



**Technical Guidance Note  
(Monitoring) M22**

**Measuring stack gas emissions  
using FTIR instruments**

**Environment Agency**

**Version 3**

**March 2012**



## **Foreword**

We have issued Technical Guidance Note (TGN) M22, within our series of monitoring TGNs, to provide guidance on the Environment Agency's requirements when using manual extractive Fourier Transform Infrared (FTIR) spectroscopic instruments to measure emissions of pollutants from industrial stacks. It is not applicable to FTIR used for continuous emissions monitoring.

FTIR instruments are widely used for stack emissions monitoring. The ability to record spectra over a wide wavelength range, and analyse these to identify a number of different pollutants in a stack gas makes FTIR a useful technique.

This TGN provides a procedure and framework to ensure the quality control and quality assurance of measurements made using FTIR as an alternative to several European standard reference methods.

## Record of amendments

Version number	Date	Section	Amendment
3	March12	4.2	Added text restricting the use of TGN M22 as an alternative method to EN 12619 or EN 13526
		4.2	Removed response time requirement for NH <sub>3</sub> , HCl and HF in Table 1
		4.2	Stated in Table 1 that the response time and annual lack of fit check is only required for determinands with applicable SRMs
		7.4.2	Included information on selecting check gas concentrations for regulatory monitoring
		7.4.2	Stated that the most reactive gas check is carried out before and after sampling
		7.4.2	Added information on reactive gases, including a table listing gases according to reactivity
		7.4.2	Deleted requirement to calculate a sample system lag time
		7.4.3.1	Clarified that the most reactive gas check is required before and after the measurement period
		7.4.3.1	Amended criteria for the reactive gas check, when using a very reactive gas
		7.4.3.1	Stated that all the results are rejected if a reactive gas check fails
		7.5	Restated that all the results are rejected if a reactive gas check fails
		7.5.1	Stated that the most reactive gas test is also carried out at the end of the measurement period
		7.6	Clarified that correction only applies to span drift
		7.7	Added note about using EN 14790 to measure water vapour
		8.3	Clarified requirement on the response time check in Table 2
		8.3	Stated in Table 2 that the annual lack of fit check is only required for determinands with applicable SRMs
		Annex A	Added definition for measurement period
		Annex A	Amended definition of monitoring campaign
		Annex A	Amended definition of reactivity
		Annex D	Deleted requirement to calculate a sample system lag time
Annex D	Amended requirement on when the lack of fit test is required		

Version number	Date	Section	Amendment
3	March 12	Annex F	Updated Table 3: Example analysis bands (Gasmeter DX4000)
			Added note about use of HCl for the reactive gas check

### **Status of this document**

This TGN may be subject to review and amendment following publication. The latest version is available on our website at:

[www.mcerts.net](http://www.mcerts.net)

### **Implementation date**

It is expected that organisations, which hold MCERTS accreditation for TGN M22, will have met the requirements of this version of TGN M22 by September 2012.

### **Feedback**

Any comments or suggested improvements to this TGN should be e-mailed to Rupert Standing at [rupert.standing@environment-agency.gov.uk](mailto:rupert.standing@environment-agency.gov.uk)

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## **1 Introduction**

### **1.1 Scope**

This Technical Guidance Note specifies the requirements for the use of manual extractive Fourier Transform Infrared (FTIR) spectroscopy for the speciation and quantification of gases monitored within stacks. The requirements described include:

- sampling configuration
- annual analyser checks
- ongoing quality assurance and quality control.

It provides a method for the use of FTIR to carry out measurements in support of the Waste Incineration Directive<sup>1</sup> (WID) and Large Combustion Plant Directive<sup>2</sup> (LCPD). It stipulates that the requirements described in these Directives and from other relevant European standards are met. It is also applicable to other applications, as it provides instrument requirements and operational procedures to ensure the production of valid monitoring results.

It applies to FTIR instruments configured for extractive sampling and measurement.

Note 1: TGN M22 is not applicable to continuous emissions monitoring systems.

TGN M22 may also be used as an alternative method (AM) to several European standard reference methods (SRMs), provided the user demonstrates equivalence according to BS/EN TS 14793<sup>3</sup>.

If this TGN is followed using an FTIR instrument certified according to MCERTS (EN 15267-3<sup>4</sup>) then the instrument may be considered suitable for use within an AM for the determinands covered by the certification, without any additional testing for compliance with the applicable SRMs.

It is recognised that FTIR may be used for carrying out investigative work on unknown or poorly characterised stack gas mixtures. This type of work is outside the scope of this TGN.

Note 2: Annex A provides a glossary of terms and definitions.

## **2 Principle of operation**

The FTIR spectrometer provides a measurement in the infrared (IR) region over a wide spectral band. Analysis of the recorded spectra enables the concentration of a wide number of determinands to be determined simultaneously. In principle any compound that has an absorption spectrum in the IR region can be measured by FTIR. Determinands that cannot be measured by FTIR are symmetrical diatomic molecules, where there is no dipole change upon vibration, such as oxygen and chlorine.

One issue with the analysis of FTIR spectra is that the spectra of different determinands may overlap, leading to potential problems with cross-interference and miss-identification. Water vapour is a common interferent, as it has absorption features across most of the IR region.

This TGN defines a “calibration range” and an MCERTS “certification range”, the former being selected by the user and the latter being stipulated by the regulation covering the plant to be monitored.

The user may calibrate the range for a given determinand over as wide a range as desired by acquiring reference spectra at appropriate concentration increments. If the instrument were used at a plant covered by WID, the certification range must be less than 1.5x the emission limit value (ELV) of each pollutant being measured (for other applications it must be less than 2.5x the ELV). Consequently, it is strongly recommended that testing carried out to demonstrate meeting of the performance requirements across the operational range is designed so that it is also possible to demonstrate meeting of the performance requirements across all likely certification ranges.

## **3 Sampling system**

### **3.1 General requirements**

Sampling shall comply with the requirements of BS EN 15259<sup>5</sup>.

A representative volume of gas is extracted from the stack at a controlled flow rate into a sampling system consisting of:

- sampling probe
- filter
- sampling line
- temperature and pressure sensors (unless already part of the FTIR analyser)
- pump

If a hot / wet system is used the probe, filter box and sampling line will generally be heated to a similar temperature as the gas cell. Furthermore, the selected temperature shall be at least 20°C above the dew point of the sampled stack gas.

Annex B gives examples of sample configurations, including systems that use sample conditioning units to remove moisture.



All parts of the sampling system positioned upstream of the FTIR analyser shall be made of materials that do not react with or absorb any of the determinands.

Note: The temperature and pressure in the FTIR gas cell are important parameters as they directly affect the determined concentrations. These parameters also help confirm that condensation of the sample in the gas cell is prevented. In contrast, the flow rate through the gas cell does not affect the determined concentration if all other parameters remain unchanged. Consequently, a continuous measurement of the flow rate is not a requirement of this TGN.

### **3.2 Apparatus requirements**

Information on materials suitable for sampling can be found under BS ISO 10396<sup>6</sup>.

#### **3.2.1 Sampling probe**

The sampling probe shall be able to convey a representative sample of the stack gas to the FTIR measurement cell. It may contain a filter to remove particulate matter. The probe shall be able to resist absorbance of determinands and be of sufficient length.

#### **3.2.2 Filter**

A filter of inert material shall be positioned after or as part of the sample probe and be of an appropriate pore size (3 µm is usually sufficient) to prevent particulate matter entering the remainder of the system. The filter shall be periodically changed or cleaned in order to ensure adequate functionality. If the filter is located outside the stack, it shall be heated to at least 20°C above the stack gas dew point temperature.

Note: An increase in the pressure drop across the filter is likely to indicate a significant number of blocked pores and the need for replacement / cleaning.

#### **3.2.3 Sampling line**

To facilitate system response time the sampling line shall be as short as is practical. The line shall be made of inert material that is able to resist the absorbance of determinands.

## **4 Determination of the characteristics of the analyser and sampling system**

### **4.1 General**

Annex C describes how the capability to measure a new determinand is added to the model / analyser calibration.

Annex D describes the methodology for carrying out performance characteristic tests.

## 4.2 Monitoring determinands covered by SRMs

The methods given below are SRMs for determinands that can be measured using FTIR. This section (4.2) is applicable to these determinands.

Applicable SRM	Substance
EN 14790	Water vapour
EN 1911	Hydrogen chloride
EN 14791	Sulphur dioxide
ISO 15713	Hydrogen fluoride
EN 14792	Oxides of nitrogen (nitric oxide and nitrogen dioxide)
EN 15058	Carbon monoxide
EN 12619	Gaseous organic carbon (total) - low concentrations
EN 13526	Gaseous organic carbon (total) – high concentrations

If the instrument has been certified under the MCERTS<sup>7</sup> scheme then it is considered suitable for use within this reference method for any determinand included in the certification, provided the instrument has been certified at an appropriate range for the application (see the *MCERTS Manual stack emission monitoring performance standard for organisations*<sup>8</sup> for information on the use of MCERTS certified instruments).

Note: for a plant covered by WID the required MCERTS certification range is less than 1.5x the ELV of each pollutant being measured. For other applications it is less than 2.5x the ELV.

The tests under *certification / commissioning* in Table 1 are completed during the certification process.

If the instrument is used to carry out quality assurance of CEMs according to EN14181 (i.e. parallel reference measurements) it must be MCERTS certified at the appropriate range.

For compliance monitoring, if the analyser does not have MCERTS certification for a determinand covered by an SRM then equivalency to the appropriate SRM shall be demonstrated by an accredited test house in accordance with BS EN/TS 14793. Furthermore, the test house shall demonstrate that the performance criteria in Table 1 under *certification / commissioning* are met.

At the time of publication, no FTIR systems have been MCERTS certified for the measurement of gaseous organic carbon (total). Therefore, TGN M22 cannot be used as an alternative method to EN 12619 or EN 13526 for quality assurance of CEMs according to EN 14181. For compliance monitoring equivalency to the SRM shall be demonstrated by an accredited test house in accordance with BS EN/TS 14793.

Table 1 – Minimum performance requirements for monitoring<sup>a</sup>

Performance characteristics	Certification / commissioning tests	Annual tests	Monitoring period tests	Performance criterion <sup>b</sup>	Performance characteristics to be included in calculation of overall uncertainty
Response time	✓	✓ <sup>c</sup>	✓	< 200 s (this does not apply to NH <sub>3</sub> , HCl and HF)	
Detection limit	✓		✓	< ± 2,0%	
Lack of fit	✓	✓ <sup>c</sup>		< ± 2,0%	✓
Zero check			✓	< ± 2,0%	
Check gas (losses and leakage in the sampling system)			✓	< ± 5,0%	
Zero drift	✓ <sup>d</sup>			< ± 2,0% / 24 h	✓
Span drift	✓ <sup>d</sup>			< ± 2,0% / 24h	✓
Sensitivity to atmospheric pressure	✓ <sup>e</sup>			< ± 3,0% / 2 kPa	✓
Sensitivity to sample gas pressure	✓ <sup>e</sup>			<± 2,0% / 3 kPa	✓
Sensitivity to ambient temperature	✓ <sup>e</sup>			<± 3,0% / 10 K	✓
Sensitivity to electrical voltage	✓ <sup>e</sup>			<± 2,0% / 10 V	✓
Interferents	✓ <sup>f</sup>			Total ± 4,0%	✓
Standard deviation of repeatability in laboratory at zero	✓ <sup>g</sup>			<± 1,0%	✓
Standard deviation of repeatability in laboratory at span level	✓ <sup>g</sup>			<± 2,0%	✓

<sup>a</sup>Table 1 applies to all determinands that a monitoring organisation has in the scope of their MCERTS accreditation for FTIR. It also applies to the measurement of water vapour by FTIR.

<sup>b</sup>Expressed as % of certification or calibration range as appropriate.

<sup>c</sup>Only required for determinands with applicable SRMs.

<sup>d</sup>If the model is certified under MCERTS for one or more determinands, tests are not required for those determinands. When monitoring a determinand, for which the analyser is not certified under MCERTS, demonstration of equivalence to the SRM by carrying out a drift test in accordance with BS EN/TS 14793 is required.

<sup>e</sup>Only required to be carried out once for one determinand, as the parameters are model specific.

<sup>f</sup>Interferents are given in EN 15267-3.

<sup>g</sup>Only one of these values shall be included in the overall uncertainty calculation. The first possibility is to choose the repeatability standard deviation obtained from laboratory tests corresponding to the closest concentration to the actual concentration in stack, or the higher (relative) standard deviation of repeatability independently of the concentration measured in stack.

#### 4.3 Monitoring determinands not covered by a SRM

Performance characteristic tests for determinands not covered by a SRM may be carried out either by an approved test house or by the user employing a suitably certified gas delivery system (a liquid injection system may be used for determinands, such as water vapour). For these determinands the minimum performance tests required under this TGN are:

- response time
- detection limit
- lack of fit
- interferents

All analysers shall undergo the tests indicated under *annual* and *monitoring period* at the frequency indicated in Table 2.

#### 5 Performance requirements of the method

The uncertainty of the measured value for the method is influenced by the performance characteristics of the analyser and by the:

- sampling line
- site specific conditions
- calibration gases used

#### 6 Establishment of the uncertainty budget

An uncertainty budget shall be established following Annex E for each determinand measured. The determined expanded uncertainty shall meet the required maximum allowable overall uncertainty where it exists for a given determinand in accordance with DD CEN/TS ISO 14956<sup>9</sup> and BS ENV 13005<sup>10</sup>.

For substances covered by the WID or LCPD the expanded uncertainty calculated by combining values of standard uncertainties associated with the performance characteristics given in Table 1 shall be less than the value specified in the relevant CEN SRMs expressed at the daily emission limit value, on dry basis and before correction to the O<sub>2</sub> reference concentration.

## **7 Field operation**

### **7.1 Sampling location**

The sampling locations shall meet the requirements of BS EN 15259 and provide representative samples. In addition, the sampling location shall be chosen with regard to safety of the personnel, accessibility and availability of electrical power. Further guidance is provided in TGN M1<sup>11</sup>.

### **7.2 Sampling point**

BS EN 15259 describes the approach for selecting a sample point(s).

### **7.3 Choice of the measuring system**

The following characteristics of stack gas shall be known before the monitoring campaign:

- temperature
- moisture content
- particulate matter loading
- the expected concentration ranges and emission limit values (where applicable)
- the expected concentration of potentially interfering substances, including at least the components listed in BS EN 15267-3.

The measuring system shall have performance data, such as MCERTS certification, to demonstrate that its suitable for each determinand in the measurement campaign.

Note: information about the application of MCERTS certified instruments for manual stack emissions monitoring are given in the *MCERTS manual stack emissions performance standard for organisations*<sup>8</sup>.

To avoid long response times, the sample line should be as short as possible. If necessary a bypass pump may be used. A heated filter appropriate to the dust loading shall be used.

### **7.4 Setting up of the analyser on site**

#### **7.4.1 General**

The complete measuring system shall be connected according to the manufacturer's instructions.

The FTIR sample cell shall be kept at the manufacturer's specified temperature and pressure. Alternatively, pressure and temperature correction is permitted if its demonstrated that the relevant performance requirements can be met whilst this correction is applied.

The sampling probe, filter, connection tube and analyser shall be stabilised at the required temperature.

After pre-heating, the flow passing through the sampling system and the analyser shall be adjusted to the chosen flow rate to be used during measurement.

Any data recording, data processing and telemetry system used in conjunction with the measuring system shall be checked for proper functioning. If any components are changed, then these checks shall be repeated. All checks shall be documented.

#### **7.4.2 Selection of check gas**

Suitable check gases shall be selected (from the target components, where possible) to demonstrate that the FTIR is capable of producing valid and accurate spectra at the frequencies and absorbance ranges that the analytical model will be using.

Note: The check gas should not be considered as a traditional span of its constituent components (as no adjustment to the analyser is made) but rather an intrinsic validation of the analyser functionality.

If check gas cylinders for the target components are not commonly available, alternative check gases that have equivalent spectral characteristics may be used. Where this approach is used the test laboratories shall justify it in the site specific protocol and monitoring report.

Check gases used for each stack matrix shall meet each of the following criteria:

- Any component specific peak greater than 10 times the LOD associated with a check gas, shall lie within  $\pm 25\%$  of the centre of a target analyte analytical wave number band. Where multiple analysis bands are used for a target analyte, this requirement shall be met for at least one of these bands.
- The absorption bands above shall exhibit peak absorbance greater than 10 times the lower detection limit but less than 1.5 absorbance units.
- At least one absorption validation gas band within the operating range of the FTIR instrument shall have an instrument-limited line width no greater than the narrowest analyte absorption band. Perform and document measurements or cite studies to determine analyte and check gas compound line widths.
- Ideally, for regulatory compliance monitoring the check gas concentration should be approximately the ELV (half hourly ELV). Due to practical considerations this may be difficult to achieve. Therefore, the check gas shall not have a concentration of over three times the ELV.

In addition, check gases must also be selected to demonstrate that the sample system is free from leaks and can transport the sample to the FTIR without any losses or contamination.

The most reactive of all the target components shall be selected for this purpose and passed through the entire sample system before and after the measurement period. However, if check gas cylinders for the most reactive target components are not commonly available, an alternative gas that has equivalent or greater reactivity may be used. Where this approach is used the test laboratories shall justify it in the site specific protocol and monitoring report.

The table below shall be used for ranking gases according to reactivity. The gases that are listed below the line in the table shall meet the criteria given in Section 7.4.3. Those gases above the line (HF, HCl and NH<sub>3</sub>) are considered to be very reactive, so the procedures in section 7.4.3.1 may be used as an alternative to section 7.4.3.

**Table of gases ordered according to reactivity<sup>1</sup>**

Gas (ranked from most to least reactive)	Section of TGN M22 applicable to use of this gas
HF <sup>2</sup> HCl NH <sub>3</sub>	7.4.3.1
SO <sub>2</sub> NO <sub>2</sub> CO <sub>2</sub> N <sub>2</sub> O NO CO	7.4.3

<sup>1</sup>In this table the term reactivity is based on experience from stack emissions monitoring. See Annex A for a definition of reactivity of gases.

<sup>2</sup>Due to the difficulty in handling HF, it is ranked as having equal reactivity to HCl.

All the check gases, wherever possible, shall be traceable and certified with a measurement uncertainty of  $\leq \pm 2\%$  of value with a 95% confidence level.

Note 2: It may be necessary to choose preliminary analytical regions, identify the minimum analyte line widths, or estimate the system noise level first. More than one compound may be needed to meet the criteria.

Note 3: Where compatibility allows, a suite of gases may be applied as a single cylinder composition.

Test laboratories shall have documented procedures, which describe how the above requirements are applied, stating the gases used, the frequencies of the specific peaks, and how these peaks apply to the gases measured.

Annex F provides examples of the selection of suitable check gases for two FTIR instruments routinely used for periodic stack emissions monitoring in the UK.

### **7.4.3 Start of monitoring period zero and check gas tests, detection limit and response time determination**

The zero gas shall be a gas containing no significant amount of the determinands, for example, purified nitrogen or synthetic air (synthetic air being N<sub>2</sub> and O<sub>2</sub>).

The zero gas shall be measured once. It is not a requirement to measure the zero gas between each check gas.

The following zero and check gas procedure shall be used:

- supply zero and check gases through the sampling system at the sampling probe, as close as possible to the nozzle (in front of the filter if possible);

Note 1: It may be necessary to use a t-piece between the test-gas cylinders and the probe, in order to avoid over-pressurizing the analyser.

- when applying the zero gas, record 10 spectra and determine the detection limit for each determinand for the monitoring period, as described in Annex D;
- when applying the zero and check gases, determine the  $T_{90}$  response time (see Annex D) based on the check gas with the slowest response (this shall be the analyser response time, the response time of the other check gas components need not be determined);
- wait at least three times the  $T_{90}$  value before taking the readings for zero and check gas;
- the performance requirements in Table 1 shall be met for the zero and check gases.

If the zero test does not meet the criteria the zero gas shall be disconnected from the front of the sample probe and connected directly to the inlet of the FTIR analyser gas cell. If the reported concentrations remain unaltered the sampling system is considered free from contamination. The zero gas shall be disconnected from the analyser inlet and reconnected to the front end of the sample probe.

Alternatively, if connecting the zero gas directly to the cell inlet results in reported concentrations that decrease to, or better than, the performance requirement, then contamination of the sampling system is indicated. The contamination shall be removed and the zero check test repeated.

A failed zero test may be due to insufficient purging and consequently, contamination from a previous sample. To eliminate this as a possible cause, purging should be continued and the test repeated. If the issue remains unresolved the noise level of the analyser should be checked. This may be done by visual examination of baseline noise differences between the current and previous zero gas spectra. If an increase in the noise is found the manufacturer should be consulted. If an increase in noise is not found the integrity of the zero gas and sampling system should be checked.

If the zero and check gas tests, including the sample system check, are passed then it may be considered that there are no significant sample losses or leakage.

#### **7.4.3.1 Checking the sampling system with a very reactive gas**

This section provides procedures for checking the sample system with a very reactive gas. This is not a check of the accuracy of the analyser but is a check of the sample system's ability to deliver the gas to the analyser without significant losses. If the measurement period is for a very reactive gas only, it is necessary to use the procedure in 7.4.3 to confirm the accuracy of the analyser. This shall be carried out using the very reactive gas being measured or an alternative gas(s). If an alternative gas is used, it is necessary to check the sample system using the very reactive gas.

For gases with a reactivity less than or similar to  $\text{SO}_2$ , the procedure in 7.4.3 shall be followed. However, for gases, such as  $\text{NH}_3$ ,  $\text{HCl}$  or  $\text{HF}$ , the check gas procedure in Section 7.4.3 may not be suitable due to the time it takes for a very reactive gas to reach  $T_{90}$ . Under these circumstances one of the following techniques may be used instead:

##### **Option 1: Dry cylinder gas injection**

When carrying out this option it is essential that an accurate cylinder gas reference value is established. To determine this value the most reactive gas is applied directly to the inlet of the analyser and a final stable result recorded. This can be determined in the laboratory or on-site. Once a gas cylinder has been checked, the frequency of repeat checks can be decided by the user.



Note 2: This method of checking eliminates issues of getting accurate concentrations of reactive gases from a cylinder.

The dry cylinder gas shall be applied to the entire sampling system and the final result obtained shall be within  $\leq \pm 10\%$  of the established cylinder value.

The gas is also applied to the entire sampling system at the end of the measurement period. The final result obtained must be within  $\leq \pm 10\%$  of the established cylinder value.

When using very reactive gases, once  $T_{90}$  has been reached, it is not necessary to wait three times  $T_{90}$  for the readings to stabilise.

Note 3: the reactive gas check shows that 90% of the reactive gas is detected by the analyser both before and after the measurement period has finished.

If this check criteria is not met the results for all the determinands monitored during the measurement period shall be rejected.

#### Option 2: Wet gas injection

The most reactive gas is applied to the entire sampling system using a wet gas injection system. The final result obtained must be within  $\leq \pm 10\%$  of the value of the gas injected into the system.

The gas is also applied to the entire sampling system at the end of the measurement period. The final result obtained shall be within  $\leq \pm 10\%$  of the value of the gas injected into the system.

When using very reactive gases, once  $T_{90}$  has been reached, it is not necessary to wait three times  $T_{90}$  for the readings to stabilise.

If this check criteria is not met the results for all the determinands monitored during the measurement period shall be rejected.

#### Option 3: Analyte spiking

A preliminary measurement of stack gases shall be carried out to obtain sample spectra to determine the concentration of target analytes.

If the reactive spike gas used is not available as a traceable and certified gas with a measurement uncertainty of  $\leq \pm 2\%$  of value with a 95% confidence level, then it may be quantified by applying the gas into the FTIR inlet only (i.e. not through the sample system), and quantifying the result.

Direct the analyte spiking check gas into the sampling system with the sample conditioning apparatus in place (if used) and co-mix with the stack gas (upstream of the particulate filter, where possible) at a known flowrate, using calibrated mass flow meters, controllers or rotameters.

The flow ratio of calibration gas to stack gas shall be between 1:10 and 9:10 for the determination of sample recovery.

Note 4: Use of a tracer gas compound such as propane blended with the calibration standard at a known concentration allows for accurate quantification of the exact dilution factor (which negates the need to calibrate accurately the mass flow meters, controllers, and rotameters). It is important to use and to calibrate accurately the mass flow meters, mass flow controllers, and rotameters used during the procedure if a tracer compound is not present in the spike gas.

The concentration of the resultant spiked gas should be sufficient to minimise the effect of any process variations on the final concentration. A maximum final spike gas concentration of 150 to 300% of the ELV should be used. For analytes without an ELV a maximum final spike concentration of 50 to 100% of the application range (i.e. the anticipated range that the analyser will be operate on during the measurement period) shall be used.

For example, if the stack gas concentration of the target analyte varies by 4ppm, this variation must be less than 30% of the final spiked concentration, to enable a pass (e.g. final spike concentration must be >13.4ppm).

The analyte spike gas shall be allowed to equilibrate fully before acquisition of the sample.

The dilution factor of the calibration gas shall be calculated using either known sample flow rate and spiked gas flow rate or from the resultant tracer gas concentration.

The bias between the observed spike value and the expected response (that is, the equivalent concentration of the spiked material plus the analyte concentration adjusted for spike dilution) shall be determined.

The percent recovery of the spiked analytes shall be calculated. Acceptable recoveries are defined as spike recoveries within 30%.

The procedure shall be carried out at the start and end of the measurement period. If this check criteria is not met the results for all the determinands monitored during the measurement period shall be rejected.

## **7.5 Emissions monitoring**

The probe is placed at the representative point(s) in the stack in accordance with BS EN 15259.

The zero gas test shall be carried out at least once per day.

If the measurement period is spread over several days, it is recommended that the check gases are measured at frequent intervals in addition to the zero gas. This reduces the risk of drift remaining undetected and exceeding the 5% limit, resulting in all the results being rejected. Also, if a check with a very reactive gas is required, routine checking reduces the risk of all the results being rejected.

### **7.5.1 End of measurement period zero and check gas tests**

Zero and check gas procedure:

- remove the probe from the stack;
- supply zero gas through the probe and wait at least three times the  $T_{90}$  value prior to taking a reading;
- the zero check performance requirement from Table 1 shall be met;
- supply the required check gases through the probe and wait at least three times the  $T_{90}$  value prior to taking a reading;
- the check gas performance requirement from Table 1 shall be met for the required check gases.
- For very reactive check gases the requirements of section 7.4.3.1 apply.

## 7.6 Determining drift across the measurement period

All the gases used to check the analyser before and after measurement period, with the exception of the very reactive gases (see section 7.4.3.1), shall pass the drift criteria below. However, if the gas with the narrowest line width selected in section 7.4.2, shows a drift between 2 – 5%, then the drift of all the measured determinands shall be corrected using this drift.

The drift of the check gases across the measurement period shall be determined from:

$$d_{CO} = \left| \frac{(c_{CO,e} - c_{CO,s})}{c_{CO,s}} \times 100 \right| \quad (1)$$

Where

$c_{CO,s}$  is the determined concentration of the check gas at the start of the measurement period;

$c_{CO,e}$  is the determined concentration of the check gas at the end of the measurement period;

$d_{CO}$  is the drift across the measurement period with respect to the check gas component.

The following criterion apply:

- < 2% drift = pass;
- ≥ 2% & < 5% drift = apply correction using the gas with the narrowest line width;
- ≥ 5% = fail.

If correction is required Annex G shall be followed.

If the drift test is failed for any gases, subject to the drift criteria above, all the data for each target component shall be rejected.

## 7.7 Verification of water vapour measurement

Water vapour is usually measured continuously with other determinands. It is often required for correcting monitoring results to reference conditions. It also affects other measurements because it has a very broad spectral range. In this TGN, water vapour is treated in the same way as other determinands.

Monitoring organisations can obtain accreditation to use FTIR as an alternative method to BS EN 14790 – *determination of the water vapour in ducts*.

Note: the MCERTS performance standard for organisations<sup>8</sup> provides further information on obtaining accreditation.

If the monitoring organisation does not meet the requirements of accreditation for water vapour measurements, they shall use BS EN 14790 to measure the water vapour for correction of monitoring results to reference conditions. This measurement shall be done for each stack at least once per day.

Note: A monitoring organisation may decide to use EN 14790 instead of this TGN to measure water vapour.

## 8 Post measurement period QA/QC checks

### 8.1 Introduction

Quality assurance and control is critically important in order to ensure that the uncertainty of the measured values are kept within the stated limits during extended automatic measurement periods in the field. This means that maintenance shall be followed as it is essential for obtaining accurate and traceable data.

### 8.2 Interference check

One absorbance spectrum shall be selected from a suitable period from each day of the monitoring period. In each analytical band the product of each reference spectrum and associated fractional concentration will be subtracted from the sample spectrum by:

$$r_i = s_i - \sum_{j=1}^m f_j R_{ij} \quad (2)$$

Where

- $r_i$  is the residual created at the  $i^{\text{th}}$  wavenumber by subtracting all scaled reference spectra from the sample spectrum;
- $s_i$  is the sample spectrum absorbance at the  $i^{\text{th}}$  wavenumber;
- $f_j$  is the ratio of the reported concentration of the  $j^{\text{th}}$  determinand to the certified value of the associated reference spectrum;
- $R_{ij}$  is the absorbance value at the  $i^{\text{th}}$  wavelength in the reference spectrum of the  $j^{\text{th}}$  determinand;
- $m$  is the number of determinands.

The above calculation assumes that the sample spectra are quantified by linear least squares scaling of reference spectra. If the analyser quantifies determinands by fitting reference spectra by some other method (perhaps involving non-linear fitting) this shall be repeated here in place of the above to create a residual. In addition, if the analyser software automatically reports a residual that is the result of the difference between the sample spectrum and the fitted reference spectra this shall be used in place of the above.

An interference check for each determinand shall be carried out using:

$$\delta_j = \left| \frac{\max(r_j)}{\max(s_j)} \right| \times 100 \quad (3)$$

Where

- $\max(r_j)$  is the maximum value across the analytical band used to quantify the  $j^{\text{th}}$  determinand in the residual spectrum;
- $\max(s_j)$  is the maximum value across the analytical band used to quantify the  $j^{\text{th}}$  determinand in the sample spectrum;
- $\delta_j$  is an indication of the percentage of information in the analytical band used for the  $j^{\text{th}}$  determinand not explained by the fitted reference spectrum.

If  $\delta_j \geq 5\%$ , then the presence of interference is indicated and Annex C shall be followed, otherwise the data shall be rejected. If  $\delta_j < 5\%$  then it is considered that no significant interference exists and that the test is passed for the  $j^{\text{th}}$  determinand.

The above interference test can sometimes be failed when concentrations close to the instruments detection limit are measured. If this occurs it shall be considered that no interference is present (and Annex C need not be followed) if it can be shown that the peak-to-peak noise in the residual is not more than twice the peak-to-peak noise in the absorption spectrum of a measurement of zero gas across the same analytical band.

### 8.3 Frequency of checks

Table 2 shows the minimum required frequency of ongoing QA / QC checks.

**Table 2 — Frequency of checks<sup>a</sup>**

Checks	Frequency	Action criteria
Cleaning or changing of particulate filters <sup>b</sup>	As required	
Regular maintenance of the monitoring	As required by manufacturer	As required
Detection limit	Each measurement period	Determined values shall be recorded for each determinand and satisfy the requirements of the measurement period
Response time	Each measurement period and at least every year for SRM determinands	Meet Table 1 performance requirement
Zero check	After repair of the instrument Each measurement period	Meet Table 1 performance requirement
Check gases	Each measurement period	Meet Table 1 performance requirement
Lack of fit	At least every year and after repair for SRM determinands	Meet Table 1 performance requirement
<sup>a</sup> If the FTIR is being used as an AM for EN 14790, then water vapour must also be checked <sup>b</sup> The particle filter shall be changed periodically depending on the dust loading at the sampling site. During this filter change the filter housing shall be cleaned. Overloading of the particle filter may increase the pressure drop and decrease the flows.		

### 8.4 Monitoring campaign data retention

All spectra and / or interferograms recorded during the monitoring campaign shall be stored with sufficient details of the acquisition parameters used so that at any future date it is possible to re-analyse the monitoring campaign spectra. These data shall be kept for a minimum of four years.

## 9 Expression of results

The results are expressed in milligrams per cubic metre, or parts per million by volume, 273 K and 101.3 kPa. Corrections for moisture and oxygen may also be required.

Calculations are provided in TGN M2<sup>12</sup>.

The uncertainty for each reported measurement of each determinand shall be determined following Annex E expressing the result to a confidence level of 95%. This uncertainty determination shall follow the methodology of the law of propagation of uncertainty as laid down in BS EN ISO 14956 and BS ENV 13005 (GUM). Many of the uncertainty sources will be common for each determinand, and therefore much of the uncertainty calculation will be common for all determinands. Table 1 defines which instrument performance characteristics should be included in the uncertainty determination. In each case the data used should be from the most recent tests.

## 10 Test report

If carrying out MCERTS accredited work the monitoring report shall be submitted in the report format specified in the *MCERTS Manual stack emission monitoring performance standard for organisations* available from [www.mcerts.net](http://www.mcerts.net).

The following shall also be included in the test report:

- the wavenumber limits of the analytical band used to analyse for each determinand;
- the detection limit determined for each determinand for the measurement period;
- any date and determinand where the interference test ( $\delta_j$ ) has been failed and details of any re-calibration carried out under Annex C.

## Bibliography

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3. DD CEN/TS 14793:2005, Stationary source emission - Intralaboratory validation procedure for an alternative method compared to a reference method.
4. EN 15267-3:2007, Air quality – Certification of automated measuring systems – Part 3: Performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources.
5. BS EN 15259:2007, Air Quality – Measurement of stationary source emissions – Requirements for measurement sections and sites and for the measurement objective, plan and report.
6. BS ISO 10396:2007, Stationary source emissions – Sampling for the automated determination of gas emission concentrations for permanently-installed monitoring systems.
7. MCERTS Performance Standards and Test Procedures for Continuous Emission Monitoring Systems. For gaseous, particulate and flow-rate monitoring systems. Environment Agency. Available from [www.mcerts.net](http://www.mcerts.net).
8. MCERTS Manual stack emission monitoring performance standard for organisations. Environment Agency. Available from [www.mcerts.net](http://www.mcerts.net)
9. BS EN ISO 14956:2002, Air quality – Evaluation of the suitability of a measurement procedure by comparison with a required measurement uncertainty.
10. BS ENV 13005:1999, Guide to the expression of uncertainty in measurement.
11. TGN M1, Sampling requirements for stack emissions monitoring, Environment Agency. Available from [www.mcerts.net](http://www.mcerts.net)
12. TGN M2, Monitoring of stack emissions to air. Environment Agency. Available from [www.mcerts.net](http://www.mcerts.net)

**Annex A: Glossary of terms and definitions**

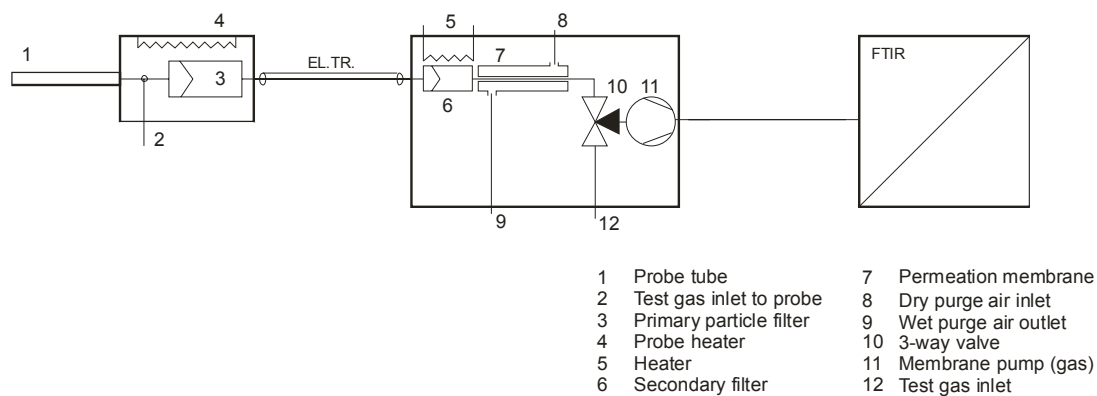
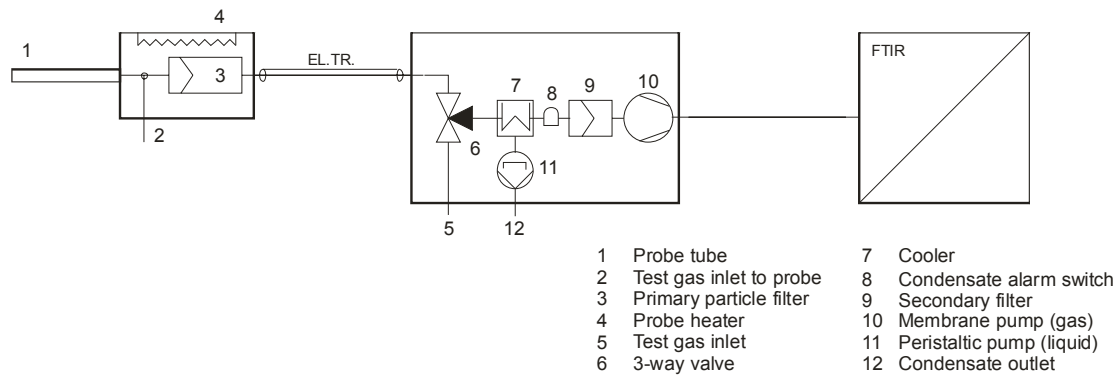
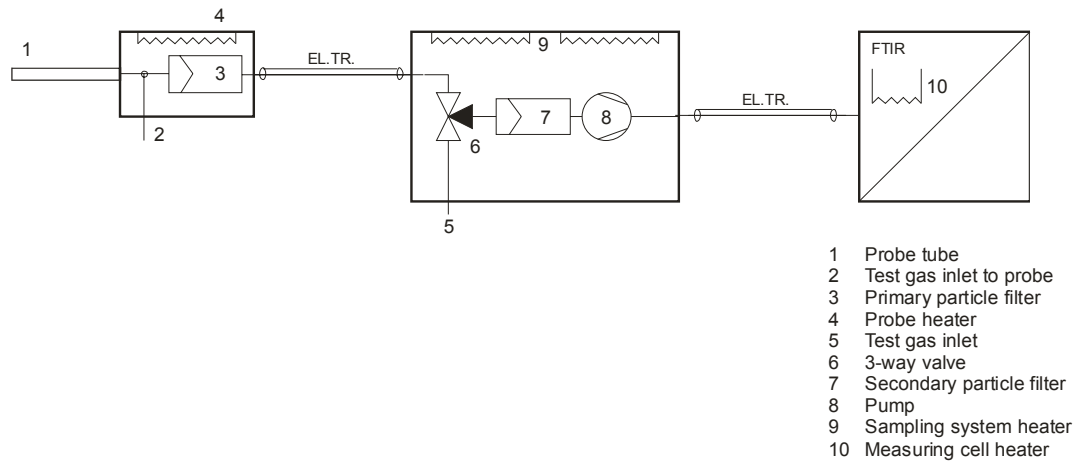
<b>Absorbance spectrum</b>	The negative of the base 10 logarithm of the transmission spectrum
<b>Analytical band</b>	The wavenumber range across which an determinand is to be analysed
<b>Alternative Method</b>	A method that has been proved equivalent to a standard reference method.
<b>Application range</b>	The upper concentration limit set for an determinand for a given monitoring period [For SRM determinands this will be determined by the relevant emission limit value. Performance requirements for the determinand will have to be met across this application range]
<b>Background spectrum</b>	The spectrum taken in the absence of spectrally absorbing determinands and the determinand(s), typically measured with pure nitrogen or synthetic air (mixture of only nitrogen and oxygen)
<b>Calibration range</b>	The upper concentration for a given determinand to which the analyser has been calibrated
<b>Check gas</b>	A traceably certified gas standard that is used periodically to verify the FTIR system's performance for a specific determinand
<b>Detection limit</b>	The minimum concentration of an determinand that can be detected by an instrument with a given (specified) statistical probability
<b>Determinand</b>	The chemical compound that is to be measured
<b>Emission limit value (ELV)</b>	Emission limit value according to EU Directives on the basis of 30 minutes, 1 hour or 1 day
<b>Error</b>	The difference between the 'true' value of the determinand and the measured value
<b>FTIR spectroscopic instrument</b>	An abbreviation for Fourier-transform infrared. An interferometric spectroscopic instrument (interferometer) that uses the infrared component of the electromagnetic spectrum for measurements. A Fourier-transform function is applied by the interferometer to obtain the absorption spectrum as a function of frequency or wavelength. [In FTIR spectroscopy the interferogram represents the detection system response versus time. The Fourier transform function produces the optical intensity as a function of frequency or wavelength]
<b>Interference</b>	When optical absorption from any other chemical compound in the sample cell occurs at a wavelength coincident with wavelength(s) used to analyse the determinand of interest resulting in an inaccurate analysis



<b>Interferogram</b>	The combined signal of all optical wavelengths recorded by the detection system during one complete scan
<b>Manufacturer</b>	Organisation producing and / or distributing the FTIR model
<b>Measurement period</b>	In this TGN, a measurement period is defined as an agreed time period. This may be for several hours or several days.  Note: A measurement period is usually defined for one emission point only. However, it is acceptable to define a measurement period for a number of different stacks, provided the same check gases are applicable.
<b>Monitoring campaign</b>	All the monitoring work carried out during a visit to a site (e.g. quarterly compliance monitoring, QAL2 on several emissions points).
<b>Performance characteristic</b>	A quantity (described by values, tolerances, ranges etc.) assigned to an FTIR in order to define its performance.
<b>Reactive gases</b>	Reactivity of gases in this TGN is ranked based on experience from stack emissions monitoring. For gases not listed in this TGN, solubility in water can be used to assess reactivity. The more soluble the gas the more reactive (i.e. difficult to sample) it is considered to be.
<b>Reference gas spectra</b>	Absorption spectra of samples of a set of gases with known concentrations as a function of wavelength (or frequency). These spectra are measured under controlled conditions of pressure and temperature with known concentration(s). The spectra are used to derive the concentrations of the determinand(s) in the industrial process
<b>Resolution</b>	The minimum separation that two spectral features can have and still be distinguished from one another
<b>Response time of the instrument</b>	The time taken for the reading for a specific determinand to reach 90% of the total reading for the determinand
<b>Sample cell</b>	The part of the FTIR instrument where the optical beam is transmitted through the sample
<b>Sample conditioning system</b>	The part of the sampling system that may remove water vapour, carbon dioxide and other undesired components
<b>Sample spectrum</b>	The absorbance spectrum of the sample extracted from the stack or flue

<b>Sampling system</b>	The entire sample chain from the industrial process to the FTIR instrument cell consisting of sample probe, sample transport, sample conditioning system, and any other necessary components
<b>Single channel spectrum</b>	The response of the FTIR as a function of wavenumber when measuring sample in the sample cell or background
<b>Standard Reference Method determinands</b>	Any determinands where the monitoring is required to conform to a European standard reference method
<b>Standard Reference Method</b>	A measurement method prescribed by European legislation, which is presumed to give the accepted reference value of the determinand.
<b>Transmission spectrum</b>	The ratio of a single channel spectrum where determinand(s) is present to a single channel spectrum where it is not, both spectra being acquired under the same conditions
<b>Uncertainty</b>	Result of the evaluation aimed at characterising the range within which the true value of an determinand is estimated to lie
<b>User</b>	The organisation employing the FTIR analyser for monitoring purposes
<b>Zero gas</b>	Gas containing no significant amount of any compounds that are IR active. Appropriate gases include pure nitrogen or synthetic air (mixture of only nitrogen and oxygen)

**Annex B: Typical sampling system configurations**



Top: Hot/wet method

Middle: Condensation/refrigeration method

Bottom: Permeation drying method

The above schematics do not represent an exhaustive list and other arrangements are possible. Also, it may be preferable to position the pump after the FTIR analyser if there are concerns over sample contamination.

For sampling saturated gas streams, such as those found downstream of wet scrubbers, probes made from polypropylene, polytetrafluoroethylene (PTFE), PFA or other non-reactive polymers may be used. Probes of this type usually incorporate a quartz-wool plug or PTFE membrane to remove water droplets from the gas stream. Polymer probes are usable with gas temperatures up to 90°C. If the stream is saturated and its acid dew point is greater than 90°C, a heated metal probe at a temperature above the dew point may be used. The temperature of the extracted gas must be maintained above the acid dew point throughout the apparatus or at least until condensate removal stage.

If permeation drying is used, care must be taken to ensure that the permeation membrane does not remove determinands of interest. Permeation gas driers using Nafion polymer membranes may also remove ammonia, amines, alcohols, aldehydes, ketones, and carboxylic acids from the gas stream together with water.

Nafion contains sulfonic acid, whose functional groups (R-SO<sub>3</sub>H) in the polymer chain, react with hydroxyl and amine groups in the same way as with H<sub>2</sub>O. Nafion also functions as an acid catalyst due to the strongly acid properties of the sulfonic acid. As a strong acid catalyst, Nafion converts organic compounds susceptible to acid catalysis into other compounds. Strictly speaking the compound is not removed from the sample gas, but it is converted into another compound instead. Acid catalysis often occurs with compounds that have:

- double or triple bonds between carbon atoms or between carbon and other atoms (alkenes, alkynes, aldehydes, ketones, carboxylic acids, nitriles, sulfoxides, etc).
- steric stress in the molecule (structural stress that can be relieved by reorganization of the molecule).

This mechanism converts aldehydes, ketones and carboxylic acids into alcohols, which leads to their elimination. Most inorganic gases are not removed by permeation tubes, and unsaturated hydrocarbons and aromatic hydrocarbons are not removed by permeation tubes unless they contain functional groups listed above.

## **Annex C: Analyser calibration**

### **1. General**

If the analyser (or rather model) has been certified under MCERTS then the new determinand shall be added to the calibration without invalidating the MCERTS certification for the remaining determinands. A new determinand shall be added to the analysers' software following the manufacturers instructions.

Alternatively, rather than using the manufacturers' software the user may employ alternative software packages to analyse spectra recorded with the FTIR instrument. However, whether using the manufacturers software or an alternative the entire system (FTIR + analysis software) shall meet all relevant performance requirements under this TGN.

Note 1: Rather than adding calibration information for the new determinand into the on-board FTIR software the user may also consider creating an off-line calibration, so that spectra recorded during a monitoring campaign can be analysed on a stand-alone PC. For example, saved spectra recorded during a monitoring campaign could be exported from the FTIR interface software (perhaps in ASCII format) and analysed in a separate software package such as MATLAB. This is a perfectly acceptable method to add and additional determinand as long as it can be demonstrated that the necessary performance requirements are met.

Note 2: The above does not preclude the manufacturer / user from changing the certified calibration and then re-submitting the model for re-certification under MCERTS.<sup>7</sup>

Note 3: Whilst calibration is referred to as requiring the use of calibration gases, other validated approaches, for instance using liquid injection, are acceptable provided they are certified as metrologically traceable and are demonstrably equivalent to calibrations using gas standards.

Note 4: Annex F describes analytical bands for some common determinands.

### **2. Zero and check gas test**

Prior to carrying out any calibration measurements the analyser shall be validated as fit for use. Zero and check gas tests shall be carried out following the same procedure as for the annual testing. The performance criteria of Table 1 shall be met.

### **3. Performance requirement testing for an additional determinand**

The performance testing required for the new determinand shall be determined with reference to Table 1 and the relevant footnotes within. The performance criteria in Table 1 shall be met following the test methodology provided in Annex D.

Note: As described in section 4 the performance tests required for the new determinand will depend on whether the analyser has any previous certification under MCERTS and whether the new determinand is an SRM determinand.

## Annex D: Performance requirement tests

### 1. General

This annex describes methodology for carrying out performance requirement tests for the characteristics listed in Table 1.

### 2. Response time

For the annual and monitoring period response time test, the methodology described in BS EN 15267-3 shall be followed, except only one rise time is required to determine the  $T_{90}$  value.

For each monitoring period the response time shall be determined.

### 3. Detection limit

Analyser models that have been certified under MCERTS<sup>7</sup> will, as a requirement, report values around zero. Many other FTIR systems will also report measured values either side of zero. In such cases the detection limit shall be determined from the noise of repeated zero measurements using Approach A. However, some FTIR systems may have a threshold below which spectroscopic peaks are not quantified. This leads to a threshold concentration, below which concentrations are forced to zero or negative concentrations are not reported due to a non-negativity constraint. In such scenarios Approach A would yield an artificially low detection limit value. It shall be established whether or not the analyser operates in this way and if it does Approach B shall be followed.

#### 3.1 Approach A

The detection limit shall be determined for the determinand by making at least 10 repeat measurements of zero gas recording 10 determined concentrations. The detection limit shall be determined from:

$$dl_j = 2 \left( \sqrt{\frac{\sum_{i=1}^n (c_{j,i} - \bar{c}_{j,i})^2}{n-1}} \right) \quad (C1)$$

Where

- $dl_j$  is the detection limit of the  $j^{\text{th}}$  determinand to a confidence level of 95%;
- $c_{j,i}$  is the reported concentration from the  $i^{\text{th}}$  measurement of the  $j^{\text{th}}$  determinand;
- $\bar{c}_{j,i}$  is the mean of all  $c_{j,i}$ ;
- $n$  is the number of measurements carried out.

Note: 10 measurements of zero gas allows the detection limit to be calculated for every determinand present in the analysers' calibration.

### 3.2 Approach B

If the analyser does operate with a cut-off then the detection limit shall be determined by making at least 10 repeat measurements of a certified reference material (CRM). The CRM concentration shall either be at 100% of calibration / certification range or at a value that is three times the concentration where the analyser is believed to cut-off or three times the expected detection limit, whichever the greatest. The detection limit shall be determined using equation (C1). When the detection limit is reported it shall be specified whether it was determined using zero gas or a CRM.

### 4. Lack of fit

The lack of fit (or linearity) shall be carried out for all determinands at least once at the point of calibration (certification / installation). Thereafter the lack of fit shall be determined as part of the annual performance testing for each analyser for those determinands with applicable SRMs. Traceable, certified standards shall be used either individually, or as a mixture, with concentrations at 100% of the respective calibration / certification ranges. Each standard shall be measured across at least five concentration points that shall include 0 and 100%. The concentration points will be separated by approximately even increments and achieved either by diluting the 100% of calibration / certification range standard by a suitably certified procedure or by acquiring a set of traceable, certified materials at the required lower concentrations.

The zero gas shall be used for the respective 0% of calibration / certification range points.

The test of linearity described in Annex C of BS EN 15267-3<sup>4</sup> shall be used. At each concentration level the  $d_{c,rel}$  (relative residual) value shall conform to the lack of fit requirement detailed in Table 1.

### 5. Zero drift

The zero gas shall be directly supplied into the analyser gas cell (*i.e.* in the absence of any sampling system) and measured using the same parameters as would be applied in a typical monitoring campaign. The reported concentration values for all determinand channels shall be recorded for two measurements of the zero gas that are 24 hours apart.

All reported concentrations across the 24 hour period shall be  $\leq \pm 2,0\%$  of the respective determinand calibration / certification range as appropriate.

### 6. Span drift

Traceable, certified standards shall be acquired individually, or as a mixture (where compatibility allows), with concentrations at the respective calibration / certification range for each determinand. The gas from each cylinder shall in turn be supplied directly into the analyser gas cell and measured using the same parameters as would be used in a typical monitoring campaign. The reported concentration values for the associated determinand channel(s) shall be recorded for two measurements of the gas that are 24 hours apart.

All reported concentrations across the 24 h period shall be  $\leq \pm 2,0\%$  of the respective determinand calibration / certification range, as appropriate.

## 7. Sensitivity to ambient pressure, ambient temperature and voltage supply

The analyser must be submitted to an approved test house and these performance characteristics tested conforming to EN 15267-3<sup>4</sup> with one selected determinand. The performance requirements in Table 1 shall be met. If the tests are passed all analysers of the same model are considered to have also satisfied the performance requirements.

## 8. Interferents

Cross-interference shall be tested by carrying out a quantification of each 100% of calibration / certification range reference spectrum (by considering it as a sample spectrum) within the calibration. In the quantification the concentrations reported by the analyser on all other channels shall be recorded. Furthermore, interference from H<sub>2</sub>O and CO<sub>2</sub> shall in all circumstances be tested using either validated reference spectra or by carrying out real measurements. Interference shall be tested with minimum concentrations of 20% and 8% for H<sub>2</sub>O and CO<sub>2</sub>, respectively.

The interference on a determinand channel from all other determinands shall be found from:

$$INT_j = \sum_{i=1}^{n-1} \frac{c_{j,i}}{CR_j} \times 100 \quad (C2)$$

Where

$INT_j$  is the total interference with the  $j^{\text{th}}$  determinand with respect to all other determinands (%);

$CR_j$  is the calibration / certification range of the  $j^{\text{th}}$  determinand;

$c_{j,i}$  is the reported concentration on the  $j^{\text{th}}$  determinand channel when quantifying the  $i^{\text{th}}$  remaining determinand;

$n$  is the number of determinands in the analyser calibration.

## 9. Standard deviation of repeatability in the laboratory at zero

The standard deviation of repeatability in the laboratory at zero shall be determined following the procedure given in EN 15267-3<sup>4</sup> and the performance requirement in Table 1 met.

## 10. Standard deviation of repeatability in laboratory at span level

The standard deviation of repeatability in the laboratory at span shall be determined following the procedure in EN 15267-3<sup>4</sup> and the performance requirement in Table 1 met.



## **Annex E: Uncertainty determination**

### **1. Required information**

The following information shall be obtained in order to complete the uncertainty calculations:

- daily average emission limit value (ELV) for the determinand (if applicable);
- atmospheric pressure variation during monitoring period;
- sample cell pressure variation during monitoring period (if unknown the value in Table 3 shall be used);
- ambient temperature variation during monitoring period;
- on-site voltage variation during monitoring period (if unknown the value in Table 3 shall be used);

### **2. Estimation of the uncertainty**

An uncertainty budget following the format given in Table 1 and in accordance with BS ENV 13005 shall be produced for each determinand measured during the monitoring period.

Table 1: Uncertainty template

Symbol	Source of uncertainty	Type	Value / $\pm$	Probability distribution	Divisor	Conversion factor (cf)	$u_i / \text{mg m}^{-3}$	$v_i$ or $v_{eff}$
$U_{fit}$	Lack of fit	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$
$U_{0,dr}$	Zero drift	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$
$U_{s,dr}$	Span drift	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$
$U_{apress}$	Sensitivity to atmospheric pressure	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100} \times \frac{\Delta P}{2}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$
$U_{spress}$	Sensitivity to sample gas pressure	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100} \times \frac{\Delta p}{3}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$
$U_{temp}$	Sensitivity to ambient temperature	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100} \times \frac{\sqrt{(T_{\max} - T_s)^2 + (T_{\min} - T_s) \times (T_{\max} - T_s) + (T_{\min} - T_s)^2}}{10}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$
$U_{volt}$	Sensitivity to electrical voltage	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100} \times \frac{\sqrt{(V_{\max} - V_s)^2 + (V_{\min} - V_s) \times (V_{\max} - V_s) + (V_{\min} - V_s)^2}}{10}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$
$u_i$	Interferents	B		rectangular	$\sqrt{3}$	$\frac{R_i}{100}$	$\frac{\text{value} \times \text{cf}}{\text{divisor}}$	$\infty$

$u_{0,r}$	Standard deviation of repeatability in laboratory at zero <sup>a</sup>	A		normal	1	$\frac{R_i}{100}$		$n - 1$
$u_{s,r}$	Standard deviation of repeatability in laboratory at span level <sup>a</sup>	A		normal	1	$\frac{R_i}{100}$		$n - 1$
$u_{cal}$	Uncertainty of CRM	B		normal	2	$\frac{R_i}{100}$		$\infty$
$u_c$	Combined uncertainty			normal			$\sqrt{\sum_{i=1}^N u_i^2}$	$\frac{u_c^4}{\sum_{i=1}^N \frac{u_i^4}{v_i}}$
$U_{95}$	Expanded uncertainty			t-distribution			$k_{95}u_c$	

<sup>a</sup> Only one of these values shall be included in the calculation: the first possibility is to choose the repeatability standard deviation obtained from laboratory tests corresponding to the closest concentration to the actual concentration in stack, or the higher (relative) standard deviation of repeatability independently of the concentration measured in stack.

Where

- $R_i$  is the certification range ( $\text{mg m}^{-3}$ ) if the determinand has been certified under MCERTS. If not then it is the calibration range;
- $\Delta P$  is the pressure deviation across the monitoring period (kPa);
- $\Delta p$  is the pressure deviation in the sample cell across the monitoring period if known (if not 5 kPa shall be used);
- $T_s$  is room temperature (298 K);
- $T_{\min}$  is the minimum ambient temperature that the analyser was exposed to during monitoring (K);
- $T_{\max}$  is the maximum ambient temperature the analyser was exposed to during monitoring (K);
- $V_s$  is the nominal site voltage (usually 110 V);
- $V_{\min}$  is the minimum value of the voltage variation expected on site during monitoring (V);
- $V_{\max}$  is the maximum value of the voltage variation expected on site during monitoring (V);
- $n$  is the number of measurements made in determining a standard deviation;
- $N$  is the number of uncertainty sources;
- $u_c$  is the combined uncertainty;
- $\nu_i$  is the degrees of freedom of the  $i^{\text{th}}$  uncertainty source;
- $\nu_{\text{eff}}$  is the effective degrees of freedom;
- $u_i$  is the uncertainty of  $i^{\text{th}}$  uncertainty source;
- $k_{95}$  is the coverage factor for a 95% confidence level.

The *value* column of Table 1 shall be populated using the determined performance for each characteristic with respect to the given determinand.

Note 1: It is necessary to produce a budget as shown in Table 1 for each determinand where uncertainty estimation is required. However, this need not be onerous as only sources  $U_{\text{apress}}$ ,  $U_{\text{spress}}$ ,  $U_{\text{temp}}$ ,  $U_{\text{volt}}$ , might vary between sites with all other parameters remaining unchanged.

The contribution of error sources with rectangular distributions shall be determined in accordance with EN ISO 14956<sup>8</sup> using:

$$u(x_i) = \sqrt{\frac{(x_{i,\max} - x_{i,\text{adj}})^2 + (x_{i,\min} - x_{i,\text{adj}})^2}{3} + (x_{i,\max} - x_{i,\text{adj}})(x_{i,\min} - x_{i,\text{adj}})} \quad (1)$$

Where

- $u(x_i)$  is the estimated uncertainty of the  $i^{\text{th}}$  error source;
- $x_{i,\text{adj}}$  is the value of influence quantity  $X_i$  at the time the analyser was calibrated;
- $x_{i,\min}$  is the minimum value of influence quantity  $X_i$  during the monitoring period;
- $x_{i,\max}$  is the maximum value of influence quantity  $X_i$  during the monitoring period.

Equation 1 is usually applicable to  $u_{temp}$  and  $u_{volt}$  as both these sources are expected to show an uneven deviation above and below the central quantity. For example, the ambient temperature during instrument calibration (assumed as 298 K) may not reside equidistant, in absolute terms, between the maximum and minimum ambient temperature encountered during the monitoring period.

For the remaining error sources it is expected that the deviation will be equal either side of the central quantity, hence:

$$\left| x_{i,max} - x_{i,adj} \right| = \left| x_{i,min} - x_{i,adj} \right| = \Delta x_i \quad (2)$$

Where

$\Delta x_i$  is the deviation in the  $i$ th uncertainty source from the value at the time the analyser was calibrated.

Consequently, for these remaining error sources (with a rectangular distribution) the uncertainty can be found from using a simplification of equation 1:

$$u(x_i) = \frac{\Delta x_i}{\sqrt{3}} \quad (3)$$

If the distribution is not rectangular  $\sqrt{3}$  shall be replaced in equation 3 with the appropriate divisor.

Values of standard uncertainty that are less than 5% of the maximum standard uncertainty may be excluded from the uncertainty budget.

Since there is more than one type A error source in Table .1 the test of significance is failed and it is necessary to determine a modified coverage factor. The effective degrees of freedom shall be determined using the Welch-Satterthwaite equation:

$$\nu_{eff} = \frac{u_c^4}{\sum_{i=1}^N \frac{u_i^4}{\nu_i}} \quad (4)$$

The degrees of freedom of type B error sources shall be considered as infinity as indicated in Table 1. The degrees of freedom of type A error sources shall be determined as shown in Table 1.

Using the determined value for  $\nu_{eff}$  the corresponding  $t_{95}$  value shall be determined using the student t table given in ENV13005<sup>9</sup> (a limited range of values are reproduced in Table 2 for quick reference). As  $t_{95} = k_{95}$  this value shall be used as the modified coverage factor.

Note 2: It is expected that  $\nu_{eff}$  shall almost always be found to be  $> 100$ , hence a coverage factor of 2 will generally be required.

**Table 2: Student t table**

$V_{eff}$	$t_{95}$	$V_{eff}$	$t_{95}$	$V_{eff}$	$t_{95}$	$V_{eff}$	$t_{95}$
1	13,97	7	2,43	18	2,15	45	2,06
2	4,53	8	2,37	20	2,13	50	2,05
3	3,31	10	2,28	25	2,11	60	2,04
4	2,87	12	2,23	30	2,09	80	2,03
5	2,65	14	2,20	35	2,07	100	2,02
6	2,52	16	2,17	40	2,06	$\infty$	2,00

In accordance with EN ISO 14956<sup>8</sup> the determined expanded uncertainty for the determinand shall meet the specified requirement.

Note 3: Generally if an uncertainty requirement exists it is derived from the appropriate EU Directive. For some determinands there may be no requirement.

### 3. Example uncertainty determination

Table 4 details an example uncertainty budget for the CO molecule using arbitrarily chosen input values shown in Table 3.

**Table 3: Arbitrarily chosen input values for an example CO uncertainty estimation**

Parameter	Value
Range ( $R_{CO}$ )	100 mg m <sup>-3</sup>
Deviation of ambient pressure across monitoring period ( $\Delta P$ )	1 kPa
Deviation of sample cell pressure across monitoring period ( $\Delta p$ )	5 kPa
Minimum ambient temperature during monitoring ( $T_{min}$ )	293 K
Maximum ambient temperature during monitoring ( $T_{max}$ )	303 K
Site voltage supply ( $V_s$ )	110 V
Minimum site voltage during monitoring ( $V_{min}$ )	93 V
Maximum site voltage during monitoring ( $V_{max}$ )	121 V
Number of measurements made during standard deviation determinations ( $n$ )	Set to 20 for both standard deviation of repeatability in laboratory at zero and also at span. Due to the requirement under MCERTS <sup>7</sup> to make 20 repeat measurements for each characteristic

Note: For the example molecule a certification range of 100 mg m<sup>-3</sup> has been arbitrarily used.

Table 4 shows that the expanded uncertainty for CO monitoring ( $U_{95,CO}$ ) is:

$$U_{95,CO} = \pm 3,6 \text{ mg m}^{-3}$$

Table 4: Example uncertainty budget

Sym.	Source of uncertainty	Value / $\pm$	Probability distribution	Divisor	Conversion factor (cf)	$u_i / \text{mg m}^{-3}$	$v_i$ or $v_{eff}$
$u_{fit}$	Lack of fit	0,7%	rectangular	$\sqrt{3}$	1	$\frac{0,7 \times 1}{\sqrt{3}} = 0,404$	$\infty$
$u_{0,dr}$	Zero drift	0,01%	rectangular	$\sqrt{3}$	1	$\frac{0,01 \times 1}{\sqrt{3}} = 5,77 \times 10^{-3}$	$\infty$
$u_{s,dr}$	Span drift	0,05%	rectangular	$\sqrt{3}$	1	$\frac{0,05 \times 1}{\sqrt{3}} = 2,89 \times 10^{-2}$	$\infty$
$u_{apress}$	Sensitivity to atmospheric pressure	0,5% / 2 kPa	rectangular	$\sqrt{3}$	$\frac{100}{100} \times \frac{1}{2} = 0,5$	$\frac{0,5 \times 0,5}{\sqrt{3}} = 0,144$	$\infty$
$u_{spress}$	Sensitivity to sample gas pressure	1% / 3 kPa	rectangular	$\sqrt{3}$	$\frac{100}{100} \times \frac{5}{3} = 1,67$	$\frac{1 \times 1,67}{\sqrt{3}} = 0,962$	$\infty$
$u_{temp}$	Sensitivity to ambient temperature	0,5% / 10 K	rectangular	$\sqrt{3}$	$\frac{100}{100} \times \frac{\sqrt{(303 - 298)^2 + (293 - 298) \times (303 - 298) + (293 - 298)^2}}{10} = 0,5$	$\frac{0,5 \times 0,5}{\sqrt{3}} = 0,144$	$\infty$
$u_{volt}$	Sensitivity to electrical voltage	0,6% / 10V	rectangular	$\sqrt{3}$	$\frac{100}{100} \times \frac{\sqrt{(121 - 110)^2 + (93 - 110) \times (121 - 110) + (93 - 110)^2}}{10} = 1,49$	$\frac{0,6 \times 1,49}{\sqrt{3}} = 0,517$	$\infty$
$u_I$	Interferents	1,4%	rectangular	$\sqrt{3}$	1	$\frac{1,4 \times 1}{\sqrt{3}} = 0,808$	$\infty$
$u_{0,r}$	Standard deviation of repeatability in laboratory at zero <sup>a</sup>	0,1%	normal	1	1	$\frac{0,1 \times 1}{1} = 0,1$	$20 - 1 = 19$

$u_{s,r}$	Standard deviation of repeatability in laboratory at span level <sup>a</sup>	0,5%	normal	1	1	$\frac{0,5 \times 1}{1} = 0,5$	$20 - 1 = 19$
$u_{cal}$	Uncertainty of CRM	2%	normal	2	1	$\frac{2 \times 1}{2} = 1$	$\infty$
$u_c$	Combined uncertainty		normal			<b>1,820</b>	<b>3331</b>
$U_{95}$	Expanded uncertainty		t-distribution ( $k_{95} = 2$ )			$2 \times 1,820 = 3,640$	

<sup>a</sup> As described previously only one of these error sources shall be included in the uncertainty budget. However, for the purposes of demonstration both sources have been included in the example.



## Annex F: Selection of check gases

It is a requirement to confirm that the FTIR instrument is able to produce spectra of sufficient accuracy, in terms of both absorbance peak height and width and that this is achievable at (or near) the wavenumber region that is used by the chosen analytical model.

In order to determine if a specific check gas is suitable for the analytical model in use, the user shall specify the central wavenumber position of the analytical band used by the model for each component.

### Example 1

The Gasett DX4000 uses a band of 2000 to 2200 for the determination of CO. The centre of this analysis band is 2100. 25% either side of this analysis band would provide a range from 1575 to 2625.

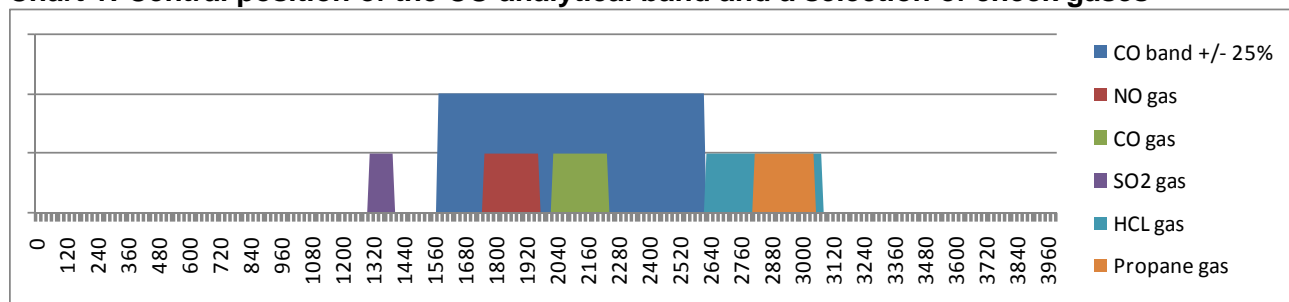
A standard spectra for CO at 100ppm has peaks that appear between 2024 and 2248. In addition a spectrum of NO at 100ppm shows peaks from 1757 to 1971.

Both CO and NO at 100ppm show absorbance peaks within the range defined above (1575 to 2625). Therefore, either gas could be used to validate the FTIR for CO.

Note: There would be other suitable gases that appear within this band as well.

Chart 1 shows the central position of the CO analytical band (+/- 25%) and a selection of typical check gases. It can be seen for this analytical band, CO or NO could be used as check gases but SO<sub>2</sub>, HCL and propane would not be suitable.

**Chart 1: Central position of the CO analytical band and a selection of check gases**



**Example 2**

The Prot-Ir 204M uses multiple bands for determination of NO (see Table 1).

**Table 1: Bands for determination of NO**

Component	Analysis band from	Analysis band to
NO band 1	1851	1864
NO band 2	1874	1913
NO band 3	2807	3159

The centre of these analysis bands with the 25% tolerance assigned is shown in Table 2.

**Table 2: Centre of NO analysis bands with a 25% tolerance assigned**

Central wavenumber position	25% below	25% above
1858	1393	2322
1893	1420	2367
2983	2237	3729

Looking at a standard spectra for NO at 100ppm, it can be seen that peaks appear between 1757 to 1971. In addition a spectra of CO at 100ppm shows peaks from 2024 and 2248.

Both NO and CO at 100ppm show absorbance peaks within at least one of the ranges defined above. Therefore, either gas could be used to validate the FTIR for NO.

Table 3 below shows common analysis bands for the Gaset DX4000 and examples of suitable check gases.

**Table 3: Example analysis bands (Gaset DX4000)**

Component	Analysis band from	Analysis band to	Central wavenumber position	25% below	25% above	Suitable check gases				
						CO	NO	SO <sub>2</sub>	HCl	C <sub>3</sub> H <sub>8</sub>
H <sub>2</sub> O	3200	3401	3300.5	2475	4126	-	-	-	Yes	Yes
CO <sub>2</sub>	926	1150	1038	779	1298	-	-	-	-	-
CO	2000	2200	2100	1575	2625	Yes	Yes	-	Yes	-
N <sub>2</sub> O	2000	2222	2111	1583	2639	Yes	Yes	-	Yes	-
NO	1875	2138	2006.5	1505	2508	Yes	Yes	-	-	-
NO <sub>2</sub>	2700	2950	2825	2119	3531	Yes	-	-	Yes	Yes
SO <sub>2</sub>	1050	1200	1125	844	1406	-	-	Yes	-	-
NH <sub>3</sub>	910	1150	1030	773	1288	-	-	Yes	-	-
HCl	2617	2880	2748.5	2061	3436	Yes	-	-	Yes	Yes
HF	3200	3400	3300	2475	4125	-	-	-	Yes	Yes

Note: Although C<sub>3</sub>H<sub>8</sub> is a suitable check gas for HCl, it would still be necessary to use HCl for a reactive gas check.

Table 4 below shows common analysis bands for the Prot-IR 204M and examples of suitable check gases.

**Table 4: Example analysis bands (Prot-IR 204M)**

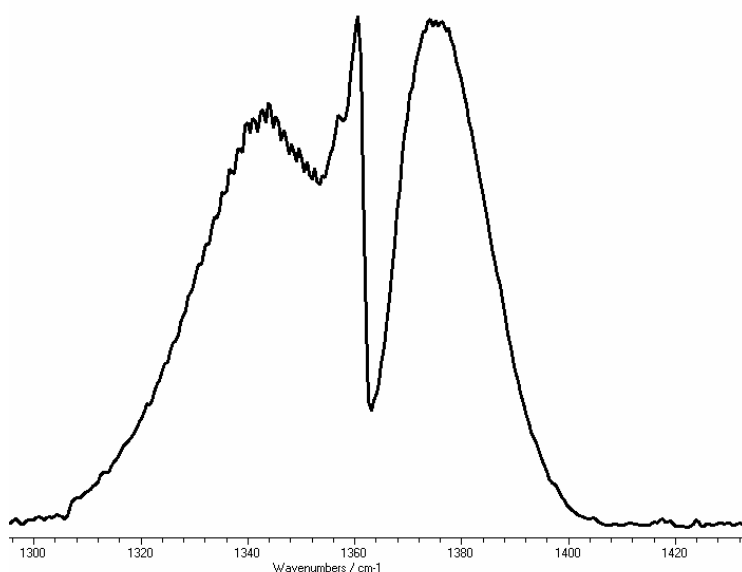
Component	Analysis band from	Analysis band to	Central wavenumber position	25% below	25% above	Suitable check gases				
						CO	NO	SO <sub>2</sub>	HCl	C <sub>3</sub> H <sub>8</sub>
H <sub>2</sub> O	2811	3031	2921	2190	3651	Yes	-	-	Yes	Yes
CO <sub>2</sub> Band 1	2291	2376	2334	1750	2917	Yes	Yes	-	Yes	Yes
CO Band 1	2133	2201	2167	1625	2709	Yes	Yes	-	Yes	-
N <sub>2</sub> O Band 1	1294	1310	1302	976	1627	Yes	Yes	Yes	Yes	Yes
N <sub>2</sub> O Band 2	2151	2223	2187	1640	2734	Yes	Yes	Yes	Yes	Yes
N <sub>2</sub> O Band 3	2527	2634	2581	1935	3226	Yes	Yes	Yes	Yes	Yes
NO Band 1	1851	1864	1858	1393	2322	Yes	Yes	Yes	Yes	Yes
NO Band 2	1874	1913	1893	1420	2367	Yes	Yes	Yes	Yes	Yes
NO Band 3	2807	3159	2983	2237	3729	Yes	Yes	Yes	Yes	Yes
NO <sub>2</sub> Band 1	1548	1677	1613	1210	2016	-	Yes	Yes	-	-
SO <sub>2</sub> Band 1	1295	1415	1355	1016	1694	-	-	Yes	-	-
NH <sub>3</sub> Band 1	899	969	934	701	1168	Yes	-	-	Yes	Yes
NH <sub>3</sub> Band 2	2811	3161	2986	2239	3732	Yes	-	-	Yes	Yes
HCl Band 1	2871	3161	3016	2262	3770	-	-	-	Yes	Yes
HF Band 1	2811	3161	2986	2239	3732	-	-	-	Yes	Yes
HF Band 2	4057	4333	4195	3146	5244	Yes	-	-	Yes	Yes

Note: Although C<sub>3</sub>H<sub>8</sub> is a suitable check gas for HCl, it would still be necessary to use HCl for a reactive gas check.

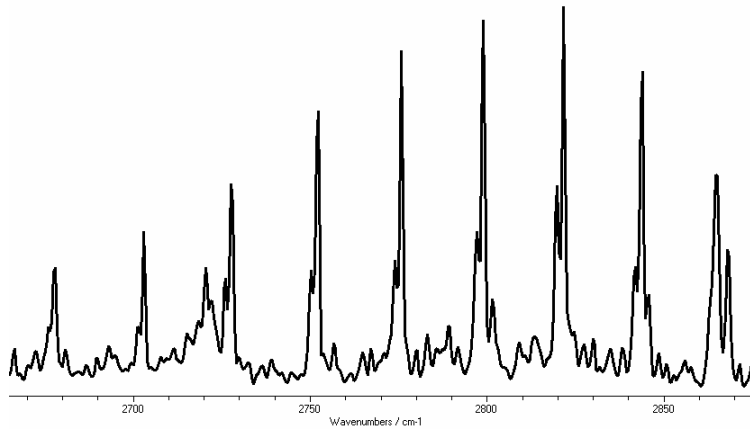
In addition to the above, the concentration of check gas selected must exhibit peak absorbance greater than ten times the LDL but less than 1.5 absorbance units.

Also, one of the selected check gases shall be demonstrated to exhibit peak widths that are no greater than those of the narrowest target analyte.

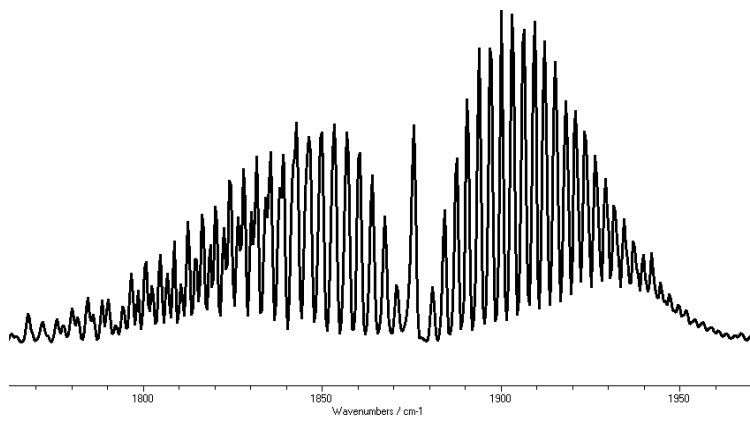
Below are some example spectra of typical gases:



SO<sub>2</sub>  
Peak width approximately 40 cm<sup>-1</sup>



HCl  
Peak width approximately  $5 \text{ cm}^{-1}$



NO  
Peak width approximately  $3 \text{ cm}^{-1}$

If these three gases were the target components for a project, one of the chosen gases would be required to have a maximum peak width no greater than NO.

## Annex G: Drift correction

### 1. General

Extractive FTIR is a ratio measurement as a single channel spectrum where determinands are present is normalised to one where they are not (the background). However, whilst drift is not expected it is none the less possible for the 100% transmission response to shift between recording the background (zero gas) and the determinand (the sample) single channel spectrum resulting in a change to the calculated absorption peak and a possible shift in the determined concentration.

Correcting for a shift is problematic since it is difficult to identify the time and the magnitude of the step change. Consequently, for the purposes of practicality it is considered under this TGN that any apparent drift that falls within the limits acceptable for correction ( $\geq 2\%$  &  $< 5\%$ ) shall be corrected based on the assumption of linear behaviour.

### 2. Correcting for drift across the monitoring period

Drift shall be corrected using the following expression (*nb.* for clarity there is an example of the correction shown in Table 1 describing the calculation stepwise):

$$c_{j,t,corr} = \frac{c_{j,t} - z_s + \left[ \frac{t}{T} (Z_e - Z_s) \right]}{\left( \frac{c_s - Z_s}{CRM - Z} \right) + \left[ \frac{t}{T} \left( \frac{c_e - Z_e}{CRM - Z} - \frac{c_s - Z_s}{CRM - Z} \right) \right]} \quad (1)$$

Where

- $c_{j,t}$  is the determined concentration of the  $j^{\text{th}}$  determinand at time  $t$ ;
- $c_{j,t,corr}$  is the determined concentration of the  $j^{\text{th}}$  determinand at time  $t$  corrected for drift;
- $t$  is the time of determination  $c_{j,t}$ ;
- $T$  is the duration of the monitoring period;
- $CRM$  is the certified concentration of the check gas;
- $Z$  is the concentration of the  $j^{\text{th}}$  determinand in the zero gas (which unless a bias has been demonstrated shall be taken as zero);
- $c_s$  is the determined concentration of the check gas at the start of the monitoring period;
- $Z_s$  is the determined concentration of when measuring the zero gas at the start of the monitoring period;
- $c_e$  is the determined concentration of the check gas at the end of the monitoring period;
- $Z_e$  is the determined concentration of check gas when measuring the zero gas at the end of the monitoring period.

## 3. Example calculation of drift correction

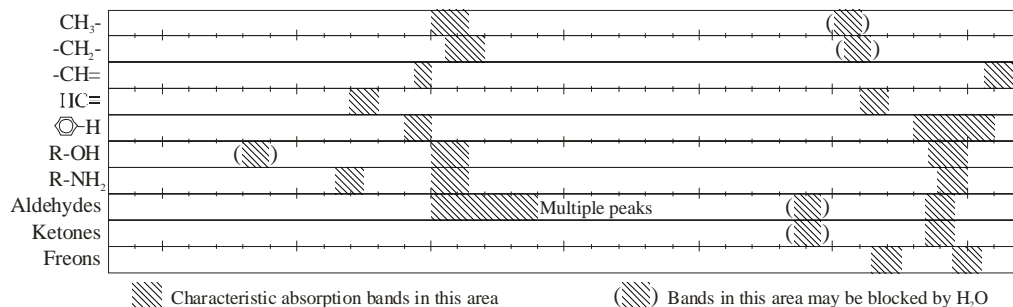
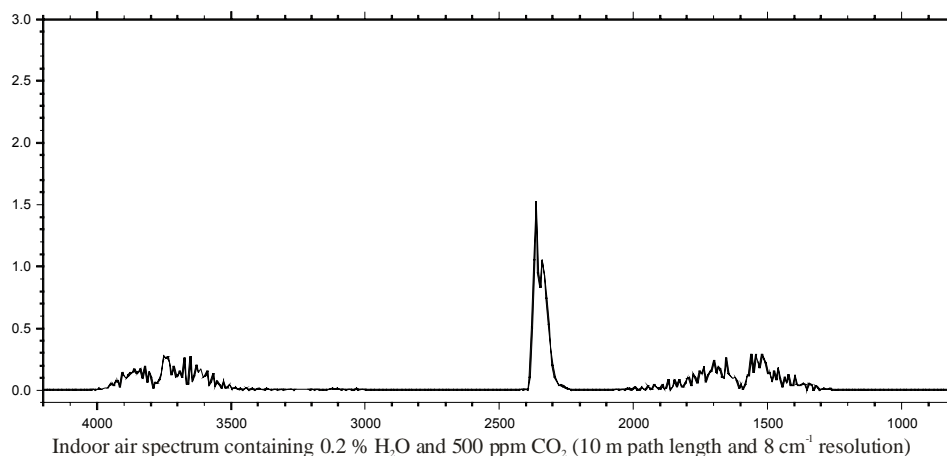
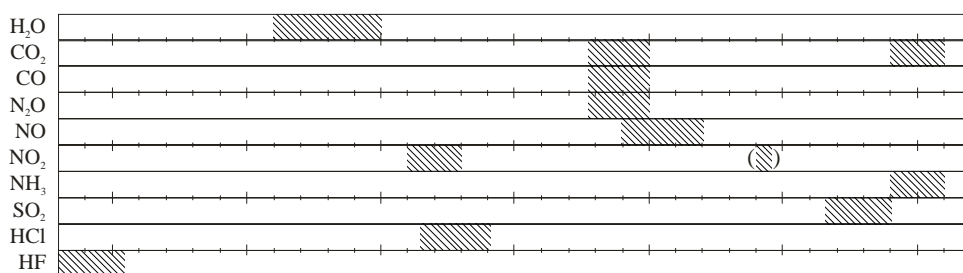
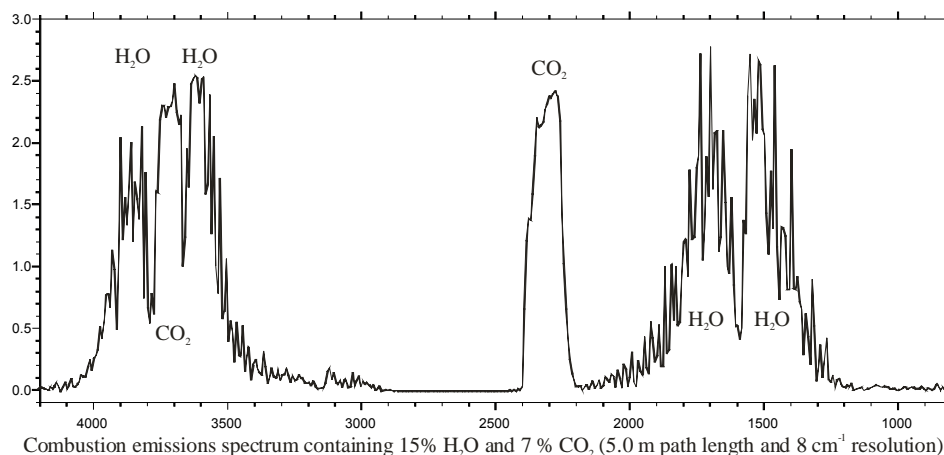
Table 1: Drift correction example detailing calculation in a stepwise approach

	A	B	C	D	E	F
1						
2		Gas	Certified conc. / ppm	Start of monitoring period determined conc. / ppm	End of monitoring period determined conc. / ppm	
3		Check gas (e.g. CO)	40	41	42	
4		Zero	0	0,2	0,4	
5						
6		Elapsed time between readings / min	420			
7		Determined conc. of $j^{\text{th}}$ determinand / ppm	120			
8		At time ( $t$ ) / min	50			
9						
10	<b>Relationship between the corrected (<math>c_{j,t,corr}</math>) and uncorrected (<math>c_{j,t}</math>) concentrations:</b>					
11		$c_{j,t,corr} = \frac{c_{j,t} - Z_t}{\gamma_t}$				
12						
13	<b>Estimated zero at time <math>t</math>:</b>					
14		<b>Zero deviation to start</b>	<b>Zero deviation to end</b>	<b>Zero drift rate / min<sup>-1</sup></b>		
15		D4-C4	E4-C4	(C15-B15)/C6		
16						
17		Estimated zero at time $t$ ( $Z_t$ ) / ppm	D4+(D15*C8)			
18						
19	<b>Determination of instrument sensitivity drift rate:</b>					
20		<b>Sensitivity change to start</b>	<b>Sensitivity change to end</b>	<b>Sensitivity drift rate / min<sup>-1</sup></b>		
21		(D3-D4)/(C3-C4)	(E3-E4)/(C3-C4)	(C21-B21)/C6		
22						
23		Sensitivity at time $t$ ( $\gamma_t$ ):	B21+(D21*C8)			
24						
25	<b>Drift corrected concentration of the <math>j^{\text{th}}</math> determinand at time <math>t</math>:</b>					
26		$c_{j,t,corr}$	(C7-C17)/C23			

Where  $\gamma_t$  is the instrument sensitivity at time  $t$ .

Using the example input data in the first few rows,  $c_{j,t,corr} = 117,154$  ppm.

### Annex H: Possible analytical bands for common determinands



**Annex I: Monitoring campaign on-site check list**

Table 1 provides an example check-list recommended for on-site use to ensure correct instigation of this TGN.

Table 1 also indicates which analyser tests shall be carried out during a monitoring period to validate performance.

**Table 1: Check-list of key on-site activities to complete during a monitoring campaign**

Activity description	Complete?
Select sampling location and point in accordance with BS EN 15259	
Determine check gases to validate analyser	
Start of campaign zero and check gas tests of analyser. Detection limit and response time determinations	
Carry out daily zero tests. [Check gas test also recommended periodically if monitoring for an extended period]	
End of monitoring period zero and check gas test	
Determine any drift across monitoring period and apply drift correction annex if necessary	
Ensure all spectra and / or interferograms recording during monitoring campaign are electronically stored. Ensure sufficient details are stored of acquisition parameters employed to allow re-analysis of the spectra at a future date	
Record all site specific conditions necessary for carrying out uncertainty estimation	