

Stanger

Science and Environment

Report for

**CEN/TC264/WG14:
Minimum Validation
Programme –
Avonmouth Field Trials**

22 October 2001

Report for

CEN/TC264/WG14: Minimum Validation Programme – Avonmouth Field Trials

Prepared by

Dr Richard Maggs
Principal Consultant

Prepared for

Dr Rudolph Neuroth
Secretariat of CEN/TC264/WG14
KRdL im VDI und DIN – Normenausschuss
Postfach 10 11 39
D-40002 Düsseldorf
GERMANY

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1 Introduction

This report summarises the results of the individual Member States participating laboratories in the CEN TC 264/WG14 Minimum Validation Programme field trials completed during the months of July – August 2001 at the Port of Avonmouth site, Bristol, UK. The following laboratories participate in the programme:

- Spain (Lab A: Instituto de Salud Carlos III)
- UK (Lab B: Stanger Science and Environment/ CRE Group/ Harwell Scientifics)
- Belgium (Lab C: VVM)
- Germany (Lab D: UBA/ Austria (UBA))

The purpose of the field trial is to perform field validations of the proposed draft standard reference method, which includes all steps covering sampling, sample preparation and analysis of samples.

An overview of the field tests is provided in CEN/TC264/WG14 Document N147Rev5. In summary, this involves all four laboratories undertaking analysis of samples by GF-AAS and two laboratories undertaking additional voluntary analysis of samples using ICP-MS. The following provides a summary with respect to the individual undertakings by the separate laboratories:

1. Lab A: GF-AAS analysis of samples obtained using quartz fibre filters (Munktell MK360 (50mm))
2. Lab B: GF-AAS and ICP-MS analysis of samples obtained using membrane filters (Sartorius Cellulose Acetate 3 µm (50mm))
3. Lab C: GF-AAS analysis of samples obtained using membrane filters (Sartorius Cellulose Acetate 3 µm (50mm))
4. Lab D: GF-AAS and ICP-MS analysis of samples obtained using quartz fibre filters (Munktell MK360 (50mm))

The undertaking of the field trials at Avonmouth, Bristol, is one of four locations (2 urban and 2 industrial) at which sampling is to be undertaken across Member States. Prior to Avonmouth the CEN array of low volume samplers, was sited at an urban location in Madrid. The sampling at Madrid was completed in May 2001 and the report recently submitted by the Spanish participant to the Secretariat of CEN TC 264/WG14.

The results presented here summarise the analysis undertaken by each participating laboratory of samples gathered during the monitoring period at Avonmouth. The next

participant in the MVP is Germany whilst Belgium are tasked with undertaking the final period of monitoring scheduled for completion by the end of 2001.

This report is structured in the following way:

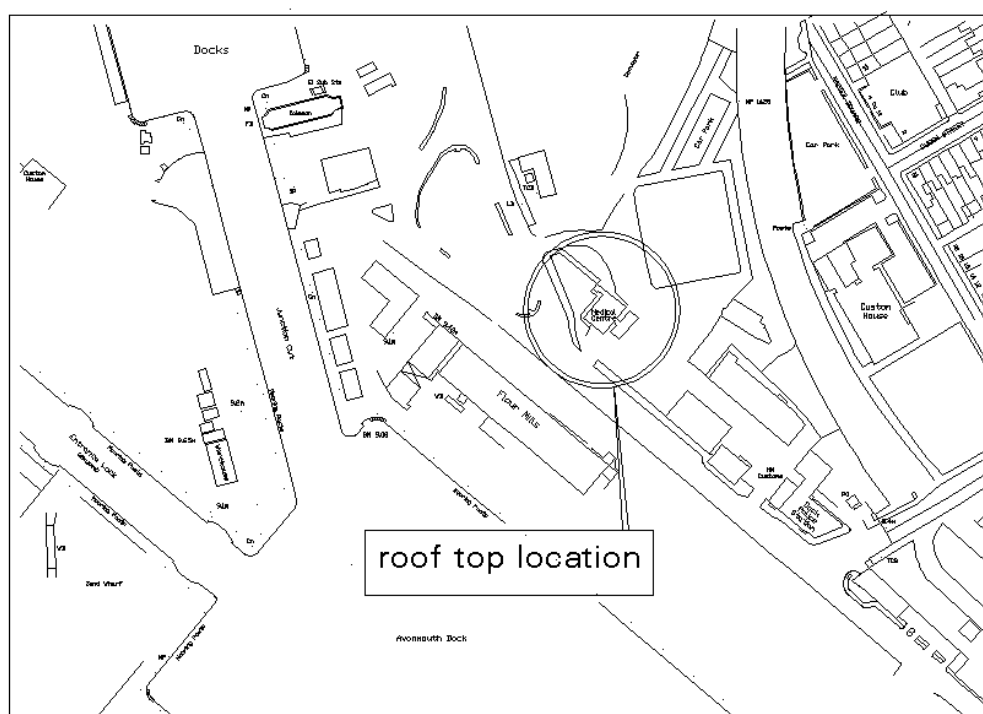
- Section 2 provides details with respect to the characteristics of the sampling location at Avonmouth, Bristol and the sampling regimes used. Information regarding any problems encountered during sampling are highlighted;
- Section 3 summarises the digestion procedures used by Member States in the preparation of the samples;
- Section 4 describes the analytical methods used in the MVP;
- Section 5 provides results related to the Avonmouth trials and is separated across two sections. Section 5.1 summarises results on the recovery rates of certified reference materials (CRMs), filter and reagent blanks, and field filter blanks, whilst Section 5.2 summarises results of the analysis of field samples;
- Section 6 provides a preliminary discussion on the results of the field trials at Avonmouth

2 Sampling site characteristics and regimes

2.1 Site characteristics

The Port of Avonmouth site is an industrial background site located in south-west England (Ordnance Survey grid reference 351245, 1781700). The specific monitoring location for the CEN array is shown in Figure 2.1. The equipment was located on top of a flat roof of a single-storey building of the Port Health Authority sites adjacent to a residential area. The area is one of mixed industrial activity (light and heavy) with a number of specific emission sources and the occurrence of fugitive dust sources. Figure 2.2 shows the CEN-array set-up on the rooftop.

Figure 2.1 Port of Avonmouth, Bristol – monitoring site¹



¹ Reproduced from the OS map with kind permission of the Ordnance Survey, © Crown Copyright. All rights reserved. Unauthorised reproduction infringes Crown Copyright and may lead to prosecution or civil proceedings. Licence number GD272671.

Figure 2.2 Port of Avonmouth, Bristol – monitoring site².



The site has been included in a previous assessment of ambient metal concentrations undertaken by the UK DEFRA and devolved administrations in order to establish the current UK position on ambient metal concentrations in anticipation of the Fourth Daughter Directive. The results are summarised at

<http://www.stanger.co.uk/airqual/metals/>

Results show annual mean metal concentrations (for year 2000) of:

- 7.7 ng/m³ for Cd,
- 2.7 ng/m³ for Ni,
- 104 ng/m³ for Pb and,
- 1.9 ng/m³ for As

² UK Partisol Plus and Partisol 2000 units are in the foreground, CEN LVS array (yellow units) located around the central electricity distribution unit

2.2 Sampling at Avonmouth

Sampling was carried out using the CEN array of low volume samplers (LVSs) at Avonmouth during the period 8 July to 2 August 2001.

Daily filter exchanges were undertaken by two operatives; Mr Andrew Edwards and Mr Nick Davey (hereby referred to as AE and ND in the Excel spreadsheets listed in Appendix 1) from Stanger Science and Environment's regional office in Bristol. In addition, Mr David Muir and Mr Dave Tuffery from Bristol City Council (referred to as DM and DT in the Excel spreadsheets listed in Appendix 1) also acted as local site operators. ND held overall on-site training and responsibility during the course of the sampling programme at Avonmouth.

Low volume samplers were calibrated and cleaned at the end of each of the four 5-day periods.

Problems were encountered with only one sampler during the course of the field trial at Avonmouth. This unit was LVS unit No. 7 (using quartz filters) and would not cease sampling according to the time set for the sampling period on 22 July 2001. Initial investigations by AE indicated that the unit would not re-set even after the mains electricity had been isolated. For this reason, samples from the spare LVS unit (No.10) were included in the analysis of quartz filters from the date of commencement of sampling (i.e. 8 July 2001) thereby replacing all samples taken from LVS unit No. 7.

Sampling data is shown in the pro-forma record sheets appended to this report (Appendix 1).

Upon completion of the field sampling at Avonmouth the National Physical Laboratory in the UK undertook a calibration check on the mass-flow controller. A copy of the full report is attached in Appendix 2. **For the purposes of this report no correction has been applied to volumetric data as a consequence of the calibration undertaken by NPL. The calibration is included for information and subsequent discussion by participants of WG14 only.**

3 Sample Preparation

Digestion procedures for the preparation of samples are outlined in CEN/TC264/WG14 Document N147. This document specifies details regarding reagents to be used and the temperature and/or pressure profiles required for the microwave system. Individual Member State laboratories due to laboratory equipment available can undertake changes to the technical details of Document N147. Where such changes are undertaken these are to be documented in the test report.

Individual summary reports submitted to Stanger Science and Environment on the analysis of field samples obtained during the Avonmouth field sampling show that all Member States followed the procedures set out in Document N147.

For each participating laboratory, the following equipment was employed for the digestion of samples:

- Lab A: Anton Paar/Perkin Elmer Multi-wave
- Lab B: CEM MARS 5 Microwave system
- Lab C: CEM MARS 5 Microwave system
- Lab D: Anton Paar/Perkin Elmer Multi-wave B30MC05A microwave system

Reagents used for digestion were in accordance with those specified in Document N147.

4 Analytical methods

The details regarding analytical methods are to be employed for the analysis of samples is also detailed in CEN/TC264/WG14 Document N147. Again, technical changes to the analytical conditions are allowed and should be justified on the basis of available equipment.

Two methods of analysis are employed and reported. All four laboratories participating in the programme are required to analyse samples by GF-AAS. In addition, two laboratories (Lab B and Lab D) have undertaken voluntary analysis of samples using ICP-MS.

For QA/QC purposes, analysis of samples includes necessary field blanks, reagent and laboratory blanks and the analysis of certified reference material (CRMs). CRMs employed in the current work are NIST 1648 (Urban Particulate Matter) and NIES No.8 (Vehicle Exhaust Particulate Matter). All QA/QC procedures outlined in Document N140Rev5 were followed.

The following provides details supplied by individual laboratories with respect to the analysis of the Avonmouth field samples.

4.1 GF-AAS

Lab A:

Measurements were made using an Atomic Absorption Spectrometer Perkin Elmer mod Analyst-100 with Graphite Furnace Perkin Elmer HGA-800 autosampler AS-72 with deuterium as background correction. The analysis was performed in accordance with Document CEN/TC264/WG14 N245.

Lab B:

Measurements were made using atomic absorption spectrometer (Varian Spectra AA-400 Zeeman). The analysis was performed in accordance with Document CEN/TC264/WG14 N245.

Lab C:

Specific details regarding calibration standards employed in the analysis of metals were not provided in the report submitted to Stanger Science and Environment for analysis of the Avonmouth samples. It is assumed that no deviations from the prescribed methodology set out in CEN/TC264/WG14 Document N147 occurred.

Lab D:

Calibration standards were prepared from single-element 1000 mg/l certified standard solutions obtained from Baker Instra-Analyzed. The standards are accurate to within 0.2% and are traceable to NIST. For Cd, standards within the range of 0.1 – 2.0ppb were used whilst for Pb, standard solutions in the range of 1 – 20ppb were employed. For As and Ni, calibration standards in the range of 1 – 20ppb were used.

4.2 ICP-MS

Lab B:

Calibration standards were prepared from a single-element 1000µg/ml certified standard solution at 0.1, 0.5, 1.0, 10, 25, 100 and 250µg/l (in the case of Pb only). A 10µg/ml multi-element stock solution was prepared by dilution of appropriate aliquots of single-element certified standard solutions. A secondary multi-element stock solution was prepared at 100 µg/l by dilution of 1 ml of the 1000µg/ml standard to 100ml using 16% nitric acid.

An internal standard stock solution was used prepared at 10 µg/ml by dilution of appropriate aliquots of the single-element certified standard solutions using 16% nitric acid. The internal standard solution contained germanium, yttrium, indium and bismuth

Lab D:

Before analysis of samples using ICP-MS, samples were diluted with water and internal standards added. For blank determinations, reported concentrations are based on original dilution (50 ml).

The same calibration procedure was used for samples, blanks and CRM solutions. The following dilution factors were applied prior to analysis:

- Samples and blanks: dilution factor 2
- NIST 1648: dilution factor 20
- NIES No.8: dilution factors 2, 4, 10

The following calibration standards were employed:

- Standard blank solution
- Operating standard – 1 solution (element conc.: 20 µg/l As, Pb, Cd, Ni)
- Operating standard – 2 solution (element conc.: 100 µg/ As, Pb, Cd, Ni)

5 Results

The following sections summarise results of the Avonmouth trials at Bristol with respect to analysis of filter and reagent blanks, field blanks and recovery rates of certified reference material. In addition, results are provided for the analysis of field samples using GF-AAS and ICP-MS (for Labs B and D).

Individual data supplied by participating laboratories for analysis of the Avonmouth samples are provided electronically in the files listed in Appendix 1.

5.1 Filter and reagent blanks, field filter blanks and CRM recoveries

Tables 5.1, 5.2, 5.3, 5.4 and 5.5 summarise the analysis of filter blanks, reagent blanks, field blanks and recovery rates of CRM for GF-AAS analysis undertaken by the four laboratories.

Table 5.1: Filter blank concentrations³ (ng/m³) (standard deviation in brackets)

	Determinant			
	Pb	Cd	As	Ni
GF-AAS				
Lab A	0.239 (0.169)	0.058 (0.026)	0.028 (0.078)	0.483 (0.297)
Lab B	1.195 (1.211)	0.488 (1.129)	-0.164 (0.065)	2.973 (4.742)
Lab C	0.312 (0.252)	-0.012 (0.032)	-0.006 (0.117)	1.098 (0.544)
Lab D	0.857 (0.257)	0.009 (0.012)	-0.122 (0.067)	1.174 (0.277)
ICP-MS				
Lab B	0.541 (0.383)	0.050 (0.052)	0.073 (0.066)	3.673 (5.106)
Lab D	1.076 (0.388)	0.028 (0.014)	-0.029 (0.057)	1.372 (0.305)

³ For a theoretical volume of 55 m³

Table 5.2: Reagent blank concentrations³ (ng/m³) (standard deviation in brackets)

	Determinant			
	Pb	Cd	As	Ni
GF-AAS				
Lab A	0.435 (0.162)	0.030 (0.008)	-0.063 (0.092)	0.765 (0.237)
Lab B	0.386 (0.267)	-0.063 (0.028)	-0.264 (0.055)	0.241 (0.501)
Lab C	0.316 (0.169)	0.027 (0.014)	-0.058 (0.056)	0.652 (0.508)
Lab D	0.200 (0.091)	0.010 (0.021)	-0.162 (0.091)	0.219 (0.124)
ICP-MS				
Lab B	0.259 (0.198)	0.067 (0.077)	-0.014 (0.009)	0.668 (0.463)
Lab D	0.310 (0.207)	0.022 (0.016)	-0.076 (0.111)	0.344 (0.324)

Table 5.3: Field filter blank concentrations³ (ng/m³) (standard deviation in brackets)

	Determinant			
	Pb	Cd	As	Ni
GF-AAS				
Lab A	1.110 (0.881)	0.025 (0.033)	0.160 (0.128)	0.858 (0.554)
Lab B	1.159 (0.723)	0.019 (0.110)	-0.236 (0.112)	0.622 (1.054)
Lab C	0.312 (0.252)	-0.012 (0.032)	-0.006 (0.117)	1.098 (0.544)
Lab D	1.544 (0.483)	0.021 (0.033)	-0.188 (0.106)	1.341 (0.282)
ICP-MS				
Lab B	0.886 (0.564)	0.382 (0.653)	0.163 (0.028)	1.010 (0.814)
Lab D	1.708 (0.447)	0.041 (0.027)	0.000 (0.095)	1.442 (0.193)

Table 5.5 CRM recovery rates (%) for NIST

	Determinant							
	Pb (6550 ± 80 mg/kg)		Cd (75 ± 7 mg/kg)		As (115 ± 10 mg/kg)		Ni (82 ± 3 mg/kg)	
GF-AAS NIST 1648								
	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
Lab A	96.8	6342	91.0	68.3	101.2	116.4	90.8	74.5
Lab B	105.5	6912	107.2	80.4	106.8	122.8	108.8	89.3
Lab C	98.3	6436	98.4	73.8	105.6	121.5	96.0	78.7
Lab D	99.1	6492	96.3	72.2	113.6	130.6	101.2	83.0
ICP-MS NIST 1648								
	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
Lab B	98.6	6459	101.2	75.9	111.7	128.5	95.1	78.0
Lab D	94.6	6194	96.5	72.4	107.8	124.0	104.6 ⁴	85.7

Table 5.5 CRM recovery rates (%) for NIES

	Determinant							
	Pb (219 ± 9 mg/kg)		Cd (1.1 ± 0.1 mg/kg)		As (2.6 ± 0.2 mg/kg)		Ni (18.5 ± 1.5 mg/kg)	
GF-AAS NIES No.8								
	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
Lab A	102.5	224	95.5	1.1	109.6	2.8	90.3	16.7
Lab B	113.8	249	94.7	1.0	85.3	2.2	91.1	16.8
Lab C	96.1	210	94.5	1.0	100.1	2.6	88.0	16.3
Lab D	102.0	223	85.2	0.9	130.7	3.4	101.5	18.8
ICP-MS NIES No.8								
	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
Lab B	102.5	224	99.2	1.1	111.8	2.9	94.3	17.4
Lab D	99.3	217	98.2	1.1	117.5	3.1	93.9	17.4

⁴ Recovery includes possible 'flyer' for fourth replicate where recovery of 145.8% was recorded

5.2 Field samples

Table 5.6 summarises the mean pollutant concentrations for Pb, Cd, As and Ni over the duration of the monitoring period for the UK Avonmouth trials (8 July 2001 – 2 August 2001) for GF-AAS and ICP-MS methods of analysis.

Figures 5.1, 5.2, 5.3 and 5.4 below show the temporal trend in occurrence of daily mean concentrations across the CEN array of co-located samplers for GFAAS and ICPMS analyses.

Actual data are shown in Appendix 1

Figure 5.1 Daily mean Lead (Pb) concentrations (ng/m³) across different co-located samplers

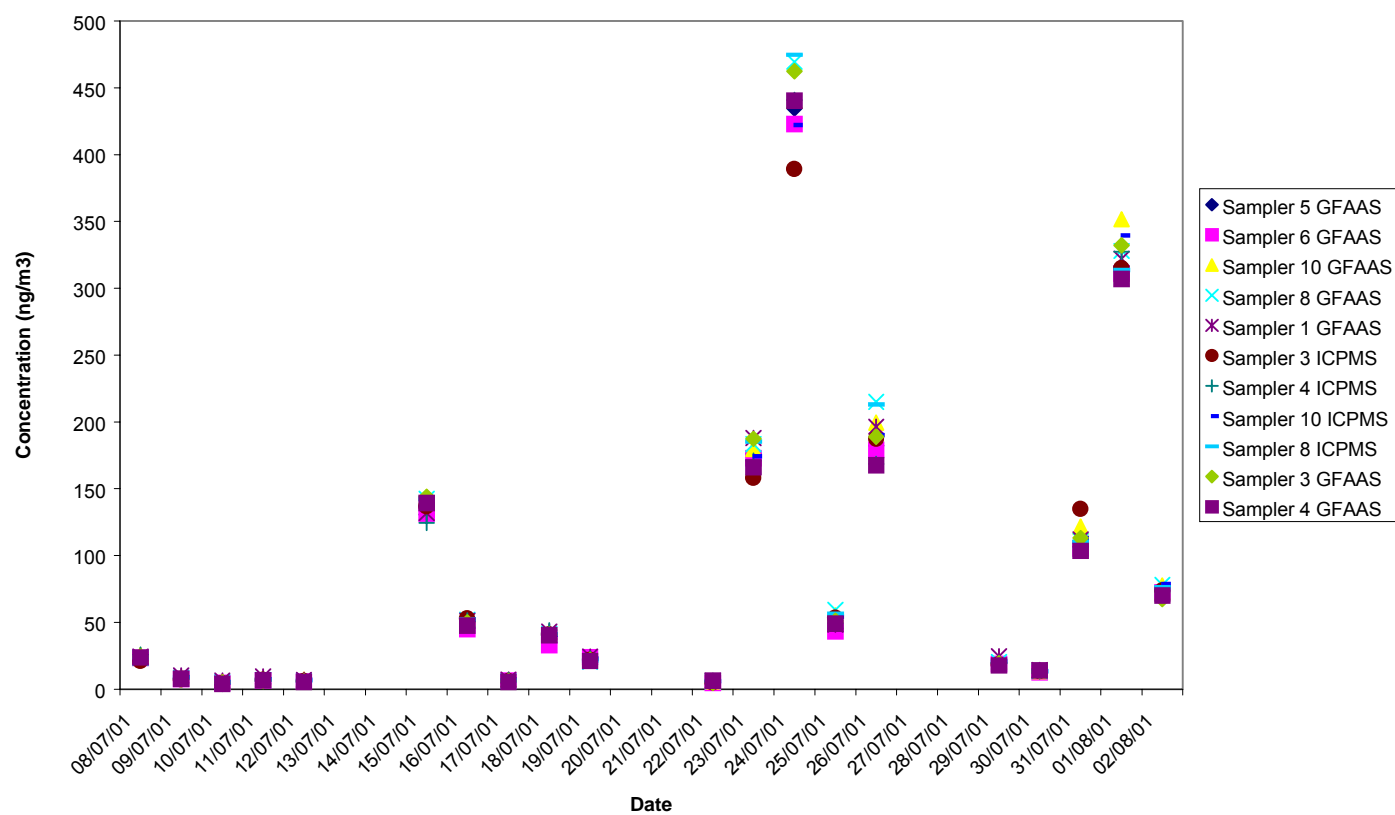


Figure 5.2. Mean daily Cadmium (Cd) concentrations (ng/m³) across co-located samplers (Avonmouth Trials)

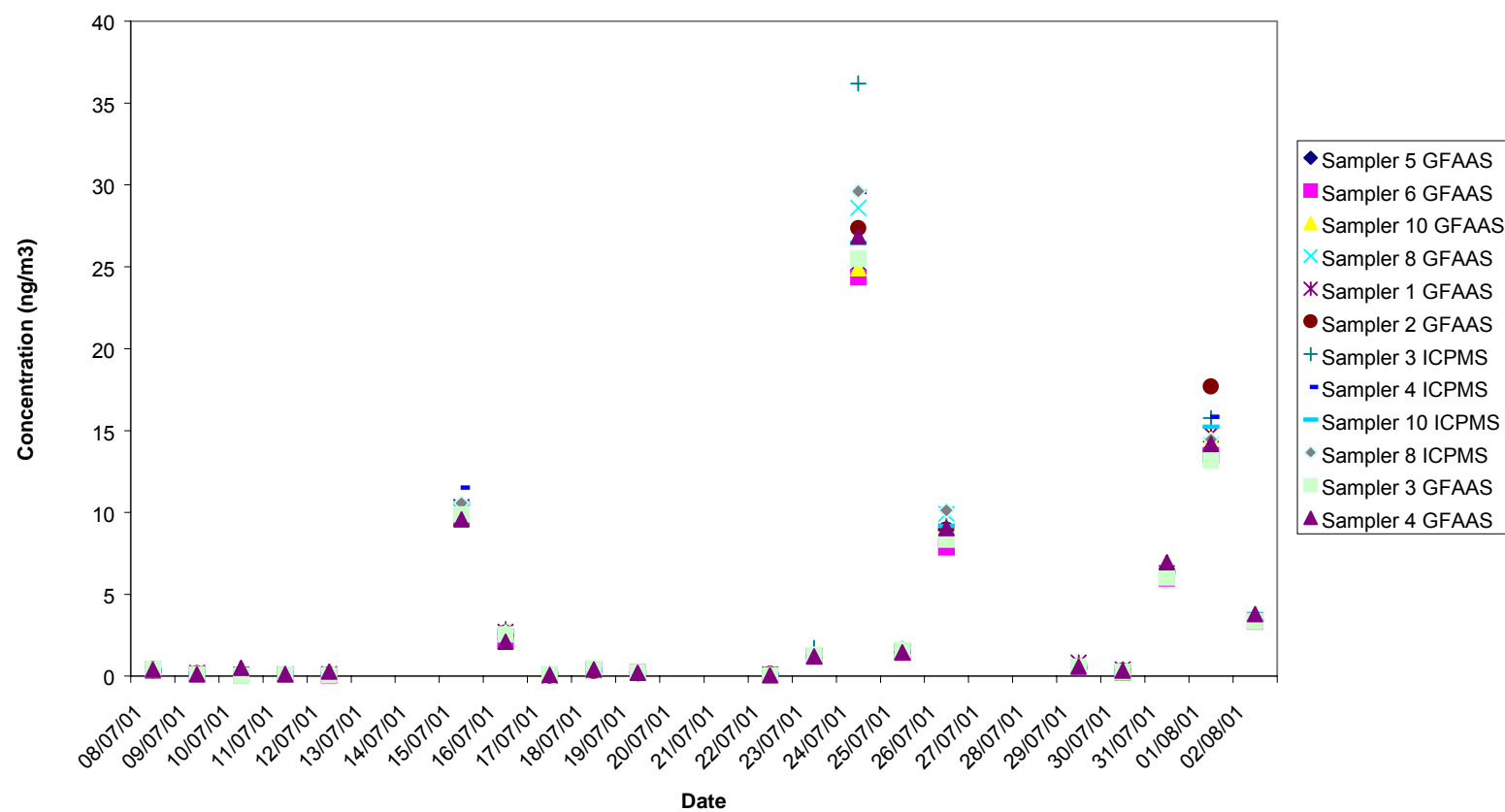


Figure 5.3. Daily mean Arsenic (As) concentrations across co-located samplers (Avonmouth Trials)

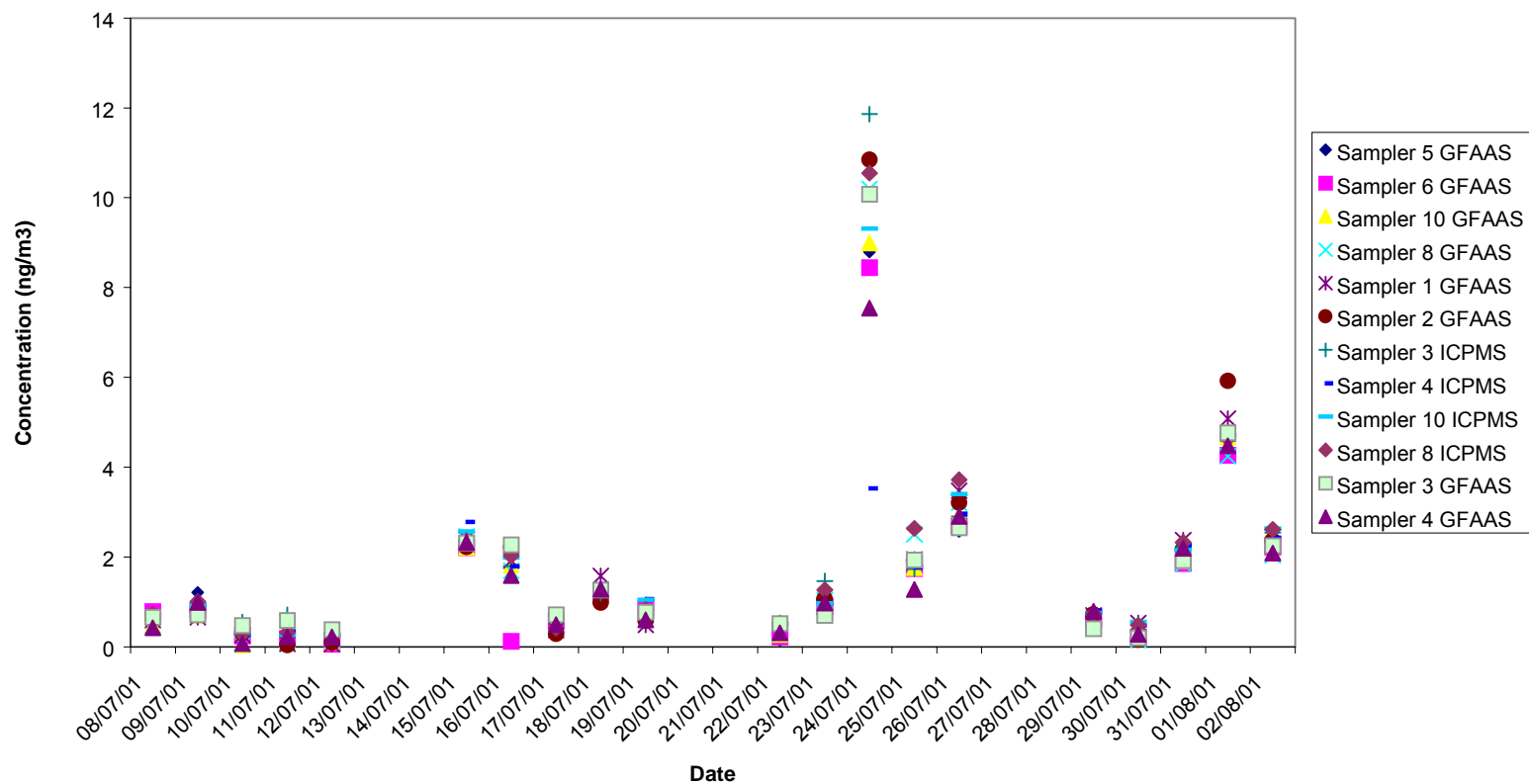


Figure 5.4 Daily mean Nickel (Ni) concentrations (ng/m3) across co-located samplers (Avonmouth Trials)

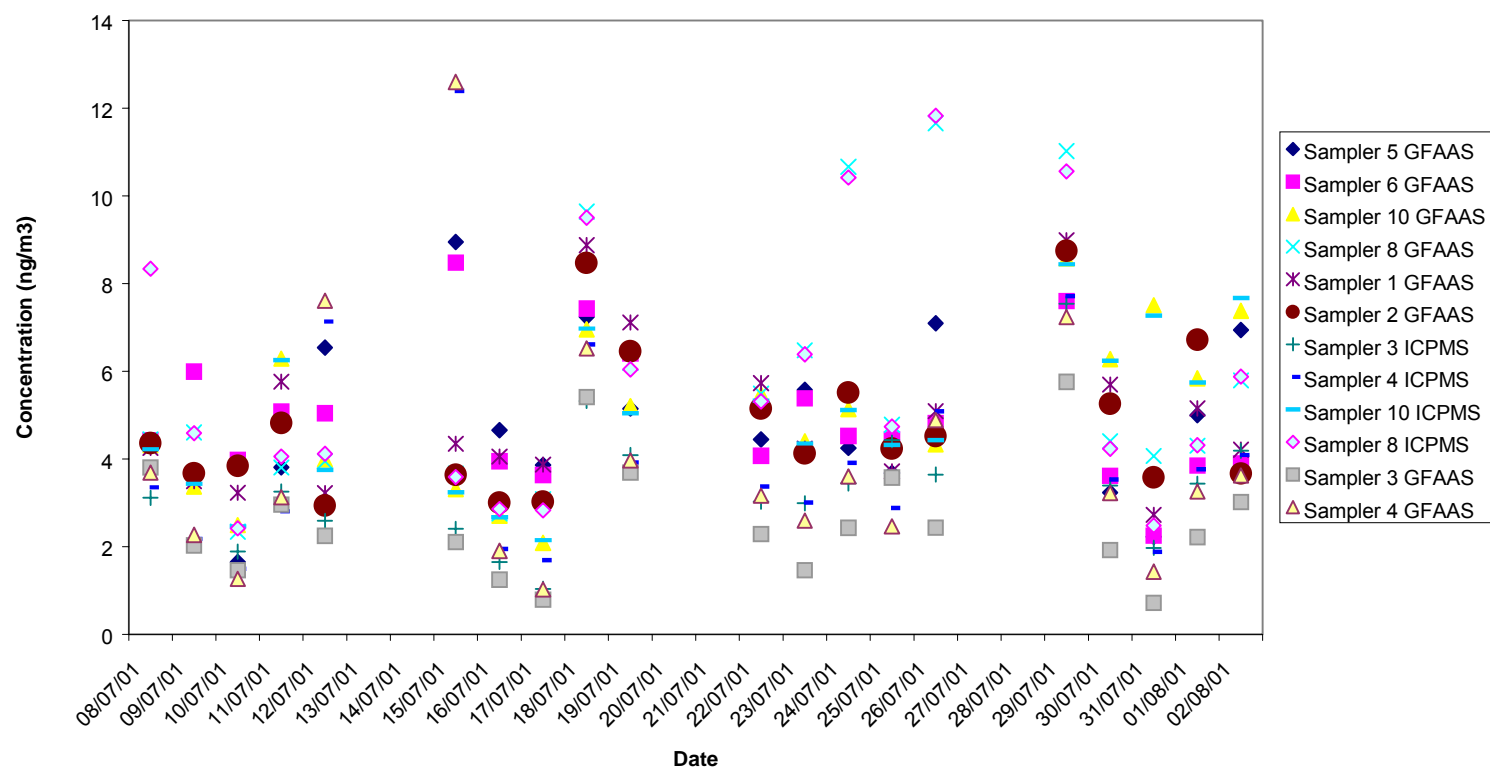


Table 5.6. Period mean pollutant concentrations (ng/m³) across co-located samplers

		Determinant (ng/m ³)			
Sampler	Lab.	Pb	Cd	As	Ni
GF-AAS					
No.1	A ⁵	68.30	2.73	1.42	4.93
No.2	A ⁶	91.12	4.06	1.92	4.78
No.3	B	87.57	3.71	1.78	2.58
No.4	B	82.13	3.91	1.55	3.97
No.5	C	83.19	3.67	1.56	5.05
No.6	C	81.49	3.59	1.51	4.94
No.8	D	89.83	3.98	1.71	5.66
No.10	D	89.38	3.80	1.64	5.00
ICP-MS					
No.3	B	82.57	4.50	1.95	3.28
No.4	B	84.07	4.14	1.53	4.14
No.8	D	89.30	4.15	1.95	5.72
No.10	D	86.32	4.01	1.84	4.96

⁵ Sample B1-18 sample lost in digestion. Period mean concentrations reported on 19 samples.

⁶ Samples B1-16 and B2-16: Arsenic concentrations not determined for sampling date 22/07/01

6 Commentary

The following provides brief detail details with respect to the main findings of the CEN WG14 field validation trials held at Avonmouth:

1. Concentrations of pollutants were similar to those previously reported in the UK programme undertaken in the year 2000. Period mean concentrations were in the following ranges: Pb; 81 - 91ng/m³; Cd 3.6 – 4.5 ng/m³; As; 1.4 – 1.9 ng/m³ and Ni: 2.6 – 5.7ng/m³. For sampler 1, lower period mean concentrations were recorded due to the loss of one sample during digestion and sample preparation. Data gathered from other co-located samplers indicate that relatively high concentrations of pollutants were observed on this day. Thus, the loss of the data had a significant impact on period mean statistics when compared to those recorded by the other samplers.
2. For Pb, Cd and As, daily mean results showed good agreement between samplers. For Ni, results across samplers showed more variability.
3. Recovery rates for certified reference materials were generally within the specified margins of tolerance set down by the Working Group.

7 Report Statement

We confirm that in preparing this report we have exercised all reasonable skill and care.

Unless specifically assigned or transferred within the terms of the agreement, the consultant asserts and retains all Copyright, and other Intellectual Property Rights, in and over the report and its contents.

Appendices

Appendix 1: Data summaries

Individual data summaries for each participating Member State are provided in the following electronic data files ('Excel' spreadsheet) using the pro-forma format supplied by CEN/TC264/WG14.

GF_AAS:

[Lab_A(GFAAS)_Bristol1and2 N147.xls]

[Lab_B(GFAAS)_Bristol3and4 N147.xls]

[Lab_C(GFAAS)_Bristol5and6 N147.xls]

[Lab_D(GFAAS)_Bristol10and8 N147.xls]

ICP-MS:

[Lab_B(ICP_MS)_Bristol3and4 N147.xls]

[Lab_D(ICP_MS)_Bristol20and8 N147.xls]

Appendix 2: National Physical Laboratory Calibration
Report: Mass Flow Meter GFM 47 S/N G16758

INTRODUCTION

A Mass-flow Meter manufactured by Aalborg, of model GFM 47 and Serial Number G16758, was calibrated gravimetrically at NPL, and then further characterised at NPL using a volumetric device traceable to NIST USA.

MEASUREMENT PROCEDURE

Metrology grade air from a cylinder of known total weight was allowed to flow in a controlled manner, at a constant flow rate for a known time, through the mass-flow meter (MFM) under test. This was achieved using a mass-flow controller (MFC) upstream of the MFM as in figure 1. The reading given by the MFM was recorded and then the total weight of the gas cylinder was re-determined. In this way it was possible to calculate a value for mass flow rate (1) where F_m is the measured mass-flow, ΔM represents the mass change of the gas cylinder and T is the flow time.

$$F_m = \frac{\Delta M}{T} \quad (1)$$

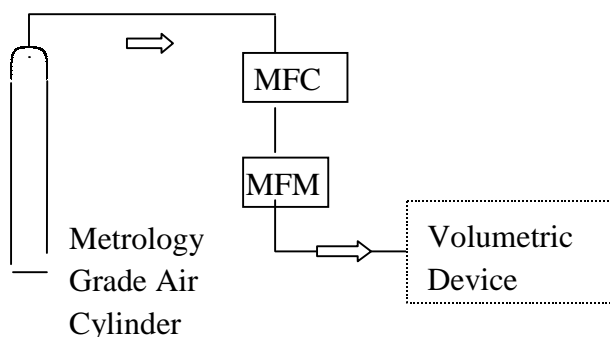
This process was carried out at eight different mass-flow values. It is possible to relate mass flow to standard volume flow as a consequence of the ideal gas law, given standard temperature and pressure (1013.25 mbar, 273.15K) and thus a modified version of (1) is shown below where F_v gives the volume flow at stp.

$$F_v = \frac{22.4 \times \Delta M}{(28.8 \times T)} \quad (2)$$

The standard flow results given in Table 1 have been calculated in this way.

Measurements were also carried out using a volumetric device in series with the calibration system as shown in Figure 1. This device, which measures flow by timing a float rising across a known volume (which is traceable to NIST USA), has an internal temperature and pressure measurement capability, providing a direct standard volume flow reading, again referenced to 1013.25 mbar and 273.15K. The measurements were used as a check on the calibration outlined above, and also to further characterise the MFM across a wider range of flows. This volumetric device has been regularly validated by comparison with gravimetric standards at NPL, in a similar manner to that outlined above for the flow-meter under test

Figure 1. A schematic of the Gravimetric and Volumetric calibration system.



RESULTS

The results of the calibration of the mass flow meter are given in Table 1 below:

Table 1 Calibration of the Mass Flow Meter (MFM).

Gravimetric Mass Flow/ grams per minute	Uncertainty in Gravimetric Flow / grams per minute	Calculated Standard Flow / litres per minute	MFM Reading / litres per minute
37.56	0.04	29.2	34.3
40.11	0.19	31.2	36.7
43.31	0.20	33.7	39.9
45.08	0.21	35.1	41.6
48.17	0.22	37.5	45.0
50.89	0.23	39.6	47.9
53.49	0.25	41.6	50.5
56.13	0.26	43.7	52.8

These results are displayed graphically in Figure 2. It is interesting to note that these results are consistent, within their respective uncertainties, with the measurements carried out using

the volumetric device, shown in Figure 3. In addition, because of the simple nature of the latter measurements, it was possible to carry out a more detailed characterisation of the MFM under test, and 16 measurements were made at flows between 1 litre.min⁻¹ and 37 litre.min⁻¹. These measurements are shown in Figure 4.

Figure 2. Gravimetric Flow Calibration

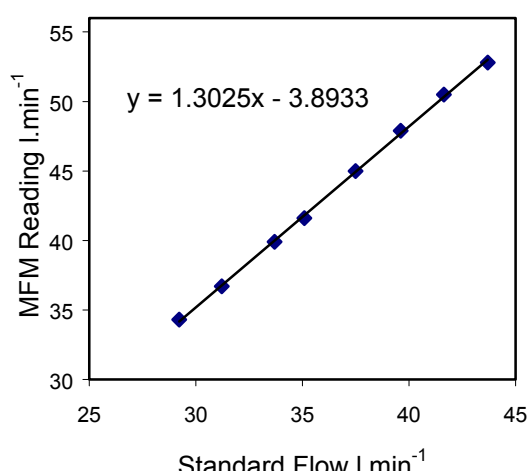


Figure 3. Volumetric Flow Calibration

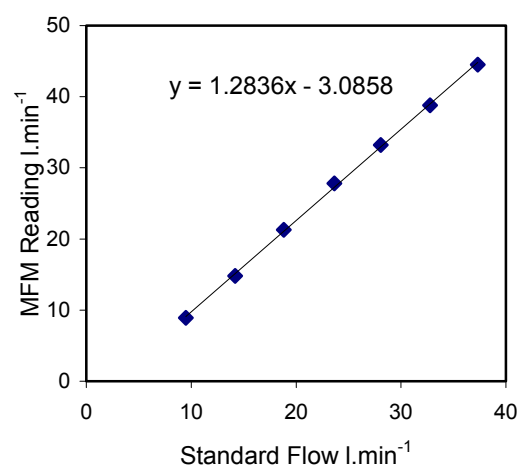
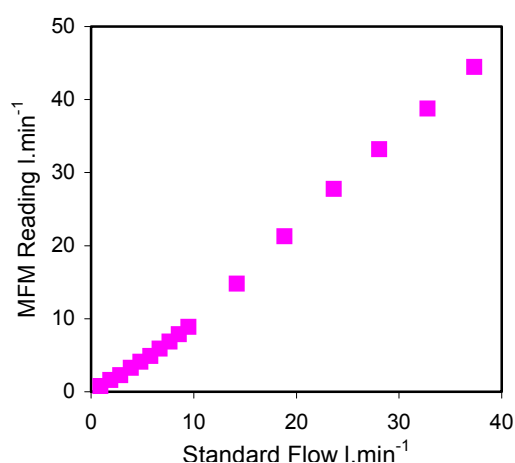


Figure 4. Detailed Volumetric Comparison



DISCUSSION

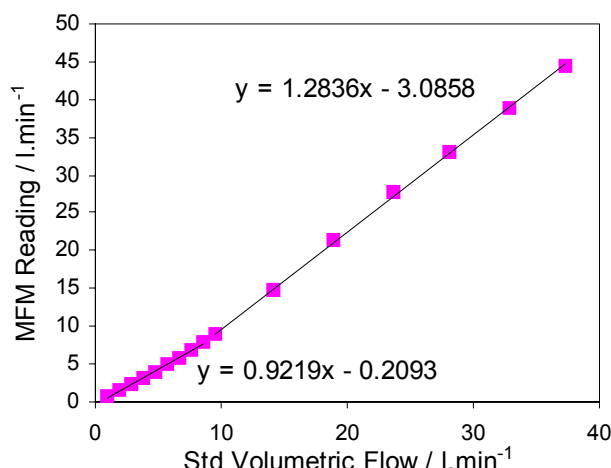
It is seen as convenient to re-state the calibration results:

$$\text{MFM} = 1.303 \times \text{Gravimetric Standard Flow} - 3.89 \text{ litres.min}^{-1}$$

This result is stated using standard conditions of 273.15K and 1013.25 mbar, and states the mass of gas flow measured as the volume that it would represent under these conditions.

It is apparent from these results that the gravimetric method of calibration compared reasonably well with the volumetric (1.5 % difference in gradient), the small discrepancy was possibly attributable to either drift of the MFM response between the two tests, or to repeatability uncertainties. (In other tests these two methods have regularly shown agreement to better than 1%.) This is important since both methods show large regression offsets as given in figs. 2 and 3 that, as we can see from figure 4, are attributable to the non-linearity that was shown by the MFM when calibrated against the volumetric device. This relationship is characterised more fully in figure 5. For illustration the deviation has been represented as two linear plots with a discontinuity at around 10 litres.min⁻¹, although in fact the response is better characterised by a smooth curve.

Figure 5. Examination of the Volumetric Comparison



In our experience thermal mass-flow devices of this type have always shown similar non-linear characteristics below approximately 10% of their full-scale flow range. Therefore with this in mind it would seem to be most sensible - if this instrument were still to be used - to calibrate it over a narrow flow range, incorporating the region at which the majority of its

flow measurements will be made. It is likely that, owing to the way in which such devices function, measurements carried out over a higher flow range (say 40 – 100 litres.min⁻¹) would reveal further non-linearities, although this need not be a problem provided that the device was accurately characterised over the flow range of interest.

CERTIFICATE

An NPL certificate summarising these results has been provided to the UK participant's project manager (Dr Richard Maggs). In this context it should be noted that NPL is the UK's national standards laboratory and these results are therefore acceptable to national accreditation bodies as being compatible with the ISO 17025 accreditation standard.