

Ambient, Indoor, Workplace Air and Stack Emissions Proficiency Testing Scheme

Version 1
Issue Date 11/04/2014

Sample Preparation Instructions – Round 1

General Instructions

Sample Storage

All samples should be stored in accordance with the instructions provided on the sample labels from the time of arrival at your laboratory. If a preservative is routinely added to the type of sample provided, as part of your laboratory procedures, a suitable aliquot should be preserved as soon as possible in the normal way. Any dilutions that result from addition of preservatives should be corrected for before submission of results.

All samples should be stored, prior to analysis, in the format provided to minimise potential from contamination. VOC samples on sorbent tubes should be stored away from other potential sources of solvent in the laboratory.

Sample Preparation

All samples should be equilibrated at room temperature $20(\pm 5)^\circ\text{C}$ before any analyses are performed. Samples should be prepared in accordance with the specific instructions for the group. These procedures should be followed exactly to ensure comparability of results

Sample Analysis

Samples should be analysed by the normal method or methods used by your laboratory. Replicate determinations can be made if this is normal laboratory procedure, although only one value per method can be submitted to LGC Standards Proficiency Testing for statistical analysis and reporting on laboratory performance.

All samples should be treated like any other samples and all normal quality control procedures should be adopted.

Results should be corrected for recovery and blank, if appropriate, and if this is the normal practice in the laboratory. **If the sample is diluted as part of the analytical process, such dilutions should be corrected for.**

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Workplace Air Samples

Sample 1 – Metals

Materials Supplied

- 4 x 25 mm diameter mixed cellulose ester filter spiked with metal solutions plus 2 x blank filters.

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
Cadmium	ug	1
Chromium	ug	1
Cobalt	ug	1
Copper	ug	1
Iron	ug	1
Manganese	ug	1
Nickel	ug	1
Lead	ug	1
Zinc	ug	1

Sample 2 – Quartz (by FTIR or XRD)

Materials Supplied

- 4 x 25 mm diameter PVC filters (GLA5000) loaded with aerosolised quartz by employing the BCIRA respirable sampler (Higgins-Dewell design). One blank filter supplied for participants using XRD technique, up to four blank filters for participants using FTIR technique.

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
Respirable grade quartz	ug	1

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Sample 5 – Volatile Organic Compounds

Materials Supplied

- 4 x charcoal filled glass sorbent tubes dynamically loaded from the gas phase (prepared in accordance with procedures set out in ISO 6145 part 4) for analysis by solvent desorption plus 4 x blank sorbent tubes. Two of these blank sorbent tubes are unopened to be used if required by participant for internal spike recovery tests

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
Benzene	ug	1
Toluene	ug	1
Xylene (all isomers)	ug	1
Ethyl benzene	ug	1

Sample 6 – Volatile Organic Compounds

Material Supplied

- 4 x charcoal filled glass sorbent tubes dynamically loaded from the gas phase (prepared in accordance with procedures set out in ISO 6145 part 4) for analysis by solvent desorption plus 4 x blank sorbent tubes. Two of these blank sorbent tubes are unopened to be used if required by participant for internal spike recovery tests.

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
1,1,1,-trichloroethane	ug	1
n-hexane	ug	1
n-butyl acetate	ug	1
Trichloroethene	ug	1
Tetrachloroethene	ug	1

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Sample 7 – Volatile Organic Compounds

Material Supplied

- 4 x Tenax TA filled sorbent tubes (Perkin Elmer type-6.4mm OD, 5 mm ID and 90 mm long) dynamically loaded from the gas phase (prepared in accordance with procedures set out in ISO 6145 part 4) for analysis by thermal desorption plus 2 x blank sorbent tubes.

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
Benzene	ug	1
Toluene	ug	1
Xylene (all isomers)	ug	1
Ethyl benzene	ug	1

Sample 8 – Formaldehyde

Materials Supplied

- 4 x 25 mm diameter glass fibre filters (GFA) spiked with formaldehyde derivative, 2,4-DNPH plus 2 x DNPH only treated blanks.

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
Formaldehyde 2,4-DNPH derivative	ug	2

Ambient Air Samples

Sample 11 – Nitrogen Dioxide

Materials Supplied

- 4 x Palmes-type diffusion tubes spiked with sodium nitrite as a surrogate for Nitrogen dioxide (NO₂)

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
NO ₂ as nitrite	ug	2

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Sample 12 – Volatile Organic Compounds

Materials Supplied

- 4 x Tenax TA filled sorbent tubes (Perkin Elmer type - 6.4mm OD, 5 mm ID and 90 mm long) dynamically loaded from the gas phase (prepared in accordance with procedures set out in ISO 6145 part 4) for analysis by thermal desorption plus 2 x blank sorbent tubes

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
Benzene	ng	1
Toluene	ng	1
Xylene (all isomers)	ng	1
Ethyl benzene	ng	1

Sample 13 – Metals at Ambient air concentrations

Materials Supplied

- 4 x 47 mm diameter quartz fibre filters spiked with metal solutions plus 2 x blank filters.

Preparation

Analyse each sample as supplied by your chosen instrumental technique for the analytes below:

Analyte	Units	DP
Cadmium	ng	1
Nickel	ng	1
Lead	ng	1
Arsenic	ng	1

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STACK EMISSION Samples

Sample 31 – Mercury

Materials Supplied

- 1 x 150-300mL sample of 2% m/m potassium permanganate/10% m/m sulphuric acid impinger solution (prepared in accordance with BS EN 13211) containing mercury.

Preparation

Determine the sample volume then analyse the sample by your chosen instrumental technique for mercury.

Analyte	Units	DP
Volume	mL	0
Mercury	mg/L	3

Sample 33 – Metals

Materials Supplied

- 1 x 150-300mL 3.3% nitric acid impinger solution (prepared in accordance with BS EN 14385) containing heavy metals.

Preparation

Determine the sample volume then analyse the sample by your chosen instrumental technique for the following analytes:

Analyte	Units	DP
Volume	mL	0
Arsenic	mg/L	3
Cadmium	mg/L	3
Chromium	mg/L	3
Lead	mg/L	3
Nickel	mg/L	3
Vanadium	mg/L	3

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Sample 34 – Sulfur dioxide

Materials Supplied

- 1 x 150-400mL hydrogen peroxide impinger solution (prepared in accordance with BS EN 14791) containing sulfur dioxide (to be determined as sulfate).

Preparation

Determine the sample volume then analyse the sample by your chosen instrumental technique for sulfur dioxide (as sulfate).

Analyte	Units	DP
Volume	mL	0
Sulfur dioxide (determined as sulfate)	mgSO ₄ /L	2

Sample 35 – Hydrogen fluoride

Materials Supplied

- 1 x 100-250mL 0.1M sodium hydroxide impinger solution (prepared in accordance with BS EN 15713) containing fluoride.

Preparation

Determine the sample volume then analyse the sample by your chosen instrumental technique for hydrogen fluoride.

Analyte	Units	DP
Volume	mL	0
Hydrogen fluoride	mgHF/L	2

Sample 38 – Metals on filters

Materials Supplied

- 1 x quartz or PTFE filter (prepared in accordance with BS EN 14385) containing heavy metals

Preparation

Analyse the sample by your routine technique for the following analytes in this sample matrix. Information on applicable methods is available in BS EN 14385.

Analyte	Units	DP
Arsenic	ug	1
Cadmium	ug	1
Chromium	ug	1
Lead	ug	1
Nickel	ug	1
Vanadium	ug	1

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Sample 39 – Rinsing solution, dust analysis (by Gravimetry)

Materials Supplied

- 1 x 250mL rinsing solution containing dissolved and suspended solids (prepared in accordance with EN 13284-1)

Preparation

- Rinse the entire contents of the sample container into a suitable dried and pre-weighed container.
- Evaporate in an oven at 120°C.
- After evaporation, place the weighing containers in the drying oven for 1 hour at 160°C.
- Cool to ambient temperature and calculate the total solids content in the sample (reporting in mg)

Analyte	Units	DP
Total Solids	mg	2