systems together.

- It is not necessary for the stack emissions monitoring organisation to carry out the performance tests for sensitivity to ambient temperature and atmospheric pressure. However, a performance evaluation based upon the technical information provided by the manufacturer shall be carried out, where possible.

Note 2: when carrying out flow measurements to support stack emission monitoring, ambient temperature and atmospheric pressure typically do not make a significant contribution to the overall uncertainty of the measurement, which means they can be excluded from the performance requirements.

- Traceable information on the remaining performance requirements in Table 3 can be provided by the manufacturer of the velocity flow determination technique. If this is not available from the manufacturer, the stack emissions monitoring organisation or another suitable organisation (for example a calibration laboratory) shall demonstrate that equipment meets these requirements.

Note 3: the standard deviation of repeatability of measurement in the laboratory and the effect of angle of sensor to flow can be carried out during the routine calibration of the velocity determination equipment.

- If an L-type Pitot meets the performance requirements of ISO 3966, then its performance will meet the requirements of Table 3.

Note 4: the uncertainty guide for L-type Pitots in ISO 3966:2008 meets the performance requirements in Table 3.

- Lowest measurable flow is the lowest value at which the sensor has been calibrated. Any use below this value shall be validated by the user before a measurement is made. The validation can be done by the stack emissions monitoring organisation or another suitable organisation (for example a calibration laboratory).

9 Measurement procedure

9.1 Site survey before testing

The requirement of obstructing no more than 5% of the stack sampling plane specified in the standard shall apply when carrying out measurements for flow only and for carrying out measurements at stacks with a stack sampling plane area greater than 1.5 m².

When carrying out flow measurements to support isokinetic sampling at stacks with an area of 1.5 m² or less, the area of the flow measurement assembly shall not obstruct more than 10% of the stack sampling plane area.

Note: several particulate monitoring systems integrate the differential pressure measurement device and the particulate sampling equipment. This means that the area of the sampling equipment is larger than the area of the flow measurement assembly, when measuring flow alone with a Pitot.

For stacks with a very small sample plane area it may not be possible to carry out isokinetic sampling and measure flow at the same time because the area of the sampling equipment may obstruct more than 10% of the stack sampling area. Under these circumstances a flow measurement with a standalone Pitot device can be made prior to carrying out isokinetic sampling.

9.2 Determination of sampling plane and number of measurement points

No additional requirements.

9.3 Checks before sampling

9.3.1 General

If a calibrated electronic pressure reading device is used, it does not need to be checked on-site before use with a liquid manometer or calibrated pressure sensor.

Electronic pressure measurement devices shall be calibrated in accordance with the test laboratory's internal calibration schedule.

The resolution in pascals of the electronic pressure measurement device shall be at least 1 Pa.

The internal diameter of the stack shall be measured (see section 7.2 of the standard).

When carrying out measurements to support isokinetic sampling and other stack emissions monitoring methods, it is not necessary to check varying flow conditions with time by using a measurement device at a fixed point.

Note: typically, velocity is monitored during isokinetic sampling, which means changes over time are accounted for when this type of sampling is carried out.

When carrying out measurements for the calibration of CEMs that measure flow, it is necessary to take account of variations in flow, as specified by the standard.

9.3.2 Pre-test leak check

A Pitot leak check shall be performed before each measurement.

The standard provides an example of how the leak check can be carried out. Stack emission monitoring organisations may follow this example, develop their own or use the procedure given in US EPA Method 2G.

9.3.3 Check on stagnation and reference pressure taps (S-type Pitot tube)

No additional requirements.

9.3.4 Tests of repeatability at a single point

A test of repeatability at a single point is usually not required when carrying out measurements to support isokinetic sampling because a single measurement device can be used.

9.3.5 Swirl or cyclonic flow

A swirl test shall be carried out at the sample plane at least once per measurement campaign.

The procedure for carrying out a swirl test using an S-type Pitot and an L Type Pitot is given in Appendix 7 of AG2 (index of Preferred Methods).

The information on velocity correction for the effect of flow direction is not needed when assessing the suitability of a sample plane's flow profile for isokinetic sampling.

9.4 Quality control

Table 4 – Performance requirements during field measurements

- Field repeatability - this is not required for measurements to support stack emissions monitoring (see 9.3.4 of this document).
- Angle of probe to measurement plane (pitch of probe) – determining this is not required for measurements to support stack emissions monitoring.
- Uncertainty in flow measurement and differential pressure reading device calibration – the relative uncertainty increases as the differential pressure decreases. Therefore, it is acceptable to demonstrate the uncertainty requirements at the full scale of detection of the differential pressure reading device. An uncertainty of $\pm 0.5\%$ or less of the full scale may be used. This uncertainty, expressed as a percentage of the full scale value, can then be applied to other readings. When using this approach, it is important to select a differential pressure reading device that has an appropriate range (see Annex H).

9.5 Measurement of flow at locations within the measurement plane

No additional requirements.

9.6 Post-measurement quality control

No additional requirements.

10 Calculation of results

10.1 General

No additional requirements.

10.2 Measurement of velocity

No additional requirements.

10.3 Determination of the mean velocity

No additional requirements.

10.4 Correction of average velocity for wall effects

The correction of average velocity for wall effects is not required for measurements to support isokinetic sampling.

10.5 Calculation of the volume flow rate from the average velocity

No additional requirements.

10.6 Conversion of results to standard conditions

No additional requirements.

10.7 Establishment of the uncertainty of results

No additional requirements.

11 Evaluation of the method

No additional requirements.

Annexes A, B, C, D, E (normative)

No additional requirements.

Annexes F, G (informative)

No additional information.

Annex H (informative) Differential pressure measurement

H.1 General

Section H.1 of the standard (EN ISO 16911-1) provides information on the relationship between flow, differential pressure, measurement range and uncertainty.

The standard states that the basic flow formula is available in CEN / TS 14793. This is a mistake. The formula is available in ISO 10780.

The example given in Annex H.1 is incorrect. For a differential pressure of 9% of manometer scale on a 0 Pa to 2500 Pa device (i.e. 225 Pa), then:

- for a liquid manometer with 1% accuracy of reading: 225 Pa equals 2.25 Pa, not 0.225
- for an electronic manometer with an accuracy of 0.5% of full scale: 0.5% of 2500 Pa is 12.5 Pa, not 1.25 Pa.

The example provided in the standard would be suitable for a lower range manometer (i.e. 0 Pa to 250 Pa).

The standard states that there are very low-resolution manometers available that can read down to a 0.001 Pa. It is not necessary to measure at this very low pressure when carrying out stack emissions monitoring. An inclined liquid manometer that is typically used for stack emissions monitoring has a resolution of about 2.5 Pa.

H.2 Liquid manometers

No additional information.

H.3 Digital manometers and other electronic devices

No additional information.

Annex I, J (informative)

No additional information.

Appendix 3 [\(GOTO CONTENTS\)](#)

Supplementary Information to EN 15259 (Measurement of stationary source emissions – Requirements for measurement sections and sites and for the measurement objective, plan and report)

Introduction

EN 15259 is important to plant designers, plant operators and organisations that measure stack emissions. By following the relevant requirements of AG2, a stack emission monitoring organisation will meet the sample planning and reporting requirements of EN 15259.

In order to meet the requirements for sampling strategy, it may be necessary to carry out a test for stack gas homogeneity. Section 8.3 of EN 15259 and this appendix provides information on meeting the requirements of the homogeneity test.

This appendix does not repeat text, tables or diagrams from EN 15259, instead it refers to the relevant sections of this standard. It is therefore essential that the reader has a copy of EN 15259.

1 Scope

No additional information

2 Normative references

No additional information

3 Definitions symbols and abbreviations

No additional information

4 Symbols and abbreviations

No additional information

5 Principles

No additional information

6 Measurement section and measurement site

No additional information

7 Measurement objective and measurement plan

No additional information

8 Sampling strategy

8.1 General

Monitoring organisations may use the results from a homogeneity test carried out by another monitoring organisation, provided the organisation that carried out the test has EN ISO/IEC 17025 accreditation for it.

Note: information on when a homogeneity test is repeated is given in section 8.3a.

8.2 Measurements of particulates and other components by grid measurement

Flow proportional sampling is not required when carrying out a grid measurement for gases.

8.3 Determination of homogeneity

a) Application of the homogeneity test

The EPA require the following risk-based assessment when a decision is being made on the need for a homogeneity test.

- The need for a homogeneity test should be determined by a qualified Stack testing practitioner, in consultation with the site operator.
- Emission testing reports shall always state if a homogeneity test has (or has not) been conducted and if this took place as part of the current or a previous test campaign.
- The following considerations shall form part of the assessment:
 - Has a homogeneity test been conducted in the past? A one-time test will generally suffice.
 - Is the emission source combustion or a non-combustion process? Non-homogenous conditions

are more likely to occur at combustion sources but under some circumstances the EPA may require a homogeneity test to be carried out on non-combustion processes.

- Sampling plane area. The test is not usually required for stacks with sampling plane areas of <1 m² (i.e. below 1.13 m in diameter for circular ducts).
- Proximity of emission test results to the emission limit value (after method uncertainty has been taken into account).
- EPA has the discretion to direct a homogeneity test in any circumstance.

Due to practical reasons the homogeneity test is carried out using instrumental measurement techniques. For determinands where the CEN / ISO standard reference method is a manual (non-instrumental) method, an alternative instrument-based method that is specified in AG2 (Index of Preferred Methods) may be used. If a suitable instrumental method is not available, the determinand may be excluded from the homogeneity test (other gases tested may be used as an alternative means of demonstrating homogeneity).

For determination of NO_x, if the NO₂ concentration is insignificant (less than 10% of the total NO_x), a homogeneity test for NO_x may be carried out by measuring NO only. If significant amounts of NO₂ are present (greater than 10% of the total NO_x), it is necessary to perform a total NO_x measurement.

Note 2: a converter may be used to convert NO₂ to NO. Total NO_x can then be measured directly. It may not be necessary to carry out a homogeneity survey for TOC, if it is established that CO is a suitable proxy for TOC.

Note 3: if TOC is at low concentrations (i.e. less than 3 mg/m³) the variability in the readings may make it difficult to apply the homogeneity test.

The homogeneity test applies to O₂ because it is used to correct the final measurement result. However, the homogeneity tests are not corrected for O₂.

The homogeneity test may apply to H₂O, if it is used to correct the final measurement result. For processes without liquid-based abatement systems, gases such as O₂ may be used as an alternative for H₂O. O₂ is not a suitable alternative for non-combustion processes, as they have fixed ambient air O₂ concentrations. SO₂ may be used as an alternative for HCl, HF and NH₃.

The homogeneity test is usually carried out at a measurement location once only. This shall be done at conditions that are representative of the normal operation of the process.

However, it may have to be repeated if there is a significant operational change in the process, such as a long term change in load, fuel, abatement technology or ductwork.

If a licensee does not have an ELV, a virtual ELV can be agreed with the EPA .

b) Responsibility on EN ISO/IEC 17025 accredited monitoring organisations

Stack emissions monitoring organisations which are accredited to EN ISO/IEC for EN 15259 shall have documented procedures which meet the requirements for the determination of homogeneity. This shall include having the required equipment and a method for calculation.

If the homogeneity test has not been carried out at a location, where it is applicable, the monitoring organisation shall inform the licensee that the homogeneity test should be carried out to confirm that the sample location is suitable for measuring gases from a single point. The SSP and monitoring report shall record if the homogeneity test has not been carried out.

c) Carrying out the test

The test is carried out using instrumental analysers. The analysers shall be verified for performance, such as by the annual systems check specified in reference methods (i.e. zero, span, linearity, response time compared to moving system etc.).

When measuring water soluble gases condensation in the probe must be avoided. For example, the full length of the probe shall be maintained at a temperature of 20K above the stack gas dew point, so that condensation of stack gas in the probe does not occur.

When measuring insoluble gases, such as NO, CO or O₂, an unheated probe may be used.

The standard states that a permanently installed CEM may be used as the reference point, provided it is calibrated in accordance with EN 14181. It is therefore necessary to confirm that the CEM has a valid up to date Quality Assurance Level 2 / Annual Surveillance Test assessment and report.

Note 1: It is generally better to use the same type of reference method for both the grid and time dependant reference measurements, since the statistical tests defined in EN 15259 require a

comparison of the standard deviations of the two sets of data.

A single instrument may be used to carry out the grid measurement and time dependent reference measurements. The time dependant reference measurements are taken for the same length of time as the grid measurements. It is important that the process is stable for both sets of measurements. This approach is acceptable if the combined uncertainty of the grid and time dependant reference measurements is less than the value specified in the standard. If this is not the case, then the measurements must be repeated with two instruments.

The standard deviation of S_{grid} is generally greater than S_{ref} because it includes additional uncertainty caused by spatial differences. If S_{ref} is less than S_{grid} , then the location is classed as being homogenous. However, it is important to check that the grid and reference measurements are compatible. For example, if S_{ref} is significantly lower than S_{grid} it could indicate a measurement error.

d) Default values in lieu of an ELV for O₂ and H₂O for calculation of U_{perm}

For O₂, a default value of 21% shall be used in lieu of an ELV. For H₂O a default value of 30% shall be used in lieu of an ELV (see Section 8.3, Note 10 of EN 15259). These default values are used in the absence of an ELV to calculate the U_{perm} value.

e) Exemption from applying test results when results are low

If the standardised measurement results are less than the maximum expanded measurement uncertainty allowed at the daily ELV for the method used, then the statistical tests to assess if the duct is homogenous do not need to be carried out. The measurement uncertainty limits are provided in AG2.

f) Using S_{grid} and S_{ref} values to exclude inconclusive results

Note 1: if the variation in the fixed point is small (i.e. low S_{ref}), it is possible that the duct will appear to be heterogeneous, even if the grid only shows a small variation (i.e. low S_{grid}). This could result in inconclusive results, which result in a "false fail" of the homogeneity test.

If the homogeneity check calculations in EN 15259 indicate that the duct is heterogeneous, the following additional checks can be applied to exclude inconclusive results (a "false fail"):

- If $S_{ref} = <2\%$ or $S_{grid} = <5\%$ of the ELV for gases except O₂ and H₂O, any sample point in the duct may be used because the duct is considered to be homogeneous (i.e. the standard deviation of the gas concentrations at the reference point is insignificant when compared to the ELV and the limit of quantification of the method).
- In the absence of an ELV for H₂O, a default value of 20% shall be used to determine if $S_{ref} = <5\%$ of the virtual ELV and a value of 30% shall be used for S_{grid} .
- If S_{ref} is $<0.2\%$ or S_{grid} is less than 0.3% for O₂, any sample point in the duct may be used.

The above checks shall only be applied after completing the determination of homogeneity procedure specified in EN 15259.

Where values are based on ELVs, the calculations may require rechecking, if ELVs for the process are lowered.

Note 2: If there is a BAT-AEL range applicable to the process the lowest value of this range could be used to complete ELV based checks. This would demonstrate that the check would still be valid, if the lowest BAT-AEL was applied in the future.

9 Measurement report

For new plants, it is recommended that a report containing details of the flow criteria and homogeneity test results is produced during the commissioning of the plant.

A determination of homogeneity report shall state whether homogeneous results were demonstrated by the procedure in EN 15259 only or whether the additional criteria for assessing inconclusive data specified by this appendix was required.

Appendix 4 [\(GOTO CONTENTS\)](#)

Supplementary Information to ISO 15713 (Stationary source emissions – Sampling and determination of gaseous fluoride content)

This appendix supplements ISO 15713. However, it does not re-state all the provisions of ISO 15713. The clause numbers in this appendix follow those of ISO 15713, although the text from the standard is not repeated in this document.

1. Scope

In order for the monitoring to be EN ISO/IEC 17025 accredited both the sampling **and** laboratory analysis shall be accredited to EN ISO/IEC 17025.

Ion chromatography may be used, instead of ion selective electrode, for the analysis of samples.

2. Normative references No

additional information

3 Terms and definitions

No additional information

4 Principle

No additional information

5 Reagents

5.1 – 5.3 No additional requirements

5.4 Calibration solutions

No additional requirements

6 Apparatus

6.1 Introduction

The standard (ISO 15713) states that materials used in the sample train (probe liner, filter, filter housing, tube connecting impingers to the probe and the impingers) shall be resistant to temperature and chemical attack from the stack gas.

The standard lists materials that are known to be suitable for the probe liner, tube connecting impingers to the probe and the impingers.

The standard does not give materials for the filter and filter housing. Therefore, manufacturer's specifications have been reviewed to select an appropriate filter and filter housing. The filter housing may be made up of the same material as the probe.

If a stack emissions monitoring organisation wishes to use materials that are not specified in this document, they shall prove to the satisfaction of their accreditation body, such as INAB or UKAS,

that the materials are resistant to chemical attack from stack gases containing gaseous fluoride.

6.2 Probe

Monel® type alloys or silica shall be used.

Note 1: Monel® type alloys have an unreactive, smooth surface that is corrosion resistant. Monel® 400 is suitable for use up to temperatures of 550°C.

Note 2: Silica refers to fused silica, which is a glass like substance made from silica sand (quartz) or silica tetrachloride. It is unreactive, chemically resistant to HF and stable at temperatures up to 900°C.

If isokinetic sampling is carried out the sample nozzle shall be made of Monel® type alloys or silica.

6.3 Filter and filter housing

The standard states that if the amount of particulate fluoride is below 10% of the total emissions then the filter can be omitted. This shall be done where possible, as it reduces the possibility of gaseous fluoride being removed by absorption onto the filter and filter holder.

If the amount of particulate fluoride in the stack gas is not known, or is not clearly below 10% of the total emissions a filter shall be used, as there is a risk of overestimating the fluoride content as soluble particle bound fluorides may enter the bubbler solution.

The filter holder shall be made of the same material as the probe or from PTFE.

HF might be outgassed from new PTFE components. Preconditioning of new PTFE components by heating shall be carried out. Heating shall be carried out to the temperature the material will be exposed to during sampling.

PTFE shall not be used above 250°C as it degrades and releases fluoride. The filter shall be made of quartz fibre.

6.4. Sampling train

The impingers shall be connected to the sampling probe using polypropylene, polyethylene or Viton® tubing.

The impingers shall be made of quartz, polypropylene or polyethylene.

6.5 - 6.11

No additional requirements

7. Sampling

7.1 - 7.5

No additional requirements

7.6. Sampling

Note: the standard describes the use of a side-stream technique when sampling isokinetically. This enables the recommended flow rate through the absorber solution to be maintained. In Ireland, this approach is not used.

Isokinetic sampling shall only be carried out when droplets are present. When isokinetic sampling is required it shall meet the requirements of EN 13284-1. When sampling isokinetically, the flow rate through the impinger may increase above that recommended by the standard. Under these circumstances it is acceptable to use a greater volume of absorber solution than specified in the standard.

7.10. Quality assurance

The standard states the leak rate measured during any leak test shall not be greater than 4% of the nominal flow rate. Sections 7.7, 7.8 and 7.9 state 2%. A 2% value shall be used.

7.11. Sample recovery

No additional requirements

7.12. Equipment field blank

The field blank shall be less than 10% of the emission limit value.

8. Analytical procedure

Ion chromatography may be used.

9. Expression of results

No additional requirements

10. Performance characteristics

No additional requirements

11. Test report

If carrying out EN ISO/IEC accredited work the monitoring report shall be submitted in the report format specified AG2.

Appendix 5 [\(GOTO CONTENTS\)](#)

Supplementary Information to EN 14385 (Stationary source emissions – determination of the total emission of As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, Tl and V)

This document supplements EN 14385:2004 (referred to in this document as EN 14385). However, it does not re-state all the provisions of EN 14385 and organisations are reminded of the need to comply with the criteria detailed in EN 14385. The clause numbers in this document follow those of EN 14385 although the text from the standard is not repeated in this document.

1. Scope

European Standard EN 14385:2004 may be used to determine other metals than those named in the scope of the standard.

In order to extend the scope of metals it is necessary for the analytical laboratory to carry out validation for the analysis of the additional metals. The filter, rinsing solutions and absorption solutions can be spiked with certified reference materials and analysed to determine recoveries and limits of detection¹.

This appendix includes two approaches. One for measuring metals including mercury and one for measuring metals without mercury.

EN 14385 allows EN 13211 - *Manual method of the determination of the concentration of total mercury* (referred to in this document as EN 13211) to be carried out using a side stream sampling technique. In Ireland, custom and practice does not normally include the use of side stream sampling techniques.

This appendix therefore includes the option of measuring mercury in the main sampling train. When carrying out this option it is important that all provisions specific to EN 13211 are met.

Measurement of metals including mercury differs from measurement of metals, according to EN 14385, in the following ways:

- a different absorber (also known as impinger) train configuration to that specified by EN 14385 is used;
- the absorbants / analytical requirements of EN 13211 are included; and
- extra analytical requirements are employed to ensure the combination of both methods does not affect the reliability of the measurement.

This approach follows the principles established by a validation exercise carried out by the Environment Agency (England)².

In order to obtain EN ISO/IEC accreditation for measuring metals including mercury it is necessary to meet the requirements of this appendix and be accredited to use EN 13211. If accreditation is sought for metals excluding mercury it is not necessary to be accredited for EN 13211. The schedule of accreditation will specify whether mercury is included or excluded.

EN 14385 has been validated on stacks with a gas composition given in Table 1 of the standard. The standard can be used on processes with other gas compositions.

¹ The analysis of tin (Sn) may be problematic due to the formation of "tin stone" (a mixed valence oxide). However, validation work carried out by a UK laboratory has shown acceptable results.

² Environment Agency (England) document: Monitoring mercury at the same time as other heavy metals (2004)

Note 1: practical experience shows that the standard can be used on a wide range of processes and stack gas conditions.

It is not acceptable to combine the measurement of metals and particulates.

2. Normative references

EN 13284-1:2001 has been replaced by EN 13284-1:2017.

3. Definitions, symbols and abbreviations

No additional information

4. Principle

No additional information

5. Apparatus, chemicals and gases

5.1 Apparatus

Commercial titanium is made to different specifications based on its purity. As the other materials present may contain the specific elements being determined during the measurement it is important that an appropriate grade of titanium is used.

Titanium will degrade in gas streams with a fluoride content of greater than 20ppm.

Titanium may also degrade at temperatures greater than 350°C.

When using this appendix to sample for metals including mercury it is important that parts of the sampling equipment in contact with the mercury containing flue gas, or with liquid containing mercury, shall be made of materials shown in Table 1 of EN 13211.

Absorbers shall be kept below 30°C. This can be done by using ice.

PTFE is not suitable for temperatures in excess of 200°C.

5.2 Chemicals and filter material

5.2.1 General

No additional information

5.2.2 Filter

A batch of filters for metals sampling are those prepared on a single occasion. This may include filters prepared prior to the sampling campaign for use exclusively on that campaign or a greater number of filters prepared on one occasion but stored for use in future monitoring campaigns.

At least one filter per batch should be analysed for metal content prior to use.

5.2.3 Hydrofluoric acid

No additional information

5.2.4 Hydrogen peroxide

No additional information

5.2.5 Nitric acid

No additional information

5.2.6 Hydrochloric acid

No additional information

5.2.7 Water

No additional information

5.2.8 Absorption solution

EN 14385 states that the absorption solution must be made freshly. The diluted nitric acid solution shall be prepared at a permanent laboratory. The addition of peroxide to the acid solution may be carried out at the laboratory or on site.

The elements being determined shall be present at a concentration below 1µg/l in the absorption solution.

EN 13211 describes two absorption solutions that may be used for the absorption of mercury.

If a potassium permanganate / sulphuric acid solution is used it shall be prepared at a permanent laboratory, as it requires the addition of concentrated hydrochloric acid.

If a potassium dichromate / nitric acid solution is used the nitric acid solution shall be made in a permanent laboratory. The potassium dichromate solid can be added to the pre-prepared nitric acid solution at the laboratory or on site.

If a potassium permanganate / sulphuric acid solution is used it is important that the solution is not mixed with the absorption solutions for other gaseous metals. Mixing of the two may lead to the nitric and sulphuric acid causing a gas release, which results in high pressure and possible explosion of the bottle. The use of an empty absorber between the different absorption solutions will reduce the likelihood of mixing occurring (see Annex B).

5.2.9 Boric acid solution

No additional information

5.2.10 Chemical blank solution for digestion

At least one digestion blank shall be prepared with each batch of samples.

5.2.11 Rinsing acid

The standard states that the rinsing acid is used for rinsing the digestion equipment. The rinsing acid is used on site to rinse the nozzle probe and filter housing (see section 8.4.2)

EN 14385 specifies the use of 25% mass content nitric acid for rinsing glassware on site. Due to health and safety reasons 5% mass content nitric acid may be used.

The elements being determined shall be present at a concentration below 1µg/l in this solution. This shall be confirmed before use.

5.2.12 Standard solutions

No additional information

5.2.13 Certified reference material

No additional information

5.2.14 Gases

No additional information

6. Sampling equipment

6.1 General requirements

When performing the determination of mercury in conjunction with EN 14385 the first three absorbers in the sample train are the same as specified in EN 14385. The fourth absorber is empty. The fifth and sixth absorbers contain absorbent solutions for gas phase mercury. It may be necessary to place an empty absorber at the start of the train and before the drier assembly (the use of these optional absorbers depends on the expected moisture content of the stack gas). An example sample train is provided in Annex B.

6.2 Isokinetic sampling equipment

No additional information

6.3 Absorbers

The absorber efficiency test compares the concentration of each element found in the third absorber with the concentration of that element in the total sample train.

Note 1: A number of metals are often measured together as a group and are summed for comparison with an ELV for total metals. For example, metals are typically reported in two groups:

- Cd and Tl
- Sb, As, Pb, Cr, Co, Cu, Mn, Ni, & V

The absorber efficiency test is applied separately to each group of metals.

If one of the individual metal elements in the last impinger (e.g. Sb) is more than 10% of the total combined metals for that individual element (i.e. the filter, probe rinse and impingers) collected, then the result for all the metals in that group shall be rejected.

However, for each group the requirement to pass the absorber efficiency check shall not apply:

- to individual metal element(s), where the individual mass of the element in the final result is less than 1% of the result for the total mass of metals in the group; or
- if the total combined result for the metals in the group is less than 30% of the ELV.

Note 2: the absorber efficiency check becomes less effective as the concentration of the pollutant in a stack gas decreases.

Note 3: For example:

- Cd and Tl combined – 0.05 mg/m³
- Sb, As, Pb, Cr, Co, Cu, Mn, Ni, V combined – 0.5 mg/m³

For Cd and Tl combined it would not be necessary to comply with the less than 10% in the last impinger criteria, if the overall combined Cd and Tl result for the sample train was below 0.015 mg/m³ (i.e. <30% of the ELV).

For combined metals (Sb, As, Pb, Cr, Co, Cu, Mn, Ni, V) it would not be necessary for each individual element to comply with the less than 10% in the last impinger criteria, if the overall combined result for the sample train was below 0.15 mg/m³ (i.e. <30% of the ELV).

For mercury, the requirements on the absorber efficiency in EN 13211 shall be followed. However, when EN 14385 and EN 13211 are combined, the mercury in the last absorber is compared to the total mercury in both the mercury absorbers and the absorbers used for metals.

7. Cleaning of the sampling equipment prior to sampling

The following is a proven cleaning procedure:

- Rinse all sampling train glassware with hot tap water and then wash in hot soapy water. Next, rinse glassware three times with tap water, followed by three additional rinses with HPLC water. Then soak all glassware in a 10 percent (v/v) nitric acid solution for a minimum of 4 hours, rinse three times with water and then rinse a final time with acetone and allow to air-dry. Cover all glassware openings until the sampling train is assembled for sampling³.

Note 1: the grade of water is defined in section 5.2.7 of the standard.

The above procedure also includes glass and titanium probe liners. However, it may not be practical to soak probe liners for 4 hours in a nitric acid solution. An alternative is to circulate the rinsing solution for 15 minutes.

It is permissible to use an alternative cleaning procedure, if an analytical laboratory provides evidence to their accreditation body that the procedure is equivalent to the procedure specified above.

Note 2: consistently low field blank results help demonstrate that an alternative cleaning procedure is effective.

Although it is acceptable to re-use glassware on different stacks, it may not be advisable due to the risk of contamination.

8. Procedure

8.1 General requirements

There are several temperature criteria which are required. These depend on the stack gas temperature; the type of equipment being used, and whether sampling for mercury is being undertaken.

EN 14385 states that the sampling probe and out-stack filter shall be maintained at a temperature of at least 20°C above stack gas temperature. However, due to limitations

³ This procedure is specified in US EPA method 29

on the equipment, it is not necessary to heat the sampling probe and out-stack filter to temperatures above 180°C.

The following applies when sampling for metals including mercury:

- the sampling probe and the out-stack filter shall be maintained at a temperature of at least 20°C above stack gas temperature (it is not necessary to heat the sampling probe and out-stack filter to temperatures above 180°C).
- when using titanium equipment, the temperature shall be maintained at 180°C or higher.

Details of the probe and filter temperature shall be included in the report.

When measuring mercury, it is important that the absorption solutions do not mix. Mixing of the solutions will result in manganese contamination.

8.2 Preparation and installation of equipment

8.2.1 Filter installation

Filters should not be installed or recovered on exposed platforms.

8.2.2 Nozzle installation

No additional information

8.2.3 Installation of absorbers

Due to the possibility of pressure caused by stored absorber solutions, it might be appropriate to consider the use of pressure-relief devices on absorber solution storage bottles.

8.2.4 Leak test

No additional information

8.2.5 Installation of equipment at chimney or duct

No additional information

8.3 Performance of the sampling

No additional information

8.4 Disassembling the equipment

Aluminium foil shall not be used to cap sample trains.

8.5 Field blanks

A blank shall be taken at each stack during a sampling campaign. A campaign is defined as one site visit.

When carrying out a sampling campaign with multiple tests over a number of days it is advisable to carry out several blanks.

When measuring mercury, it is necessary to carry out analysis of mercury upstream of the mercury absorbers. This analysis shall also form part of the field blank.

8.6 Requirements for storage of the samples

Samples shall be stored during transport in a cool dark place, such as a cool box packed with ice or plastic freezer packs. An appropriate temperature measurement / indication device shall be used to confirm that the temperature is maintained below 25°C.

Note: this approach is based on the requirements in US EPA M29 and VDI 3869.

8.7 Pre-treatment before analysis

No additional information

8.7.1 General

No additional information

8.7.2 Pre-cleaning of the digestion equipment

No additional information

8.7.3 Treatment of CRM and filter

No additional information

8.7.3.1 General procedure

No additional information

8.7.3.2 Heating in oven or heating plate

No additional information

8.7.3.3 Heating in microwave oven

When analysing metals including mercury the requirements of EN 13211 shall be met.

Note: EN 13211 specifies that the filter should be heated during digestion for a greater length of time than specified in EN 14385.

8.7.4 Pre-treatment of absorption solutions

No additional information

8.7.5 Pre-treatment of rinsing solutions

When measuring mercury, it is necessary to carry out the pre-treatment according to EN 14385. However, before carrying pre-treatment for metals analysis a portion shall be quantitatively removed for mercury analysis.

8.8 Analysis

When measuring mercury, it is necessary to meet the requirements of EN 13211. For the filter and probe wash the following approach shall be used:

- the filter shall be digested/heated and analysed as one sample following the requirements in EN 14385. This gives the result for both the mercury and metals on the filter;
- the probe rinse volume shall be measured. This shall be split into two samples A and B (B being a measured small aliquot);
- Sample A shall be digested/heated and analysed according to EN 14385. This gives the result for metals in the sample A portion of the probe rinse. The metals concentration in sample A shall be used to determine the metals mass in sample B. The results from samples A and B shall be combined to give the total metals in the probe rinse.
- Sample B shall be analysed according to EN 13211. This gives the probe rinse mercury result for sample B. The mercury concentration in B shall be used to determine the mercury mass in Sample A. The results from samples A and B shall be combined to give the total mercury in the probe rinse.

Besides these requirements it is also necessary to analyse the absorber solutions in the first three absorbers, which are primarily in place to capture other gas phase metals. In total five

absorbers shall be measured for mercury by taking a known volume from each. The analytical method for measuring all five absorbers is the same. At least once per sampling campaign it shall be proven that less than 5% of the total gas phase mercury absorbed is in the final absorber (see section 7.33 of EN 13211).

Linearity shall be checked, and the correlation coefficient should be 0.98 or better. Analyte additions can be used as an alternative calibration technique and internal standards may be added to correct for transport effects and instrument drift.

Suitable quality checks (QCs) should be prepared and measured with each batch of samples. For filters, a suitable certified reference material (CRM) can be used if available. If no CRMs are available, then a spiked filter may be exposed to the digestion process instead. For adsorption and rinsing solutions, spiked solutions can be used. For all QCs a recovery of 100 ± 20 % of the expected or certified value is acceptable. If any QC results are outside this range, then this must be highlighted in the final report. If recoveries are outside the required range sampling and analysis stages might have to be repeated.

Note: adsorption and rinsing solutions are tested as part of the blank solution for digestion and, consequently, do not need to be done separately.

9. Expression of results

If results are at, or below, the limit of detection then they shall be reported as being at the detection limit.

10. Test report

No additional information

Annex A: Examples of absorption vessels Example B

Greenburg-Smith impingers shall be used. **Annex B: Types of**

isokinetic equipment

The examples shown below are shown with 'out-of-stack' filtration.

Figure 1: Sample train for metals (excluding mercury)

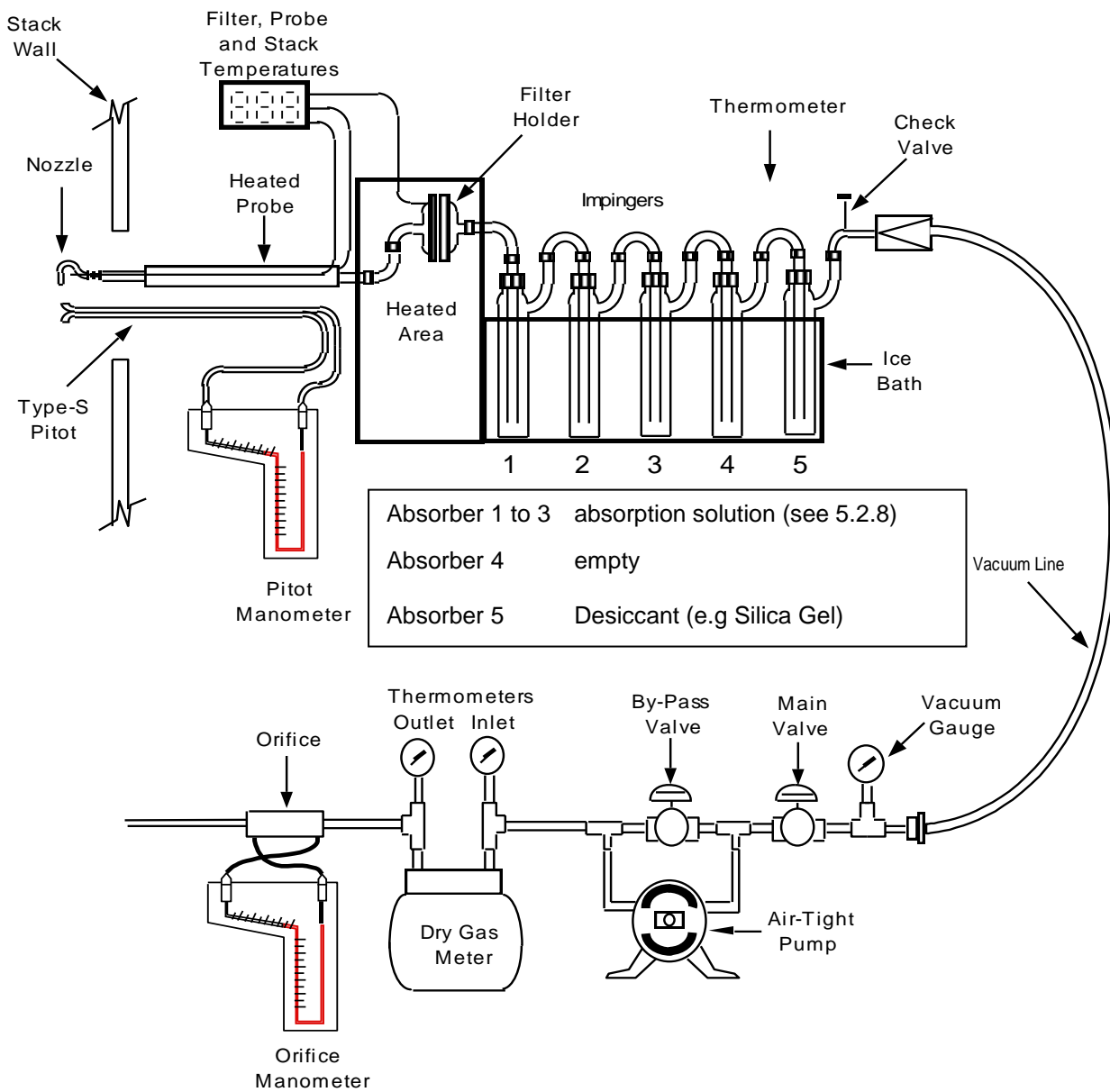
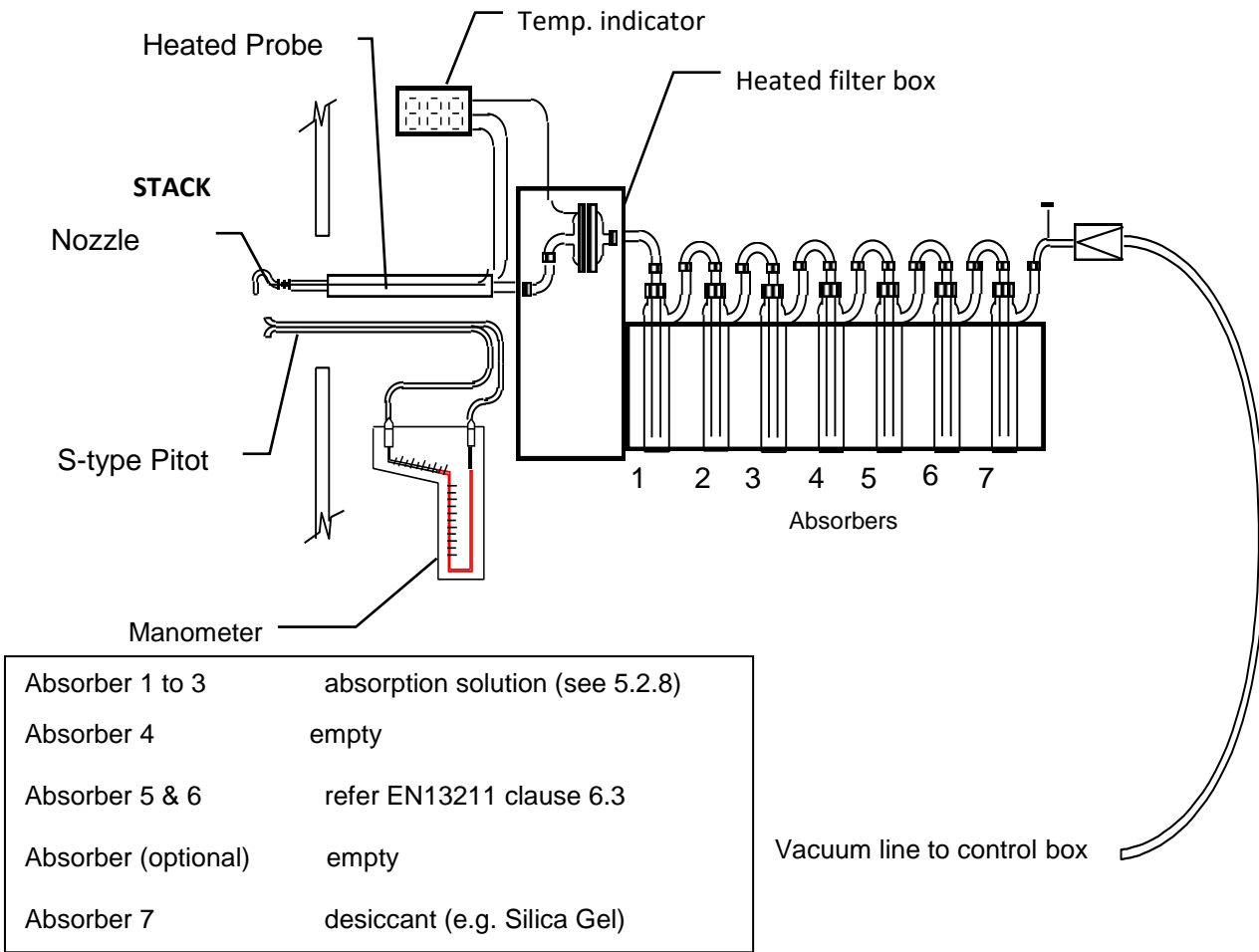


Figure 2: Sample train for metals and mercury



Note: Absorber Nos 1-3 are analysed for all metals including mercury. Absorbers Nos 5 and 6 are analysed for mercury only. The amount of mercury in the final absorber should be less than 5% of the total amount of mercury in all absorbers or correspond to less than $2 \mu\text{g}/\text{m}^3$ whichever is the greater.

Annex C: Pre-cleaning procedures of the sampling equipment at the laboratory and determination of the absorption efficiency

No additional information

Annex D: Measurement results of two field tests

No additional information

Annex E: pre-tests for determination of the efficiency, of the digestion and of the performance of the analytical procedure

No additional information

Annex ZA: Relationship with EU Directives

No additional information

Appendix 6 [\(GOTO CONTENTS\)](#)

Supplementary Information to EN 13725 (Air Quality – Determination of odour concentration by dynamic olfactometry)

This appendix supplements EN 13725:2003 (referred to as EN 13725 in this appendix). However, it does not re-state the provisions of EN 13725 and organisations are reminded of the need to comply with the criteria detailed in EN 13725 in addition to those defined in this document.

1 Scope

This appendix focuses on the collection of odour samples from stack gas emissions. The samples are subsequently analysed by delayed dilution olfactometry, in accordance with EN 13725.

The technique referred to as direct olfactometry in EN 13725 is not currently applied in Ireland and is not discussed further within this document.

2 Normative references

The following referenced documents are essential for the interpretation and application of this appendix:

- EN 13725, Air quality: Determination of odour concentration by dynamic olfactometry
- ISO 5492, Sensory analysis; vocabulary
- EN15259 - Stationary source emissions – Requirements for the measurement sections and sites and for the measurement objective, plan and report
- EN 16911-1 – Stationary source emissions – Manual and automatic determination of velocity and volume flow rate in ducts Part 1: manual reference method
- AG2 Index of Preferred Methods Appendix 2 or EN 16911-1 - Supplementary Information to EN 16911-1
- Guidance Note on Site Safety Requirements for Air Emissions Monitoring (AG1). Environmental Protection Agency (OEE)
- Emission Monitoring Guidance Note (AG2). Environmental Protection Agency (OEE)

3 Terms, definitions, and symbols

No additional requirements to EN 13725

4 Principle of measurement (sampling)

No additional requirements to EN 13725

5 Performance quality requirements

5.1 General

The performance of the technique in terms of quality is influenced by a range of quantifiable sources of uncertainty. The component and overall uncertainty of the techniques used to collect and analyse odour samples shall be demonstrated and recorded.

The following quantifiable sources of uncertainty are associated with odour sampling:

- uncertainty of measurement of volume flow, pressure and temperature in accordance with EN 16911-1

Note: the uncertainty associated with flow affects the odour emission rate not the odour concentration.

- uncertainty of odour analysis according to EN 13725
- uncertainty associated with determining the dilution ratio during pre-dilution

The following sources of uncertainty are also associated with the technique, but are not directly quantifiable:

- The uncertainty associated with temporal variation in odour concentration within the stack, due to changes in process conditions.

Note: this is likely to have the largest effect on the uncertainty.

- The uncertainty associated with the conservation of the sample whilst the sample is drawn from the sampled gas flow to the sample container (odour sampling bag).
- The uncertainty associated with the conservation of the sample during transportation and storage.
- The uncertainty associated with non-compliance with the sample location (e.g. positioning of sample plane or sample point in locations that are not representative of the stack gas).

It is important that the sample location allows representative sampling. Further information is provided in Guidance Note on Site Safety Requirements for Air Emissions Monitoring (AG1).

It is very difficult to assess the effect of a non-compliant sample location, so by convention it shall not be included in the reported measurement uncertainty. However, it shall clearly state in the measurement report that the location was non-compliant (Emission Monitoring Guidance Note, AG2).

5.2 Accuracy – statistical model

No additional requirements to EN 13725.

5.3 Overall sensory quality requirements

No additional requirements to EN 13725.

5.4 Quality requirements for dilution apparatus

No additional requirements to EN 13725.

5.5 Quality requirements for dilution apparatus

No additional requirements to EN 13725.

6 Materials, gases and panel members

6.1 General properties of materials

No additional requirements to EN 13725.

6.2 Sampling equipment

6.2.1 General

No additional requirements to EN 13725.

6.2.2 Materials for sampling equipment

No additional requirements to EN 13725.

6.2.3 Conditioning of sampling equipment

No additional requirements to EN 13725.

6.2.4 Cleaning and re-use of equipment

Sample lines and dilution probes shall not be reused prior to cleaning and shall only be reused for collection of samples from a single source, where the odour is expected to remain at a stable concentration (i.e. the process is under the same stable operating conditions).

The cleaning procedure depends on the type of odorous contamination and sampling equipment used. EN 13725 provides an example of a cleaning procedure in section 6.2.4. The following are examples of other cleaning procedures that could be applied, especially for equipment that cannot be taken apart (e.g. swage lock valves):

- cleaning in an ultrasonic bath with 3% hydrogen peroxide solution
- flushing with 3% hydrogen peroxide solution
- heating to at least 100°C for more than three hours

For equipment that can be taken apart, soaking with a cleaning product, followed by manual scrubbing may be used.

Detergents, bleach and dishwasher cleaning products that have an odour shall not be used.

Note: odourless non-foaming soap is a suitable cleaning product (e.g. Decon 90).

Following cleaning, the equipment parts shall be flushed with neutral gas during drying. The odour neutrality of the equipment shall be confirmed by checking the air used after flushing for odour neutrality.

6.3 Sample container

6.3.1 Materials for sample container (bags)

EN 13725 lists materials that are suitable for sample containers. The following may also be used:

- electropolished stainless steel
- glass.

EN 13725 includes a note which states that polyvinylfluoride (PVF, Tedlar) may not be suitable because it has the potential to release solvents. These bags shall not be used unless the material has been tested for suitability.

Materials shall be tested for suitability by assessing if they are odourless and if they can hold a mixture of odorants with minimal changes for periods of storage (see 6.3.2).

6.3.2 Conditioning and testing of sample containers

It is recommended that sample containers are conditioned on-site immediately prior to use by flushing with a sample of the stack gas. This can be done by partially filling the container with sample and then evacuating the container manually or using a pump to ensure it is completely empty prior to sample collection.

If pre-dilution equipment is used, it is a requirement that the sample train is flushed with the diluted sample at the target dilution factor. The duration of flushing shall be determined on the basis of the length of the sampling train. The flushing shall be carried out with the diluted sample at the target dilution factor.

6.3.3 Cleaning and re-use of sample containers.

No additional requirements to EN 13725.

6.4 Gases

6.4.1 Neutral gas for pre-dilution

No additional requirements to EN 13725.

6.4.2 Reference material: odorant (n-butanol)

No additional requirements to EN 13725.

6.4.3 Reference material for calibration of dilution equipment

No additional requirements to EN 13725.

6.5 Dilution apparatus

Appropriate materials shall be used for those parts of the dilution apparatus that are in contact with the odour sample. The materials described in section 6.2.2 of EN 13725 are suitable.

6.6 Environment for observation by assessors

No additional requirements to EN 13725.

6.7 Panel

No additional requirements to EN 13725.

7 Sampling

7.1 General

Design of sampling surveys and pre-site planning

(i) Health and Safety

Specific detailed consideration shall be given to ensuring safe access for the sample team to collect samples, the safety of staff involved in sample transportation, and the safety of the analysis panel.

Information on sampling facilities and health and safety are provided in the Guidance Note on Site Safety Requirements for Air Emissions Monitoring (AG1).and the Source Testing Association (STA) booklet 'Risk Assessment Guide: Industrial- emission monitoring'¹.

Note 1: There are many hazards associated with stack emissions monitoring. For example, working at height, substances in stack gases and hot surfaces or hot stack gases.

Olfactometry analysis involves presentation of odour samples to a panel of human assessors. The estimated concentrations of chemicals and substances contained within samples shall, where possible, be made clear on chain-of-custody forms provided by the analytical laboratory. If there is a risk to the safety of the laboratory operator or panellist, then samples containing potentially hazardous (e.g. carcinogenic) components may not be analysed.

(ii) Sample ports (for stack sampling)

The sampling point requirements for stack sampling are defined in Guidance Note on Site Safety Requirements for Air Emissions Monitoring (AG1).

(iii) Site Specific Protocol (Measurement Plan)

Planning should be conducted in accordance with the Emission Monitoring Guidance Note (AG2).

This should also include detailed consideration of:

- The number and type of odour sources to be sampled
- Access requirements
- Physical and chemical characteristics of emissions
- Potential for process variability
- Number of samples to be collected, duration and frequency of sampling

iv) Sample volume

The volume of sample required for analysis is determined by the:

- Requirements of the analytical laboratory
- Objectives of the survey
- Maximum acceptable measurement uncertainty required

v) Sampling duration and sample numbers

The duration of sampling and number of samples collected should be defined on the basis of the:

- Survey objectives
- Maximum allowable measurement uncertainty
- Potential for variations in odour emissions, due to process factors.

The typical duration for collection of a single odour sample, using the lung principle is between 4 to 30 minutes. For stable processes, with stable odour emissions, triplicate samples are recommended. Typically sampling may produce an average triplicate sample over a 30-minute time period (e.g. 3 times 10 minutes). If fewer samples are collected, the justification for this shall be clearly explained in the test report.

Sample time shall be adapted (reduced or extended) depending upon the characteristics of the stack gas emissions being sampled. This may also have implications on the total number of odour samples collected. More samples and longer samples times are required for variable processes.

A stable process may have variable odour concentrations, which may affect sample number and duration.

For assessing compliance with licence conditions, the uncertainty of the measurement technique shall be taken into account when defining the number of replicate samples to be collected.

vi) Assessing the need for sample pre-dilution

The requirement for pre-dilution shall be determined prior to sampling, by consideration of the:

- Likely chemical components and concentrations of the sampled gas

Note 1: If pre-dilution is undertaken because of the likely chemical components and concentrations of the sampled gas, it can be carried out at the point of sampling or in the laboratory after sampling. However, for highly flammable or explosive gases it is recommended that dilution is carried out on site, rather than transporting to laboratory and doing it there.

- Moisture content, pressure and temperature of sampled gas
- Ambient temperature and pressure during collection, transport and storage.

The pre-dilution factor applied shall be sufficiently large to prevent condensation during collection, storage or transport of the samples.

Note 2: It is important to consider the dilution factor carefully because the uncertainty of the method increases as the dilution factor increases.

7.2 Choice of sampling method

7.2.1 Sampling for direct olfactometry

This technique is not currently applied in Ireland and is not covered by this appendix.

7.2.2 Sampling for delayed olfactometry

Delayed olfactometry is the analytical technique used in the UK and Ireland.

Sample collection should be conducted using the lung principle or via a pre-dilution device. The direct pumping technique referenced in EN13725 shall not be conducted due to the risk of contamination of the sample.

7.3 Sampling procedures

7.3.1 Sample collection method (lung principle)

The following sample procedure shall be followed:

1. The volume flow rate, temperature and humidity of the air stream (for stack sources) shall be measured. Volume flow and temperature shall be measured in accordance with EN 16911-1.
2. The sample train shall be assembled to ensure there are no routes of leakage or ingress of dilution air.
3. The sample probe shall be positioned in the stack at a representative point.
4. The sample conditioning requirements of section 6.2.3 and 6.3.2 shall be followed.
5. The sampling vessel shall be evacuated, and the odour sample collected in the sampling container (bag).
6. Once the sampling container has been filled to the desired level the container shall be removed from the sample train, sealed and packaged for transport to the analysis laboratory.

For stacks under negative pressure, the stack sample port shall be sealed prior to undertaking sampling, to prevent ingress of dilution air.

Sample lines shall be kept as short as possible (to minimise the possibility of absorption). Sample lines and probes shall be changed between odour sources or between samples, if the characteristics of the odour source are likely to change with time.

Condensation in the extraction tube and sample line, in the dilution equipment and in the sample bag shall be prevented. If condensation occurs, the sample shall be rejected.

If a sample is rejected because of condensation, the site-specific protocol shall be modified in order to prevent it occurring again. This may require the use of dilution or increased dilution, if a dilution system was used in the first place.

If probes and sample lines are reused, they shall be cleaned and checked for odour- neutrality.

Filters shall be used if the particulate loading adversely affects the operation of the sample train.

7.3.2 Pre-dilution during sampling

Note: Pre-dilution refers to the mixing of a neutral odourless gas (i.e. nitrogen) at the time of sampling.

Post-dilution refers to dilution carried out after sampling by the analytical laboratory.

7.3.2.1 General

Pre-dilution of the sample is applied for the following reasons:

- If there is a risk of condensation of the sample in the sample line and sample container during collection
- If there is a risk of condensation of the sample during storage and transport
- To reduce the concentration of toxic or flammable components prior to transport to the laboratory
- To reduce the concentration of the odour to within the range of the olfactometer used by the analytical laboratory.

7.3.2.2 Equipment and procedures for static pre-dilution during sampling

For sources under negative or positive pressure it is recommended that dynamic pre- dilution should be used.

Caution should be applied when using static dilution to reduce the temperature of the sample to below dew point, as it can be difficult to entirely eliminate the risk of condensation during mixing of the sample with neutral gas in a sample container.

7.3.2.3 Equipment and procedures for dynamic pre-dilution during sampling

Note 1: although referred to as dynamic dilution, the actual dilution rate is fixed during the test. Alternatively, it can be referred to as dilution at source.

Dynamic pre-dilution is preferably performed in a pre-dilution unit (e.g. injector), which can be situated in the gas flow to ensure that the pre-dilution gas and sample gas are under the same conditions in terms of temperature and pressure. If pre-dilution is performed outside the gas flow, the probe and sampling line shall be heated until pre-dilution is carried out to prevent condensation. In such circumstances, it may also be necessary to equalise the pressure between the probe and duct, depending upon the design of the dilution probe.

If the stack gas emissions are at ambient temperatures it is unlikely that condensation will occur. This means the pre-dilution unit does not have to be in the stack. This approach can be used when the size of the access port is too small for the head.

An estimated dilution ratio of the equipment shall be determined by means of the sample flow rate and dilution settings. It is also dependent on the temperature of the dilution equipment.

The accuracy of the dilution ratio shall be checked after collection of each sample. This is achieved either by calibration of the dilution probe using the procedure described in section 5.4 of EN 13725 or by measurement of the concentration of a defined indicator gas that is present within the undiluted and diluted odour sample (e.g. oxygen, carbon monoxide, carbon dioxide).

Olfactometry analysis involves presentation of odour samples to a panel of human assessors. The type and concentration of any tracer gas (e.g. carbon monoxide) shall be made clear on the chain-of-custody

form provided by the analytical laboratory. Samples with high concentrations of potentially hazardous components may not be analysed where there is a risk to the safety of the laboratory operator or panellist.

Note 2: a potential error in the calibrated flow meters approach is the impact of stack gas pressure on the dilution factor. One proven way of determining the dilution factor is to measure stack gas oxygen and oxygen measured in the odour bag.

This indicator gas method of determining the pre-dilution factor can only be applied if the indicator concentration in the pre-diluted sample differs from that in the waste gas and the pre-dilution gas. A pre-dilution gas shall be used that does not contain the indicator gas (e.g. nitrogen).

The concentration of the selected indicator gas shall be determined during sampling of the waste gas and immediately after sample collection for each of the pre-diluted samples. If the indicator gas concentration in the waste gas is constant and no concentration variations are expected, a measurement prior to and following sample collection shall be sufficient for determination of the marker concentration in the waste gas. If the indicator gas concentration in the waste gas is not constant over time (e.g. combustion gas process), the indicator gas concentration shall be determined over the entire sampling period.

The pre-dilution factor Z is determined using the following equation:

$$Z_V = \frac{C_{\text{Ind, waste gas}} - C_{\text{Ind, predilution gas}}}{C_{\text{Ind, sample(pred.)}} - C_{\text{Ind, predilution gas}}} \quad (1)$$

If the indicator gas is not contained in the pre-dilution gas, the following simplified equation applies:

$$Z_V = \frac{C_{\text{Ind, waste gas}}}{C_{\text{Ind, sample(pred.)}}} \quad (2)$$

Key

Z_V = Pre-dilution factor

C = Concentration

Ind = Indicator concentration Sample (pred.) = Pre-diluted sample

The analyser employed to measure the indicator gas concentration shall be checked each day of the measurement with a zero gas and a span gas at an appropriate concentration as per standard gas reference methods. The zero and span drift criteria used in these methods shall be applied.

Note 3: If oxygen is used to determine the pre-dilution factor, the check can be carried out with ambient air (20.9% O_2) and nitrogen (0% O_2).

When using oxygen as a tracer gas, the procedure specified EN 14789² shall be followed (other reference methods are applicable if carbon monoxide or carbon dioxide are used as tracer gases).

The analyser shall have an expanded uncertainty of $\pm 3\%$ or less.

The limit of detection of the indicator gas analyser will impact on the dilution factor that can be achieved. Care should be taken to ensure that the sample is not over-diluted as this can lead to an over-estimation of odour concentration if the dilution reduces the concentration of the sample to below the limit of detection of the analytical technique.

All odour concentration data shall be expressed in accordance with the reference conditions described in EN 13725 (i.e. 20°C, 101.3 kPa on a wet basis).

Note 4: For ducts under high negative or positive pressure the use of pre-dilution equipment may not be possible or adversely impacted. This can be confirmed prior to undertaking sampling.

7.3.3 Transport and storage before measurement

All samples shall be clearly labelled with a unique sample identification code on collection. The samples shall be protected. Storage and transportation in a rigid container is recommended, as it prevents mechanical damage.

Consideration and control shall be applied to the following factors to preserve the integrity of the sample during transport:

- Prevention of condensation due to changes in the internal and external temperature of the sample container
- Leakage or air ingress due to pressure changes (e.g. when transported by air)
- Photochemical reactions (samples shall not be exposed to direct sunlight or strong daylight).

A full chain of custody shall be maintained for the odour sample bags from procurement from the supplier through use on site and arrival at the analysis laboratory. The chain of custody shall include details of how the transport and storage issues are controlled and mitigated.

EN 13725 stipulates a maximum temperature during storage of 25°C. This temperature may be exceeded (for example, when working in hot conditions) provided the deviation from the standard is justified in the final monitoring report. As this is unavoidable when working in hot conditions, it is acceptable to claim accreditation, in spite of this deviation.

7.4 Sampling strategy

No additional requirements to EN 13725.

8 Presentation of odorants to assessors

No additional requirements to EN 13725.

9 Data recording, calculation and reporting

9.1 Minimum data recording requirements

No additional requirements to EN 13725.

9.2 Calculation of odour concentration of a sample from a set of panel member responses

No additional requirements to EN 13725.

9.3 Calculation of odour flow from odour concentration and odour flow rate

No additional requirements to EN 13725.

9.4 Calculation of odour abatement efficiency

No additional requirements to EN 13725.

9.5 Presentation and minimum requirements for reporting and archiving results

No additional requirements to EN 13725.

9.5.1 The minimum data for reporting

If carrying out accredited work, the monitoring report shall be submitted in the report format specified in the Emission Monitoring Guidance Note (AG2).

9.5.2 The minimum data for archiving

No additional requirements to EN 13725.

Annex A (normative) Working conditions and working platform for sampling

Sample locations and facilities should meet the requirements of EN 15259 and Guidance Note on Site Safety Requirements for Air Emissions Monitoring (AG1).

Annex B (informative) Physiological principles

No additional information to EN 13725.

Annex C (informative) Example of calculation of instrumental accuracy and instability

No additional information to EN 13725.

Annex D (informative) Example of calculation of odour measurements within one laboratory

No additional information to EN 13725.

Annex E (informative) Example of calculations for panel selection

No additional information to EN 13725.

Annex F (informative) Example of the calculation of the odour concentration from a set of panel member responses

No additional information to EN 13725.

Annex G (informative) Example of the calculation used to determine the number of odour concentration measurements required to achieve a defined precision

No additional information to EN 13725.

Annex H (informative) Example of the calculation used to determine the number of odour concentration measurements required to detect a difference between two means

No additional requirements to EN 13725.

Annex I (informative) Example of the calculation of the odour flow rate (standard conditions) for a wet emission

Unlike in stack emissions monitoring, odour emissions are standardised at 20°C (293K), rather than 0°C (273k).

Annex J (informative) Sampling strategy

No additional information to EN 13725.

References

1. Source Testing Association (STA) booklet 'Risk Assessment Guide: Industrial-emission monitoring'.
2. EN 14789:2017 Stationary Source Emissions – Determination of volume concentration of oxygen.

Appendix 7 [\(GOTO CONTENTS\)](#)

Supplementary Information to EN 13284-1 (Stationary source emissions – Determination of low range mass concentration of dust)

This appendix supplements EN 13284-1⁽¹⁾. However, it does not re-state all the provisions of EN 13284-1 and organisations are reminded of the need to comply with the criteria detailed in EN 13284-1. The clause numbers in this document follow those of EN 13284-1, although the text from the standard is not repeated in this document. EN 13284-1 remains the authoritative document and in cases of dispute, the accreditation body will adjudicate on unresolved matters.

1 Scope

No additional requirements to EN 13284-1.

2 Normative references

No additional requirements to EN 13284-1.

3 Terms and Definitions

No additional requirements to EN 13284-1.

4 Symbols and abbreviations

No additional requirements to EN 13284-1.

5 Principle

No additional requirements to EN 13284-1.

6 Measurement planning and sampling strategy

6.1 Measurement planning

No additional requirements to EN 13284-1.

6.2 Sampling strategy

- a) The maximum (angle) of flow with regard to the stack axis shall be measured and recorded for each sample point (see Box 1 below). If the angle of flow at any of the sample points is greater than 15° with regard to the stack axis, the flow criteria are not in compliance with the standard. In the absence of an alternative, compliant sampling location, the sample points at which the angle of flow is not compliant shall not be sampled. This sampling deviation shall be included in the sample report.
- b) The standard states that the minimum velocity of the stack gas must generate a differential pressure larger than 5Pa if a Pitot tube is used to carry out the measurement. However, it is possible to carry out sampling at lower velocities using an alternative measurement device, for example an anemometer, provided that the velocity of the stack gas is above the limit of detection of the measurement device and it has been calibrated within the region of the expected velocity.
- c) If local negative flow is present, then the sampling location is not in compliance with the standard. If an alternative location cannot be found, then the sample point(s) in areas of negative flow shall not be sampled. This sampling deviation shall be included in the sampling report.

If requirements (a) – (c) of the standard regarding the angle of flow and velocity cannot be met the sampling location is not compliant with the standard. The client shall be informed that the location does not comply with the standard (see 10.2 of the standard). An alternative compliant location shall be sought.

If, after consultation with the client, a suitable sampling location cannot be found then the number of sampling points on the sampling plane shall be doubled (up to a maximum of 20 sample points is normally sufficient). When the number of sample points is doubled a traverse shall be repeated to determine which ones are compliant. The sampling points, which have negative flow, or the angle of flow is not less than 15° shall not be sampled.

It must be stated in the monitoring report that the flow criteria are not in compliance with the standard.

If a suitable sample location cannot be found, the estimation of the uncertainty relating to the results of a specific stack emission measurement exercise is complicated and not possible to quantify. This means that the effect of the sample location on the measurement uncertainty shall not be taken into account when reporting the uncertainty. Under these circumstances, explanatory remarks justifying the deviations from the standard shall be included in the monitoring report and it shall be stated clearly in the report that the sample location requirements were not met and, therefore, that the results were not produced in compliance with the standard.

An approach to conducting a 'swirl test' with an S type Pitot tube is given below:

1. *Level and zero the manometer*
2. *Connect a S Type Pitot tube to the manometer and leak-check the system*
3. *Position the S Type Pitot tube at each traverse point, in succession, so that the planes of the face openings of the Pitot tube are perpendicular to the stack cross-sectional plane (when the S Type Pitot tube is in this position, it is at 0° reference)*
4. *Note the differential pressure reading at each traverse point*
5. *If a null (zero) Pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point*
6. *If the Pitot reading is not zero at 0° reference, rotate the Pitot tube (up to $\pm 90^{\circ}$ yaw angle), until a null reading is obtained*
7. *Determine that the angle of rotation is less than 15° at each sample point*
8. *Apply the swirl test to each sample point*
9. *If the value of rotation is less than 15° at each sample point, the overall flow condition in the stack is acceptable.*

Note: US EPA M1⁽²⁾ and ISO 10780⁽³⁾ both provide details of how to carry out a swirl tests using an S Type Pitot.

Conducting a swirl test with an L Type Pitot

The following approach is reproduced from ISO 10780:1994

The type L Pitot provides an accurate measure of gas velocity when the Pitot tube is aligned within 15° of the direction of gas flow. However, the pressure difference between the two pressure-sensing orifices decreases sharply when misalignment exceeds 15° , until a negative response occurs when the head is at 90° to the flow. This provides a simple method for estimating gas flow direction and may be used to test for the presence of swirling flow within the duct.

7 Equipment and materials

No additional requirements to EN 13284-1.

8 Weighing procedure

No additional requirements to EN 13284-1.

9 Sampling procedure

There are two approaches to obtaining a water vapour concentration:

Option 1: The water vapour concentration is measured before particulate monitoring using EN 14790⁽⁴⁾. This concentration is used to determine the isokinetic rate during the test. It is also used when calculating the measurement result(s), provided the water vapour conditions are stable and well characterised.

Note 1: if the water vapour concentration is variable, there is a risk that the water vapour for the process being monitored may change. This may lead to the isokinetic rate during the test being incorrect, which may mean the result(s) does not comply with EN 13284-1.

Note 2: CEMs data may show that a process has stable water vapour conditions.

Option 2: The water vapour concentration is determined before the particulate test and also during the particulate test. The water vapour concentration determined before the test is used to estimate the isokinetic rate required during the particulate test. The water vapour concentration measured during the particulate test is used for determining the water vapour to be applied when calculating the measurement result(s).

When using Option 2 is it important that the water vapour concentration used to calculate the isokinetic rate is reliable. This concentration can be obtained in the following ways:

- carry out sampling in accordance with EN 14790 (this approach shall be used on a stack where the water vapour concentration is unknown or where the water vapour concentration is variable);
- use an expected water vapour concentration based on previous monitoring campaigns or where it is known that the water vapour concentration is at ambient conditions (this shall only be done if the water vapour conditions are stable and well characterised).

Note 3: if the water vapour concentration is estimated before the particulate test using information from previous monitoring campaigns, there is a risk that the water vapour for the process being monitored may have changed. This may lead to the isokinetic rate during the test(s) not meeting the criteria specified in EN 13284-1. For stable and well characterised stack gas emissions, where it is known that the water vapour concentration is at ambient conditions or has been previously confirmed to be less than 5% volume/volume concentration, it is not necessary to determine the water vapour concentration by measurement.

If droplets are present in stack gas emissions, Option 1 shall be followed, using Annex A of EN 14790 to determine the water vapour concentration.

Note 4: the approach used in Annex A is for the measurement of water vapour only. It does not include the droplets.

10 Calculation

No additional requirements to EN 13284-1.

11 Measurement report

No additional requirements to EN 13284-1.

Annexes A - H

No additional requirements to EN 13284-1.

References

- (1) EN 13284-1:2017 - Determination of low range mass concentration of dust – Part 1:
Manual gravimetric method
- (2) US EPA Method 1 Determination of stack gas velocity and volumetric flow rate (Type S Pitot tube).
Available from the US EPA website.
- (3) ISO 10780:1994 Stationary source emissions – Measurement of velocity and volume flowrate of gas streams in ducts.
- (4) EN 14790:2017 Stationary source emissions – determination of water vapour in ducts.