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REFERENCE : O-96013
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DANIDA

Environmental Information and Monitoring Programme (EIMP) Air Quality Monitoring Component

Mission 11 Report

EIMP Environmental Information
and Monitoring Programme

NILU Norwegian Institute
for Air Research

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Environmental Information and Monitoring Programme (EIMP). Air Quality Monitoring Component

Mission 11 Report

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1 Introduction

The 11th mission to Egypt covered the period 21.2- 4.3. 1999. The mission was undertaken by senior scientist Oddvar Røyset in cooperation with Bjarne Sivertsen.

Oddvar Røyset, chemical analysis expert, performed the work carried out in this mission. Of the work programme activities A - I, the following tasks were covered:

E. Training

Follow up the procedures implemented during the 10. Mission, and on the job training at CEHM, Cairo University, for personnel from CEHM and NIS

F. QA/QC

Update SOPs and develop new SOPs to be used in the EIMP Air Quality Manual

H. Reference laboratory

Make recommendations to the preparation of QA standards for SO_4, NO_2 and lead to be used at CEHM

The work in this mission was mainly focused towards the follow up of the methods implemented during the 10. Mission. The work also included on the job training of the staff at the Centre for Environmental Hazards and Mitigation (CEHM) at the Cairo University and the Reflab. Water at Ain Shams University and NIS. The follow up work included training included sampling and chemical analysis methods for NO_2, SO_2, TSP, PM_{10}, passive sampling (NO_2, SO_2) as well as a method for collection of dustfall from the air. New training included implementation of a method for determination of lead in air, based on EPA-procedures, and on quality control and data storage procedures.

2 A. Institutional support

No activity during this mission.

3 B. Design of monitoring programme

No activity during this mission.

4 C. Procurement of equipment, hardware and software

No activity during this mission.

5 D. Data management

No activity during this mission.

6 E. Training

On the job training of the staff at Centre for Environmental Hazards Mitigation (CEHM) at the Cairo University, Giza and NIS. The training waas focused on procedures for data storage, data presentation, data evaluation and quality control for the determination of SO_2, NO_2, TSP/PM_{10} and dustfall (using dust buckets).

The training included one person from the NIS.

The training programme had the activities listed below and was performed by Oddvar Røyset. The preliminary training program schedule is given in appendices.

6.1 22. 2. 1999 - EIMP office

Meeting with Ulla Lund.

- Status of new equipment ordered.
- Determination of lead in air. Procedures recommended by CAIP (Cairo Air Improvement project) were evaluated. It was decided to recommend the procedures from Environment Protection Agency (EPA;
    - 40CFR Ch I(7-1-96), Part 50, appendix G (digestion by nitric acid and determination by flame atomic absorption spectrometry)
    - Method 6010B (ICP-AES, inductively coupled plasma atomic emission spectromentry)
- Arrange meeting with Saad Hassan form Ain Shams Reflab water to discuss QA-QC procedures for SO_4, NO_2, lead.

6.2 23.2.99 - EIMP Office

Prepared procedures for data storage and lead in air.

Meeting with Anwar Ahmed regarding status for new equipment. The status for the new equipment is:

Equipment type		Status pr. 23.2.99
Autosampler for the Dionex DX 100 Ion Chromatograph (recommended the Gilson 222XL type or equivalents)	ca 75000	Under evaluation by dr. Ahmed Soliman/Anwar Ahmed to find a model with the correct specifications.
Water treatment system for production of pure water	ca 50000	In Cairo.
New microbalance for TSP with larger weighing chamber to fit for 10"x8" highvolum filters	ca 25000	It was decided to use a Sartorius balance in the store.
Laboratory shaking machine for the extraction of NO2 tubes	13000-15000	Cairo airport 27.2.99.
Computer	7000	Received Nov. 1998.
Desiccator	1300	Cairo airport 27.2.99.
Filtration equipment for dustfall	3000	Cairo airport 27.2.99.
Volumetric flasks of 1000 ml, 10 units	1500	Cairo airport 27.2.99.

6.3 24.2. 99 - CEHM Cairo University

Discussed possible problems of the analysis procedures implemented in October/November 1998.

Lecture on procedures for data storage and security.

6.4 25.2.99 - EIMP office

Evaluation of data produced by CEHM..

Investigations of the system for VOC.

Evaluations of procedures for lead in air.

6.5 28.2.99 - CEHM, Cairo University

Lecture on procedures for lead in air.

40CFR Ch I(7-1-96), Part 50, appendix G (digestion by nitric acid and determination by flame atomic absorption spectrometry).

Method 6010B (ICP-AES, inductively coupled plasma atomic emission spectrometry.

Discussion of quality control procedures.

6.6 29.2.99 - CEHM Cairo University

Discussions of quality of data produced by the laboratory from November 1998 to January 1999.

- Data for Alex for SO_2 is lower than expected. No obvious reason is found
- Some strange values for Alex in February 1999, high Cl-values (possible seaspray, or burning of Cl-containing plastic trash in the vicinity of the stations).

Method comparison between NILU and CEHM for the determination of NO_2 and SO_2 by passive samplers.

The passive samples for comparison were taken by 2 parallel colocated passive samplers at the Cairo, Abu Zabel station from 21.10.98 to 02.11.98. The following results were obtained for filters leached into 5 ml of solution. The agreement was satisfactory, as the deviations were within 25 %.

		NILU	CEHM	CEHM/NILU
NO_2	µg NO_2/ml	1.13	0.93	0.82
SO_2	µg SO_2-S/ml	1.2	1.49	1.24

Delivered updated Excel templates for the storage and graphical presentation of data for SO_2, NO_2, SO_2-passive, NO_2-passive, TSP/PM_{10}, lead, dustfall.

Further discussions about the Gilson autosampler. Discussions of need for possible new equipment.

6.7 3.3.99 - CEHM, Cairo University,

Lecture on quality control.

Discussions regarding quality of data for SO2 and NO2 in Cairo and Alexandria.

EIMP office

New equipment needs.

Updated EIMP Air quality manual with new procedures.

Prepared lecture on quality control procedures

Writing mission report

Investigation on VOC equipment.

7 F. QA/QC

7.1 SOPs

Follow up and training have been performed for the 8 methods where SOPs was developed, as listed below:

Action	Parameter	Procedure name
Follow up	SO_2	Procedure for sampling and analysis of SO_2 in air by use of a filterpack sampler
Follow up	NO_2	Procedure for sampling and analysis of NO_2 in air. Iodide absorption method
Follow up	Passive SO_2 and NO_2	Procedure for sampling and analysis of NO_2 and SO_2 in air by the use of passive samplers.
Follow up	TSP, PM_{10}	Procedure for sampling and analysis of suspended particulates in air by the use of a highvolume sampler
Follow up	Dustfall	Procedure for sampling and analysis of dust fallout from the air
Updated	QA-QC	EIMP Air Quality QA-QC-procedures
New	Lead	Recommendation for EPA 40CFR50G, EPA 6010B
New	Data	Data storage

7.2 QA/QC samples and presentation

The level of quality control needed was discussed with Ulla Lund, and it was decided that three types of quality control samples was needed, one for $SO_4^=$, NO_2^- and lead. The two former is recommended to prepare locally by Reflab water, while for lead a commercial standard sample from Spex industries, USA, was recommended. For lead two additonal certified reference materials from NIST (Urban fly ash and Urban particulate matter) was recommended.

The CEHM -laboratory had access to a specially developed program for presentation of quality assurance data. The program is developed by VKI in Denmark and has the name Quality. Personnel from the laboratory have got training on the use of this program, but the program has yet not been implemented for routine use.

8 G. Monitoring

No activity during this mission.

9 H. Reference laboratory

Training of 1 person from the NIS was performed

10 I. Component Co-ordination

No activity during this mission.

Appendix A

People and colleagues

People and colleagues

The following persons participated in the training program

Name	Participation during 11. mission	Location
Dr. Ahmed Soliman Abd Ellah, laboratory manager	X	CEHM
Dr Amany Taher, ass. laboratory manager	X	CEHM
Hany Nabil	X	CEHM
Dr. Gehad Genidy	X	CEHM
Mohammed Abd El Maugood		CEHM
Shireen Ali		CEHM
Kamla Moustafa	X	CEHM
Moustafa Morad	X	CEHM
Mona Moneer		Ain Shams University
Wagdi Mahmoud Khedr		Ain Shams University
Basma Salia	X	NIS

In addition I also had the pleasure to meet

Dr. Tarek El Araby, manager of CEHM	CEHM
Dr. Hesham Mohamed El Araby, manager of information and data analysis laboratory	CEHM

The address for mailing is
Dr. Ahmed Soliman Abd Ellah
Department of chemistry
Faculty of Science
Cairo University
Giza
EGYPT
Tel. 202 567 4843 job (202 518 7785 private)
email : ahmedsoliman α frcu.eun.eg

Appendix B

E. Training
E1 Preliminary time schedule for training
E2 Lectures given

E1 Preliminary time schedule for training Oddvar Røyset, NILU, Norway, chemical analysis expert.

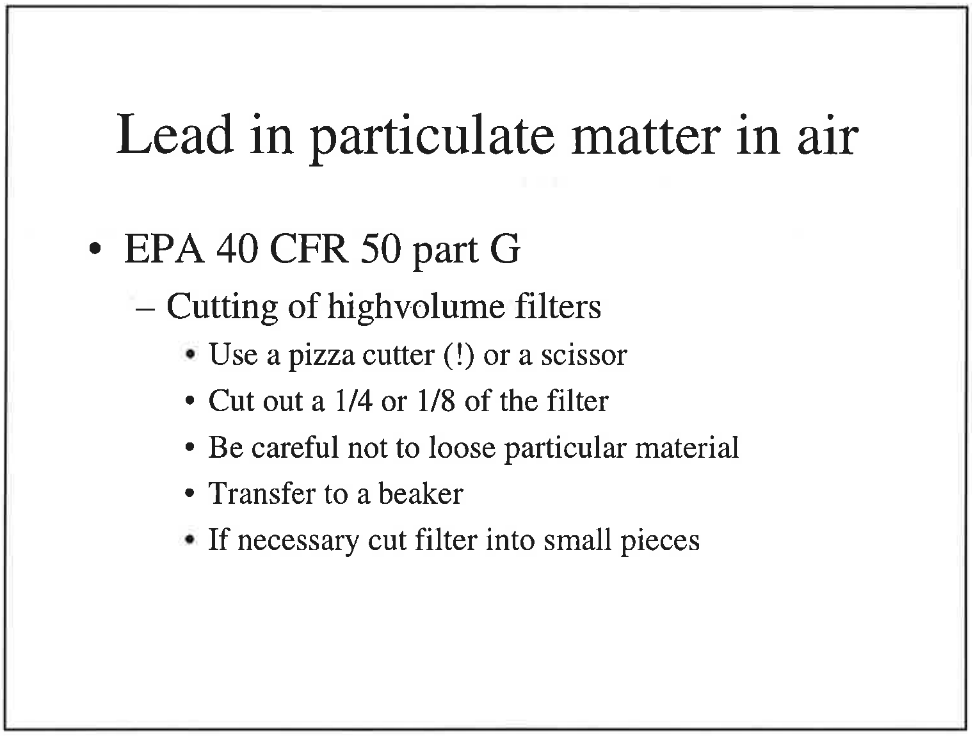
Sunday 21.2	Arrival in Cairo at about 17:50 with Lufthansa	
Monday 22.2 11:30	Discussions at EIMP office • Status on new equipment ordered. • QA/QC procedures of Ain Shams, how to proceed • Procedure for lead in air - go through EPA docs. and decide which procedure to recommend	Ulla Ulla Ulla
14:00	• Air section meeting 14:00. Planning of activities	LM,BS,OR, MF, JS
	• Setting up my PC in EIMP network.	EIMP experts
Tuesday 23.2. 09:00 09:13:00 14:00 - 16:00	EIMP office. • Status new equipment • Further planning and preparation of procedures • Visit at CEHM. Staff meeting at 14:00. Making agreements with CEHM staff regarding ORs program. NB. Need a whiteboard and overhead	Anwar

Wednesday 24.2.
10:00-15:00
	CEHM Cairo University
	Discuss with Ahmed Soliman and co-workers.
	• Status of procedures implemented in Nov. 1998. Discuss possible problems regarding the methods for
	  • SO_2 - Problems with leakage of filterholders for SO_2.
	  • NO_2, passive sampling, dustfall, TSP/PM_{10}.
	• Status of new equipment ordered November.
	• The new microbalance for TSP/PM_{10}
		OR
		Ahmed Soliman
		Armany Taher
		CEHM coworkers
		Ain Shams
		MM,WMK
		NIS (Basma)

Thursday 25.2 10:00-15:00	Visit at CEHM. Further discussions. • Data storage and data presentations. • Excel reporting tools. It would be fine if all analysis of SO2/NO2/Dustfall/TSP/Passive sampling were entered into Excel and printed out. • Evaluation of data, using data graphs for evaluating quality of analysis. • Data storage procedures (Archives for site forms, worksheets, chromatograms, storage of data files from ion chromatographs - file structure, backup).	OR Ahmed Soliman Armany Taher CEHM coworkers Ain Shams MM,WMK NIS (Basma)

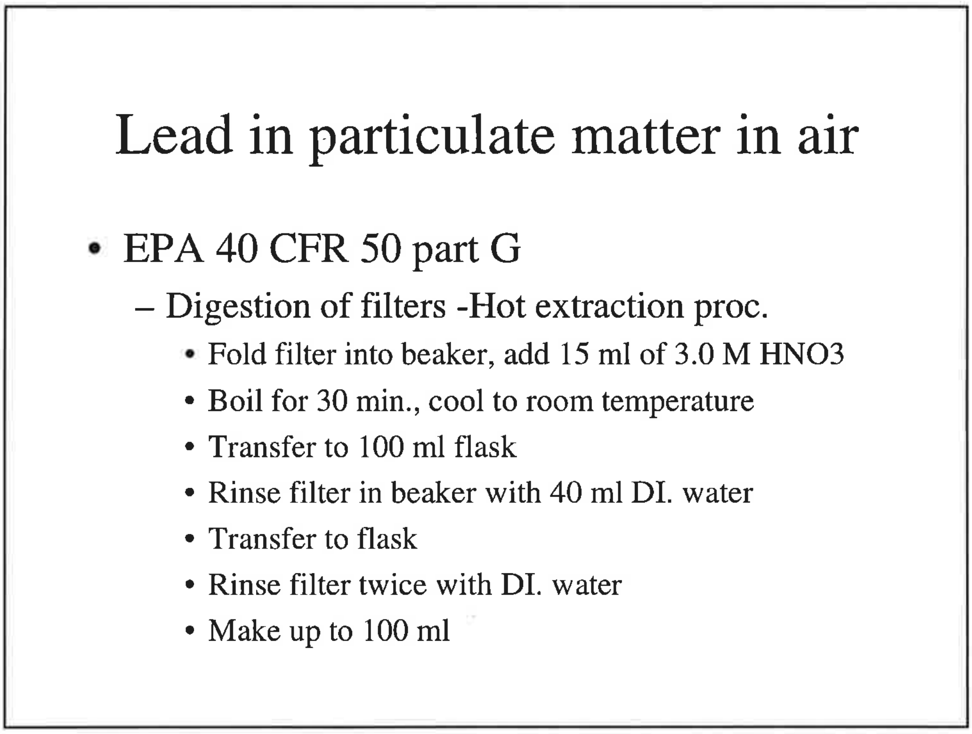
Sunday 28.2 10:00-15:00	Visit at CEHM. Procedure for determinations of lead (Pb). Go through • EPA 40CFR-50 Part G (digestion and flame AAS-analysis) • EPA 6010B (ICP-AES-analysis)	OR, Ahmed Soliman Armany Taher, CEHM coworkers, Ain Shams MM,WMK NIS (Basma)
Monday 1. 3 10:00 -11:00	EIMP office. Meeting with Saad Hassan from Ain Shams Reflab Water QA-QC procedures for SO_4, NO_2, lead (NIST 1648b). Writing mission report.	Ulla, Saad Hassan
Tuesday 2.3 10:00 - 14:00	Visit at CEHM • QA-QC Procedures for SO4, NO2, lead. • The use of the Quality data program from VKI (Denmark). • Questions about GC method of VOC (canisters, injection)	OR, Ahmed Soliman Armany Taher, CEHM coworkers, Ain Shams MM,WMKNIS (Basma)
Tuesday 2. 3	EIMP office. Writing mission report.	
Wednesday 3. 3 10:00 - 12:00	Visit CEHM. Closure of visit and summing up. Future work.	
Thursday 4.3	Departure Cairo (03:35) to Oslo by Lufhansa	

E2 Lectures given



Lead in particulate matter in air

- EPA 40 CFR 50 part G
  - Cutting of highvolume filters
    - Use a pizza cutter (!) or a scissor
    - Cut out a 1/4 or 1/8 of the filter
    - Be careful not to loose particular material
    - Transfer to a beaker
    - If necessary cut filter into small pieces



Lead in particulate matter in air

- EPA 40 CFR 50 part G
  - Digestion of filters -Hot extraction proc.
    - Fold filter into beaker, add 15 ml of 3.0 M HNO3
    - Boil for 30 min., cool to room temperature
    - Transfer to 100 ml flask
    - Rinse filter in beaker with 40 ml DI. water
    - Transfer to flask
    - Rinse filter twice with DI. water
    - Make up to 100 ml

Lead in particulate matter in air

- EPA 40 CFR 50 part G
  - Analysis by ICP-AES or FAAS
    - Standards 0.1 - 10 µg Pb/ml
      - Prepare from $Pb(NO_3)_2$ salt to 1000 µg Pb/ml stocks
      - Or commercial available standards 1000 µg Pb/ml
    - Matched with the same HNO3-conc as samples
    - Wavelength ICP-AES 220.353 nm
    - Wavelength FAAS 283.3 or 217.0 nm

Lead in particulate matter in air

- EPA 40 CFR 50 part G
  - Quality control
    - Standards from Spex Industries, USA
      - 1000 µg Pb/ml in 1 % HNO3
    - Reference materials from NIST
      - NIST 1633a Coal fly ash

Lead in particulate matter in air

- EPA 40 CFR 50 part G
  - Calculations

$$C_{air}[\mu g Pb / m3] = \frac{(C_{Pb} - F_{bl}) \cdot 100ml}{V_a \cdot F_p}$$

Data Storage

- Storage time 5 years
- Data must be available for inspection and easy acces
- Stable storage media
  - Tape
  - CD-ROM
  - ZIP-drive diskettes
  - Mirror harddisk (extra harddisk in PC)

Data storage

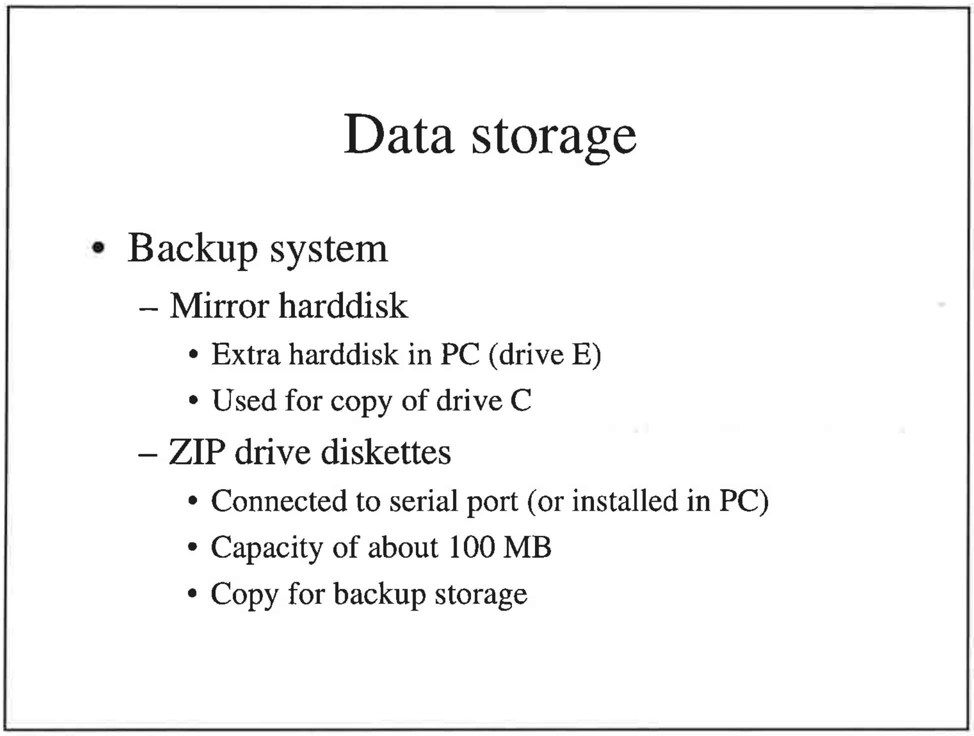
- Storage of forms and printouts
- Use binders
  - Site forms
  - Worksheets for instruments
  - Cromatogram printouts

Data storage

- Dionex Peaknet datastorage
  - Major directory YYMM (9901, 9902 etc.)
  - Data directory Comp_MMDD (SO2_0201 etc.)
  - Example directory C:\9901
    - C:\9901\SO2_0201\Peaknet analysisfiles
    - C:\9901\NO2_0203\Peaknet analysisfiles
    - C:\9901\0205\Peaknet analysisfiles

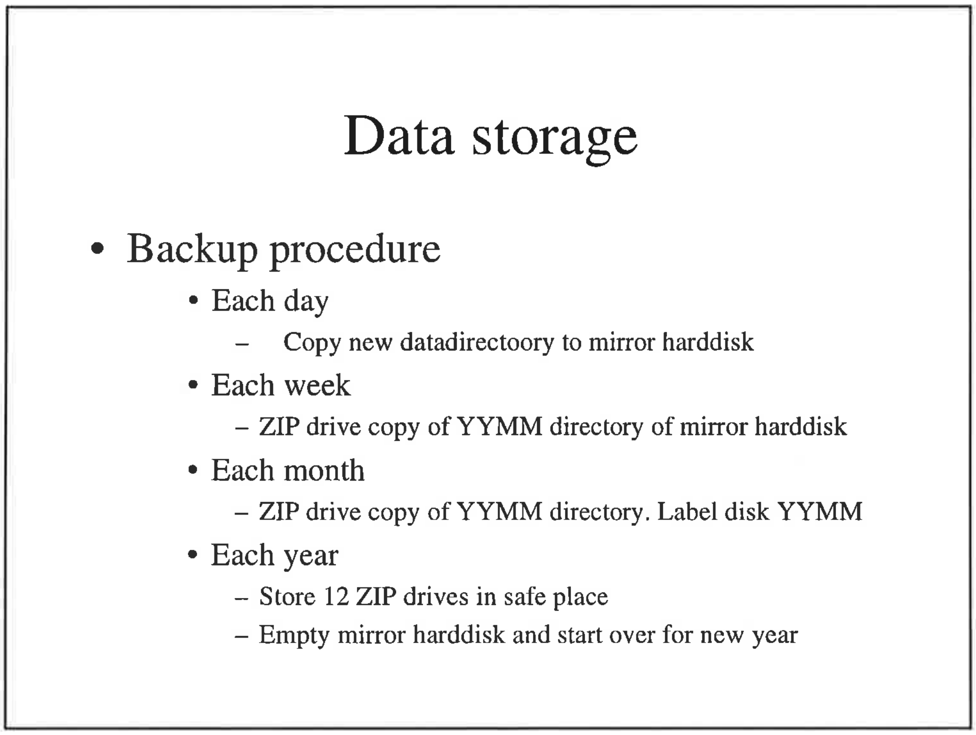
Data storage

- Peaknet database
  - Peaknet stores calcultated results in an MS Access database
  - Stored in "C:\peaknet\database\peaknet.mdb"
  - Each year, make a copy of the database
    - name "peaknet_9901_9912.mdb"
  - Empty peaknet.mdb database each year
  - Backup the database, together with the results on the ZIP drive



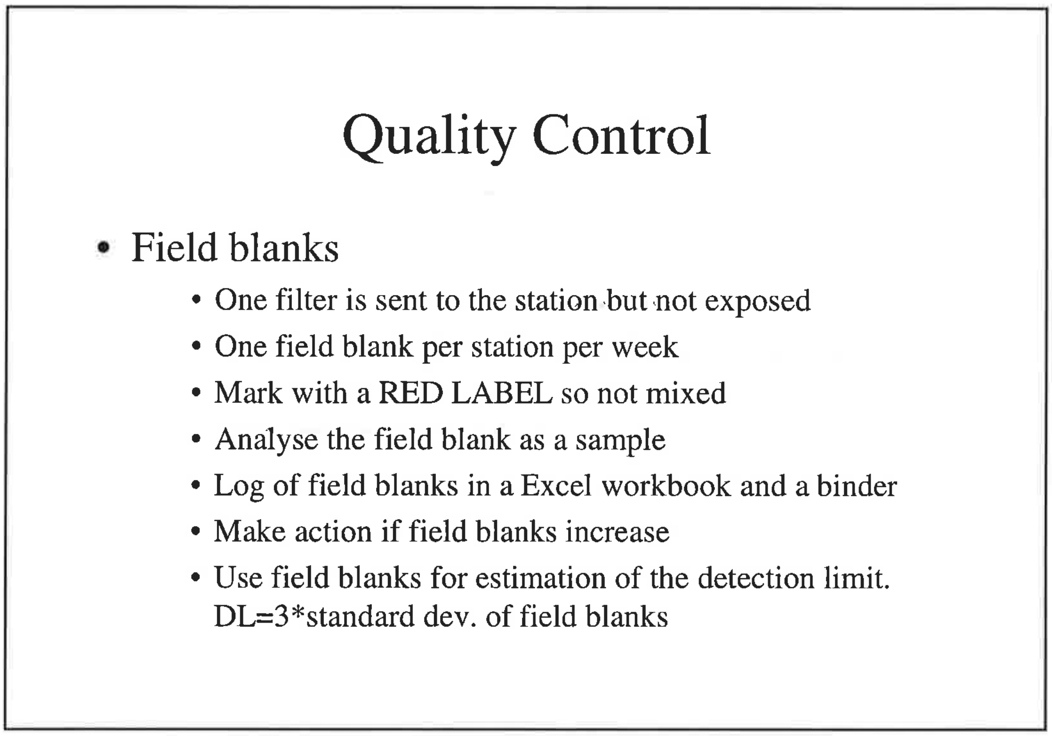
Data storage

- Backup system
  - Mirror harddisk
    - Extra harddisk in PC (drive E)
    - Used for copy of drive C
  - ZIP drive diskettes
    - Connected to serial port (or installed in PC)
    - Capacity of about 100 MB
    - Copy for backup storage



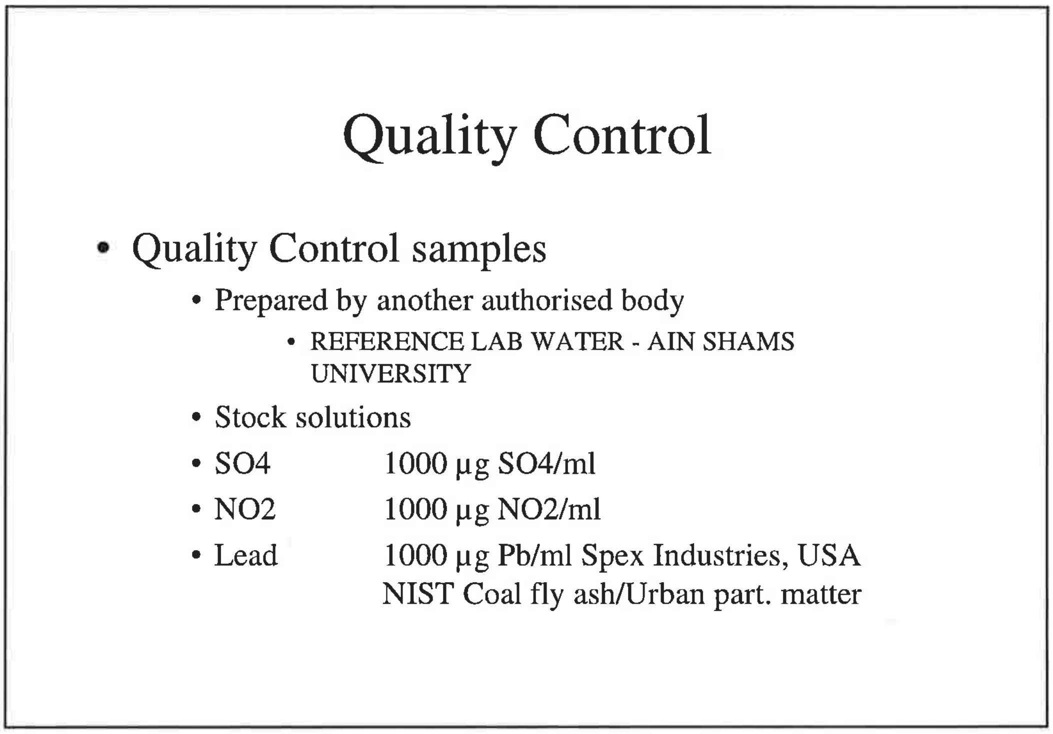
Data storage

- Backup procedure
  - Each day
    - Copy new datadirectoory to mirror harddisk
  - Each week
    - ZIP drive copy of YYMM directory of mirror harddisk
  - Each month
    - ZIP drive copy of YYMM directory. Label disk YYMM
  - Each year
    - Store 12 ZIP drives in safe place
    - Empty mirror harddisk and start over for new year



Quality Control

- Field blanks
    - One filter is sent to the station but not exposed
    - One field blank per station per week
    - Mark with a RED LABEL so not mixed
    - Analyse the field blank as a sample
    - Log of field blanks in a Excel workbook and a binder
    - Make action if field blanks increase
    - Use field blanks for estimation of the detection limit. DL=3*standard dev. of field blanks



Quality Control

- Quality Control samples
    - Prepared by another authorised body
        - REFERENCE LAB WATER - AIN SHAMS UNIVERSITY
    - Stock solutions
    - SO4 1000 µg SO4/ml
    - NO2 1000 µg NO2/ml
    - Lead 1000 µg Pb/ml Spex Industries, USA
      NIST Coal fly ash/Urban part. matter

Quality Control

- Quality control samples
  - Daily working QC-samples
    - SO4 1.00 and 10.0 µg SO4/ml
    - NO2 1.00 and 10.0 µg NO2/ml
    - Lead 1.00 and 10.0 µg Pb/ml
    - Lead digests from NIST reference materials
  - Analyse at least TWICE each day where analysis are performed

Quality Control

- Quality control samples
- Presentation
  - X-Charts Plot of accuracy
    - Plot of results for QC-samples versus time
  - R-Charts Plot of precision
    - Plots of results of difference between parallel 1 and parallel 2 of each QC sample versus time
- Presentation by “Quality program” developed by VKI, Denmark

Quality Control

- Quality control samples
- Action limits
  - Warning limit 2*st.dev. of QC-sample
  - Action limit 3*st.dev. of QC-sample
- Action if
  - 1 result outside Action limit
  - 3 following results outside Warning limit

Quality Control

- Graphical presentation of results
  - High values
    - Contamination ?
    - Sampled for more than one day ?
    - Prefilter black ?
  - Low values
    - Power failure ?
    - Impregnation of filter not OK ?
    - Filter damaged, filter broken ?

Quality Control

- Graphical presentation of results
  - Compare NO_2 and at the same station
  - Compare with neighbouring stations where available
  - Compare with monitor results, daily averages
  - Compare with other environmental parameters
    - Traffic
    - Wind speed and wind direction

Quality Control

- If problems occur
- Make notes
    - in the site forms, which are archived
    - in the Excel analysis workbook
- Make action as soon as possible
- Contact Site responsible person
- Contact laboratory manager
- Fix problems - gather experience on how to avoid problems in the future

Quality Control

- Audit trail logs
  - Archives must be kept in good order
  - All electronic data must be stored in a safe way
- Data availability for at least 5 years for all data of relevance for the quality of the results delivered

Quality Control

- Air Quality Manual
  - Available to users !
  - Always updated with correct version of procedures!
    - Issue no.
    - Revision date
    - Printed date
  - Outdated procedures must be removed from the laboratory and the Air Quality Manual!

Appendix C

F QA/QC
EIMP Air Quality Manual. Standard Operational Procedures for Wet Chemistry analysis methods. New methods implemented during the 11. Mission.

Standard Operational Procedures for Wet Chemistry analysis methods

Determination of lead in suspended particulate matter collected from ambient air	Page 1 of 2 pages
	Date: 01.03.99
Printed date: 99.03.08	Issue no: 001

Determination of lead in suspended particulate matter collected from ambient air

Oddvar Røyset
Norwegian Institute for Air Research, NILU, Norway.

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Determination of lead in suspended particulate matter collected from ambient air	Page 2 of 2 pages
	Date: 01.03.99
Printed date: 99.03.08	Issue no: 001

1. Introduction

The determination of lead in suspended particulate matter in ambient and urban air is performed according to procedures developed by the U.S Environmental Protection Agency -EPA.

The preparation of filters, digestion of filters, are carried out according to the procedure:

40 CFR, Part 50, Appendix G (40CFRCh.I(7-1-96 Edition)). Reference method for the determination of lead in suspended particulate matter collected from ambient air.

The determination of lead in the solution achieved from the digestion, may be achieved in two ways.

Determination by flame AAS according to the method above (40CFRCh.I(7-1-96 Edition)). Alternatively the determination may be performed with inductively coupled plasma atomic emission spectrometry (ICP-AES) according to the method:

Method 6010B Inductively coupled plasma atomic emission spectrometry. Revision 2, December 1996. US EPA.

The latter method is recommended since CEHM have a modern ICP-AES instrument.

2. Quality control

The digestion of particulate matter should be checked by a certified reference material. This material should be digested according to the procedure in (40CFRCh.I(7-1-96 Edition)). Digest 100 mg of the material with 15 ml of the 3 M HNO3 solution used for the digestion of the filters., and dilute to 100 ml. Determine the amount of lead and check against the certified values. The following reference materials from US National Institute for Standards and Technology (NIST) are recommended:

NIST Coal fly ash
NIST Urban particulate matter.

USEPA METHOD

APPENDIX G TO PART 50—REFERENCE METHOD FOR THE DETERMINATION OF LEAD IN SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR

1. *Principle and applicability.*

1.1 Ambient air suspended particulate matter is collected on a glass-fiber filter for 24 hours using a high volume air sampler. The analysis of the 24-hour samples may be performed for either individual samples or composites of the samples collected over a calendar month or quarter, provided that the compositing procedure has been approved in accordance with section 2.8 of appendix C to part 58 of this chapter—*Modifications of methods by users.* (Guidance or assistance in requesting approval under Section 2.8 can be obtained from the address given in section 2.7 of appendix C to Part 58 of this chapter.)

1.2 Lead in the particulate matter is solubilized by extraction with nitric acid (HNO_3), facilitated by heat or by a mixture of HNO_3 and hydrochloric acid (HCl) facilitated by ultrasonication.

1.3 The lead content of the sample is analyzed by atomic absorption spectrometry using an air-acetylene flame, the 283.3 or 217.0 nm lead absorption line, and the optimum instrumental conditions recommended by the manufacturer.

1.4 The ultrasonication extraction with HNO_3/HCl will extract metals other than lead from ambient particulate matter.

2. *Range, sensitivity, and lower detectable limit.* The values given below are typical of the methods capabilities. Absolute values will vary for individual situations depending on the type of instrument used, the lead line, and operating conditions.

2.1 *Range.* The typical range of the method is 0.07 to 7.5 µg Pb/m^3 assuming an upper linear range of analysis of 15 µg/ml and an air volume of 2,400 m^3.

2.2 *Sensitivity.* Typical sensitivities for a 1 percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 µg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

2.3 *Lower detectable limit (LDL).* A typical LDL is 0.07 µg Pb/m^3. The above value was calculated by doubling the between-laboratory standard deviation obtained for the lowest measurable lead concentration in a collaborative test of the method.*(15)* An air volume of 2,400 m^3 was assumed.

3. *Interferences.* Two types of interferences are possible: chemical and light scattering.

3.1 *Chemical.* Reports on the absence *(1, 2, 3, 4, 5)* of chemical interferences far outweigh those reporting their presence, *(6)* therefore, no correction for chemical interferences is given here. If the analyst suspects that the sample matrix is causing a chemical interference, the interference can be verified and corrected for by carrying out the

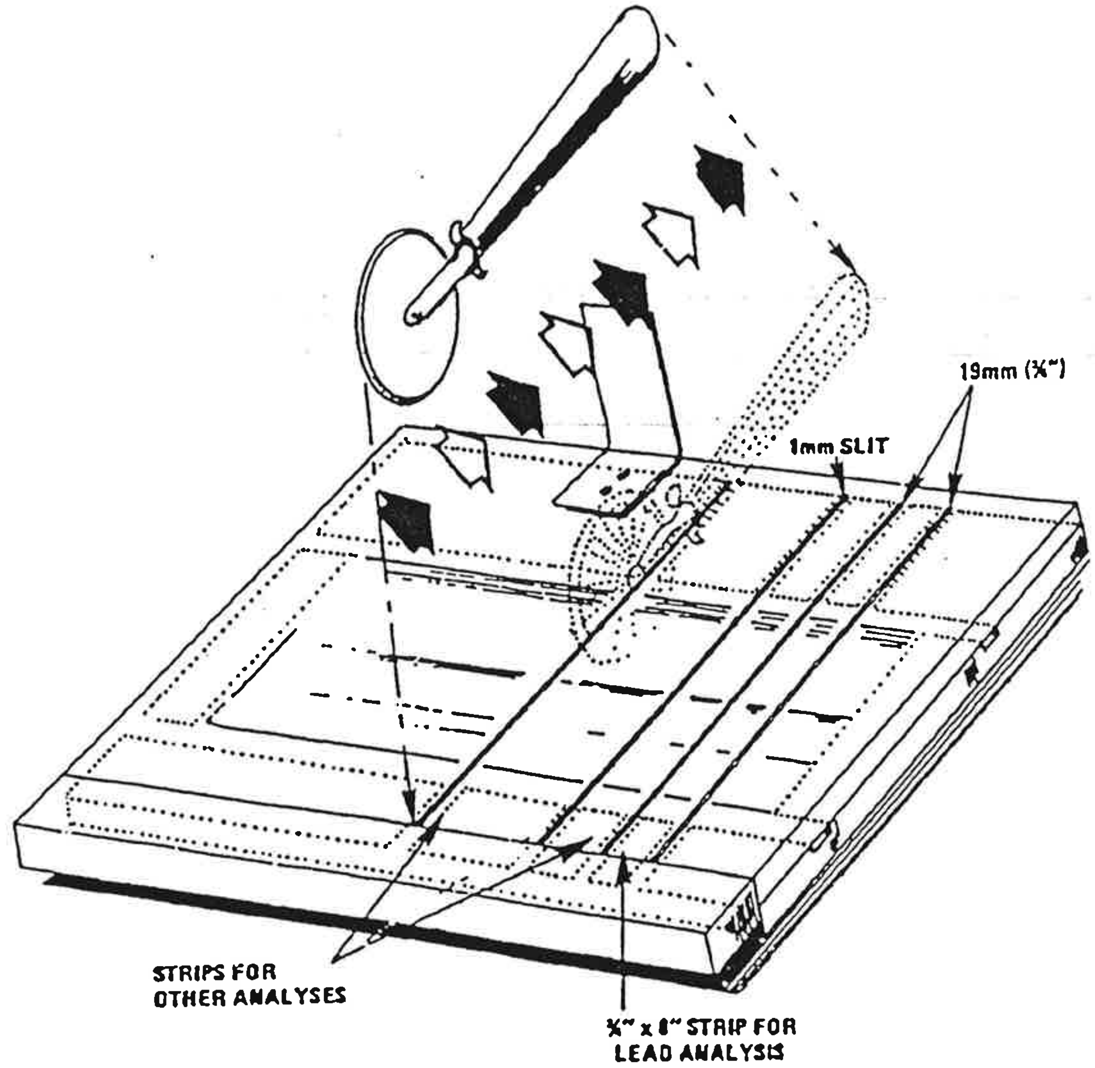

Figure 2

(Secs. 109, 301(a) of the Clean Air Act, as amended (42 U.S.C. 7409, 7601(a)); secs. 110, 301(a) and 319 of the Clean Air Act (42 U.S.C. 7410, 7601(a), 7619))

[43 FR 46258, Oct. 5, 1978; 44 FR 37915, June 29, 1979, as amended at 46 FR 44163, Sept. 3, 1981; 52 FR 24664, July 1, 1987] analysiswith and without the method of standard additions.(*7*)

3.2 *Light scattering.* Nonatomic absorption or light scattering, produced by high concentrations of dissolved solids in the sample, can produce a significant interference, especially at low lead concentrations. (*2*) The interference is greater at the 217.0 nm line than at the 283.3 nm line. No interference was observed using the 283.3 nm line with a similar method.(*1*)

Light scattering interferences can, however, be corrected for instrumentally. Since the dissolved solids can vary depending on the origin of the sample, the correction may be necessary, especially when using the 217.0 nm line. Dual beam instruments with a continuum source give the most accurate correction. A less accurate correction can be obtained by using a nonabsorbing lead line that is near the lead analytical line. Information on use of these correction techniques can be obtained from instrument manufacturers' manuals.

If instrumental correction is not feasible, the interference can be eliminated by use of the ammonium pyrrolidinecarbodithioate-methylisobutyl ketone, chelation-solvent extraction technique of sample preparation.(*8*)

4. *Precision and bias.*

4.1 The high-volume sampling procedure used to collect ambient air particulate matter has a between-laboratory relative standard deviation of 3.7 percent over the range 80 to 125 μg/m³.(*9*) The combined extraction-analysis procedure has an average within-laboratory relative standard deviation of 5 to 6 percent over the range 1.5 to 15 μg Pb/ml, and an average between laboratory relative standard deviation of 7 to 9 percent over the same range. These values include use of either extraction procedure.

4.2 Single laboratory experiments and collaborative testing indicate that there is no significant difference in lead recovery between the hot and ultrasonic extraction procedures.(*15*)

5. *Apparatus.*

5.1 *Sampling.*

5.1.1 *High-Volume Sampler.* Use and calibrate the sampler as described in appendix B to this part.

5.2 *Analysis.*

5.2.1 *Atomic absorption spectrophotometer.* Equipped with lead hollow cathode or electrodeless discharge lamp.

5.2.1.1 *Acetylene.* The grade recommended by the instrument manufacturer should be used. Change cylinder when pressure drops below 50–100 psig.

5.2.1.2 *Air.* Filtered to remove particulate, oil, and water.

5.2.2 *Glassware.* Class A borosilicate glassware should be used throughout the analysis.

5.2.2.1 *Beakers.* 30 and 150 ml. graduated, Pyrex.

5.2.2.2 *Volumetric flasks.* 100-ml.

5.2.2.3 *Pipettes.* To deliver 50, 30, 15, 8, 4, 2, 1 ml.

5.2.2.4 *Cleaning.* All glassware should be scrupulously cleaned. The following procedure is suggested. Wash with laboratory detergent, rinse, soak for 4 hours in 20 percent (w/w) HNO_3, rinse 3 times with distilled-deionized water, and dry in a dust free manner.

5.2.3 *Hot plate.*

5.2.4. *Ultrasonication water bath, unheated.* Commercially available laboratory ultrasonic cleaning baths of 450 watts or higher "cleaning power," i.e., actual ultrasonic power output to the bath have been found satisfactory.

5.2.5 *Template.* To aid in sectioning the glass-fiber filter. See figure 1 for dimensions.

5.2.6 *Pizza cutter.* Thin wheel. Thickness 1mm.

5.2.7 *Watch glass.*

5.2.8 *Polyethylene bottles.* For storage of samples. Linear polyethylene gives better storage stability than other polyethylenes and is preferred.

5.2.9 Parafilm "M".[1] American Can Co., Marathon Products, Neenah, Wis., or equivalent.

6. *Reagents.*

6.1 *Sampling.*

6.1.1 *Glass fiber filters.* The specifications given below are intended to aid the user in obtaining high quality filters with reproducible properties. These specifications have been met by EPA contractors.

6.1.1.1 *Lead content.* The absolute lead content of filters is not critical, but low values are, of course, desirable. EPA typically obtains filters with a lead content of 75 μg/filter.

It is important that the variation in lead content from filter to filter, within a given batch, be small.

6.1.1.2 *Testing.*

6.1.1.2.1 For large batches of filters (>500 filters) select at random 20 to 30 filters from a given batch. For small batches (>500 filters) a lesser number of filters may be taken. Cut one ¾″x8″ strip from each filter anywhere in the filter. Analyze all strips, separately, according to the directions in sections 7 and 8.

6.1.1.2.2 Calculate the total lead in each filter as

$$F_b = \mu g\ Pb/ml \times \frac{100\,ml}{strip} \times \frac{12\,strips}{filter}$$

where:

F_b=Amount of lead per 72 square inches of filter, μg.

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[1] Mention of commercial products does not imply endorsement by the U.S. Environmental Protection Agency.

6.1.1.2.3 Calculate the mean, F_b, of the values and the relative standard deviation (standard deviation/mean × 100). If the relative standard deviation is high enough so that, in the analysts opinion, subtraction of F_b, (section 10.3) may result in a significant error in the µg Pb/m³, the batch should be rejected.

6.1.1.2.4 For acceptable batches, use the value of F_b to correct all lead analyses (section 10.3) of particulate matter collected using that batch of filters. If the analyses are below the LDL (section 2.3) no correction is necessary.

6.2 *Analysis.*

6.2.1 Concentrated (15.6 *M*) HNO_3. ACS reagent grade HNO_3 and commercially available redistilled HNO_3 has found to have sufficiently low lead concentrations.

6.2.2 Concentrated (11.7 *M*) HCl. ACS reagent grade.

6.2.3 *Distilled-deionized water.* (D.I. water).

6.2.4 3 *M* HNO_3. This solution is used in the hot extraction procedure. To prepare, add 192 ml of concentrated HNO_3 to D.I. water in a 1 *l* volumetric flask. Shake well, cool, and dilute to volume with D.I. water. *Caution:* Nitric acid fumes are toxic. Prepare in a well ventilated fume hood.

6.2.5 0.45 *M* HNO_3. This solution is used as the matrix for calibration standards when using the hot extraction procedure. To prepare, add 29 ml of concentrated HNO_3 to D.I. water in a 1 *l* volumetric flask. Shake well, cool, and dilute to volume with D.I. water.

6.2.6 2.6 *M* HNO_3+0 to 0.9 *M* HCl. This solution is used in the ultrasonic extraction procedure. The concentration of HCl can be varied from 0 to 0.9 *M*. Directions are given for preparation of a 2.6 *M* HNO_3+0.9 *M* HCl solution. Place 167 ml of concentrated HNO_3 into a 1 *l* volumetric flask and add 77 ml of concentrated HCl. Stir 4 to 6 hours, dilute to nearly 1 *l* with D.I. water, cool to room temperature, and dilute to 1 *l*.

6.2.7 0.40 *M* HNO_3 + X *M* HCl. This solution is used as the matrix for calibration standards when using the ultrasonic extraction procedure. To prepare, add 26 ml of concentrated HNO_3, plus the ml of HCl required, to a 1 *l* volumetric flask. Dilute to nearly 1 *l* with D.I. water, cool to room temperature, and dilute to 1 *l*. The amount of HCl required can be determined from the following equation:

$$y = \frac{77\,\text{ml} \times 0.15x}{0.9\,\text{M}}$$

where:

y = ml of concentrated HCl required.
x = molarity of HCl in 6.2.6.
0.15 = dilution factor in 7.2.2.

6.2.8 Lead nitrate, $Pb(NO_3)_2$. ACS reagent grade, purity 99.0 percent. Heat for 4 hours at 120° C and cool in a desiccator.

6.3 *Calibration standards.*

6.3.1 Master standard, 1000 µg Pb/ml in HNO_3. Dissolve 1.598 g of $Pb(NO_3)_2$ in 0.45 *M* HNO_3 contained in a 1 *l* volumetric flask and dilute to volume with 0.45 *M* HNO_3.

6.3.2 Master standard, 1000 µg Pb/ml in HNO_3/HCl. Prepare as in section 6.3.1 except use the HNO_3/HCl solution in section 6.2.7.

Store standards in a polyethylene bottle. Commercially available certified lead standard solutions may also be used.

7. *Procedure.*

7.1 *Sampling.* Collect samples for 24 hours using the procedure described in reference *10* with glass-fiber filters meeting the specifications in section 6.1.1. Transport collected samples to the laboratory taking care to minimize contamination and loss of sample. *(16)*.

7.2 *Sample preparation.*

7.2.1 *Hot extraction procedure.*

7.2.1.1 Cut a ¾″ x 8″ strip from the exposed filter using a template and a pizza cutter as described in Figures 1 and 2. Other cutting procedures may be used.

Lead in ambient particulate matter collected on glass fiber filters has been shown to be uniformly distributed across the filter.[1,3,11] Another study[12] has shown that when sampling near a roadway, strip position contributes significantly to the overall variability associated with lead analyses. Therefore, when sampling near a roadway, additional strips should be analyzed to minimize this variability.

7.2.1.2 Fold the strip in half twice and place in a 150-ml beaker. Add 15 ml of 3 *M* HNO_3 to cover the sample. The acid should completely cover the sample. Cover the beaker with a watch glass.

7.2.1.3 Place beaker on the hot-plate, contained in a fume hood, and boil gently for 30 min. Do not let the sample evaporate to dryness. *Caution:* Nitric acid fumes are toxic.

7.2.1.4 Remove beaker from hot plate and cool to near room temperature.

7.2.1.5 Quantitatively transfer the sample as follows:

7.2.1.5.1 Rinse watch glass and sides of beaker with D.I. water.

7.2.1.5.2 Decant extract and rinsings into a 100-ml volumetric flask.

7.2.1.5.3 Add D.I. water to 40 ml mark on beaker, cover with watch glass, and set aside for a minimum of 30 minutes. This is a critical step and cannot be omitted since it allows the HNO_3 trapped in the filter to diffuse into the rinse water.

7.2.1.5.4 Decant the water from the filter into the volumetric flask.

7.2.1.5.5 Rinse filter and beaker twice with D.I. water and add rinsings to volumetric flask until total volume is 80 to 85 ml.

7.2.1.5.6 Stopper flask and shake vigorously. Set aside for approximately 5 minutes or until foam has dissipated.

7.2.1.5.7 Bring solution to volume with D.I. water. Mix thoroughly.

7.2.1.5.8 Allow solution to settle for one hour before proceeding with analysis.

7.2.1.5.9 If sample is to be stored for subsequent analysis, transfer to a linear polyethylene bottle.

7.2.2 *Ultrasonic extraction procedure.*

7.2.2.1 Cut a ¾″x 8″ strip from the exposed filter as described in section 7.2.1.1.

7.2.2.2 Fold the strip in half twice and place in a 30 ml beaker. Add 15 ml of the HNO_3/HCl solution in section 6.2.6. The acid should completely cover the sample. Cover the beaker with parafilm.

The parafilm should be placed over the beaker such that none of the parafilm is in contact with water in the ultrasonic bath. Otherwise, rinsing of the parafilm (section 7.2.2.4.1) may contaminate the sample.

7.2.2.3 Place the beaker in the ultrasonication bath and operate for 30 minutes.

7.2.2.4 Quantitatively transfer the sample as follows:

7.2.2.4.1 Rinse parafilm and sides of beaker with D.I. water.

7.2.2.4.2 Decant extract and rinsings into a 100 ml volumetric flask.

7.2.2.4.3 Add 20 ml D.I. water to cover the filter strip, cover with parafilm, and set aside for a minimum of 30 minutes. This is a critical step and cannot be omitted. The sample is then processed as in sections 7.2.1.5.4 through 7.2.1.5.9.

NOTE: Samples prepared by the hot extraction procedure are now in 0.45 M HNO_3. Samples prepared by the ultrasonication procedure are in 0.40 M HNO_3 + X M HCl.

8. *Analysis.*

8.1 Set the wavelength of the monochromator at 283.3 or 217.0 nm. Set or align other instrumental operating conditions as recommended by the manufacturer.

8.2 The sample can be analyzed directly from the volumetric flask, or an appropriate amount of sample decanted into a sample analysis tube. In either case, care should be taken not to disturb the settled solids.

8.3 Aspirate samples, calibration standards and blanks (section 9.2) into the flame and record the equilibrium absorbance.

8.4 Determine the lead concentration in µg Pb/ml, from the calibration curve, section 9.3.

8.5 Samples that exceed the linear calibration range should be diluted with acid of the same concentration as the calibration standards and reanalyzed.

9. *Calibration.*

9.1 Working standard, 20 µg Pb/ml. Prepared by diluting 2.0 ml of the master standard (section 6.3.1 if the hot acid extraction was used or section 6.3.2 if the ultrasonic extraction procedure was used) to 100 ml with acid of the same concentration as used in preparing the master standard.

9.2 *Calibration standards.* Prepare daily by diluting the working standard, with the same acid matrix, as indicated below. Other lead concentrations may be used.

Volume of 20 µg/ml working standard, ml	Final volume, ml	Concentration µg Pb/ml
0	100	0
1.0	200	0.1
2.0	200	0.2
2.0	100	0.4
4.0	100	0.8
8.0	100	1.6
15.0	100	3.0
30.0	100	6.0
50.0	100	10.0
100.0	100	20.0

9.3 *Preparation of calibration curve.* Since the working range of analysis will vary depending on which lead line is used and the type of instrument, no one set of instructions for preparation of a calibration curve can be given. Select standards (plus the reagent blank), in the same acid concentration as the samples, to cover the linear absorption range indicated by the instrument manufacturer. Measure the absorbance of the blank and standards as in section 8.0. Repeat until good agreement is obtained between replicates. Plot absorbance (y-axis) versus concentration in µg Pb/ml (x-axis). Draw (or compute) a straight line through the linear portion of the curve. Do not force the calibration curve through zero. Other calibration procedures may be used.

To determine stability of the calibration curve, remeasure—alternately—one of the following calibration standards for every 10th sample analyzed: Concentration ≤ 1µg Pb/ml; concentration ≤ 10 µg Pb/ml. If either standard deviates by more than 5 percent from the value predicted by the calibration curve, recalibrate and repeat the previous 10 analyses.

10. *Calculation.*

10.1 *Measured air volume.* Calculate the measured air volume at Standard Temperature and Pressure as described in Reference 10.

10.2 *Lead concentration.* Calculate lead concentration in the air sample.

$$C = \frac{(\mu g\ Pb/ml \times 100\ ml/strip \times 12\ strips/filter) - F_b}{V_{STP}}$$

where:

C=Concentration, µg Pb/sm³.

µg Pb/ml=Lead concentration determined from section 8.

100 ml/strip=Total sample volume.

12 strips=Total useable filter area, 8″ × 9″. Exposed area of one strip, ¾″ × 7″.

Filter=Total area of one strip, ¾″ × 8″.

F_b=Lead concentration of blank filter, µg, from section 6.1.1.2.3.

V_{STP}=Air volume from section 10.2.

11. *Quality control.*

¾″ × 8″ glass fiber filter strips containing 80 to 2000 µg Pb/strip (as lead salts) and blank strips with zero Pb content should be used to determine if the method—as being used—has any bias. Quality control charts should be established to monitor differences between measured and true values. The frequency of such checks will depend on the local quality control program.

To minimize the possibility of generating unreliable data, the user should follow practices established for assuring the quality of air pollution data, *(13)* and take part in EPA's semiannual audit program for lead analyses.

12. *Trouble shooting.*

1. During extraction of lead by the hot extraction procedure, it is important to keep the sample covered so that corrosion products—formed on fume hood surfaces which may contain lead—are not deposited in the extract.

2. The sample acid concentration should minimize corrosion of the nebulizer. However, different nebulizers may require lower acid concentrations. Lower concentrations can be used provided samples and standards have the same acid concentration.

3. Ashing of particulate samples has been found, by EPA and contractor laboratories, to be unnecessary in lead analyses by atomic absorption. Therefore, this step was omitted from the method.

4. Filtration of extracted samples, to remove particulate matter, was specifically excluded from sample preparation, because some analysts have observed losses of lead due to filtration.

5. If suspended solids should clog the nebulizer during analysis of samples, centrifuge the sample to remove the solids.

13. *Authority.*

(Secs. 109 and 301(a), Clean Air Act, as amended (42 U.S.C. 7409, 7601(a)))

14. *References.*

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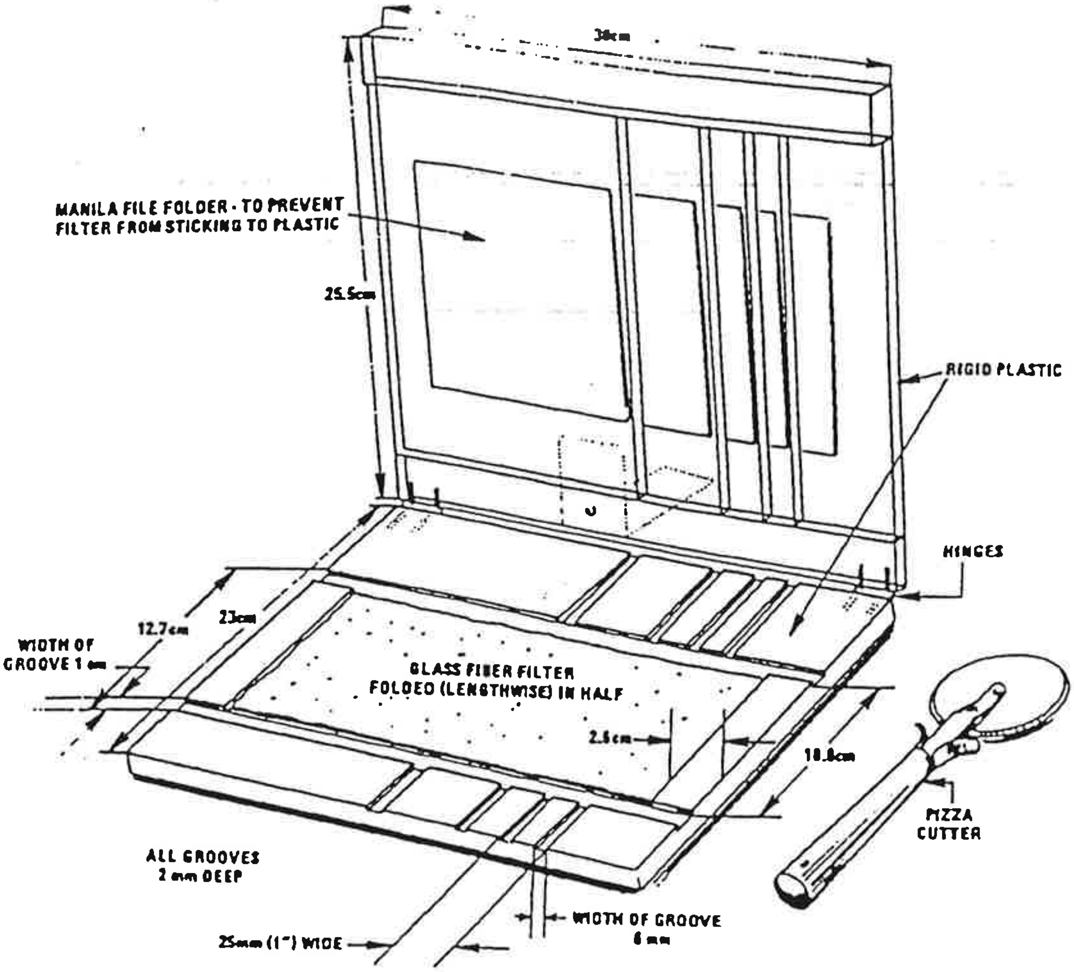

Figure 1

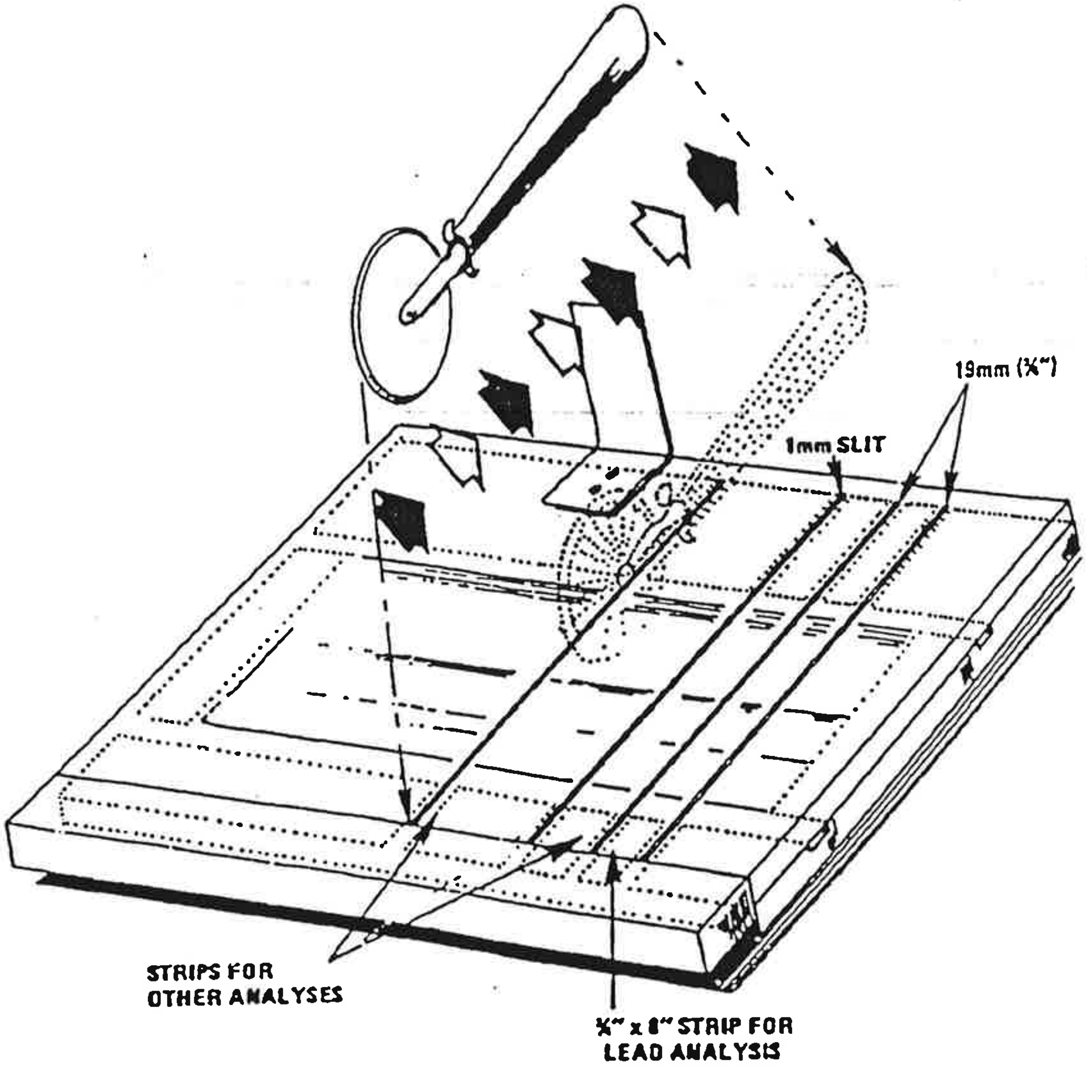

Figure 2

(Secs. 109, 301(a) of the Clean Air Act, as amended (42 U.S.C. 7409, 7601(a)); secs. 110, 301(a) and 319 of the Clean Air Act (42 U.S.C. 7410, 7601(a), 7619))

[43 FR 46258, Oct. 5, 1978; 44 FR 37915, June 29, 1979, as amended at 46 FR 44163, Sept. 3, 1981; 52 FR 24664, July 1, 1987]

METHOD 6010B

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards. Refer to Chapter Three for the appropriate digestion procedures.

1.2 Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths and estimated instrumental detection limits for the elements in clean aqueous matrices. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements and matrices other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 8.0) is demonstrated.

1.3 Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

1.4 Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Chapter Three). When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 3.0 should also be recognized and appropriate corrections made; tests for their presence are described in Section 8.5. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

3.0 INTERFERENCES

3.1 Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

3.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

3.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

3.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction **require** the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

3.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary.

3.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

3.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences (Table 2) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

3.1.7 Users of sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.

3.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

3.1.9 When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within the 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

3.1.10 When interelement corrections are not used, verification of absence of interferences is required.

3.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

3.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is ≥ 20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

3.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 8.5.1 will help determine if a physical interference is present.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

3.4 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

3.5 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

3.6 The dashes in Table 2 indicate that no measurable interferences were observed even at higher interferant concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

4.0 APPARATUS AND MATERIALS

4.1 Inductively coupled argon plasma emission spectrometer:

4.1.1 Computer-controlled emission spectrometer with background correction.

4.1.2 Radio-frequency generator compliant with FCC regulations.

4.1.3 Optional mass flow controller for argon nebulizer gas supply.

4.1.4 Optional peristaltic pump.

4.1.5 Optional Autosampler.

4.1.6 Argon gas supply - high purity.

4.2 Volumetric flasks of suitable precision and accuracy.

4.3 Volumetric pipets of suitable precision and accuracy.

5.0 REAGENTS

5.1 Reagent or trace metals grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

5.1.1 Hydrochloric acid (conc), HCl.

5.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriately sized beaker.

5.1.3 Nitric acid (conc), HNO_3.

5.1.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an appropriately sized beaker.

5.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

5.3 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All salts must be dried for 1 hour at 105°C, unless otherwise specified.

Note: This section does not apply when analyzing samples that have been prepared by Method 3040.

CAUTION: Many metal salts are extremely toxic if inhaled or swallowed. Wash hands thoroughly after handling.

Typical stock solution preparation procedures follow. Concentrations are calculated based upon the weight of pure metal added, or with the use of the element fraction and the weight of the metal salt added.

For metals:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg)}}{\text{volume (L)}}$$

For metal salts:

$$\text{Concentration (ppm)} = \frac{\text{weight (mg) x mole fraction}}{\text{volume (L)}}$$

5.3.1 Aluminum solution, stock, 1 mL = 1000 µg Al: Dissolve 1.000 g of aluminum metal, weighed accurately to at least four significant figures, in an acid mixture of 4.0 mL of (1:1) HCl and 1.0 mL of concentrated $HN0_3$ in a beaker. Warm beaker slowly to effect solution. When dissolution is complete, transfer solution quantitatively to a 1-liter flask, add an additional 10.0 mL of (1:1) HCl and dilute to volume with reagent water.

NOTE: Weight of analyte is expressed to four significant figures for consistency with the weights below because rounding to two decimal places can contribute up to 4 % error for some of the compounds.

5.3.2 Antimony solution, stock, 1 mL = 1000 µg Sb: Dissolve 2.6673 g $K(SbO)C_4H_4O_6$ (element fraction Sb = 0.3749), weighed accurately to at least four significant figures, in water, add 10 mL (1:1) HCl, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.3 Arsenic solution, stock, 1 mL = 1000 µg As: Dissolve 1.3203 g of As_2O_3 (element fraction As = 0.7574), weighed accurately to at least four significant figures, in 100 mL of water containing 0.4 g NaOH. Acidify the solution with 2 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.4 Barium solution, stock, 1 mL = 1000 µg Ba: Dissolve 1.5163 g $BaCl_2$ (element fraction Ba = 0.6595), dried at 250°C for 2 hours, weighed accurately to at least four significant figures, in 10 mL water with 1 mL (1:1) HCl. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.5 Beryllium solution, stock, 1 mL = 1000 µg Be: Do not dry. Dissolve 19.6463 g $BeSO_4 \cdot 4H_2O$ (element fraction Be = 0.0509), weighed accurately to at least four significant figures, in water, add 10.0 mL concentrated HNO_3, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.6 Boron solution, stock, 1 mL = 1000 µg B: Do not dry. Dissolve 5.716 g anhydrous H_3BO_3 (B fraction = 0.1749), weighed accurately to at least four significant figures, in reagent water and dilute in a 1-L volumetric flask with reagent water. Transfer immediately after mixing in a clean polytetrafluoroethylene (PTFE) bottle to minimize any leaching of boron from the glass volumetric container. Use of a non-glass volumetric flask is recommended to avoid boron contamination from glassware.

5.3.7 Cadmium solution, stock, 1 mL = 1000 µg Cd: Dissolve 1.1423 g CdO (element fraction Cd = 0.8754), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.8 Calcium solution, stock, 1 mL = 1000 µg Ca: Suspend 2.4969 g $CaCO_3$ (element Ca fraction = 0.4005), dried at 180°C for 1 hour before weighing, weighed accurately to at least four significant figures, in water and dissolve cautiously with a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.9 Chromium solution, stock, 1 mL = 1000 µg Cr: Dissolve 1.9231 g CrO_3 (element fraction Cr = 0.5200), weighed accurately to at least four significant figures, in water. When solution is complete, acidify with 10 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.10 Cobalt solution, stock, 1 mL = 1000 µg Co: Dissolve 1.00 g of cobalt metal, weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.11 Copper solution, stock, 1 mL = 1000 µg Cu: Dissolve 1.2564 g CuO (element fraction Cu = 0.7989), weighed accurately to at least four significant figures), in a minimum amount of (1:1) HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.12 Iron solution, stock, 1 mL = 1000 µg Fe: Dissolve 1.4298 g Fe_2O_3 (element fraction Fe = 0.6994), weighed accurately to at least four significant figures, in a warm mixture of 20 mL (1:1) HCl and 2 mL of concentrated HNO_3. Cool, add an additional 5.0 mL of concentrated HNO_3, and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.13 Lead solution, stock, 1 mL = 1000 µg Pb: Dissolve 1.5985 g $Pb(NO_3)_2$ (element fraction Pb = 0.6256), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10 mL (1:1) HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.14 Lithium solution, stock, 1 mL = 1000 µg Li: Dissolve 5.3248 g lithium carbonate (element fraction Li = 0.1878), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HCl and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.15 Magnesium solution, stock, 1 mL = 1000 µg Mg: Dissolve 1.6584 g MgO (element fraction Mg = 0.6030), weighed accurately to at least four significant figures, in a minimum amount of (1:1) HNO_3. Add 10.0 mL (1:1) concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.16 Manganese solution, stock, 1 mL = 1000 µg Mn: Dissolve 1.00 g of manganese metal, weighed accurately to at least four significant figures, in acid mixture (10 mL concentrated HCl and 1 mL concentrated HNO_3) and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.28 Tin solution, stock, 1 mL = 1000 µg Sn: Dissolve 1.000 g Sn shot, weighed accurately to at least 4 significant figures, in 200 mL (1:1) HCl with heating to effect dissolution. Let solution cool and dilute with (1:1) HCl in a 1-L volumetric flask.

5.3.29 Vanadium solution, stock, 1 mL = 1000 µg V: Dissolve 2.2957 g NH_4VO_3 (element fraction V = 0.4356), weighed accurately to at least four significant figures, in a minimum amount of concentrated HNO_3. Heat to increase rate of dissolution. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.3.30 Zinc solution, stock, 1 mL = 1000 µg Zn: Dissolve 1.2447 g ZnO (element fraction Zn = 0.8034), weighed accurately to at least four significant figures, in a minimum amount of dilute HNO_3. Add 10.0 mL concentrated HNO_3 and dilute to volume in a 1,000 mL volumetric flask with water.

5.4 Mixed calibration standard solutions - Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks (see Table 3). Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage. Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging. Some typical calibration standard combinations are listed in Table 3.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.

5.5 Two types of blanks are required for the analysis for samples prepared by any method other than 3040. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing.

5.5.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial and continuing calibration blank determinations (see Sections 7.3 and 7.4).

5.5.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

5.6 The Initial Calibration Verification (ICV) is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument (see Section 8.6.1 for use).

5.7 The Continuing Calibration Verification (CCV)) should be prepared in the same acid matrix using the same standards used for calibration at a concentration near the mid-point of the calibration curve (see Section 8.6.1 for use).

5.8 The interference check solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest, particularly those with known interferences at 0.5 to 1 mg/L. In the absence of measurable analyte, overcorrection could go undetected because a negative value could be reported as zero. If the particular instrument will display overcorrection as a negative number, this spiking procedure will not be necessary.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material in Chapter Three, Inorganic Analytes, Sections 3.1 through 3.3.

7.0 PROCEDURE

7.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must either use an internal standard or be matrix matched with the standards. Solubilization and digestion procedures are presented in Sample Preparation Methods (Chapter Three, Inorganic Analytes).

7.2 Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration). Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer.

7.2.1 Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges, the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis. These documented data must be kept on file and be available for review by the data user or auditor.

7.2.2 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Because of differences among various makes and models of spectrometers, specific instrument operating conditions cannot be provided. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user. The analyst should follow the instructions provided by the instrument manufacturer unless other conditions provide similar or better performance for a task. Operating conditions for aqueous solutions usually vary from 1100 to 1200 watts forward power, 14 to 18 mm viewing height, 15 to 19 liters/min argon coolant flow, 0.6 to 1.5 L/min argon nebulizer flow, 1 to 1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments. For an axial plasma, the conditions will usually vary from 1100-1500 watts forward power, 15-19 liters/min argon coolant flow, 0.6-1.5 L/min argon nebulizer flow, 1-1.8 mL/min sample pumping rate with a 1 minute preflush time and measurement time near 1 second per wavelength peak for sequential instruments and 10 seconds per sample for simultaneous instruments. Reproduction of the Cu/Mn intensity ratio at 324.754 nm and 257.610 nm respectively, by adjusting the argon aerosol flow has been recommended as a way to achieve repeatable interference correction factors.

7.2.3 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

7.2.3.1 Ignite the radial plasma and select an appropriate incident RF power. Allow the instrument to become thermally stable before beginning, about 30 to 60 minutes of operation. While aspirating a 1000 ug/L solution of yttrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the load coil. Record the nebulizer gas flow rate or pressure setting for future reference. The yttrium solution can also be used for coarse optical alignment of the torch by observing the overlay of the blue light over the entrance slit to the optical system.

7.2.3.2 After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min by aspirating a known volume of calibration blank for a period of at least three minutes. Divide the volume aspirated by the time in minutes and record the uptake rate; set the peristaltic pump to deliver the rate in a steady even flow.

7.2.3.3 Profile the instrument to align it optically as it will be used during analysis. The following procedure can be used for both horizontal and vertical optimization in the radial mode, but is written for vertical. Aspirate a solution containing 10 ug/L of several selected elements. These elements can be As, Se, Tl or Pb as the least sensitive of the elements and most needing to be optimize or others representing analytical judgement (V, Cr, Cu, Li and Mn are also used with success). Collect intensity data at the wavelength peak for each analyte at 1 mm intervals from 14 to 18 mm above the load coil. (This region of the plasma is referred to as the analytical zone.) Repeat the process using the calibration blank. Determine the net signal to blank intensity ratio for each analyte for each viewing height setting. Choose the height for viewing the plasma that provides the best net intensity ratios for the elements analyzed or the highest intensity ratio for the least sensitive element. For optimization in the axial mode, follow the instrument manufacturer's instructions.

7.2.3.4 The instrument operating condition finally selected as being optimum should provide the lowest reliable instrument detection limits and method detection limits.

7.2.3.5 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and viewing height should be re-optimized.

7.2.3.6 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within ± one reporting limit from zero. The upper control limit is the analyte instrument detection limit. Once established the entire routine must be periodically verified every six months. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine should be kept on file. Special cases where continual verification is required are described elsewhere.

7.2.3.7 Before daily calibration and after the instrument warmup period, the nebulizer gas flow rate must be reset to the determined optimized flow. If a mass flow controller is being used, it should be set to the recorded optimized flow rate, In order to maintain valid spectral interelement correction routines the nebulizer gas flow rate should be the same (< 2% change) from day to day.

7.2.4 For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements.

7.2.5 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

7.2.5.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit. Refer to Chapter One for additional guidance on the performance of MDL studies.

7.2.5.2 Determination of limits using reagent water represent a best case situation and do not represent possible matrix effects of real world samples.

7.2.5.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate. Successful analysis of samples with added analytes or using method of standard additions can give confidence in the method detection limit values determined in reagent water.

7.2.5.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses from a minimum for three, preferably five, different concentration standards across the range. One of these should be near the upper limit of the range. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst should also be aware that if an interelement correction from an analyte above the linear range exists, a second analyte where the interelement correction has been applied may be inaccurately reported. New dynamic ranges should be determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range should be checked every six months. For those analytes that are known interferences, and are present at above the linear range, the analyst should ensure that the interelement correction has not been inaccurately applied.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit should have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These non-linear response curves should be revalidated and recalculated every six months. These curves are much more sensitive to changes in operating conditions than the linear lines and should be checked whenever there have been moderate equipment changes.

7.2.6 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

7.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 5.4. Flush the system with the calibration blank (Section 5.5.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve must consist of a minimum of a blank and a standard.

7.4 For all analytes and determinations, the laboratory must analyze an ICV (Section 5.6), a calibration blank (Section 5.5.1), and a continuing calibration verification (CCV) (Section 5.7) immediately following daily calibration. A calibration blank and either a calibration verification (CCV) or an ICV must be analyzed after every tenth sample and at the end of the sample run. Analysis of the check standard and calibration verification must verify that the instrument is within ± 10% of calibration with relative standard deviation < 5% from replicate (minimum of two) integrations. If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV, CCV or check standard must be reanalyzed. The analysis data of the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

7.5 Rinse the system with the calibration blank solution (Section 5.5.1) before the analysis of each sample. The rinse time will be one minute. Each laboratory may establish a reduction in this rinse time through a suitable demonstration.

7.6 Calculations: If dilutions were performed, the appropriate factors must be applied to sample values. All results should be reported with up to three significant figures.

7.7 The MSA should be used if an interference is suspected or a new matrix is encountered. When the method of standard additions is used, standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix. It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. This technique is valid in the linear range when the interference effect is constant over the range, the added analyte responds the same as the endogenous analyte, and the signal is corrected for additive interferences. The simplest version of this technique is the single addition method. This procedure calls for two identical aliquots of the sample solution to be taken. To the first aliquot, a small volume of standard is added; while to the second aliquot, a volume of acid blank is added equal to the standard addition. The sample concentration is calculated by: multiplying the intensity value for the unfortified aliquot by the volume (Liters) and concentration (mg/L or mg/kg) of the standard addition to make the numerator; the difference in intensities for the fortified sample and unfortified sample is multiplied by the volume (Liters) of the sample aliquot for the denominator. The quotient is the sample concentration.

For more than one fortified portion of the prepared sample, linear regression analysis can be applied using a computer or calculator program to obtain the concentration of the sample solution.

NOTE: Refer to Method 7000 for a more detailed discussion of the MSA.

7.8 An alternative to using the method of standard additions is the internal standard technique. Add one or more elements not in the samples and verified not to cause an interelement spectral interference to the samples, standards and blanks; yttrium or scandium are often used. The concentration should be sufficient for optimum precision but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. This technique is very useful in overcoming matrix interferences especially in high solids matrices.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection. All quality control measures described in Chapter One should be followed.

8.2 Dilute and reanalyze samples that exceed the linear calibration range or use an alternate, less sensitive line for which quality control data is already established.

8.3 Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample (refer to Chapter One).

8.4 Analyze matrix spiked duplicate samples at a frequency of one per matrix batch. A matrix duplicate sample is a sample brought through the entire sample preparation and analytical process in duplicate.

8.4.1.1 The relative percent difference between spiked matrix duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (replicate).

(A control limit of ± 20% RPD or within the documented historical acceptance limits for each matrix shall be used for sample values greater than ten times the instrument detection limit.)

8.4.1.2 The spiked sample or spiked duplicate sample recovery is to be within ± 25% of the actual value or within the documented historical acceptance limits for each matrix.

8.5 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in Sections 8.5.1 and 8.5.2, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

8.5.1 Dilution Test: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

8.5.2 Post Digestion Spike Addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

8.6 Check the instrument standardization by analyzing appropriate QC samples as follows.

8.6.1 Verify calibration with the Continuing Calibration Verification (CCV) Standard immediately following daily calibration, after every ten samples, and at the end of an analytical run. Check calibration with an ICV following the initial calibration (Section 5.6). At the laboratory's discretion, an ICV may be used in lieu of the continuing calibration verifications. If used in this manner, the ICV should be at a concentration near the mid-point of the calibration curve. Use a calibration blank (Section 5.5.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.

8.6.1.1 The results of the ICV and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.6.1.2 The results of the check standard are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.6.1.3 The results of the calibration blank are to agree within three times the IDL. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

8.6.2 Verify the interelement and background correction factors at the beginning of each analytical run. Do this by analyzing the interference check sample (Section 5.8). Results should be within ± 20% of the true value.

9.0 METHOD PERFORMANCE

9.1 In an EPA round-robin Phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been spiked with various metal concentrates. Table 4 lists the true values, the mean reported values, and the mean percent relative standard deviations.

9.2 Performance data for aqueous solutions and solid samples from a multilaboratory study (9) are provided in Tables 5 and 6.

10.0 REFERENCES

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TABLE 1
RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Detection Element	Wavelength[a] (nm)	Estimated IDL[b] (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227x2	17
Molybdenum	202.030	5.3
Nickel	231.604x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO_2)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856x2	1.2

[a] The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted (e.g., in the case of an interference) if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see Section 3.1). In time, other elements may be added as more information becomes available and as required.

[b] The estimated instrumental detection limits shown are provided as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[c] Highly dependent on operating conditions and plasma position.

TABLE 2
POTENTIAL INTERFERENCES
ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM INTERFERENCE AT THE 100-mg/L LEVEL[c]

		Interferant[a,b]									
Analyte	Wavelength (nm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al - 1000 mg/L
Ca - 1000 mg/L
Cr - 200 mg/L
Cu - 200 mg/L
Fe - 1000 mg/L
Mg - 1000 mg/L
Mn - 200 mg/L
Tl - 200 mg/L
V - 200 mg/L

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

[c] Interferences will be affected by background choice and other interferences may be present.

TABLE 3
MIXED STANDARD SOLUTIONS

Solution	Elements
I	Be, Cd, Mn, Pb, Se and Zn
II	Ba, Co, Cu, Fe, and V
III	As, Mo
IV	Al, Ca, Cr, K, Na, Ni,Li, and Sr
V	Ag (see "NOTE" to Section 5.4), Mg, Sb, and Tl
VI	P

TABLE 4. ICP PRECISION AND ACCURACY DATA[a]

Element	Sample No. 1				Sample No. 2				Sample No. 3			
	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b] (%)	Accuracy[d] (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b]	Accuracy[d] (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b] (%)	Accuracy[d] (%)
Be	750	733	6.2	98	20	20	9.8	100	180	176	5.2	98
Mn	350	345	2.7	99	15	15	6.7	100	100	99	3.3	99
V	750	749	1.8	100	70	69	2.9	99	170	169	1.1	99
As	200	208	7.5	104	22	19	23	86	60	63	17	105
Cr	150	149	3.8	99	10	10	18	100	50	50	3.3	100
Cu	250	235	5.1	94	11	11	40	100	70	67	7.9	96
Fe	600	594	3.0	99	20	19	15	95	180	178	6.0	99
Al	700	696	5.6	99	60	62	33	103	160	161	13	101
Cd	50	48	12	96	2.5	2.9	16	116	14	13	16	93
Co	700	512	10	73	20	20	4.1	100	120	108	21	90
Ni	250	245	5.8	98	30	28	11	93	60	55	14	92
Pb	250	236	16	94	24	30	32	125	80	80	14	100
Zn	200	201	5.6	100	16	19	45	119	80	82	9.4	102
Se[c]	40	32	21.9	80	6	8.5	42	142	10	8.5	8.3	85

[a] Not all elements were analyzed by all laboratories.
[b] RSD = relative standard deviation.
[c] Results for Se are from two laboratories.
[d] Accuracy is expressed as the mean concentration divided by the true concentration times 100.

TABLE 5

ICP-AES PRECISION AND ACCURACY FOR AQUEOUS SOLUTIONS[a]

Element	Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Accuracy[c] (%)
Al	14.8	8	6.3	100
Sb	15.1	8	7.7	102
As	14.7	7	6.4	99
Ba	3.66	7	3.1	99
Be	3.78	8	5.8	102
Cd	3.61	8	7.0	97
Ca	15.0	8	7.4	101
Cr	3.75	8	8.2	101
Co	3.52	8	5.9	95
Cu	3.58	8	5.6	97
Fe	14.8	8	5.9	100
Pb	14.4	7	5.9	97
Mg	14.1	8	6.5	96
Mn	3.70	8	4.3	100
Mo	3.70	8	6.9	100
Ni	3.70	7	5.7	100
K	14.1	8	6.6	95
Se	15.3	8	7.5	104
Ag	3.69	6	9.1	100
Na	14.0	8	4.2	95
Tl	15.1	7	8.5	102
V	3.51	8	6.6	95
Zn	3.57	8	8.3	96

[a] these performance values are independent of sample preparation because the labs analyzed portions of the same solutions

[b] N = Number of measurements for mean and relative standard deviation (RSD).

[c] Accuracy is expressed as a percentage of the nominal value for each analyte in acidified, multi-element solutions.

TABLE 6

ICP-AES PRECISION AND BIAS FOR SOLID WASTE DIGESTS[a]

	Spiked Coal Fly Ash (NIST-SRM 1633a)				Spiked Electroplating Sludge			
Element	Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Bias[c] (%AAS)	Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Bias[c] (%AAS)
Al	330	8	16	104	127	8	13	110
Sb	3.4	6	73	96	5.3	7	24	120
As	21	8	83	270	5.2	7	8.6	87
Ba	133	8	8.7	101	1.6	8	20	58
Be	4.0	8	57	460	0.9	7	9.9	110
Cd	0.97	6	5.7	101	2.9	7	9.9	90
Ca	87	6	5.6	208	954	7	7.0	97
Cr	2.1	7	36	106	154	7	7.8	93
Co	1.2	6	21	94	1.0	7	11	85
Cu	1.9	6	9.7	118	156	8	7.8	97
Fe	602	8	8.8	102	603	7	5.6	98
Pb	4.6	7	22	94	25	7	5.6	98
Mg	15	8	15	110	35	8	20	84
Mn	1.8	7	14	104	5.9	7	9.6	95
Mo	891	8	19	105	1.4	7	36	110
Ni	1.6	6	8.1	91	9.5	7	9.6	90
K	46	8	4.2	98	51	8	5.8	82
Se	6.4	5	16	73	8.7	7	13	101
Ag	1.4	3	17	140	0.75	7	19	270
Na	20	8	49	130	1380	8	9.8	95
Tl	6.7	4	22	260	5.0	7	20	180
V	1010	5	7.5	100	1.2	6	11	80
Zn	2.2	6	7.6	93	266	7	2.5	101

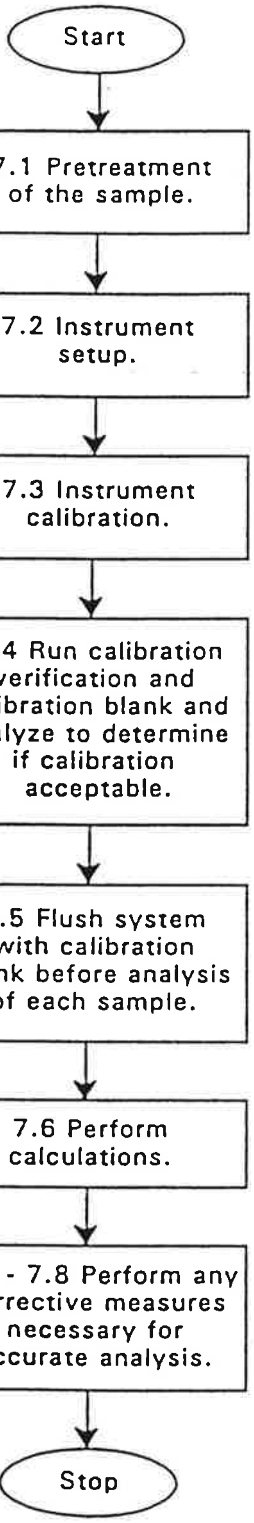
[a] These performance values are independent of sample preparation because the labs analyzed portions of the same digests.

[b] N = Number of measurements for mean and relative standard deviation (RSD).

[c] Bias for the ICP-AES data is expressed as a percentage of atomic absorption spectroscopy (AA) data for the same digests.

METHOD 6010B

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY



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Procedure for storage of data from chemical analysis in the EIMP project

Oddvar Røyset
Norwegian Institute for Air Research, NILU, Norway.

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Procedure for storage of data from chemical analysis in the EIMP project
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1. Introduction

This procedure describes recommendation for the storage of various data for chemical monitoring systems used within the EIMP project.

1.1 Storage time for archives

It is generally recommended in quality systems that all data should be archived and be available for inspection for at least -5- years. Exceptions for this rule may be where electronic media stores the same data in a safe way.

2. Archives of forms

2.1 Site forms

The site forms shall be stored in binders. It is recommended to save the forms for each site in separate binders. In each binder it is advisable to separate the site forms for different sampling systems.

2.2 Chromatograms

The daily printouts of chromatograms from the ion chromatographs should be stored in binders. If these data are also stored electronically (on tapes or CDs) these paper printouts may not be stored for more than one year.

2.3 Worksheet for chemical analysis

It is recommended to gather the results for the daily analysis on worksheets ("Laboratory worksheeet Ion Chromatography") before they are entered into Excel or laboratory database (LIMS) systems. The Laboratory worksheets should be filled in during the quality assurance control of the chromatograms from the ion chromatographs (or printout from other types of instrumentation).

2.4 Excel data storage and reporting forms

When all data have been properly quality controlled they are entered into the Excel data storage and reporting forms. It is recommended to store the Excel worksheet with data for each year a separate directories on the computer, i.e. "Data1998" etc. The following Excel workbooks are recommended:

Table 1
Recommended workbooks for storage of data

Report type	Excel file name	Comments
Report SO_2	SO2_template.xls	The Report SO_2 is a Excel workbook with one sheet for data and one for graphical presentation for each site.
Report NO_2	NO2_template.xls	As above
Report Passive NO_2	NO2_passive_template.xls	As above
Report Passive SO_2	SO2_passive_template.xls	As above
Report Dustfall	Dustfall_template.xls	As above
Report PM_{10}	PM10_template.xls	As above
Report Lead	Lead_template.xls	As above
Field blanks	Fieldblank_template.xls	Prepare one sheet for each component in one Excel workbook

2.5 Field blanks

It is recommended to make a separate log of the results for the field blanks, using a separate binder named "Field blanks". The data should also be stored in an Excel Workbook with graphical presentation (see Table 1). It is of high importance to have a good documentation of these to get a quick overview of the blank values of the sampling and analysis systems.

3. Electronic storage of data

3.1 Electronic storage of data from the Dionex Peaknet chomatography system

It is recommended to prepare a directory structure which facilitates easy access to the rawdata (data for each sample) files from the Dionex Peaknet chromatography system. A directory structure which is efficient is to make one catalogue (directory) for each month containing the year and month, (i.e. YYMM, 9901, 9902, 9903 etc.). Under each of these monthly catalogues a daily catalogue is made where the results for the single day is stored. For convenience these catalogues may be named MMDD plus additional information of choice. The directory structure for February 1999 may thus look like:

Table 2. Example of directory structure

YYMM-catalogue	MMDD-catalogues	Alternative names
9902		
	0201	SO2_0201
	0203	NO2_0203
	...	
	0228	

The analysis results for each sample is in Peaknet stored in an Microsoft Access database under the directory Peaknet/database with the name Peaknet.mdb. During analysis this database aggregates data and may eventually become rather. It is recommended to store this database under a separate name which is used for archiving old data. Depending on the production of data, this may be done on monthly, quarterly or yearly basis. For the EIMP program it is probably sufficient to do the archiving of this database on a yearly basis.The Peaknet.mdb file is thus copied to another name, such as Peaknet_9801_9806.mdb (to take care of data from January to June 1998), or Peaknet_9901_9912.mdb (for the whole year). The original Peaknet database is now emptied for data and new data may now be stored there.

3.2 Permanent electronic storage of data

All data of relevance which are captured non PCs must be stored on permanent storage media. Such media may be an extra harddisk, a tape, a CD or a high capacity floppydisk (a ZIP drive). For convenience we recommend a system consisting of an extra harddisk and a ZIP drive, which facilitates an appropriate data safety with a low cost of work.

3.2.1 Extra Harddisk on Peaknet PC

The production of data from the Peaknet system is rather large. About 100 MB per month is needed, so that a harddisk of 1500-2000 MB is necessary to cover one year. It is recommended to install an extra harddisk on thePeaknet PC (named for example E)

3.2.2 ZIP drive

A ZIP drive is a high capacity floppydisk with a capacity of at least 100 MB per disk. The ZIPdrive may be connected to a serial port on the PC or directly inside the PC, and named drive F. It may be advisable to connect it onto the serial port so that the ZIP drive may be moved between different PCs.

3.3 Procedure for electronic storage

The following procedure should facilitate a good data safety:

Make the same data directories for Peaknet data on the backup harddisk of the PC, i.e. 9901/SO2_0201, 9901/SO2_0202 etc.

For good data safety it is important to copy the Peaknet data directory on the C harddisk to the same directory on the backup harddisk (E) each day.

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At the end of the month the whole directory for that month (i.e. 9901 etc.) is copied to the ZIP drive. Make sure that all the subdirectories is copied to the ZIP drive. The ZIP drive is labeled with month and year and stored in a safe place.

At the end of the year the data are thus stored on 12 separate ZIP drives. The backup harddisk of the PC (E) contains also the 12 monthly catalogues. Check that the monthly ZIP drives contains all the data on the backup harddisk and store the ZIP drives in a safe place.

The data on the backup harddisk may now be deleted, and the backup harddisk is now free to be used for the data created in the future year.

Appendix D

Equipment needs at CEHM

EIMP

Environmental Information
and Monitoring Programme
EEAA - Danida - COWI
30 Misr-Helwan Str. Maadi, Cairo, Egypt
Tel: 202 525 6442, Fax: 202 526 6447

Memo

To:	**Bjarne Sivertsen**
Copy to:	**Mohammed Fathy, Ahmed El Seoud, Joergen Simonsen, Ulla Lund, Anwar Ahmed**
From	**Oddvar Røyset**
Subject	**Equipment needs at CEHM**
Date:	**03.03.1999**

Equipment needs at CEHM of Cairo University

During my second visit at the CEHM at Cairo University I noticed some shortages of equipment. The equipment listed below, is strongly recommended for the measurements of SO2, NO2 , TSP, PM10 and dustfall measurements for the EIMP project.

Equipment type	**Priority**	**Approximate price DKK**
Vacuum pump for VOC canisters	1	max 40000
Injection device for Gas Chromatograph for VOC collected in canisters	1	not available by 03.03.99
Printer	1	ca 5 000
New PC for Ion chromatographs with mirror harddisk, ZIPdrive for backup, ethernetcard, CD write and read, Windows95/98	1	ca 15 000
Vacuum pump for filtration device for dustfall measurements	1	not available by 03.03.99
Upgrade of Peaknet software from 4.0 to version 5.1.	1	not available by 03.03.99

Comments

Vacuum pump for VOC canisters

A vacuum pump is needed for the evacuation of VOC canisters. The pump must be of a special quality - oil free pump (to avoid hydrocarbon contamination of canisters by oil from the pump).

A suitable pump is the type
Vacuubrand MD 4 Vario
Vacuubrand MD 4C Vario

Injection device for Gas Chromatograph for VOC collected in canisters

The Gas Chromatograph used for VOC analysis of VOC canister samples must have an injection device appropriate for injection of gas samples. The detailed specifications of this is not clear at 03.03.99.

Printer

There is a strong need for a printer which should be attached to Ahmed Solimans PC, or the PC where the data for the analysis are stored in the excel worksheet laboratory database. For quality control the graphs for the data achieved for the different stations should be plotted on a weekly basis in order to quickly sort out possible problems.
A laserprinter or equivalent is recommended.

PC for ion chromatographs

The PC on the ion chromatograph is old and do not have facilities for proper data security routines. I recommend strongly to get a new PC for this purpose. The new PC for Ion chromatographs should contain:
17" monitor
CD-drive with read and write
Harddisk of at least 2 GB
Extra harddisk of at least 2 GB (mirror harddisk)
ZIPdrive for backup
Ethernetcard for communication with ion chromatographs
Windows95/98

With the use of a mirror harddisk and a ZIP drive it is possible to develop good routines for data storage and security.

Vacuum pump for filtration device for dustfall measurements

The laboratory wishes to get a small lowcost vacuumpump for the filtration device used for dustfall samples.

Upgrade of Peaknet software

The current version of the Peaknet chromatography software used at the CEHM laboratory is v.4.0. It is recommended to upgrade to the latest version, v. 5.1., as this version have many new useful features.

Appendix E

VOC method

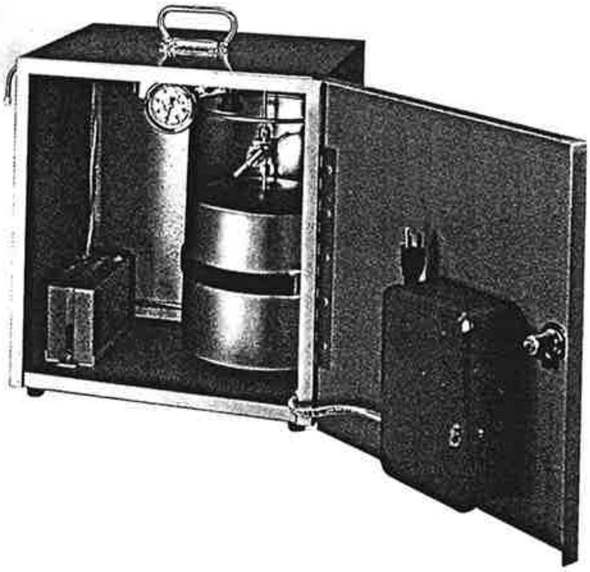
Instrumentation at CEHM

HP 6890 Series Gaschromatograph.
HP Purge and trap Concentrator for VOCs in water samples.
N2 generator for generating GC carrier gas.
Thermo Environmental Instruments mod 640 VOC steel canister sampler.

Thermo Environmental Instruments Inc.

VOLATILE ORGANIC CANISTER SAMPLER

MODEL 640



- **Twenty-four hour sample; approximately three liters under negative pressure**
- **Sampling is initiated and terminated at programmed time(s)**
- **Evacuated canister fills as pressure equalizes**
- **Completely D/C powered and portable**
- **Optional critical orifices for sample periods of 1, 3 or 8 hours**

The Thermo Environmental Instruments Inc. Volatile Organic Canister Sampler (VOCS) is a portable, automatic sampler. It offers a quick and accurate method for sampling trace-level volatile organic compounds in ambient air. The sampler uses an evacuated canister, programmable timer and a latching solenoid valve. The VOCS is convenient for sampling at hazardous waste sites, leaking underground tanks or for high-rise structures, among other uses.

The VOCS is lightweight and easily transported from one site to another. The user only has to set the timer and the unit operates automatically. The valve automatically latches open at the programmed time and air is pushed through the unit by the atmospheric pressure. The critical flow orifice is sized to provide an appropriate sample flow. At the end of the sample cycle, the valve latches closed and the sample is ready for laboratory analysis.

The SUMMA® passivated six-liter stainless-steel canister is fabricated to the highest standards of cleanliness with specially prepared interior surfaces treated by the SUMMA® polishing process. Only Nupro® stainless-steel valves are used in the T.E.I. VOCS, ensuring an organic-free sample train.

The components of the sampler are available separately for purposes of manual grab sampling or to meet unique customer needs.

Intermediate Flow Sampler Specifications

SYSTEM SPECIFICATIONS

Flow Rate:	Volumetric standard 2.1 cm^3/min (3l /24 hr).
Shelter:	Clear anodized aluminum, 2 mm (0.080 in) thick.
Flow Controller:	Critical orifice.
Canister:	Six liter, stainless steel, SUMMA® polished.
Sample Train:	Stainless-steel.
Vacuum Gauge:	0-30 in Hg.
Charging Circuit:	120 VAC/60 Hz or 220 VAC/50 Hz.
Power:	No A/C power required for sampling, completely D/C powered.
Digital Programmable Timer:	Seven-day solid-state digital programmable timer with 20 set points to permit custom scheduling sampling periods.
Assembled Dimensions:	41 cm (16 in - Height) x 38.8 cm (15 1/4 in Width) x 30.5 cm (12 in Depth) - 0.0443 m^3 (1.56 ft^3).
Shipping Dimensions:	51 cm (20 in Height) x 51 cm (20 in Width) x 63.8 cm (25 in Depth) - 0.16 m^3 (5.79 ft^3).
Net Weight/Shipping Weight:	13 kg (28.5 lb) / 17.5 kg (38.5 lb).

OPTIONS

Critical Orifice:	Fixed flow rate orifices available: 2.1 cm^3/min (24-hour sample period); 6.3 cm^3/min (8-hour sample period); 16.7 cm^3/min (3-hour sample period); 50.0 cm^3/min (1-hour sample period)

Thermo Environmental Instruments Inc.

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NILU

Norwegian Institute for Air Research (NILU)
P.O. Box 100, N-2027 Kjeller – Norway

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Environmental Information and Monitoring Programme (EIMP).
Air Quality Monitoring Component

Mission 11 Report

PROJECT LEADER

Bjarne Sivertsen

NILU PROJECT NO.

AUTHOR(S)

Oddvar Røyset and Bjarne Sivertsen

CLASSIFICATION *

A

CONTRACT REF.

REPORT PREPARED FOR:
COWI/EIMP
EEAA Building, 30 Misr Helwan Street
Maadi, Cairo, Egypt

ABSTRACT
The eleventh mission to Egypt on the DANIDA EIMP programme included the final training in handling and analyses of inorganic air quality samples. QA/QC procedures, manuals and data bases were introduced and discussed. Training was a major part of the mission.

NORWEGIAN TITLE

Overvåkingsprogram for luftkvalitet i Egypt

KEYWORDS

Air Quality	Chemical analyses	Training

ABSTRACT (in Norwegian)

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B *Restricted distribution*
C *Classified (not to be distributed)*