DETAILED SAMPLING PROTOCOLS / METHODOLOGIES

In all cases, where analysis of collected samples is required, the analysis will be by a subcontract laboratory (ECL has no accredited in-house analysis capability). Analysis is carried out by the sub-contract laboratory indicated on the analysis certificates in the report. The UKAS/MCERTs accreditation status of the analysis is also indicated on the certificates. In addition to the survey samples, appropriate field blanks and efficiency checks are submitted as part of the technical procedure.

Part 1 – Accredited Methods

Pressure, Temperature and Velocity - Compliance (UKAS/MCERTS)

Testing is carried out using a sampling system in accordance with **BS EN ISO 16911-1:2013 & MID** and In-house technical procedure **ECL/TPD/022A.**

Temperature is recorded using a thermocouple and digital temperature reader.

Velocity and pressure are recorded using an “L” type / “S” type pitot and digital / inclined manometer, data being recorded in Pascals / mm H_2O / m/s.

Pressure, Temperature and Velocity – 14181 Sampling (UKAS/MCERTS)

Testing is carried out using a sampling system in accordance with **BS EN ISO 16911-1:2013, CEN TR 17078:2017 (taking into account the requirements of BS EN ISO 16911-2:2013 & MID)** and In-house technical procedure **ECL/TPD/022B.**

Temperature is recorded using a thermocouple and digital temperature reader.

Velocity and pressure are recorded using an “L” type / “S” type pitot and digital manometer, data being recorded in Pascals / mm H_2O / m/s.

Particulates (UKAS/MCERTS)

Testing is carried out using a Manual Stack Sampling system in accordance with **BS EN 13284-1:2017 & MID** and in-house technical procedure **ECL/TPD/027 (or 27a - STACKMITE CONTROL UNIT ONLY)**.

Isokinetic particulate sampling is achieved when the velocity of gas entering the sampling nozzle is exactly equal to the velocity of the approaching gas stream within the stack.

For out-stack sampling, a measured volume of sample gas is withdrawn from the stack isokinetically through a sampling nozzle and heated sampling probe, and then through a 37mm, 47mm or 110mm pre-weighed and pre-blown GFA or Quartz filter positioned in a heated housing.

For in-stack sampling, a measured volume of sample gas is withdrawn from the stack isokinetically through a sampling nozzle and through a 37mm, 47mm, 85mm or 110mm pre-weighed and pre-blown GFA or Quartz filter positioned in an unheated housing inserted into the stack.

Particulate matter is collected on the filter. Following testing the front half of the filter housing, probe (out-stack sampling only) and the sample nozzle are rinsed to remove any particulate matter which, may have impacted on the surfaces during testing. The dry residue of the Acetone & DI water used for rinses is **<1mg/l.** The filters and rinses are subsequently analysed to determine the amount of particulate matter captured.

The standard pre-sample conditioning temperature of the filters is 180°C and the standard post-sample conditioning temperature is 160°C. (Any modifications to this are noted in section 2 of this report). Apparent weights are corrected, if required, based on the weights of three control filters and evaporating basins which are weighed (pre and post sampling) with each batch of filters & rinses.

Total Metals (Including but not limited to)
As, Cd, Cr, Co, Cu, Mn, Ni, Pb, Sb, Tl and V (UKAS/MCERTS)

Determination of the total emissions of **Total Metals** is carried out using a Universal Stack Sampling system and heated titanium sample probe in accordance with **BS EN 14385:2004 & MID** and In-house technical procedure **ECL/TPD/028**.

In this method the stack gases are filtered (110mm out-stack, heated quartz filter) to remove particulate matter then the gases are passed through a series of impingers. The first three impingers each containing 140ml of **3.3 % Nitric Acid / 1.5 % Hydrogen Peroxide** the fourth impinger is empty and finally through an impinger containing a measured quantity of silica gel.

Filters and probe washes are spiked with internal standard and brought into HF/Nitric acid solution by microwave digestion. Excess HF is neutralised with Boric acid. Aliquots of impingers are taken, spiked with internal standard and diluted to 50mls. Aliquots of the solutions are analysed by ICP/MS.

Total Mercury (Hg) (UKAS/MCERTS)

Determination of the total emissions of **Mercury** is carried out using a Universal Stack Sampling system and heated titanium sample probe in accordance with **BS EN 13211:2001** and In-house technical procedure **ECL/TPD/030**.

In this method the stack gases are filtered (110mm out-stack heated, quartz filter) to remove particulate matter then the gases are passed through a series of impingers. The **first two** impingers each containing 140ml of **Acidified Permanganate or Acidified Dichromate** the **third** impinger is empty and finally through an impinger containing a measured quantity of silica gel.

Filters and probe washes are brought into HF/Nitric acid solution by microwave digestion. Excess HF is neutralised with Boric acid. Impingers are diluted with HCl and decolourised with ascorbic acid. Aliquots of the solutions are analysed by Continuous Flow CVAFS with Tin Chloride reductant.

Dioxins & Furans - (Filter / Condenser Method) **(UKAS/MCERTS)**

Determination of **Dioxins & Furans (Chlorinated & Brominated congeners)** is carried out using a Universal Stack Sampling system in accordance with **BS EN 1948-1 to 3:2006 & MID** and In-house technical procedure **ECL/TPD/031**.

In this method the stack gases are sampled via a heated titanium probe and are then filtered (110mm out-stack, heated quartz or GFA filter) to remove particulate matter. The filtered gases are then passed through a cooling condenser and spiked XAD absorber, a verification absorption stage is not used. The gases the pass through a condensate flask and a series of silica gel filled impingers.

The gases are cooled via the condenser where the cooled gases and condensate percolate through the XAD absorber accumulate in the condensate flask before being dried by the silica impingers.

Prior to every sampling campaign, the sample train glassware (upstream of the silica impingers) is rinsed with solvents and then muffled at 450 °C for three hours, before being carefully capped for transport to site. If any of the glassware is re-used on site (for further samples) additional rinses of the glassware are taken prior to the re-use and kept for analysis in case of high results being obtained for the following sample.

For Chlorinated Dioxin sampling, the sorbent trap is filled with approx 70g of Amberlite XAD-2 resin, it is spiked with 1.0ng of 13C12 1,2,3,7,8-PeCDF & 1,2,3,7,8,9-HxCDF and 2.0ng of 13C12 1,2,3,4,7,8,9-HpCDF.

For Brominated Dioxin sampling, the sorbent trap is filled with approx 70g of Amberlite XAD-2 resin, it is spiked with 1.0ng of 13C12 2,4,6,8-TBDF.

PCBs - (Filter / Condenser Method) **(UKAS/MCERTS)**

Determination of **PCBs** is carried out using a Universal Stack Sampling system in accordance with **BS EN 1948-1&4:2010** and In-house technical procedure **ECL/TPD/031**.

In this method the stack gases are sampled via a heated titanium probe and are then filtered (110mm out-stack, heated quartz or GFA filter) to remove particulate matter. The filtered gases are then passed through a cooling condenser and spiked XAD absorber, a verification absorption stage is not used. The gases the pass through a condensate flask and a series of silica gel filled impingers.

The gases are cooled via the condenser where the cooled gases and condensate percolate through the XAD absorber accumulate in the condensate flask before being dried by the silica impingers.

Prior to every sampling campaign, the sample train glassware (upstream of the silica impingers) is rinsed with solvents and then muffled at 450 °C for three hours, before being carefully capped for transport to site. (The probe cannot be muffled, so is heated to 180 °C for three hours using its own heater). If the probe or any of the glassware is re-used on site (for further samples) additional rinses of the glassware are taken prior to the re-use and kept for analysis in case of high results being obtained for the following sample.

The sorbent trap is filled with approx 70g of Amberlite XAD-2 resin, it is spiked with 1.0ng of 13C12 PCB 60 & PCB 159.

TVOC as Carbon (UKAS/MCERTS)

Testing is carried out using an MCERTS Certified Signal 3030PM FID and heated gas sample line, with reference to the manufacturer's operation handbook, **BS EN 12619:2013** and in-house technical procedure **ECL/TPD/032A**.

The analyser is calibrated on site using certified propane span gases, (made up in synthetic air) which are traceable to ISO 17025 standard. (with uncertainty <2%).

Zero measurements are performed using synthetic air zero gas, with TVOC content less than 0.2 mg/m^3 (or purity greater than 99.998%).

The analyser is calibrated directly into the sample inlet and then checked through the entire sampling system (including sampling probe, heated filter and heated gas transport lines). Data is corrected by molecular weight to TVOCs as total carbon.

Data is recorded as minute averages over each test period. The data is presented in the Figures Section and the minute averaged data is detailed in the Tables Section.

TVOC as Carbon (UKAS/MCERTS)

Testing is carried out using an MCERTS Certified Sick F3006 FID and heated gas sample line, with reference to the manufacturer's operation handbook, **BS EN 12619:2013** and in-house technical procedure **ECL/TPD/032B**.

The analyser is calibrated on site using certified propane span gases, (made up in synthetic air) which are traceable to ISO 17025 standard. (with uncertainty <2%).

Zero measurements are performed using synthetic air zero gas, with TVOC content less than 0.2 mg/m^3 (or purity greater than 99.998%).

The analyser is calibrated directly into the sample inlet and then checked through the entire sampling system (including sampling probe, heated filter and heated gas transport lines). Data is corrected by molecular weight to TVOCs as total carbon.

Data is recorded as minute averages over each test period. The data is presented in the Figures Section and the minute averaged data is detailed in the Tables Section.

TVOC as Carbon (UKAS/MCERTS)

Testing is carried out using an MCERTS Certified SK-Elektronik UPAS-FID PT and heated gas sample line, with reference to the manufacturer's operation handbook, **BS EN 12619:2013** and in-house technical procedure **ECL/TPD/032C**.

The analyser is calibrated on site using certified propane span gases, (made up in synthetic air) which are traceable to ISO 17025 standard. (with uncertainty <2%).

Zero measurements are performed using synthetic air zero gas, with TVOC content less than 0.2 mg/m^3 (or purity greater than 99.998%).

The analyser is calibrated directly into the sample inlet and then checked through the entire sampling system (including sampling probe, heated filter and heated gas transport lines). Data is corrected by molecular weight to TVOCs as total carbon.

Data is recorded as minute averages over each test period. The data is presented in the Figures Section and the minute averaged data is detailed in the Tables Section**.**

Combustion Gases (NO_x, SO_2, CO, O_2 & CO_2) (UKAS/MCERTS)

Measurements of combustion gases are carried out using an MCERTS Certified **Horiba PG 250** stack gas analyser. Continuous monitoring of emissions is undertaken over each test period recording minute averaged data (one measurement every 60 seconds). The measurement techniques for each determinand are as follows:

Determinand	Technique	SRM
• NO_x	Chemiluminescence	BS EN 14792: 2017
• SO_2	Non dispersive infrared	CEN/TS 17021: 2017
• CO	Non-dispersive infrared	BS EN 15058: 2017
• O_2	Galvanic / Zirconia	BS EN 14789: 2017
• CO_2	Non-dispersive infrared	PD CEN/TS 17405:2020

The analyser is set up with reference to the manufacturers operator handbook and the in-house technical procedure **ECL/TPD/033c**. The analyser is calibrated on site using certified gases which are traceable to ISO 17025. (with uncertainty <2%). Zero measurements are performed using Nitrogen. The analyser is calibrated directly into the sample inlet and then checked through the entire sampling system (including sampling probe, heated & unheated gas transport lines and gas drying/ conditioning system).

Data is presented graphically in the Figures Section, and the minute averaged data is given in the Tables Section.

Combustion Gases (NO_x, CO, O_2 & CO_2) (UKAS/MCERTS)

Measurements of combustion gases are carried out using an MCERTS Certified **Horiba PG 350(E)** stack gas analyser. Continuous monitoring of emissions is undertaken over each test period recording minute averaged data (one measurement every 60 seconds). The measurement techniques for each determinand are as follows:

Determinand	Technique	SRM
NO_x	Chemiluminescence	BS EN 14792: 2017
CO	Non-dispersive infrared	BS EN 15058: 2017
O_2	Paramagnetic Sensor	BS EN 14789: 2017
CO_2	Non-dispersive infrared	PD CEN/TS 17405:2020

The analyser is set up with reference to the manufacturers operator handbook and the in-house technical procedure **ECL/TPD/033d**. The analyser is calibrated on site using certified gases which are traceable to ISO 17025. (with uncertainty <2%). Zero measurements are performed using Nitrogen. The analyser is calibrated directly into the sample inlet and then checked through the entire sampling system (including sampling probe, heated & unheated gas transport lines and gas drying/ conditioning system).

Data is presented graphically in the Figures Section, and the minute averaged data is given in the Tables Section.

Combustion Gases (NO_x, CO, O_2 & CO_2) (UKAS/MCERTS)

Measurements of combustion gases are carried out using an MCERTS Certified **Horiba PG 350 (EU P-AMS)** stack gas analyser. Continuous monitoring of emissions is undertaken over each test period recording minute averaged data (one measurement every 60 seconds). The measurement techniques for each determinand are as follows:

Determinand	Technique	SRM
• NO_x	Chemiluminescence	BS EN 14792: 2017
• CO	Non-dispersive infrared	BS EN 15058: 2017
• O_2	Paramagnetic Sensor	BS EN 14789: 2017
• CO_2	Non-dispersive infrared	PD CEN/TS 17405:2020

The analyser is set up with reference to the manufacturers operator handbook and the in-house technical procedure **ECL/TPD/033D**. The analyser is calibrated on site using certified gases which are traceable to ISO 17025. (with uncertainty <2%). Zero measurements are performed using Nitrogen. The analyser is calibrated directly into the sample inlet and then checked through the entire sampling system (including sampling probe, heated & unheated gas transport lines and gas drying/ conditioning system).

Data is presented graphically in the Figures Section, and the minute averaged data is given in the Tables Section.

Halogen Halides & Halogens (UKAS/MCERTS)

Testing is carried out (isokinetically or non-isokinetically) using a Universal Stack Sampling system in accordance with **US EPA Method 26a, US EPA Method 26** and In-house technical procedure **ECL/TPD/035**. Non-isokinetic sampling can only take place if there are no droplets present in the stack gas.

In this method the stack gases are filtered to remove particulate matter then the gases are passed through a series of impingers. The first two impingers each containing 100ml of 0.1N Sulphuric Acid (0.1N H_2SO_4) the second set of impingers each containing 100ml of 0.1N Sodium Hydroxide (0.1N NaOH) and finally through an impinger containing a measured quantity of silica gel.

The first two impingers containing the 0.1N Sulphuric Acid are analysed for concentrations of Hydrogen Chloride and Hydrogen Fluoride by IC (Ion Chromatography). The second set of impingers containing 0.1N Sodium Hydroxide are analysed by IC (Ion Chromatography) for the concentrations of Chlorine.

Hydrogen Chloride (UKAS/MCERTS)

Testing is carried out (isokinetically or non-isokinetically) using a Universal Stack Sampling system in accordance with **BS EN 1911:2010** and In-house technical procedure **ECL/TPD/081**. Non-isokinetic sampling can only take place if there are no droplets present in the stack gas.

In this method the stack gases are filtered to remove particulate matter then the gases are passed through a series of impingers. The first two or three impingers each containing 100ml of De-ionised Water and finally through an impinger containing a measured quantity of silica gel.

The impingers containing the de-ionised water are analysed for concentrations of Hydrogen Chloride by IC (Ion Chromatography).

Polycyclic Aromatic Hydrocarbons (PAH) - (UKAS/MCERTS)

(Heated Filter / Condenser / Absorber Method)

Determination of **PAH** is carried out using a Universal Stack Sampling system in accordance with **BS ISO 11338:2003** and In-house technical procedure **ECL/TPD/037**.

In this method the stack gases are sampled via a heated titanium probe and are then filtered (110mm out-stack, heated quartz or GFA filter) to remove particulate matter. The filtered gases are then passed through a cooling condenser and spiked XAD absorber, a verification absorption stage is not used. The gases the pass through a condensate flask and a series of silica gel filled impingers.

The gases are cooled via the condenser where the cooled gases and condensate percolate through the XAD absorber accumulate in the condensate flask before being dried by the silica impingers.

The sorbent trap is filled with approx 70g of Amberlite XAD-2 resin, it is spiked with 1µg of d10-Fluorene.

Ammonia (UKAS ONLY)

Testing is carried out (isokinetically or non-isokinetically) using a Universal Stack Sampling system in accordance with **US EPA Method 26a, US EPA Method 26** and In-house technical procedure **ECL/TPD/038**. Non-isokinetic sampling can only take place if there are no droplets present in the stack gas.

In this method the stack gases are filtered to remove particulate matter then the gases are passed through a series of impingers. The first two impingers each containing 100ml of 0.1N Sulphuric Acid (0.1N H_2SO_4). The third impinger is left empty and finally through an impinger containing a measured quantity of silica gel.

The first two impingers containing the 0.1N Sulphuric Acid are analysed for concentrations of Ammonia by IC (Ion Chromatography).

Ammonia (UKAS/MCERTS)

Testing is carried out (isokinetically or non-isokinetically) using a Universal Stack Sampling system in accordance with **BS EN ISO 21877:2019** and In-house technical procedure **ECL/TPD/034**. Non-isokinetic sampling can only take place if there are no droplets present in the stack gas.

In this method the stack gases are filtered (quartz filter) to remove particulate matter then the gases are passed through a series of impingers. The first two impingers each contain 140ml of 0.1N Sulphuric Acid (0.1N H_2SO_4). The third impinger is left empty and the final impinger contains a measured quantity of silica gel.

The first two impingers containing the 0.1N Sulphuric Acid (and the empty impinger) are analysed for concentrations of Ammonia by IC (Ion Chromatography).

Sulphur Dioxide (UKAS/MCERTS)

Testing is carried out (isokinetically or non-isokinetically) using a Universal Stack Sampling system in accordance with **BS EN 14791:2017** and In-house technical procedure **ECL/TPD/039**. Non-isokinetic sampling can only take place if there are no droplets present in the stack gas.

In this method the stack gases are filtered to remove particulate matter then the gases are passed through a series of impingers. The first two impingers each contain 140ml of 3% Hydrogen Peroxide (3% H_2O_2). The third impinger is left empty and the final impinger contains a measured quantity of silica gel.

The first two impingers containing the 3% Hydrogen Peroxide (and the empty impinger) are analysed for concentrations of Sulphur Dioxide by IC (Ion Chromatography).

Homogeneity (UKAS/MCERTS)

Testing for homogeneity is carried out in accordance with section 8.3 of **BS EN 15259:2007** and In-house technical procedure **ECL/TPD/040**.

If the following determinands have ELVs in an operators permit then a homogeneity test is applicable for each of them. **NOx, CO, TOC**. (Other parameters may need homogeneity testing too). If the NO_2 concentration is less than 10% of the total NOx, then the homogeneity test for NOx may be carried out measuring NO only. 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. (if the TOC concentration is below 3mg/m^3 then the variability in the readings may make it difficult to apply the homogeneity test. The homogeneity test applies to oxygen because it is used to correct final results. However, the homogeneity tests are not oxygen corrected. The homogeneity test may apply to H_2O, if it is used to correct the final measurement result. For processes WITHOUT liquid based abatement systems, gases such as O_2 may be used as an alternative for H_2O.

Due to practical reasons, the homogeneity test is carried out using instrumental measurement techniques. For determinands, where the CEN/ISO SRM is a non-instrumental method, an alternative instrument based method may be used.

Water Vapour (UKAS/MCERTS)

Testing is carried out using a Universal Stack Sampling system in accordance with **BS EN 14790:2017** and In-house technical procedure **ECL/TPD/082**.

In this method the stack gases are filtered (in-stack unheated filter or out-stack heated filter) to remove particulate matter. The gases are then passed through a **heated probe** and then to a cooled moisture trapping unit. All unheated parts of the sample train (outside the sample port) which come into contact with stack gas are weighed pre and post sampling in order to determine the weight gain.

After each test, a visual inspection of the last impinger is made to confirm that at least 50% of the silica gel column has not changed colour. This indicates satisfactory collection of water vapour.

Hydrogen Fluoride (UKAS/MCERTS)

Testing is carried out (isokinetically or non-isokinetically) using a Universal Stack Sampling system in accordance with **PD CEN/TS 17340:2020** and In-house technical procedure **ECL/TPD/081**. Non-isokinetic sampling can only take place if there are no droplets present in the stack gas.

In this method the stack gases are filtered to remove particulate matter then the gases are passed through a series of impingers. The first two or three impingers each containing 100ml of De-ionised Water and finally through an impinger containing a measured quantity of silica gel.

The impingers containing the DI Water are analysed for concentrations of Hydrogen Fluoride by IC (Ion Chromatography).

Speciated VOC (UKAS/MCERTS)

Non-continuous sampling for **Speciated VOC** is carried out in accordance with **CEN/TS 13649:2014** and In-house technical procedure **ECL/TPD/084.** In this method a metered volume of stack gas is extracted through a standard charcoal sorbent tube/ thermal desorption tube.

Due to restrictions set out in CEN/TS 13649:2014, MCERTS/UKAS accreditation can only be claimed when the target parameters are organic compounds, the sorbent tube used is a standard charcoal tube/ thermal desorption tube and when laboratory analysis is UKAS accredited and carried out by GC. If other tubes are used, or if analysis is by other means than GC, then usually only UKAS accreditation can be claimed (for organic compounds only), as long as the laboratory analysis is UKAS accredited. (MCERTS accreditation may still be claimed if prior approval is given for the modifications by the Environment Agency – details will be given in section 2 of this report).

(Exceptions to this restriction are allowed for compounds specifically listed in EA TGN M2 as being suitable for sampling based on CEN/TS 13649:2014).

Laboratory analysis **cannot** be UKAS accredited for “Total VOC” or “TOP 10 compounds”.

For the subcontract laboratory to claim UKAS accreditation for analysis, the internal recovery of a spiking compound (desorption efficiency from tube) needs to be above 80%. If it falls below 80% this will be noted on the analysis certificate.

If greater than 5% of the total amount of any of the target species is found in the back up portion of the sorbent tube, this will be noted on the analysis certificate.

QAL 2 & AST sampling and reporting (UKAS/MCERTS)

QAL 2 & AST sampling and reporting is carried out in accordance with **BS EN 14181:2014, MID 14181 & (TGN M20)** and In-house technical procedures **ECL/TPD/087, 088, 089 & 090.**

Oxygen (UKAS/MCERTS)

Measurements of Oxygen are carried out using a Testo 350XL electrochemical cell combustion gas analyser which has been validated to meet the performance requirements of **BS EN 14789:2017** Continuous monitoring of emissions is undertaken over each test period recording minute averaged data.

The analyser is set up with reference to the manufacturers operator handbook and the in-house technical procedure **ECL/TPD/086**. The analyser is calibrated on-site using certified gases which are traceable to ISO 17025 (with uncertainty <2%). (for emissions streams where oxygen is above 15%, dry ambient air can be used to calibrate the analyser). Zero measurements are performed using Nitrogen. The analyser is calibrated directly into the sample inlet (which is up-stream of the built-in chiller system) and then checked through the entire sampling system (including sampling probe, short heated/ unheated gas transport lines and external gas conditioning systems as required).

Combustion Gases (NO, N_2O, SO_2, CO & O_2) (UKAS/MCERTS) FTIR 204/M

Measurements of combustion gases are carried out using an MCERTS Certified Protea 204M FTIR stack gas analyser, with a 20m heated sample line. (The 40 mg/m^3 range for N_2O has been validated to meet the performance requirements of **BS EN 21258).** Continuous monitoring of emissions is undertaken over each test period recording an average of several spectra recorded over approximately one minute intervals. The measurement techniques for each determinand are as follows:

Determinand	Technique	SRM
• NO, N_2O, SO_2 & CO	Fourier Transform Infra Red	**PD CEN/TS 17337:2019**
• O_2	Zirconia Cell	**AM BS EN 14789:2017**

The analyser is set up with reference to the manufacturer's operator handbook and the in-house technical procedure **ECL/TPD/097**. Direct response checks and system losses checks are carried out on site for all gases. The analysers response test is carried out through the entire sampling system annually. All tests are carried out using certified gases which are traceable to ISO 17025 (with uncertainty <2%). Zero measurements are performed using Nitrogen. Data is presented graphically in the Figures Section, and the minute averaged data is given in the Tables Section.

The data recording & manipulation software contains built-in checks on the analysis, including the interference test. This check is done in real-time after each analysis and the software is set to read Pass or Fail (green or red) depending on the outcome. Any red (failed) data is rejected. The limits of detection for each determinand are as follows:

Determinand	Limit of Detection
• NO	1.2 mg/m^3
• N_2O	0.2 mg/m^3
• SO_2	0.5 mg/m^3
• CO	0.8 mg/m^3
• O_2	0.2% (v/v)

Combustion Gases (NO, NO_2, N_2O, SO_2, NH_3, CO, CO2 & O_2) (UKAS/MCERTS) AtmosFIR AFS-B2T

Measurements of combustion gases are carried out using an MCERTS Certified Protea AtmsoFIR (AFS-B2T) stack gas analyser, with a heated sample line. Continuous monitoring of emissions is undertaken over each test period recording an average of several spectra recorded over approximately one minute intervals. The measurement techniques for each determinand are as follows:

Determinand	**Technique**	**SRM**
• NO, N_2O, NO_2, SO_2 CO, CO2 & NH_3	Fourier Transform Infra Red	**PD CEN/TS 17337:2019**
• O_2	Zirconia Cell	**AM BS EN 14789:2017**

The analyser is set up with reference to the manufacturer's operator handbook and the in-house technical procedure **ECL/TPD/098**. Direct response checks and system losses checks are carried out on site for all gases. The analysers response test is carried out through the entire sampling system annually. All tests are carried out using certified gases which are traceable to ISO 17025 (with uncertainty <2%). Zero measurements are performed using Nitrogen. Data is presented graphically in the Figures Section, and the minute averaged data is given in the Tables Section.

The data recording & manipulation software contains built-in checks on the analysis, including the interference test. This check is done in real-time after each analysis and the software is set to read Pass or Fail (green or red) depending on the outcome. Any red (failed) data is rejected. The limits of detection for each determinand are as follows:

Determinand	**Limit of Detection**
• NO	1.0 mg/m^3
• NO_2	0.6 mg/m^3
• N_2O	0.4 mg/m^3
• SO_2	0.6 mg/m^3
• CO	0.6 mg/m^3
• CO_2	0.05% (v/v)
• O_2	0.2% (v/v)
• Ammonia	0.1 mg/m^3

Oil Mist & Cyclohexane Extractable Materials (in-stack sampling only) (Oil Mist, Tar & Bitumen Fume UKAS/MCERTS). (Rubber Fume & Other Parameters UKAS Only).

Testing is carried out using a Manual Stack Sampling system in accordance with **BS EN 13284-1:2017 & TGN M2** and In-house technical procedure **ECL/TPD/94 (or 94a STACKMITE CONTROL UNIT ONLY)**.

Isokinetic sampling is achieved when the velocity of gas entering the sampling nozzle is exactly equal to the velocity of the approaching gas stream within the stack.

A measured volume of sample gas is withdrawn from the stack isokinetically through a sampling nozzle and through a 37mm, 47mm or 110mm pre-cyclohexane extracted and pre-weighed GFA or Quartz filter, positioned in an unheated housing inserted into the stack.

Oil Mist (and other cyclohexane extractable material) is collected on the filter. Following testing the front half of the filter housing and the sample nozzle are rinsed with acetone to remove any material which may have impacted on the surfaces during testing.

The filters are subsequently analysed gravimetrically based on MDHS 84, to determine the amount of cyclohexane extractable material captured. The rinses are analysed to BS EN 13284-1, to determine the total particulate matter collected.

PM10 & PM 2.5 (UKAS/MCERTS)

Testing is carried out using a Manual Stack Sampling system in accordance with **BS EN ISO 23210:2009** and In-house technical procedure **ECL/TPD/095.**

Sampling is made using the TCR Tecora model MSSI impactor. This sampling system has been validated as detailed in the following ISO document.

ISO/TC 146/SC 1/WG 20 N83

Isokinetic sampling is achieved when the velocity of gas entering the sampling nozzle is exactly equal to the velocity of the approaching gas stream within the stack.

A measured volume of sample gas is withdrawn from a single representative sample point in the stack at a fixed sample rate (90 – 130% of isokinetic rate) through a sampling nozzle and then through an unheated 2-stage impactor head inserted into the stack. The impactor head contains two 47mm quartz impactor plates and one 47mm quartz back-up filter. These plates and filters are pre-blown and pre-weighed in accordance with **BS EN 13284-1:2017.**

Particulate matter is separated by size fractionation onto the impactor plates and back up filter. The plates and filters are subsequently analysed to determine the amount of particulate matter captured in each size range.

The standard pre-sample conditioning temperature of the filters is 180°C and the standard post-sample conditioning temperature is 160°C. (Any modifications to this are noted in section 2 of this report). Apparent weights are corrected, if required, based on the weights of three control filters and evaporating basins which are weighed (pre and post sampling) with each batch of filters & rinses.

The impactor head is rinsed between samples, but no rinses are collected for analysis as part of this sampling procedure.

Isocyanates

Non-continuous sampling for Isocyanates is carried out **isokinetically,** in accordance with **USEPA CTM 36, TGN M2** and In-house technical procedure **ECL/TPD/096 or 096A**.

For in-stack sampling, a measured volume of sample gas is withdrawn from the stack, through a titanium sampling nozzle and through a 1-(2-pyridyl)piperazine (1,2-PP) pre-treated 37mm or 47mm filter, positioned in an unheated titanium filter housing inserted into the stack. Isocyanates are collected on the filter.

For out-stack sampling, a measured volume of sample gas is withdrawn from the stack isokinetically through a titanium or glass sampling nozzle and heated/ unheated titanium or glass sampling probe, and then through a 1-(2-pyridyl)piperazine (1,2-PP) pre-treated 37mm or 47mm filter, positioned in a heated/ unheated glass or titanium filter housing.

Following testing, the front half of the filter housing, (the probe for out stack sampling) and the sample nozzle are rinsed with Acetone & a mix of 80% Acetonitrile / 20% DMSO to remove any isocyanate which may have impacted on the surfaces during testing.

The filter and rinse(s) are then combined into the same container, before subsequent analysis by HPLC to determine the amount of isocyanate captured.

DETAILED SAMPLING PROTOCOLS / METHODOLOGIES

Part 2 – Non-Accredited Methods

Sulphuric Acid Mist

Testing is carried out using a Universal Stack Sampling system in accordance with **US EPA Method 8.** In this method stack gas is extracted through a heated probe and through an impinger containing 80% Isopropanol. The sample is then passed through a glass fibre filter and then through two impingers containing 3% Hydrogen Peroxide and finally through an impinger containing a measured quantity of silica gel.

The 80% isopropanol and glass fibre filter samples can be analysed for sulphuric acid mist and sulphur trioxide. The 3% Hydrogen Peroxide can be analysed for sulphur dioxide.

ECL does not hold UKAS accreditation for US EPA Method 8.

Formaldehyde

Testing is carried out isokinetically using a Manual Stack Sampling system in accordance with **US EPA Method 316**. In this method the stack gases are filtered to remove particulate matter then the gases are passed through a series of impingers containing Deionised Water. The first three impingers contain 100ml of De-ionised Water and finally through an impinger containing a measured quantity of silica gel.

The impingers containing the de-ionised water are analysed for concentrations of Formaldehyde by HPLC.

ECL does not hold UKAS accreditation for US EPA Method 316.

Hydrogen Cyanide

Testing is carried out isokinetically using a Manual Stack Sampling system in accordance with **USEPA OTM-29** In this method the stack gases are filtered to remove particulate matter then the gases are passed through a series of impingers. The first three impingers each containing 100ml of 0.1m Sodium Hydroxide (NaOH) and finally through an impinger containing a measured quantity of silica gel.

The impingers containing the NaOH are analysed for concentrations of Hydrogen Cyanide by Colorimetry.

ECL does not hold UKAS accreditation for US EPA OTM-29.

Odour (Threshold)

Measurements of Odour (Threshold) are carried out using an Air Spectrum vacuum chamber which has been validated to meet the performance requirements of **BS EN 13725:2003.**

Sampling involves capturing a sample of stack gas in an inert nalophan bag housed inside a vacuum chamber. By evacuating air from inside the vacuum chamber using an air-to-air pump, the bag, connected via PTFE tubing to a probe sited in the stack seeks to remain in pressure equilibrium with the air at the end of the sealed system and thus fills with stack gas.

All bags are pre-conditioned with a small sample of stack gas before being emptied and filled with the sample gas. Samples were taken in triplicate from each of the emission points, in accordance with the monitoring standard.

ECL does not hold UKAS accreditation for BS EN 13725:2003.

Particulate Phase Chrome (vi)

Testing is carried out using a Manual Stack Sampling system based on **BS EN 13284-1:2017.**

Isokinetic sampling is achieved when the velocity of gas entering the sampling nozzle is exactly equal to the velocity of the approaching gas stream within the stack.

A measured volume of sample gas is withdrawn from the stack isokinetically, through a sampling nozzle and through a NaOH treated 37mm PVC filter, positioned in an unheated housing inserted into the stack.

The filters are subsequently analysed based on **NIOSH 7605/7600,** to determine the amount of Chromium (vi) captured.

ECL does not hold UKAS accreditation for this sampling method.