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DANIDA

Environmental Information and Monitoring Programme (EIMP)

Air Quality Monitoring Component

Mission 10 Report

EIMP Environmental Information and Monitoring Programme

NILU Norwegian Institute for Air Research

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

Mission 10 Report

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1 Introduction

The 10th mission to Egypt covered the period 18 October to 5 November 1998. The mission was undertaken by senior scientist Oddvar Røyset in co-operation with Bjarne Sivertsen.

The eight mission to Egypt covered the period February -May 1998. The mission was undertaken by Leif Marsteen (3 February to 12 May 1998) and Bjarne Sivertsen (16 March to 15 May 1998).

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
    On the job training at CEHM and Reference laboratory
QA/QC
    Develop SOPs to be used in the EIMP Air Quality Manual
Reference laboratory
    Develop QA standards to be used at CEHM

The work undertaken in this mission 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. The 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.

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

The main purpose of the mission was on the job training of the staff at Centre for Environmental Hazards Mitigation (CEHM) at the Cairo University, Giza, as well as from the Reflab water from Ain Shams University. The training mainly focused on the use of sampling equipment for SO_2, NO_2, TSP, PM_{10}, lead, as well as a method for the collection of dustfall using dust buckets.

The training also included two persons from the Reflab water from the Ain Shams University.

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

19.10.1998. Visit at CEHM at Cairo university

The first days visit included presentations for the staff of the planned training programme, as well as a tour through the laboratory. Different topics were briefly discussed and the following remarks were made:

The ion chromatographs were not equipped with autosamplers. This is necessary for the routine analysis of samples for the EIMP project. When the project is running as planned about 200 samples has to be analysed each week. It is not possible to analyse more than about 4 samples per hour. To carry out this number of samples it is necessary to run the instrument during the evening and nights. This can only be achieved by the use of an autosampler (else the operators have to work overtime during the evening many days a week). The cost of an autosampler is about US $ 10000 -12000. It is recommended to buy one autosampler in the beginning and install it on the DX 100 IC instrument.

he laboratory also lacked dispensers for the extraction solutions of the filters. At least 2 of 10 ml dispensers is needed. Also adjustable pipettes were lacking. One with a volume of 2-10 ml is recommended. This is lowcost equipment which can be purchased immediately.

The laboratory had equipment for pressurised air, but this equipment was not yet in operation. This is recommended for the NO_2 samplers (for extraction and drying).

The balance used for the weighing of highvolume-filters lacked a stand for the filter. It is recommended to make a U-shaped stand to place on the balance, where the filters may rest during weighing.

It is also recommended to measure the relative humidity and the temperature of the weighing room daily. The results for this should be documented in a separate logbook for the weighing room.

The highvolume filters used were of quarts and were reported to be very brittle. It is recommended to try glass fibre filters, as this is more robust.

20.10.98. CEHM Cairo University

Started training of the staff: The background of the EIMP project was presented. A lecture was given on the manual SO_2 using a filterpack. Training was given on mounting the NILU filterpack. The impregnation and extraction solutions for the filters were prepared. Training was given on impregnation of filters and mounting the filters into the filterpack and labelling the filterpack properly.

21.10.98. CEHM Cairo University

The staff from Ain Shams University arrived and they were given a lecture on the theory of the SO_2 method. Continued training on the SO_2 method. Unpacked exposed filters from 8-13.10. from Nasr City, put them into extraction tubes, extracted them and mixed. The site form were lacking. Analysis of the extracts by IC was planned to perform, but the IC suddenly got a problem with the software which had to be solved by service engineers from Dionex. The IC analysis were postponed.

Started training on the NO_2 method. A lecture on the theory was given. The impregnation solution was prepared and a set of samplers was impregnated. A new procedure for drying the samplers were tried out as they did not have vacuum desiccator equipment as recommended in the procedure. The new procedure included drying in a laboratory oven at 50 °C overnight. The need for a 2-10 ml adjustable pipette for extraction of filters was repeated.

22.10.98. CEHM Cairo University

Further training on the use of SO_2 and NO_2 sampling equipment. The new drying procedure for NO_2 tubes proved to work satisfactory. It was therefor decided that the tubes could be dried in a laboratory oven overnight instead of using vacuum drying as recommended by the original procedure.

25.10.98. CEHM Cairo University

Lecture on passive sampling theory. Trained staff on the impregnation, and mounting of passive samplers for SO_2 and NO_2.

Demonstrated the use of various report templates made for SO_2, NO_2, TSP,

PM_{10}, passive sampling (NO_2, SO_2) and lead. Detailed training given on the use of the templates for SO_2 and NO_2.

26.-27. 10.98. CEHM Cairo University

Chemical analysis of prepared samples for NO_2 and SO_2.

26- 27.98. EIMP office

Development of SOP for the dustfall method by Oddvar Røyset.

28.10.98. CEHM Cairo University

Training on the evaluation of results of the SO_2 and NO_2 samples. The laboratory had already finished the analysis for first week at the Nasr City site in Cairo. The chromatograms were evaluated together with dr. Ahmed Soliman Abd Ellah. The analysis seemed to be of good quality. The calibration graphs were linear between 0 - 20 µg/ml of both SO_4 and NO_2. Also the chromatograms using the AS4A column were satisfactory. The NO_2 at a retention time of about 1.2 min, were effectively separated from the front peak (which was low). The iodide peak eluted after about 4 minutes and the signal reached baseline again after about 7 minutes. A total analysis time of 10 minutes is enough for the NO_2 application.

The results for SO_2 seemed also to be good with a good separation of SO_4 on the AS4A column. However, there was some initial problems with the SO_2 samples, as stronger gas evaluation than expected occurred. This may make the results from these first samples questionable.

The calculated air concentrations of NO_2 at Nasr City varied between 1 -100 µg NO_2/m^3. The calculated air concentrations of SO_2 at Nasr City varied between 1 -10 µg SO_2/m^3. This value is surprisingly low. Some of the results are probably wrong due to problems with electricity at the Nasr City field station as it was reported that the electricity had been away for several periods during the week the samples were exposed. There was also a problem with the tubes used for extraction, as the pressure became very high so that the stoppers were blown out. New tube types with screwcap are being investigated.

In the EIMP program dust fallout has also been included. The SOP for the determination of dustfall were therefore presented for dr. Soliman Abd Ellah, dr. Armani Taheer and Hani Nabil. Some forms for field and laboratory use were also presented.

A new adjustable 1-10 ml pipette had been purchased. Except for having a little bit thick tip, it was found useful for NO_2. It was decided to look for a thinner tip.

CEHM Cairo University

Analysis of samples for SO_2 for Nasr City and Tabbin South from the week of about 20 - 27.10. 1998.

01.11.98. CEHM Cairo University

Staff meeting .

Evaluation of the results for SO_2 and NO_2 for the week 20-27.10 of Nasr City and Tabbin South. The results for Tabbin South were between 50-170 µg SO_2/m^3, which is at the level expected, indicating that the chemical analysis is of satisfactory quality.

At Nasr City the NO_2 were between 10-100 µg NO_2/m^3. SO_2 were between 10-100 µg SO_2/m^3. These results are within a reasonable range. There was still some problems with the electricity supply at the stations which may preclude some results, but the general impression is that we are in the right concentration range.

02.10.98. EIMP office

Preparing the final version of the EIMP Air Quality Manual. Standard Operational Procedures for Wet Chemistry Analysis Methods.

03.10.98. CEHM Cairo University

Concluding visit for this mission.

The EIMP Air Quality Manual was handed over to Dr, Ahmed Soliman Abd Ellah.

Four passive samplers from Abu Zabel (duplicates of SO_2 and NO_2) were delivered to be analysed at the laboratory. An extra parallel of these should also be analysed at NILU.

Some final topics were discussed.

- The Quality program arranged by Ain Shams University.
- Time for next visit in January - February 1999. The exact time for the next visit will be decided later.

04.10.98 EIMP Office

The mission report was finalised. A draft copy was given to Bjarne Sivertsen, Mohammed Fathy, and Morten C Andersen.

The Quality program which should be performed by Ain Shams University was discussed with Ulla Lund, who should relay the necessary messages to the responsible persons at Ain Shams.

Concluding remarks

The general experience after about 3 weeks, is that the laboratory have got good experience with the equipment needed for the analysis of SO_2 and NO_2 for both active and passive sampling. The staff is very interested in the topic of air analysis and have during the short training period gained experience with all parts of the equipment and procedures needed for the treatment of the samples. The ion chromatographic equipment is also working well, although the need for a autosampler is strong. The laboratory had already worked for about one year with TSP measurements at one site. There was a problems with the balance which had a too small weighing chamber to fit the highvolume filters. A balance with a larger weighing chamber is recommended.
Some shortages and problems still remains to be solved:

The Whatman 40 filters have long delivery times, NILU will try sending a small amount of filters while waiting.

The equipment for NO_2 free air was still lacking. This is recommended for the NO_2 method.

For the dustfall procedure, a filtration equipment is needed.

For the lead procedure, an ASTM method was used for the digestion and preparation of the filters. (Digestion prepared in open beakers on a hotplate). It was recommended not to change this procedure. It is recommended that a copy of the ASTM method is added to The EIMP Air Quality Manual or to the laboratories general Quality Manual. The methods for lead used has the references EPA (40 CFR50) for the digestion and EPA (10CFR50) for the AAS.

For SO_2 analysis the laboratory has earlier used standards prepared as sulphate (SO_4). It is recommended that the laboratory continues using standards prepared as sulphate and not as sulphur. The procedures in the EIMP Air Quality Manual has been adjusted to take this into account.

7 F. QA/QC

7.1 SOPs

Training have been performed for 6 methods where SOPs have been developed. This includes the 6 methods given below:

Parameter	*Procedure name*
SO_2	Procedure for sampling and analysis of SO_2 in air by use of a filterpack sampler.
NO_2	Procedure for sampling and analysis of NO_2 in air. Iodide absorption method.
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.
TSP, PM_{10}	Procedure for sampling and analysis of suspended particulates in air by the use of a highvolume sampler.
Dustfall	Procedure for sampling and analysis of dust fallout from the air.
QA-QC	EIMP Air Quality QA-QC-procedures.

7.2 QA/QC samples

The level of quality control needed was discussed with Ulla Lund, and it was decided that two quality control samples was needed. One should contain of 10.00 and the other 1.00 µg/ml of both $SO_4^{=}$ and NO_2^{-}. On the basis of these samples , X- and R-charts should be prepared by the laboratory. The QC-samples should be prepared by the Ain Shams University, and delivered to CEHM of the Cairo University to control the ion chromatographic analysis of sulphate and nitrite. Details about the preparation and handling of the QC-samples is described in a separate procedure “EIMP Air Quality QA-QC-procedures”. The 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.

8 G. Monitoring

No activity during this mission.

9 H. Reference laboratory

Training of 2 person from the Ain Shams University were performed in parallell with the training of the staff at CEHM. Procedures for the preparation of reference solutions of sulphate and nitrite will be handed over by Dr. Ulla Lund.

10 I. Component Co-ordination

No activity during this mission.

Appendix A

People and colleagues

Appendix A. People and colleagues

The following persons participated in the training program

Name	*Location*
Dr. Ahmed Soliman Abd Ellah, laboratory manager	CEHM
Dr Amany Taher, ass. laboratory manager	CEHM
Hany Nabil	CEHM
Dr. Gehad Genidy	CEHM
Mohammed Abd El Maugood	CEHM
Shireen Ali	CEHM
Kamla Moustafa	CEHM
Moustafa Morad	CEHM
Mona Moneer	Ain Shams University
Wagdi Mahmoud Khedr	Ain Shams University

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

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Appendix B

Training

B1 Preliminary time schedule
B2 Lectures given

B1 Preliminary time schedule

Time schedule for Oddvar Røyset, NILU, Norway, chemical analysis expert.
On the job training of chemical laboratory staff at CEHM, Cairo university and Reflab water, Ain Shams university
Preliminary program for training

Sunday	18.10.	Arrival Cairo
Monday	19.10.	Visit Cairo University
Tuesday	20.10.	SO_2 method , theory and practical exercises
Wednesday	21.10.	SO_2 method , practical exercises, chemical analyses by IC
Thursday	22.10.	NO_2 method, theory and practical exercises
Friday	23.10.	Free
Saturday	24.10.	Free
Sunday	25.10.	NO_2 method, practical exercises, chemical analyses by IC
Monday	26.10.	Passive sampling for SO_2 and NO_2, theory, practical exercises
Tuesday	27.10.	Passive sampling for SO_2 and NO_2, practical exercises, analysis by IC
Wednesday	28.10.	SO_2 practical exercises and analysis by IC, calculation and data storage spreadsheets
Thursday	29.10.	NO_2, practical exercises and analysis by IC, calculation and data storage spreadsheets
Friday	30.10.	Free
Saturday	31.10.	Free
Sunday	1.11	If desired we go through the Highvolume sampler method. Weighing . Lead analysis.
Monday	2.11	Practical exercises and summing up problems in the SO_2 method
Tuesday	3.11	Practical exercises and summing up problems in the NO_2 method
Wednesday	4.11	Closure
Thursday	5.11.	Departure back to Norwa

B2 Lectures given

SO_2 in urban air

1. Introduction

3 - 300 µg SO_2/m^3

2. Principle

$KOH + H_2O + SO_2 => SO_3^{=}$
$SO_3^{=} + H_2O_2 => SO_4^{=}$
$SO_4^{=}$ is determined by ion chromatography

Few interferences

SO_2 in urban air

Sampling

3.1 Air sampler

NILU FK Sequential sampler
7 samples are exposed simultaneously

3.2 Filters

Whatman 40 filters

3.3 Filterholder

NILU inline filterholder

3.4 Cleaning of filters

Usually not necessary for urban air

SO_2 in urban air - sampling

3.5 Impregnation of filters

Impregnation solution
1 M KOH in methanol

Impregnation of filters
300 µl per filter

Control of impregnated filters
Check that blanks are acceptable low

Storage of impregnated filters
Caution: Filters pick up SO_2
Plastic bags with zipclosure
Dessicator

SO_2 in urban air - sampling

3.6 Mounting and dismounting of filter packs

Assembly and Dismounting
Filterholder tool

3.7 Extraction of exposed filters

10 ml 0.3 % H_2O_2
15 ml single use plastic tubes with stoppers

3.8 Field blank samples

marked with red labels
one per week at some stations

SO_2 in urban air

4. Chemical analysis

4.1 Ion chromatography

Dionex with autosampler
AS4A or AS9-SC column
ASRS suppressor

4.2 Calibration

4.2.1 Stock solutions
1000 µg SO_4-S/ml
4.2.2 Working standards
0 - 10 µg SO_4-S/ml
4.2.3 Calibration graphs
Area integration of peaks

4.3 Quality assurance

Field blanks
Caution : Units of SO_4 may confuse:
µg SO_4-S/ml
µg $SO_4^{=}$/ml

SO_2 in urban air

5. Calculations

5.1 Calculating the air concentration

$$C(SO_2 - S) = \frac{a \cdot v}{Av}$$

Caution : Correct unit for SO_2

$$C(SO_2) = C(SO_2 - S) \bullet 2$$

6. Reports

Caution :Correct units for SO_2

Passive sampler for SO_2/NO_2

Overview of the IVL-type passive samplers described in this method.

Gas	Impregnation solution	Air volume 30 days exposure	Maximum concentration 30 days exposure
SO_2	KOH	0.96 m3	1600 µg SO_2/m^3
NO_2	NaI + NaOH	0.83 m3	1100 µg NO_2/m^3

The passive sampler used is developed by Martin Ferm at the IVL-institute in Sweden

Passive sampler for SO_2/NO_2

NO_2Iodide absorbent
SO_2KOH absorbent

Impregnation the same as for the active NO_2 and SO_2 methods!

Extraction of the absorbents (impregnated filters) the same as for the active NO_2 and SO_2 methods

Chemical analysis the same as for for the active NO_2 and SO_2 methods!
I.e. ion chromatography

Concentration range for exposure times of 2 - 4 weeks the same as for the active NO_2 and SO_2 methods!
I.e. 1 -10 µg/ml in an extract of 5 ml.

The uptake of gases by the passive sampler depends of diffusion theory of gases.
Diffusion theory is a bit complicated,
But the calculations turns out simple in the end.

Passive sampler for SO_2/NO_2

Filters

Absorbent filterWhatman 40 24 mm
PrefilterMillipore FALP 1 µm 24 mm

Impregnation

50 µl of impregnation solution to each filter

Storage of impregnated samplers

Must be closed to air!
Use small plastic vials which are tightly capped

Extraction

5.0 ml per filter
Use 10 -15 ml plastic tubes

Analysis

Ion chromatography for both SO_2 and NO_2

Passive sampler theory

Amount of gas collected on a passive sampler

$$x = t \cdot C \cdot D \cdot \frac{A}{L}$$

The concentration is expressed by

$$C = \frac{x \cdot L}{t \cdot D \cdot A}$$

where

C	concentration of the sampled gas in µg/cm^3
x	amount of gass collected in µg (concentration in extraction solution * extraction volume)
t	time of exposure in seconds
D	diffusion coeffecient of the gas in cm^2 s^{-1}
A	total area of the sampler in cm^2
L	length of diffusion path of the sampler in cm

Passive sampler theory

Altering the equation for the passive sampler to achieve the uptake rate

$$C = \frac{x \cdot L}{t \cdot D \cdot A} = \frac{x}{tD\frac{A}{L}} = \frac{x}{t \cdot \frac{D}{\frac{L}{A}}}$$

The uptak rate U contains all the constants!

$$U = \frac{D}{\frac{L}{A}} =$$

The unit depends on the dimensions of the constants, either

cm^3/sec
m^3/day

Passive sampler theory

Using the uptake rate in the passive sampler equation, the equation becomes very simple

$$C = \frac{x}{t \cdot U}$$

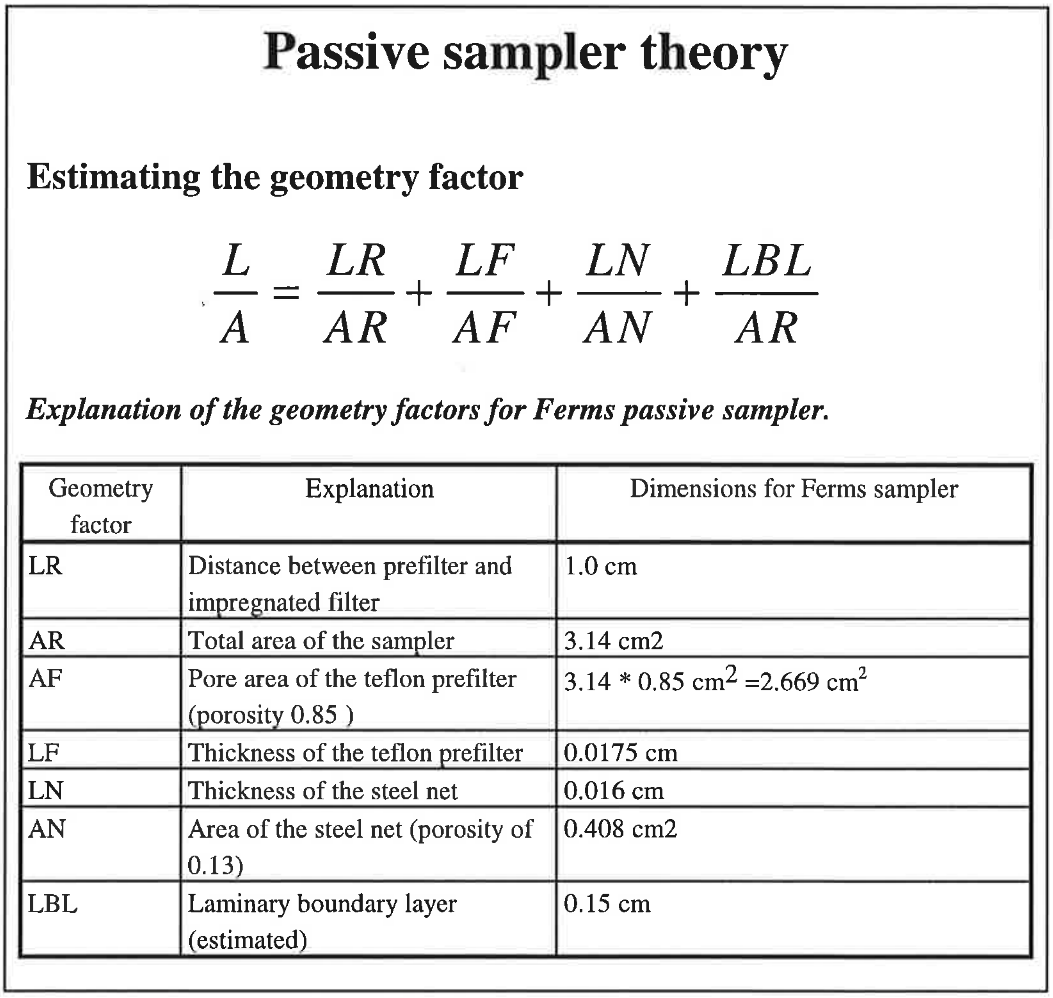
Most convenient to use the unit m^3/day(24h) for U

Equation for NO_2 :U=0.0323 m^3/day

$$C(NO_2) = \frac{x}{t \cdot 0.0323}$$

Equation for SO_2U=0.0277 m^3/day

$$C(SO_2) = \frac{x}{t \cdot 0.0277}$$



Passive sampler theory

Estimating the geometry factor

$$\frac{L}{A} = \frac{LR}{AR} + \frac{LF}{AF} + \frac{LN}{AN} + \frac{LBL}{AR}$$

Explanation of the geometry factors for Ferms passive sampler.

Geometry factor	Explanation	Dimensions for Ferms sampler
LR	Distance between prefilter and impregnated filter	1.0 cm
AR	Total area of the sampler	3.14 cm2
AF	Pore area of the teflon prefilter (porosity 0.85)	3.14 * 0.85 cm^2 =2.669 cm^2
LF	Thickness of the teflon prefilter	0.0175 cm
LN	Thickness of the steel net	0.016 cm
AN	Area of the steel net (porosity of 0.13)	0.408 cm2
LBL	Laminary boundary layer (estimated)	0.15 cm

Passive sampler theory

$$\frac{L}{A} = \frac{LR}{AR} + \frac{LF}{AF} + \frac{LN}{AN} + \frac{LBL}{AR}$$

Estimation of the total geometry factor for Ferms passive sampler

Factor	Input values	Geometry factor cm^{-1}
L_R / A_R	1.0 /3.14	0.318
L_F / A_F	0.0175 /2.669	0.00785
L_N / A_N	0.016/0.408	0.039
L_{BL} / A_R	0.15 / 3.14	0.0477
SUM L/A		**0.412 cm^{-1}**
SUM L/A		**41.2 m^{-1}**

$D(NO_2) = 0.0000154\ cm^2/sec$
$D(NO_2) = 1.33\ m^2/day$

$$U = \frac{D}{L} = \frac{1.33 m^2/day}{41.2} = 0.0322\ m^3/day$$

NO_2 in urban air

1. Introduction

Urban air 1 -300 µg NO_2/m^3

2. Principle

The iodide absorption method

$$I^- + NO_2 => NO_2^- + I_2$$

NO_2 determined with Ion hromatography

Sampling efficiency and interferences
Efficiency is good
Interferences HNO_2 + PAN

3. Sampling

3.1 Sampling equipment

NILU FK sequential sampler
7 samplers may be exposed

3.3 Preparation of the samplers

1. Impregnation solution
   NaI/NaOH/methanol
2. Cleaning of exposed samplers
3. Source of NO_2 free air
4. Impregnation and preparation of the glass sinters

3.5 Extraction of exposed glass filter samplers

Extraction solution 1 mM TEA

3.6 Field blanks

4. Chemical analysis

4.1 Ion chromatography equipment

Dionex IC with autosampler
Column AS9-SC
ASRS supressor

4.2 Calibration

1. Stock solution
   1000 µg NO_2^-/ml

2. Working standards
   0 - 10 µg NO_2^-/ml

3. Calibration graphs and integration
   Area integration
   Linear fit

4.3 Quality assurance

Field blanks

5. Calculations

5.1 Calculation of the air concentration

$$C(NO_2) = \frac{a \cdot v}{Av}$$

Reports

Appendix C

EIMP Air Quality Manual Standard Operational Procedures for Wet Chemistry Analysis Methods

Contents

Parameter	*Procedure name*
	Forms and Excel templates
SO_2	Procedures for sampling and analysis of SO_2 in air by use of a filterpack sampler
NO_2	Procedure for sampling and analysis of NO_2 in air. Iodide absorbtion method
Passive SO_2 and NO_2	Procedure for sampling and analysis of NO_2 and SO_2 in air by use of passive samplers
TSP, PM_{10}, Lead	Procedure for sampling and analysis of suspended particulates in air by the use of a high volume sampler
Dustfall	Procedure for sampling and analysis of dustdallout from the air
QA/QC	EIMP Air Quality QA/QC-procedures

EIMP
Site form

Year	FIELD STATION FORM Sequential FK-Sampler	Amount of tubes left
	Iodide method - NO_2	

Station code	Station name:	Observer

From		To		Hours	Channel	Air volume	REMARKS
Date	Time	Date	Time	exposed	No.	m^3	
					1		
					2		
					3		
					4		
					5		
					6		
					7		
					8		

READING OF THE GAS METER AT START AND STOP

	Date	Time	Total	Reading of gas meter - m^3
	YY/MM/DD	Hour:min.	number	NB ! 3 decimals behind comma
START			of hours	⊥⊥⊥⊥⊥⊥⊥,⊥⊥⊥⊥
STOP			exposed	⊥⊥⊥⊥⊥⊥⊥,⊥⊥⊥⊥
				Total air volume: ,

Instruction for reporting

Use ballpoint pen. If the remark column is too small, write on the backside with column x) as reference for the date.
When changes are done, cross over the old value and write a new value beside.

Samples received laboratory		REMARKS FOR THE LABORATORY	Proof reading	
Date	Sign		Date	Sign

EIMP
Site form

Year	**FIELD STATION FORM** **Sequential FK-Sampler** **Filterpack method - SO_2**	

Station code	**Station name**	**Observer**

From		**To**		**Ch.**	**Air flow L/h**		**Air volume**	**REMARKS**	
Date	**Time**	**Date**	**Time**	**No**	**Start**	**Stop**	**m^3**		**X**
				1					1
				2					2
				3					3
				4					4
				5					5
				6					6
				7					7
				8					8

Instruction for reporting

Use ballpoint pen.
If the remark column is too small, write on the backside with column x) as reference for the date.
When changes are done, cross over the old value and write a new value beside.

Samples received laboratory		**REMARKS FOR THE LAB. -- ONLY LAB. FILLS IN HERE**	**Proof read**	
Date	**Sign.**		**Date**	**Sign.**

EIMP
Site form

Year	**FIELD STATION FORM**
	HIGHVOLUME SAMPLER

Station code:	**Station name:**	**Observer:**

From		**To**		**Flow meter**		**Air**	**Remarks**	
Date	**Time**	**Date**	**Time**	**reading, m^3**		**volume**		
				Start	**Stop**	**m^3**		**X**
								1
								2
								3
								4
								5
								6
								7
								8

Instruction for reporting

Use ballpoint pen.
If the remark column is too small, write on the backside with column x) as reference for the date.
When changes are done, cross over the old value and write a new value beside.

Samples received laboratory		**REMARKS FOR THE LAB. -- ONLY LAB. FILLS IN HERE**	**Proof read**	
Date	**Sign.**		**Date**	**Sign.**

EIMP
Site form

Year	**FIELD STATION FORM**
	Passive samplers - NO_2/SO_2

Station code:	**Station name:**	**Observer:**

From		**To**		**Sampler type**	**Site location Position**	**Sampler no**	**Remarks**	**Ref**
Date	**Time**	**Date**	**Time**	NO_2/SO_2				x
								1
								2
								3
								4
								5
								6
								7
								8
								9
								10
								11
								12

Instruction for reporting

Use ballpoint pen.
If the remark column is too small, write on the backside with column x) as reference for the date.
When changes are done, cross over the old value and write a new value beside.

Samples received laboratory		REMARKS FOR THE LABORATORY	Proof reading	
Date	Sign		Date	Sign

WORKSHEET FOR WEIGTH OF HIGHVOLUME FILTERS

Project no:__________________ Field station name:______________________ Filter type:____________________

Weight name_________________________

Unexposed filters					Exposed filters			DUST	Remarks
Filter no.	Date exposed	Date weighed	Sign	Weight unexposed - mg	Date weighed	Sign	Weight exposed - mg	Exposed - unexposed - µg	

WORKSHEET FORM - WEIGTH OF WATERINSOLUBLE DUSTFALL

Project no:__________________ **Field station name:**_______________________ **Filter type:**____________________

Weight name__________________________

Unexposed filters				Exposed filters			Difference	Remarks
Filter no.	Date weighed	Sign	Weight unexposed - mg	Date weighed	Sign	Weight exposed - mg	Exposed - unexposed - mg	

WORKSHEET FORM - WEIGHT OF WATERSOLUBLE DUSTFALL

Project no:________________ **Field station name:**______________________________________

Weight name_______________________

Empty beaker				Beaker after evaporation			Difference	Total volume	Remarks
Beaker no.	Date weighed	Sign	Weight empty mg	Date weighed	Sign	Weight after mg	After - empty mg	of solution ml	

LABORATORY WORKSHEET
ION CHROMATOGRAPHY

Seq nr	Sample identification					NO_2^- µg NO_2^- /ml	$SO_4^=$ µg $SO_4^=$/ml	Remarks
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								
31								
32								
33								
34								
35								
36								
37								
38								
39								
40								

Analysis date:____________ Sign:___________

LABORATORY WORKSHEET
ION CHROMATOGRAPHY

Seq nr	Sample identification					NO_2^- µg NO_2^- /ml	$SO_4^=$ µg $SO_4^=$/ml	Remarks

Analysis date:__________ Sign:__________

SO2 Report

DATA REPORTING FORM FOR SO_2										
Field station name		Date from	Date to	Time from	Time to		Air volume m^3	Extract conc $\mu g\ SO_4/ml$	Extract volume ml	Air conc SO_2 $\mu g\ SO_2/m^3$
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34
							2	1	10	3.34

NO2 report

REPORTING FORM FOR NO2										
Field station name		Date from	Date to	Time from	Time to		Air volume m^3	Extract conc	Extract volume	Air conc NO_2
								µg NO_2/ml	ml	µg NO_2/m^3
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00
							2	1	50	25.00

TSP report

DATA REPORTING FORM FOR TSP									
Field station		Date	Date	Time	Time		Air volume	Weigth	Air conc
name		from	to	from	to		m^3	TSP	TSP
								µg	µg TSP/m^3
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44

PM10 report

DATA REPORTING FORM FOR PM10									
Field station name		Date from	Date to	Time from	Time to		Air volume m^3	Weigth PM10 µg	Air conc PM10 µg $PM10/m^3$
								10000	#VALUE!
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44
							1440	10000	69.44

Pb report

DATA REPORTING FORM FOR LEAD (Pb)											
Field station		Date	Date	Time	Time		Air volume	Extract	Extract	Filter	Air conc
name		from	to	from	to		m^3	conc	volume	part	Pb
								µg Pb/ml	ml		µg Pb/m3
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08
							1440	0.3	200	0.5	0.08

Passive SO2

Form for reporting SO$_2$ by the use of a passive sampler									
Sample identification	Date	Date	Time	Time	Days	Extraction	Extraction	Uptake	SO$_2$
Field station name						conc	volume	rate	
	from	to	from	to		µg SO$_4$/ml	ml	m^3/day	µg SO$_2$/m^3
					20	2.0	5.0	0.0277	12.0
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!
							5.0	0.0277	#DIV/0!

Passive NO2

Form for reporting NO2 by the use of a passive sampler									
Sample identification	Date	Date	Time	Time	Days	Extraction	Extraction	Uptake	NO_2
Field station name						conc	volume	rate	
	from	to	from	to		µg NO_2/ml	ml	m^3/day	µg NO_2/m^3
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!
							5.0	0.0323	#DIV/0!

Passive SO2 worksheet

Form for reporting SO_2 by the use of a passive sampler

Sample identification	Date	Date	Time	Time	Days	Extraction	Extraction	Uptake	SO_2
Field station name						conc	volume	rate	
	from	to	from	to		µg SO_4/ml	ml	m^3/day	µg SO_2/m^3
					20	2.0	5.0	0.0277	12.0
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	
							5.0	0.0277	

Passive NO2 worksheet

Form for reporting NO2 by the use of a passive sampler									
Sample identification	Date	Date	Time	Time	Days	Extraction	Extraction	Uptake	NO_2
Field station name						conc	volume	rate	
	from	to	from	to		µg NO_2/ml	ml	m^2/day	µg NO_2/m^3
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	
							5.0	0.0323	

Procedure for sampling and analysis of SO_2 in air by use of a filterpack sampler

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Procedure for sampling and analysis of SO_2 in air by use of a filterpack sampler

1. Introduction

This procedure describes a method for sampling and chemical analysis of SO_2 in air by use of a 2 stage filterpack sampler. The method is applicable for ambient and urban air in the range 1 - 300 µg SO_2 m^{-3} for sampling periods of 1 day (24h).

2. Principle

The filterpack consist of 2 filters coupled in series. The first filter is an untreated paper filter which collects particles (and particulate sulphate). The second filter is impregnated with KOH and collects SO_2 .

The impregnated filter is extracted with a dilute solution of hydrogen peroxide which transforms the collected SO_2 to sulphate (SO_4^{2-}). This is determined with ion chromatography using a standard anion separation column and an ion chromatography system from Dionex Corp. , USA.

The particulate sulphate collected on the first filter can be determined if desired by extraction of the filter by water. The extracted sulphate is determination by ion chromatography as for SO_2. In the method described in this procedure, the particulate sulphate is not determined

The absorption capacity of the filtersampler is about 10000 µg SO_2. Using the recommended air volume of 3.6 m3 per 24 h, this capacity corresponds to about 3000 µg SO_2 m^{-3}. Taking precautions not to expose the filter to more than 10% of the total capacity, the filter pack can be used with small risks for breakthrough up to 300 µg SO_2 m^{-3} for 24 h sampling periods. In situations of mixed exposure to other acid gases (HCl, HNO3 etc.) this must be taken into consideration when calculating the capacity and breakthrough risk.

3. Sampling

3.1 Air sampler

A NILU FK sequential sampler equipped for 8 filter packs is used. Normal sampling flow for this sampler is 2.5 l/min (corresponding to 0.15 m^3 pr h or 3.6 m^3 per 24h).

3.2 Filters

Both the particulate and impregnated filter is a 47 mm Whatman 40 (W40) paper filter manufactured by Whatman, UK.

The filters must regularly be checked for blank values of sulphate. The blank value should normally not exceed 5 µg SO_4 per filter.

3.3 Filterholder

The filterholder is a standard NILU filterholder with 2 stages. The front part of the filterholder is the inline type (tube in front, not open face). A 40 mm opening clamp ring is used for both filter stages.

During use the clamp ring and filter support plate (thr grid plate) is removed and cleaned each time the filter is changed. The cleaning procedure is soaking in demineaAralized water. Rinse afterwards in demineralized water and dry in a clean environment.

New cleaned clamp rings and support plates are put in when new filters are mounted into the filter holder.

3.4 Cleaning of filters

Normally the paper filters from Whatman (Whatman 40) are clean enough for sampling of SO_2 without any cleaning procedure before use. The blank value should normally not exceed 5 µg SO_4 per filter. The blank is normally monitored by the field blank filters. If the field blanks become higher than recommended in 3.8., blank values of the filters must be checked, and cleaning of filters before use must be considered.

3.5 Impregnation of filters

3.5.1 Impregnation solution

The impregnation solution consists of 1.0 M KOH and 10 % glycerol dissolved in methanol. The chemicals must be of analytical grade. KOH is used as pellets (MERCK 5033 is a suitable brand).

The solution is prepared by adding about 70 ml methanol to a 100 ml volumetric flask, add 5.6 g of KOH pellets and shake until dissolved. Add 10 ml glycerol and dilute to the mark with methanol.

This solution should be prepared freshly each time filters are to be impregnated. Keep tightly closed in the flask until used.

3.5.2 Impregnation of filters

The Whatman 40 filters are placed on suitable stands before impregnation. Suitable stands are plastic bottle caps with a diameter of about 40 mm. The filters are impregnated with 300 µl impregnation solution (corresponding to an amount of 0.3 meq KOH). The impregnation is preferably performed with a micropipette. Drip the solution onto the filter in a circular way so that the solution is equally distributed over the whole filter area. It is important that the whole filter area is wetted with impregnation solution. Let the filters dry (max. 30 min). Put the filters into plastic bags with zip closure.

During impregnation and packing of the filters, the filters should only be touched by clean forceps. If field blanks become high, it should also be considered to use protective gloves.

3.5.3 Control of impregnated filters

The blanks of impregnated filters should be monitored before use. 3 filters of each bath of impregnation are ramdomly chosen and extracted as described in section 3.7.

The batch blank value should be below 5 µg SO_4 pr filter (preferably below 1).

3.5.4 Storage of impregnated filters

Store the closed plastic bags with filters in a desiccator which are tightly closed. Mark the bag with date for impregnation. Impregnated filters should not be stored for more than 3 months before use.

3.6 Mounting and dismounting of filter packs

3.6.1 Assembly

During this operation plastic gloves should be used and the filters should only be touched with clean forceps. The filterpack should be assembled and dismounted in the laboratory only.

The first (upper) particulate filter is a Whatman 40 filter which is untreated. The second stage is the impregnated filter.

Put the filters onto the support grid plates in the filter holder, put the clamp ring on top and mount the appropriate parts of the filterholder. Close the filter pack with the closure wrench tool to the torque specified by the manufacturer to prevent leaks.

After the assembly, the filterholder must be closed with plastic stoppers in both ends. It is recommended that each filter pack should be tagged with the site code, and the date for start and stop of the exposure. The closed filterpack is placed in plastic bags with zip closure to prevent contamination.

3.6.2 Dismounting

Exposed filterpacks are opened with the filterpack wrench tool. The first particulate filter is removed. If there are no requirements for analysis of this filter, it is discarded, otherwise it must be prepared and extracted according to the specified procedure for the determination of the desired components.

The second impregnated filter is placed in a clean plastic vial with a minimum volume of 15 ml. Single use polystyrene vials of about 15 ml volume are appropriate. If the filters are not extracted immediately, the vials must be closed. The filters must be extracted within the same day. Tag the vials with the necessary identification.

3.7 Extraction of exposed filters

The extraction solution for impregnated filters is 0.3 % hydrogen peroxide.

This solution is prepared by 10 ml of 30 % H_2O_2 (Merck 30% p.a. perhydrol or equivalents) which is added to 1000 ml demineralized water. This solution should not be stored more than a month. It is recommended to check the blank value of this solution, it should not be above 0.05 μg SO_4 ml^{-1}.

The solution is placed in a laboratory dispenser which can dispense exactly 10.0 ml solution to each vial tube with exposed filters. It is recommended to check the accuracy of the dispenser using a microbalance. Weigh the dispensed amount, it must dispense exactly 10.0 (+/- 0.1) g (ml) of solution.

The tubes are closed and left for some hours. Usually small amounts of gas is formed inside the tube. It is therefore recommended to gently open the stopper of the tube to release the overpressure within a few hours. The tubes must be shaken to ensure complete extraction of the filter. This is done by turning the tube upside down at least ten times. Ensure that the stopper of the tube do not fall out while doing this. An ultrasonic bath may be used for extraction, but this may release much fibres from the filter into the solution, making clogging of transfer tubes of the ion chromatograph a problem during analysis.

The vials with filters are stored in a refrigerator until analysis. The closed tubes with the samples may be stored up to 2 months in a refrigerator, but it is advised to analyse them as soon as possible.

3.8 Field blank samples

For each station it is recommended to prepare one field blank sample once a month. The field blank sample is marked properly and shipped together with the samples to the field site, but not taken out of the plastic bag at the site. The field blank sample is returned to the laboratory and extracted and analysed as the rest of the samples. To prevent mixture with normal samples, the filter pack with the field blank sample should be tagged with a red label.

A separate log is made with the result for the field blank samples. An acceptable field blank level must be established on experience, but a first guide to a target value is 5 μg SO_4 per sample. If field blanks increases, the procedures must be examined to sort out possible contamination sources.

To keep cross contamination between stations low and thereby field blanks low, it is recommended to use the same filterpack equipment repeatedly at each station and not mix filterpack equipment from highly exposed stations with equipment from lower exposed sites.

4. Chemical analysis

4.1 Ion chromatography equipment an columns

The chemical analysis of sulphate in the extracts are performed by ion chromatography using standard conditions. This procedure is based on the use of ion chromatography equipment manufactured by Dionex Corp., USA.

The Dionex ion chromatograph is set up as recommended in the instructions from Dionex. The instrument should preferably be equipped with an autosampler for unattended analysis of series of samples.

For this application a anion separation column of the type Dionex AS4A is recommended. This is a general purpose rugged column which copes well with the matrix used. Sulphate is normally eluted after about 6 - 10 min depending on the eluent strength.

Other columns from Dionex may be used (such as AS9A, AS14 etc). These have similiar properties as AS4A. Refer to the column manuals for optimum conditions.

A normal anion supresssor system from Dionex must be used together with this column system. This may be a normal chemically based supressor or a self regenerating (an electrolytical) supressor. The latter is the simplest and most convenient in use.

Both the column and the supressor must be operated according to the instructions given in the manuals supplied with the systems from Dionex.

4.2 Calibration

4.2.1 Stock solutions based on sulphate (SO4)

Stock solutions of sulphate standards may be purchased from Dionex. These contains normally 1000 μg SO_4 ml^{-1} as sulphate (SO4).

A 1000 μg SO_4 ml^{-1} solution may be prepared as follows. Dry pro analysis quality of the salt Na_2SO_4 (Merck p.a. or equivalents) at 105 °C for at least 1 hour and cool in a desiccator. Weigh accurately 1.479 g Na_2SO_4 and transfer to a 1000 ml volumetric flask. Dilute to volume (1000 ml) with demineralised water. This solution contains 1000 μg SO_4 ml^{-1}. It must be noted that this solution is prepared as sulphate (SO4) and not as sulphur (S)! The stock solution must be stored in tightly closed PP or glass bottles. When stored cool in a refrigerator, the stock solution is stable for at least one year.

4.2.2 Stock solutions based on sulphur (S)

If the labortory expresses concentrations of standards as sulphur (S), a 1000 μg S ml^{-1} solution may be prepared as follows. Dry pro analysis quality of the salt Na_2SO_4 (Merck p.a. or equivalents) as in 4.2.1. Weigh accurately 4.439 g Na_2SO_4 and transfer to a 1000 ml volumetric flask. Dilute to volume (1000 ml) with demineralized water. This solution contains 1000 µg S ml^{-1}. This solution is prepared as sulphur (S) and not as sulphate (SO4)! The stock solution must be stored in tightly closed PP or glass bottles. When stored cool in a refrigerator, the stock solution is stable for at least one year.

4.2.3 Working standards based on sulphate (SO4)

For the preparation of calibration standards, specifications are given for sulphate based standards only. Calibration standards are prepared by dilution from the stock solution containing 1000 µg $SO_4^{=}$ ml^{-1}. To cover the normal working range of this sampling and analysis method, working standards in the range 0 - 30 µg SO_4 ml^{-1} is normally appropriate

Typical working standards of 5.0, 10.0, 20.0, 30.0 SO_4 ml^{-1} is used. When stored in tightly closed bottles in a refrigerator (4 °C), these working standards are stable for at least 2 months.

If sample extracts contains more than 30 µg SO_4 ml^{-1} they should be diluted.

4.2.4 Calibration graphs and integration

Calibration graphs should be prepared by area integration of the peaks (not peak heights). Area integration must also be used for the samples. This is recommended as the matrix used may distort peak shapes somewhat. Normally linear fit of calibration graphs is recommended in the range 0 -30 µg SO_4 ml^{-1}.

4.3 Quality assurance

The following quality control procedures must be followed:

- Regular analysis of field blanks must be performed. Actions must be taken if the field blank increases above acceptable levels.
- Always check that the correct units of S is used throughout all analysis and calculations, ie. that either the unit S, SO2 or SO4 is used. Necessary formulas for transformation between units must be established and carefully evaluated.
- Before the calculations of the concentrations of the samples, it must be checked that the calibration graph fit to the model equations is good enough (either linear or curved fit).
- All chemicals, standards etc. should be used within storage limitations.

5. Calculations

5.1 Calculating the air concentration for sulphate based standards

If the standards used are based on sulphate ($SO_4^{=}$), the concentration of SO_2 in air is calculated by:

$$Ac(SO_2) = \frac{Ec(SO_4) \cdot Ev \cdot 0.667}{Av}.$$

47
51
57

$Ac(SO_2)$	air concentration of SO_2 [$\mu g\ SO_2\ m^{-3}$]
$Ec(SO_4)$	concentration of $SO_4^=$ in extracts from filters determined by ion chromatography [$\mu g\ SO_4^=\ ml^{-1}$]
Ev	extraction volume of filters [ml] (typical 10 ml)
Av	volume of sampled air [m^3]
0.667	the conversion factor between SO_4 and SO_2

5.2 Calculating the air concentration for sulphur(S) based standards

If the standards used are based on sulphur (S), the concentration of SO_2 in air is calculated by:

$$Ac(SO_2) = \frac{Ec(S)\cdot Ev\cdot 2}{Av}.$$

where

$Ac(SO_2)$	air concentration of SO_2 [$\mu g\ SO_2\ m^{-3}$]
Ec(S)	concentration of sulphur (S) in extracts from filters determined by ion chromatography [$\mu g\ S\ ml^{-1}$]
Ev	extraction volume of filters [ml] (typical 10 ml)
Av	volume of sampled air [m^3]
2	the conversion factor between S and SO_2

6. Reports

The data are expressed in the desired unit (as explained in chapter 5). The sample identification and SO_2 results are transferred to the final data storage and retrieval system which is used for the project.

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Procedure for sampling and analysis of NO_2 in air. Iodide absorption method

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Procedure for sampling and analysis of NO_2 in air. Iodide absorption method

1. Introduction

This procedure describes a method for the determination of NO_2 in air by the use of the iodide absorbent method, using a glass sinter sampler. The method is applicable for urban and ambient air in the concentration range 1 - 300 $\mu g\ NO_2\ m^{-3}$ for sampling periods of 1 day (24h).

2. Principle

2.1 Methods for determining NO_2

Several methods, both manual and continuous, have been used for the measurement of nitrogen dioxide in ambient air. In urban air, the chemiluminesence method have replaced the manual absorption solution methods, and is introduced as an ISO standard (ISO,1985a). The chemiluminesence method for NO_2 is based on reduction to NO by a heated catalytic converter and calculation of the concentration as the difference between ($NO+NO_2$) and NO (the signal without converter). For clean air sites commercial monitors are usually not sensitive enough, and since other reducible nitrogen compounds (eg. HNO_3 and PAN) may exist in the same concentration level as NO_2, the method is not specific. However monitors with selective photolytic converters may be used if the sensitivity is adequate.

Also the liquid phase NO_2-luminol chemiluminescence reaction have been used in a commercial monitor for nitrogen dioxide at low levels (Schiff et al., 1986). This monitor has been shown to give almost interference-free values for NO_2 (eg. Gehrig and Baumann, 1993). However a small interference from ozone has been observed by Kelly et al. (1990) and Hesterberg and Neftel (1993) found nonlinearities under 1 ppb in addition to other systematical errors due to pressure variations in the inlet system and temperature changes in the reaction chamber. Since this monitor work with a liquid phase reaction, it needs more regular service than the ordinary chemiluminescent instruments.

The manual absorbing solution method based on direct Griess reaction during sampling (Saltzman method) has also been appointed an ISO standard (ISO, 1985b). This method is sensitive and more selective than the chemiluminescence method, but the colour to be measured spectrophotometrically is developed during sampling, and the measurement have to be performed immediately after sampling due to instability. This makes the method unsuitable if the exposed absorbing solution have to be transported to a chemical laboratory far from the sampling site, particular if temperature and light exposure cannot be controlled.

Other absorbing solutions have been used in which nitrogen dioxide is absorbed and transformed to nitrite (EMEP, 1977). These methods are usually not sensitive enough in background areas, and also have the problem of instability of the exposed absorption solution during transport when the temperature and sunlight cannot be controlled. There has also been considerable uncertainty about the absorption efficiency of the absorbing solutions, and to which extent this varies with concentration. The use of experimentally determined absorption efficiencies have shown to be inadequate (Fähnrich et al., 1993).

A new manual method based on absorption of nitrogen dioxide on a sodium iodide impregnated glass-sinter has been developed by Ferm and Sjödin (1993). Due to the reasons mentioned above, only the sodium iodide method has been included in this manual.

2.2 The iodide absorption method

This method (Ferm and Sjödin, 1993) is based on the same principle as the method developed by Pavlenko and Volberg (1979, 1991). This method is recommended at EMEP stations with low concentrations of NO_2 and where the analysis has to be performed in a laboratory far from the sampling site.

Ambient air with a flow rate of about 0.5 l/min is drawn through an air intake (inverted funnel) and a glass filter impregnated with sodium iodide (NaI) and sodium hydroxide (NaOH). Nitrogen dioxide is absorbed in the filter and the iodide reduces NO_2 to nitrite (NO_2^-). The hydroxide is converted to carbonate during sampling due to uptake of carbon dioxide. The nitrite formed on the glass filter is extracted with deionized water. After extraction the nitrite concentration can be determined photometrically by the Griess method (ISO, 1985b). In this method, however, the dermination of the collected nitrite is performed by ion chromatography.

This method can be used for measurement of nitrogen dioxide on a 24 h basis in ambient air within the range 0.1-10 $\mu g\ NO_2/m^3$, and also in urban air up to 300 $\mu g\ NO_2/m^3$ (air sample of 0.7 m^3). Exposed samples are stable for several weeks and can be transferred to a laboratory for chemical analysis. The extraction volume may be adjusted according to the concentration level expected. At low concentrations at background sites an extraction volume of about 5 ml has found to be appropriate. In urban air measurements the sample may be extracted with a volume of 10 ml or even larger. The large extraction volume facilitates the use of ion chromatography as measurement method. The iodide peak is reduced to an acceptable low level so that interference with the determination of nitrite is avoided.

2.2.1 Sampling efficiency and interferences

The sampling efficiency at a flowrate of 0.5 l/min and a relative humidity of 15% is higher than 98%. With a relative humidity higher than 60%, the sampling efficiency is higher than 98% even at a flowrate of 4 l/min. (Ferm pers. comm.).

Interference studies showed negligible formation of nitrate on the NaI/$NaCO_3$-substrate. Nitric oxide formation was also never observed behind the filter and no oxidation of nitrite by ozone is found. The absorption of PAN (peroxyacetyl nitrate) and the subsequent formation of nitrite on the alkaline NaI filter have been demonstrated with about 20% absorption (Ferm and Sjödin, 1993). This cause a positive interference which can be severe if the PAN-concentration is higher than the NO_2-concentration. This may happen in very remote areas, but not at most of the EMEP-sites.

3. Sampling

3.1 Sampling equipment

A NILU FK sequential sampler equipped for 8 samplers is used. The sampling flow is 0.5 l min^{-1} (corresponding to about 0.7 m^3 in 24h).

Figure 1 shows the components of a suggested sampling system. The Figure shows an automated system, a simpler set-up with manual exchange of samples may also be used. The main componets are as follows:

3.1.1 Air inlet

An inverted funnel made of PTFE teflon, polypropylene, borosilicate or polyethylene should be used in order to prevent entrance of precipitation at the sampling point.

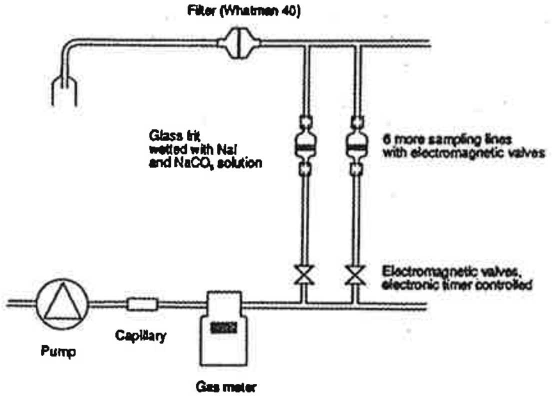

Figure 1: Sampling system for nitrogen dioxide.

3.1.2 Tubing

The sampling tube connection between the air inlet and the absorption system should be as short as possible, and made of PTFE teflon, polypropylene, borosilicate glass or polyethylene.

3.1.3 Filterholder with prefilter

A filterholder with a filter should be used in front of the absorption system in order to remove particulate matter,. The filter must be inert to NO_2. A teflon membrane filter with a pore size 1–2 µm or a Whatman 40 cellulose filter or equivalent may be used. The filterholder and the connections to the sampling line must be air-tight. The prefilter can be used for one week.

3.1.4 Absorption system

A 4 mm thick sintered glass filter 25 mm i.d. with a porosity of 40–60 µm enclosed in a glass bulb as shown in Figure 2 is used as a substrate for the impregnation. The glass bulbs should be connected to the sampling line using short pieces of silicon tubing. During transport the silicon tubing must be closed by pieces of glass, plastic rods or appropriate caps made of PP, PE or PTFE teflon.

The sinter glass filter in bulb may be purchased from NILU Products AS, 2007 Kjeller, Norway, telephone 47 63 898000, fax 47 63 898050.

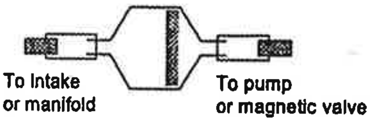

Figure 2: Sintered glass filter in a glass bulb.

3.2 Site requirements

The site requirements for nitrogen dioxide are as for sulphur dioxide with respect to regional location and point sources. As nitrogen dioxide is taken up by vegetation, the air intake should not be sheltered by vegetation, but be freely exposed. The air intake should be 2–5 m above the ground. For urban air measurements the sampling site must be placed so that it is representative for the urban area or street canyon to be investigated. The pump and sampling equipment should be placed in a room where the temperature is controlled at 25 °C ± 5 °C.

3.3 Preparation of the absorption system

3.3.1 Impregnation solution

The impregnation solution consist of 7.9 g NaI and 0.88 g NaOH in 100 ml methanol or ethanol. The solution should be made fresh for every new batch of filters to be prepared, due to possible uptake of nitrogen dioxide from laboratory air.

3.3.2 Cleaning of exposed samplers

Used samplers must be carefully cleaned before use. They may be left overnight soaked in deionized water to release old marking labels etc. After that the samplers are then cleaned by flushing deionized water into the back end of the tube (in the opposite direction of the sampling flow). At 0.5 - 1 liter of deionized water should be flushed through each tube to ensure a proper cleaning. After cleaning the samplers should be dried in a laboratory oven at 100 - 105 °C.

3.3.3 Source of NO_2 free air

A source of NO_2 free air should be available in the laboratory. This should be produced from a central pressurized air distribution system in the laboratory, or by using a small air pump. The air should be cleaned for NO_2 by placing a NaI impregnated filter sampler in the line. A new impregnated sampler should be placed in the line each day the system is used to make sure that the capacity of this rinsing sampler is not overloaded.

3.3.4 Impregnation and preparation of the glass sinters

500 µl impregnation solution is added to carefully cleaned glass sinters through the back end of the sampler (it is easier to wet the complete surface of the glass sinter by adding from back - the short end- of the sampler.

The sinters may be dried with a flow of NO_2-free air (as specified in 3.3.3). Blow the air in from the short end (back end) while holding it upwards. Use a low flow so that sputtering of the methanol solvent do not occur (only low bubbling).

The manual drying by compressed air is a time consuming process. A recommended alternative is to place the samplers in a large vacuum desiccator. Evacuate the desiccator for about 2 hours at 50 - 60°C. This will evaporate the solvent completely.

For urban air measurements it may be sufficient that the tubes may be dried by being left open to air. Place the the samplers in a suitable stand with the short end up in a vertical position. Leave the samplers with both ends open to air for about 30 min at room temperature in the laboratory.

After the drying the glass bulbs should be closed in both ends as soon as possible. Well closed and protected impregnated sinters can be stored for several weeks before, as well as after sampling, preferably in a cool place.

3.4 Sampling procedure

Assemble the sampling equipment at the site as shown in Figure 1, and make sure that the glass bulbs are coded with site name and date. Control that the equipment is leak free. Check the initial flowrate with the gas meter. It should be about 0.5 l/min. Record the gas meter reading at the start and again at the stop. For a 24-hour sample the total air volume should be about 0.72 m^3. Using the NILU sequential sampler taking daily samples for one week, the total volume for one week is recorded (for seven samples). The air volume for each sample is then found by dividing by seven, provided the flow rate is kept constant. When using a sequential sampler is it important to check that the right glass bulb is actually exposed.

After sampling the glass bulbs must be closed in both ends and sent to the laboratory for chemical analysis. The site forms must be filled in with adequate information about gas flows, start and stop of sampling etc.

3.5 Extraction of glass filter samplers

3.5.1 Extraction solution

The extraction solution is 0.001 M triethanolamine. This is prepared by 133 µl of triethnolamine added to 1.0 L of deionized water. (The triethanolamine is added to reduce the iodine formed in the reaction with nitrogen dioxide to iodide).

3.5.2 Extraction of samples

Open the front end of the glass bulb and add 10.0 ml of the extraction solution. Leave the front end open and place the samplers in a laboratory shaking machine. A shaking machine with a capacity of about 50 glass sinter samplers is recommended for the extraction and shaking.

After the shaking is finished, remove the sampler from the shaking machine and transfer the sample to a plastic tube (a (PS) polystyrene 15 ml reagent tube is recommended. This should fit to the autosampler of the ion chromatograph). The tubes may be placed in a suitable stand. Hold the glass sinter sampler with the front end up over the tube and remove the stopper in the back end of the sampler. Quickly place the back end of the sampler into the tube and let the sample drip into the plastic tube. To speed up the transfer and to ensure that the sample is completetely transferred, blow NO_2free air into the front end of the sampler to flush the solution into the receiving tube. The air should be cleaned for NO_2 as specified in 3.3.3.

Make sure that the plastic tubes used for the final storage of the samples, are properly identified with field station name, date of exposure etc.

3.6 Field blanks

All steps in the described procedure which could contaminate the samples should be controlled regularly and be properly documented.

It should be regularly checked that the impregnation solution has a low and negligible content of NO_2^-. This is checked by adding 50 ml of deionized water to 0.5 ml of impregnation solution. The blank value of this solution should be less than 0.01 µg NO_2^-/ml.

When a new batch of impregnated filters have been produced, 5% of the filters should be leached as the exposed samples. The leaching solution should be analysed in the usual way for NO_2^-.The amount of NO_2^- found should be less than 0.02 µg NO_2^-/filter.

In every batch of impregnated glass sinter samplers s sent to the sampling site, one field blank sample must be included. For daily sampling, one field blank per station per week is recommended. The field blank in 50 ml extraction solution should be below 0.05 µg NO_2^-/ml. If the field blanks are stable and well below this limit, the frequency of field blanks may be reduced. This must be evaluated after some experience is achieved.

4. Chemical analysis

4.1 Ion chromatography equipment

The chemical analysis of nitrite in the extracts is performed by ion chromatography using standard conditions. This procedure is based on the use of ion chromatography equipment manufactured by Dionex Corp., USA.

The Dionex ion chromatograph is set up as recommended in the instructions from Dionex. The instrument should preferably be equipped with an autosampler for unattended analysis of series of samples.

For this application a anion separation column of the type Dionex AS9-SC is recommended. This is a general purpose rugged column which copes well with the matrix used. Nitrite is normally eluted after about 1 - 2 min depending on the eluent strength. The iodide peak is large, but elutes after 4 -8 min. A total analysis time of about 10 min. is sufficient.

Other columns from Dionex may be used (such as AS4A, AS14 etc). These have similiar properties as AS9. The iodide peak may elute at different times compared to AS9-SC. Refer to the column manuals for optimum conditions.

A normal anion suppressor system from Dionex must be used together with this column system. This may be a normal chemically based suppressor or a self regenerating (an electrolytical) suppressor. The latter is the simplest and most convenient in use.

Both the column and the suppressor must be operated according to the instructions given in the manuals supplied with the systems from Dionex. The analysis time must be chosen so that the iodide peak is completely eluted out of the system and the signal has reached the baseline level. An analysis time of about 15 minutes is typical for the analysis of nitrite in this matrix.

4.2 Calibration

4.2.1 Stock solution

Stock solutions may be purchased from Dionex or other commercial vendors. They must contain 1000 µg NO_2^-/ml.

A stock solution with the same concentration may be produced by using $NaNO_2$ salt. Dry the $NaNO_2$ at 105 ° C for 1 h and cool in a desiccator. Weigh accurately 1.500 g of $NaNO_2$, add to a 1000 ml volumetric flask and dilute to volume with deionized water.

4.2.2 Working standards

Working standards should cover the concentration range 0 - 10 µg NO_2^-/ml. Typical concentration levels for standards may be 0.5, 1.0, 2.5, 5.0, 10.0 µg NO_2^-/ml. This covers the concentration range in air of about 1 - 150 µg NO_2^-/m^3.

4.2.3 Calibration graphs and integration

Calibration graphs should be prepared by area integration of the peaks (not peak heights). Area integration must also be used for the samples. This is recommended as the matrix used may distort peak shapes somewhat. Normally linear fit of calibration graphs is recommended in the range 0 - 10 µg NO_2^-/ml.

4.3 Quality assurance

The following quality control procedures must be followed:

- Regular analysis of field blanks must be performed. Actions must be taken if the field blank increases above acceptable levels.
- Always check that the correct units of N is used throughout all analysis and calculations, ie. that always the unit NO_2 is used. Necessary formulas for transformation between units must be established and carefully evaluated.
- Before the calculations of the concentrations of the samples, it must be checked that the calibration graph fit to the model equations is good enough (either linear or curved fit).
- All chemicals, standards etc. should be used within storage limitations.
- The analysis time must be so long that the tail of the iodide peak has reached baseline before the next sample is injected.
- an external standard traceable to an internationally acceptable reference material such as NIST should be regularly analysed and the results should agreen within +/- 10 % from the true value.

5. Calculations

5.1 Calculation of the air concentration

The concentration C of nitrogen dioxide in the air sample expressed as $\mu g\ NO_2/m^3$ is given by:

$$C(NO_2) = \frac{Ec \cdot Ev}{Av}$$

where

C(NO2)	concentration of nitrogen dioxide in the air expressed as $\mu g\ NO_2/m^3$
Ec	the concentration of NO_2^- in $\mu g\ NO_2/ml$ in the extraction solution
Ev	the volume of the extraction solution, normally 10 ml
Av	the volume of the sampled air in cubic meter.

6. Reports

The data ere expressed in the desired units as explained in chapter 5. The sample identification and the NO2 results together with results for QA-samples, are transferred to the final data storage and retrieval system which is used for the project.

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Procedure for sampling and analysis of NO_2 and SO_2 in air by the use of passive samplers.

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Table of contents:

Procedure for sampling and analysis of NO_2 and SO_2 in air by the use of passive samplers.

1. Introduction

This procedure describes a method the sampling and analysis of NO_2 and SO_2 in air using passive samplers of the type developed by Ferm at the IVL-institute (Ferm, 1991). The passive samplers are exposed typically from 1 - 4 weeks, but shorter exposure times down to 1 day may be used at exposed sites such as in street canyons and near emission sources of the gases. The working range of the method is in the area 1 - 200 µg /m3 for both NO_2 and SO_2. At high concentrations (above 200 µg/m^3) care must be taken to avoid overloading the capacity of the sampler. In such cases the exposure times should be reduced to prevent overloading the capacity of the sampler.

2. Principle

A passive sampler is a device which collect gases by diffusion as the driving force. The passive sampler is designed as a tube and a concentration gradient is established from the front of the tube to the active surface in the end of the tube. The active surface is normally an efficient absorbent so that the concentration on the surface is close ro zero (ideally it should be zero). The concentration gradient over the sampler (the distance from the front to absorbent surface) is thus equal to the concentration in the surrounding air.

The sampling velocity of a passive sampler is estimated from Ficks first law of diffusion. By using the diffusion coeffecient of the gas and the geometry factors of the sampler (the dimensions of the sampler) the uptake rate may be estimated. The concentration of the gas in air may then be calculated by measuring the amount of gas absorbed on the absorbent (the active surface) in the sampler, the exposure time and the uptake rate estimated.

In this method passive samplers developed by the IVL-institute in Gothenbourg, Sweden, is used, as desribed by Ferm (1991). Table 1 shows some figures of merit for the sampler. The maximum capacity for a 30 day exposure period is 1100 and 1600 µg /m3 for NO_2 and SO_2. To make sure that overloading not occurs it is recommended not to expose the sampler for more than 10 % of the total capacity, i.e. concentration levels around 100 - 200 µg /m^3. As the amount of gas collected increases with the exposure time, higher concentrations may be tolerated at shorter exposure times.

Table 1: Overview of the IVL-type passive samplers described in this method.

Gas	Impregnation solution	Amount of absorbent added to a sampler (at 50 µl)	Maximal capacity	Maximal capacity	Maximum concentration 30 days exposure
SO_2	KOH	50 µmol KOH	25 µmol SO_2	1600 µg SO_2	1600 µg SO_2/m^3
NO_2	NaI + NaOH	25 µmol NaI	25 µmol NO_2	1100 µg NO_2	1100 µg NO_2/m^3

3. Sampling

3.1 The passive sampler

The passive sampler is purchased from NILU products AS, N2007-Kjeller, Norway. A complete passive sampler consists of the following parts seen from the bottom side (where the absorben is placed):

Bottom lid (bottom cap, colour coded, blue (NO$_2$) and red (SO$_2$)
Impregnated filter
Passive sampler tube
Teflon filter
Steel net
Front lid (cap with 20 mm hole)

The bottom lid (cap) is colour coded to distinguish between NO$_2$ and SO$_2$ samplers. The following colour code is used

- Red SO$_2$-sampler
- Blue NO$_2$-sampler

A detailed description of the sampler is given by Ferm (1991) and Røyset (1988).

The passive samplers are placed in transport boxes made of PE or PP. These may be purchased from NILU products AS, N 2007-Kjeller, Norway, together with the passive sampler.

The impregnated filter of the sampler is a Whatman 40 paperfilter with 24 mm diameter. This filter may be purchased as a special order from Whatman Ltd, UK. Alternatively they may be purchased from NILU products AS, N2007-Kjeller, Norway.

In the front of the sampler is placed a steel net and a teflon membrane filter. These are used to prevent turbulence inside the sampler and the collection of particles. This is done to facilitate laminar diffusion in the inside of the sampler tube, which is necessary for Ficks law of diffusion to apply.

The steel net is a part of the sampler and is reused each time the sampler is mounted. The steel net are delivered together with the sampler when purchased from NILU products AS, N-2007-Kjeller, Norway. They may also be purchased as an extra component.

The teflon prefilter is of the type Millipore Fluoropore membrane filters with pore size 1.0 µm FA and a diameter of 25 mm (Millipore code FALP 02500). This is purchased from Millipore. This filter is of single use and is changed each time the sampler is mounted.

3.2 Impregnation solutions

The impregnation solutions are the same as for the active sampling methods described for SO$_2$ and NO$_2$.

Tabell 3.1: Composition of impregnation solutions for the passive samplers.

Sampler	Impregnation solution	Preparation of impregnation solution
SO_2	5.6 g KOH, 10.0 ml glycerol to 100 ml methanol	KOH is dissolved dissolved in methanol using ultrasonic shaking, add glycerol and dilute to volume (100 ml) by methanol. The solution must be prepared new each time a batch of samplers is prepared.
NO_2	7.8 g NaI (sodium iodide) 0.87 g NaOH to 100 ml methanol	NaI and NaOH is dissolved in methanol using ultrasonic shaking and diluted to 100 ml by methanol. The solution must be prepared fresh each time a new batch of samplers are prepared.

3.3 Preparation of paperfilters and passive samplers

3.3.1 Cleaning of paperfilters

Normally the Whatman 40 filters are clean enough to be used without any rinsing or pretreatment. The quality of the filters are monitored by the field blank samples. If the field blank samples become higher than normal, the purity of the filters must be checked. In such a case the filters may be rinsed by a solution of 0.1 M K_2CO_3 dissolved in deionized water. However, since the filters are difficult to dry without becoming folded, washing should be avoided if possible.

Tabell 3.2: Desired blank values of impregnated filters in 5 ml extraction solution

Sampler	Desired blank values of impregnated filters in 5 ml extraction solution
SO2	< 0.05 µg SO_4-S /ml
NO2	< 0.05 µg NO_2^-/ml

3.3.2 Cleaning of passive samplers

All the parts of the passive sampler (except the teflon filter) is cleaned by soaking overnigth in demineralized water. This must be done each time the sampler have been exposed, and before it is mounted with new absorbents. Dry the parts of the sampler before use.

3.4 Impregnation of filters

The impregnation is performed by placing the paper filter in to the cap of the passive sampler. 50 µl of the impregnation solution is carefully added to the filter with a micropipette. Make sure that the whole filter is wetted with the impregnation solution. Make sure that the tip of the micropipette is cleaned properly before it is used for impregnation (rinse it several times with impregnation solution).

Let the impregnated filters dry on the cap for maximum 10 minutes and mount then the passive sampler. Immediately after mounting, place the mounted sampler in a transport box which is properly closed.

3.5 Sampling using the passive sampler

It is important that the passive sampler is stored in a closed transport box until exposure.

For exposure the sampler is taken out of the transport box and placed on a suitable surface by double sided adhesive tape. It is desirable that the passive sampler is placed with the opening pointing downwards to prevent exposure to dustfall and rain. Inside buildings it is recommended to place the sampler so that the opening is facing downwards. It is also recommended to place the sampler under a shielding plate (a small roof) where the sampler is facing downwards. A suitable shielding plate may be:

- a circular plate with rim along the sides like a "freesbee"
- a plastic plate bended in a right angle where one side is a roof (where the passive sampler is mounted facing downwards) and the other side is used for mounting to the surface. The mounting to the surface may be done by metal screws (to wooden surfaces) or alternatively by double sided adhesive tape.

Expose the sampler for the desired time period (1 day, 1 week, 2 weeks, 4 weeks). Immediately after the sampling is stopped, place the sampler into the transport box again and close it properly. Tag the transport box with site name, start and stop date, and type of sampler (SO_2, NO_2).

Fill in a field form for the passive sampler (FIELD FORM PASSIVE SAMPLER). Make sure to fill in all information in the field form, especially start and stop date and time, site name, position and type of sampler. The field form must follow the sampler back to the laboratory and be properly archived in the laboratory so that the information is not lost.

3.6 Extraction of passive samplers

3.6.1 Extraction solutions

The extraction solutions are the same as for the corresponding methods for the active samplers for SO_2 using the filterpack sampler and for NO_2 using the iodide method. The composition of the extraction solutions are summarized in Table 3.3. It is desirable to place the extraction solution into a dispenser. Make sure that the correct volume is dispensed, by checking the volume gravimetrically on a microbalance (it should be 5.00 +/-0.05 ml). Alternatively, pipettes may be used for adding the extraction solution.

Table 3.3: Extraction solutions for the SO_2 and NO_2 passive samplers.

Samper type	Extraction solution	Preparation of extraction solution	Extraction volume
NO_2	1 mM triethanolamine (TEA) in water	133 μl of triethanolamine is added to 1000 ml of deionized water	5.0 ml
SO_2	0.3 % H_2O_2 in watern	10 ml 30% H_2O_2 is added to 1000 ml deionized water	5.0 ml

3.6.2 Extraction of passive samplers

Take the passive sampler out of the transport box. Remove the bottom cap from the passive sampler. With a forceps take out the filter from the bottom cap and put it into a plastic vial (a vial vith a volume of 10 - 15 ml is appropriate). Add the extraction solution as specified in Table 3.3 (5.00 ml) as soon as possible to the vial. Put the stopper in the plastic vial and shake carefully to extract the ions in the filter into the solution. (Turning carefully upside down 10 times is sufficient). Store the vials in a refrigerator at 4° C until analysis.

In the vials for the extracts from the SO_2 samplers small amounts og gas may be formed during extraction. If this is the case, open the stopper carefully to release the overpressure, and close again.

The teflon prefilter in the sampler is discarded each time the sampler has been used.

3.7 Field blank samples

It is important to monitor the quality of the passive samplers by taking field blank samples. These should be placed in a transport box, and taken to the field site, stored there if possible during the measurement period and taken back to the laboratory for analysis. If this is not possible, field blank samples should be stored in the laboratory for the sampling period.

It is recommended to take at least one field blank sample for each sampling campaign, or one field blank sample per month for continious measurement programs.

The level of the field blank should be recorded on separate forms and continiously evaluated. The field blanks should not exceed the levels indicated in Table 3.2. If the field blanks increase above the levels recommended, the procedure must be examined to eliminate possible contamination sources.

4. Chemical analysis

4.1 Ion chromatography equipment and columns

The chemical analysis of sulphate and nitrite in the extracts from the passive samplers are performed by ion chromatography using standard conditions. This procedure is based on the use of ion chromatography equipment manufactured by Dionex Corp., USA.

The Dionex ion chromatograph is set up as recommended in the instructions from Dionex. The instrument should preferably be equipped with an autosampler for unattended analysis of series of samples.

For this applications a anion separation column of the type Dionex AS4A is recommended. This is a general purpose rugged column which copes well with the matrix used. Sulphate is normally eluted after about 6 - 10 min depending on the eluent strength.

Other columns from Dionex may be used (such as AS9A, AS14 etc). These have similiar properties as AS4A. Refer to the column manuals for optimum conditions.

A normal anion supresssor system from Dionex must be used together with this column system. This may be a normal chemically based supressor or a self regenerating (an electrolytical) supressor. The latter is the simplest and most convenient in use.

Both the column and the supressor must be operated according to the instructions given in the manuals supplied with the systems from Dionex.

4.2 Analysis of NO_2

The chemical analysis of NO_2 in the extracts are performed with ion chromatography in the same way as for the method described for the sampling of NO2 by the iodide method. The concentration of iodide in the extraction solution is at the same level as for the active sampling method. Care must thus be taken to adjust the total analysis time so that the iodide is eluted completely before the next sample in injected. Normally a total analysis time of about 15 minutes is sufficient.

The concentration of the NO_2 in the extracts from the passive samplers is normally at the same level as for the active method so that working standards solutions in the range 0 - 10 µg NO_2^- /ml may be sufficient. However, the working range for the standards must be chosen on experience. At high concentrations in the air and long exposure times, the concentrations in the extracts may be higher than 10 µg NO_2^-/ml.

4.3 Analysis of SO_2

The chemical analysis of SO_2 in the extracts are performed with ion chromatography in the same way as for the method described for the sampling of SO_2 by the impregnated filter method. The concentration of KOH/carbonate in the extraction solution is 10 times lower,making the front peak much lower than inn the extracts from the impregnated filter method. Normally a total analysis time of about 10 minutes is sufficient to elute the sulphate peak completely.

The concentration of the SO_2 in the extracts from the passive samplers is normally in the same range or somewhat lower than for the active method. The same working standards solutions in the range 0 - 30 µg SO_4/ml may be sufficient. However, the working range for the standards must be chosen on experience. At high concentrations in the air and long exposure times, the concentrations in the extracts may be higher than 30 µg SO_4/ml.

For SO_2 it is important that the correct type of standards are used. Some laboratories uses standards prepared as $SO_4^{=}$ while others uses standards prepared as S (SO_4-S). Care must be taken so that the same units and respectable conversion factors are used throughout.

4.4 Quality assurance

Make sure that the following points are properly monitored during samplling analysis

- all froms are properly filled in
- the field blanks are continiously monitored
- the calibration graphs for the ion chromatograph fits to the mode used (linear, quadratic etc)
- all chemicals, standards etc are used within storage limitations.
- the correct units for SO_2 is used (SO_2 or S) and that the apropriate standards are used.

5. Calculations

5.1 Estimating the uptake rate for the passive sampler

The uptake mechanism for gases in a passiv sampler has been described by Ferm (1991) and Anda (1991) for the derivation of the formulas necessary. The uptake is estimated based on Ficks law of diffusion. By integration of Ficks law and combining with the contants derived from the dimensions of the samplers a general fomula for the concentration of the gas in air may be calculated by the formula:

$$C = \frac{x \cdot L}{t \cdot D \cdot A} \qquad (1)$$

where

C	concentration of the sampled gas in $\mu g/m^3$
x	amount of gass collected in μg (concentration in extraction solution * extraction volume)
t	time of exposure in seconds
D	diffusion coeffecient of the gas in $cm^2\ s^{-1}$
A	total area of the sampler
L	length of diffusion path of the sampler in cm

The L/A term depends of the geometry of the sampler and the resistances to diffusion in the diffusion path. For the Ferm sampler this may be expressed by:

$$\frac{L}{A} = \frac{LR}{AR} + \frac{LF}{AF} + \frac{LN}{AN} + \frac{LBL}{AR} \qquad (2)$$

The dimensions of the different geometry factors are given in the table below.

Tabell 5.1: Explanation of the geometry factors for Ferms passive sampler.

Geometry factor	Explanations	Dimensions for Ferms sampler
LR	Distance between prefilter and impregnated filter	1.0 cm
AR	Total area of the sampler	3.14 cm2
AF	Pore area pof the teflon prefilter (porosity 0.85)	3.14 * 0.85 cm^2 =2.669 cm2
LF	Thickness of the teflon prefilter	0.0175 cm
LN	Thickness of the steel net	0.016 cm
AN	Area of the steel net (porosity of 0.13)	0.408 cm2
LBL	Laminary boundary layer (estimated)	0.15 cm

Tabell 5.2: Estimation of the total geometry factor for Ferms passive sampler

Factor	Input values	Geometry factor cm^{-1}
L_R / A_R	1.0 /3.14	0.318
L_F / A_F	0.0175 /2.669	0.00785
L_N / A_N	0.016/0.408	0.039
L_{BL} / A_R	0.15 / 3.14	0.0477
SUM L/A		**0.412**

The diffusion coeffecient is given in the unit m^2/sec. If the time is given in the unit days (24 hours), the unit of the diffusion coeffecients must be recalculated to the unit m^2/day by multiplying the diffusion coeffecient by 86400 [sec/day]. These units are given in i table 5.3.

The uptake rate for the passive sampler may be derived form the general formula for the passive sampler, by putting in the constants for the geometry and the diffusion coeffecient.

The general formula may be rewritten as

$$C = \frac{x}{t \cdot \frac{D \cdot A}{L}} \qquad (3)$$

which means that the constants D, A and L may be combined to calculate an uptake rate (U) by the expression:

$$U = \frac{D}{\frac{L}{A}} \qquad (4)$$

The geometry factor (L/A) of 0.412 cm^{-1} is changed to 41.2 m^{-1} and the diffusion coeffecient is changed to the unit m^2/day. Then the units of the uptake U rate will be m^3/day (24h). In the case of NO_2 the uptake rate U may be estimated as:

$$U = \frac{1.33 \cdot m2 / day}{\frac{41.2}{m}} = 0.0323 \cdot m3 / day$$

The concentration of NO_2 in air may thus be calculated by the formula

$$C(NO2) = \frac{x}{t \cdot 0.0323} \qquad (5)$$

where

C	concentration of the sampled gas in µg NO_2 /m^3
x	amount of gas collected in µg (concentration in extraction solution multiplied by the extraction volume)
t	time of exposure in days (24h units)
0.0323	this constant has the unit m^3/day (24h)

Corresponding calculations may be performed for SO_2. All the constants and the uptake rates for NO_2 and SO_2 are gathered in Table 5.3 below.

Tablell 5.3: Diffusion coeffecients and estimates for the uptake rate for Ferms passiv sampler

Gas	Molecular weigth	Diffusion coeffecient D	Diffusion coeffecient D	Geometry factor	Uptake rate
			86400 sec/day		(D / 41.2)
		[m^2/sec]	[m^2/day] [m^2/24h]	m^{-1}	m^3/day (m^3/24h)
NO_2	46	1.54 * 10-5	1.33	41.2	0.0323
SO_2	64	1.32 * 10-5	1.140	41.2	0.0277

5.2 Calculation of the NO_2 concentration in air

As explained in chapter 5.1 the calculation of the air concentration of NO_2 is performed by transforming the general formula for the passive sampler using the geometry and the diffusion coeffecient for NO_2. The amount of NO_2 (x in formula (1)) is is calculated as the product of the concentration and the extract volume, so that the air concentration of NO_2 is then calculated by the formula:

$$C(NO_2) = \frac{m(NO_2) \cdot v}{t \cdot 0.0323} \qquad (6)$$

where

$C(NO_2)$	concentration of the sampled gas in the unit µg NO_2 /m^3
m(NO_2)	concentration of NO_2^- determined in the extract from the filter in the unit µg NO_2^-/ml
v	extraction volume of the filter in ml
t	time of exposure in days (24h units)
0.0323	the uptake rate for NO_2 which has the unit m^3/day (24h)

It must be noticed that the concentration of NO_2 is expressed as µg NO_2 /m^3 by this expression.

5.3 Calculation of the SO_2 concentration in air

As explained in chapter 5.1 the calculation of the air concentration of SO_2 is performed by transforming the general formula for the passive sampler using the geometry and the diffusion coeffecient for SO_2. The amount of SO_2 (x in formula (1)) is is calculated as the product of the concentration and the extract volume, so that the air concentration of SO_2 is then calculated by the formula:

$$C(SO_2) = \frac{m(SO_4) \cdot v \cdot 0.667}{t \cdot 0.0277} \qquad (7)$$

where

$C(SO_2)$	concentration of the sampled gas in the unit µg SO_2-S /m^3
$m(SO_4)$	concentration of SO_4 determined in the extract from the filter in the unit µg SO_4 /ml
v	extraction volume of the filter in ml
0.667	conversion factor from SO_4 to SO_2
t	time of exposure in days (24h units)
0.0277	the uptake rate for SO_2 which has the unit m^3/day (24h)

It must be noticed that the concentration of SO_2 is expressed as µg SO_2/m^3 by this expression.

If the calibration standard for ion chromatography are expressed as sulphur (S) and not as sulphate, the approapriate conversion factors between sulphur and SO_2 must be used. Details about how this is done, is described in the "Procedure for sampling and analysis of SO_2 in air by the use of a filterpack sampler".

6. Reports

The data for SO_2 and NO_2 must be expressed in the correct units as explained in Chapter 5. The sample identification and the results for SO_2 and NO_2 in the correct units are transformed to the final data storage and retrieval system which is used for the project.

7. References

M. Ferm (1991) A sensitive diffusional sampler. IVL-report, B-1020, IVL, Gothenbourg, Sweden.

O.Anda (1991), Passive prøvetakere, NILU TR 10/91, Norwegian Institute for Air Research, NILU, Kjeller, Norway

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Procedure for sampling and analysis of suspended particulates in air by the use of a highvolume sampler

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Procedure for sampling and analysis of suspended particulates in air by the use of a highvolume sampler

1. Introduction

This method describes the use of a highvolume sampler for the collection of suspended particulates (TSP and PM10) in air by the use of a highvolume sampler. The method is designed for sampling periods of 24 h at concentrations in the range 2 - 750 μg m^{-3}. Particulate lead may also be determined in the dust collected on the filter down to a concentration of at least 0.1 μg Pb m^{-3}.

2. Principles

Suspended particulates in air is collected by the use of a high volume sampler with an air flow rate of 1 m^3 min^{-1}. A a large rectangular filter (size 203x254 mm) is used and a sampling period of 24 h is a typical value. The highvolume sampler may collect different fractions depending on what air intake (hood) is used. The two most common fractions are:

- TSP - the total suspended particulate fraction includes particulates with a size up to 25 - 50 μm.
- PM10 - the suspended particulate matter fraction with particulates smaller than 10 μm. A hood with a cutoff at 10 μm in used for this fraction.

The collected particulates are determined gravimetrically by the difference between the weight of unexposed and exposed filters. The weighing procedure is the same for both fractions above.

If desired, lead in the particulates may be determined after nitric acid digestion of the filter, using flame atomic absorption spectrometry.

3. Sampling

3.1 Use of the highvolume sampler

The highvolume sampler is of the type Sierra Anderson. The sampler must be used according to the instructions from the manufacturer. It must be euipped with the correct air intake (hood) depending on what fraction of particulates in air which shal be collected. The two different intakes used in the EIMP project are

- Total suspended particulates (TSP) intake. This collects particulates in up to 25 - 50μm.
- PM10 intake (PM10). This collects particulates less than 10 μm.

The flowrate of the highvolume sampler is about 1 m^3 min^{-3}. The flowrate must be determined at start and stop of the measurement period. This is filled into the filed station form. The flow rate measurements are the same for both the TSP and PM10-fractions.

3.2 Filters

For both the TSP- and PM10-fractions the size of the filterholder of the sampler and the filter type and dimensions are the same.

The size of the filters is 203 x 254 mm (8x10 inches) to fit into the sampler. Glass micro fiber filters or quartz micro fiber filters may be used. Quartz filters are recommended if lead shall be determined in the collected particulates afterwards. If only the amount of collected particulate matter shall be determined, glass micro fiber filters are adequate. These are less expensive than the quartz filters. The glass micro fiber filters are usually more robust and are generally recommended where applicable. The specifications of the filters from Whatman are:
- Glass micro fiber filter: Whatman EPM 2000 8”x10” sheets.
- Quartz micro fiber filters: Whatman QM-A quartz filters 8”x10” sheets.

Both types of filters may be purchased from Whatman Ltd, Springfiled mill, Maidstone, Kent, ME14 2LE, UK. Quartz micro fiber filters may also be of the type Munktell MK 360 from Stora AB, Sweden.

3.3 Mounting/dismounting the filters in the sampler

During mounting and dismounting of the filters in the sampler it is recommended that the operator should use disposable plastic gloves to prevent contamination of the filters. It is also recommended only to touch the filters along the edges. This is particularly important if lead shall be determined afterwards.

Take the filter out of the plastic storage bag and mount it in the sampler. Using glass micro fiber filters, the rough side shall face upwards. The plastic bag which contains the number of the filter, must be stored until the filter is exposed. The exposed filter is then put back into the same bag. If the filter number is written on the edge of the filter, make sure that this side faces down.

After exposure take the filter out of the sampler and fold it into two equal parts (lenghtwise) so that the exposed sides is in. Take care not to lose collected particulates. Put it into the same plastic bag as it was sent out in (which contains the number of the filter). Mark the bag with date of exposure and field station name.

Fill in the field station form for the highvolume sampler with information about exposure date, time and flowrate reading at start and stop of exposure.

3.4 Field blanks

It is recommended to take field blank samples for quality control. The field blank filters should be handled as the ordinary filters. They are taken to the field station and mounted into the filterholder, but taken out again immediately folded and put back into the transport plastic bag. The field blank samples are weighed. Actions must be taken if the filters increases or decreases significantly in weight during the transport to the field station and back. In the case of the determinination of lead, the value found in the field blank filter must be significantly lower than from the exposed fillters. Acceptable values must be established on experience.

4. Analysis

4.1 Requirements for the laboratory environment of the weighing room

The laboratory weighing room where conditioning and weighing of filters are performed should have control of temperature and humidity. The temperature of the weighing room should be between 15 - 30 °C with less than +/- 3 °C variation. The humidity should be less than 50 % relative humidity with less than +/- 5 % variation.

Control of temperature is less critical than humidity. If it is problematic to maintain the limits, the control of humidity should be focused first.

4.2 Laboratory balance

A microbalance with a sensitivity of 0.1 mg should be used. For quality control the balance should be checked with a sertified gravimetric standard in the area 1 - 10 gram.

If possible use a suitable stand on the microbalance where the relatively large filters may rest during weighing.

4.3 Weighing unexposed filters

Leave the filters om a clean surface such as a plate of clean laboratory paper during conditioning.

During handling of filters it is recommended to use disposable gloves and if possible the filters should only be touched by tweezers along the edges.

The unexposed filters must be preconditioned under controlled temperature and humidity in 24 h before weighing. This is best achieved by placing the filters in the weighing room where the temperature and humidity is controlled as specified in 4.1.

Weigh the unexposed filter unfolded. Put the filter into a plastic bag with zip closure. Fill in the filter weighing form. It is recommended to have one form for each station. Each filter must have a unique number which is filled into the form. This number must also be marked on the plastic bag where filters are stored. The number may also be marked on the filter. The mark should be made at one corner outside the exposed area and the side which faces downwards in the filterholder. Use a filttip pen or a pencil.

4.4 Weighing exposed filters

The exposed filters (which are folded) are carefully taken out of the plastic bag and left for conditioning in the weighing room for 24 h. The filters must be handled so that parts of the collected dust is not lost. The filters are then weighed and the results are filled into the weighing form. The amount of dust is then calculated as the weight difference between the exposed and unexposed filters. While raw weight of the filters are expressed in mg, the weight difference is expressed in µg.

5. Calculations

The air concentration of suspended particulates in air is determined by the formula for both the TSP and PM10 fraction:

$$SPconc = \frac{W}{Av}$$

where

SPconc	concentration of particulates in µg m^{-3} (for both TSP- and PM10-fraction)
W	weigth of particulates collected on the filter in µg
Av	total volume of sampled air in m^3

If the air volume is determined by the measurements of flowrates at start and stop, the air volume (Av) is determined as the average flowrate multiplied by the exposure time:

$$Av = \frac{(Fs + Fe)}{2} \cdot N$$

where

Fs	flowrate at start in $m^3 h^{-1}$
Fe	flowrate at end in $m^3 h^{-1}$
N	number of hours sampled in h

6. Reports

The data are reported using the report forms designed for the EIMP-project.

7. References

US EPA (1977). Test method, section 2.2. Reference method for the determination of suspended particulates in the atmosphere (high volume method).

Quality assurance handbook for air pollution measurement systems, Volume II - Ambient air specific methods. EPA-600/4-77-027a. , Environmental Protection Agency, (EPA), USA.

8. Appendix. Determination of particulate lead collected with highvolume filters

8.1 General

The lead in the particulates collected on the filter may be determined after digestion of the filtermaterial with nitric acid. The lead in the nitric acid extract of the filter can then be determined by atomic absorption spectrometry.

At a concentration of 0.1 µg Pb m^{-3} in air and 24 h samples, the concentration of the extract produced by the digestion, is about 0.35 µg Pb ml^{-1}. At a concentration level in air between 0.1 - 1 µg Pb ml^{-1}, the concentration in the extracts from the filters will thus be between 0.3 - 3 µg Pb ml^{-1}. At this concentration level flame atomic absorption spectrometry (FAAS) is of sufficient sensitivity for the determination of lead.

For the determination of lead it is important to clean all equipment very carefully. All equipment which is used for the treatment of samples and standards, should be rinsed by soaking in 1 % nitric acid and rinsed with deionized water before use.

8.2 Digestion of filters

8.2.1 Reagents

The digestion solution is nitric acid which is diluted 1:1 with deionized water. Make this solution by adding equal parts of deionized water and concentrated nitric acid into a bottle and mix. It is recommended to use high purity nitric acid for this purpose such as Merck suprapure or equivalents if the concentrations are low (approximately 1 µg Pb m^{-3}). Otherwise Merck pro analysis quality or equivalents may be sufficient. The need for the relatively expensived high purity acid may be checked, by analyzing the concentration in a 1:10 dilution of the acid. The blank level should be below 0.01 µg Pb /ml.

Make a solution of 1 % nitric acid (1:100) by diluting 1 part of concentrated nitric acid to 99 parts of deionized water.

8.2.2 Digestion equipment

The digestion is performed in 250 ml glass bottles of the type Duran. The pouring ring should be made ETFE (a red termoplast which withstand temperatures to 180°C). The cap should also be made of the special red termoplast type covered by teflon inside. This is available as special equipment for the Duran bottles. The Duran bottles are made by Schott glaswerke, Postbox 2480, W-6500 Mainz, Germany.

For the digestion an autoclave is used. The autoclave should be equipped with a pressure relief valve which opens at 2 atmospheres. When this is heated with water inside, a temperature of 120 °C is achieved at a pressure of 2 atmospheres. With this it is possible to heat the samples to 120°C at a pressure of 2 atmospheres. A laboratory type autoclave which can be heated to this temperature and this pressure is recommended.

8.2.3 Digestion procedure

Take the exposed filter out from the plastic storage bag. Cut the filter in two equal parts lengthwise the filter (across the folding). Make sure that no particulates are lost. One part is used for the digestion, the other part is put back into the plastic bag.

Cut the filter into small pieces so that they fit the opening of the Duran bottle and put the filter pieces into the bottle. Add the 1:1 nitric acid digestion solution to the bottle so that the filters are covered by solution. 40 ml are usually sufficient. Close the digestion bottle with the cap.

Put the bottles into the autoclave. Add deionized water into the autoclave at a height of 2 -3 cm from the bottom. Heat the autoclave for 1 hour at a temperature of 120 °C and 2 atm. Afterwards, cool the autoclave to room temperature and make sure that the pressure is released before opening.

Remove the bottles with samples from the autoclave. Transfer the solution in the digestion bottles to a 200 ml volumetric flask (without removing the filtermaterial). Rinse the filtermaterial by adding 40 ml 1 % nitric acid to the digestion flask. Shake the solution so that the filtermaterial is thoroughly washed and transfer the solution to the same volumetric flask. Perform this rinsing 2 times. After the second rinsing the 200 ml receiving volumetric flask is filled to volume by 1 % nitric acid. The filtermaterial in discarded.

8.3 Determination of lead by flame AAS

As the typical concentrations will be in the range 0.3 - 3 µg Pb ml^{-1} in the extracts from the filters, standards in the range 0.1 - 10 µg Pb ml^{-1} is needed. Typical standard concentrations may be 0.1, 1.0, 5.0 and 10 µg Pb ml^{-1}. These should be prepared in 10 % nitric acid to match the acid content (matrix) of the samples.

Set up the flame AAS instrument as recommended by the manufacturer. Calibrate with the standards and determine the concentration of lead in the extracts from the filters.

If the concentrations are below 0.1 µg Pb ml^{-1} in the extracts from the filters, it is recommended to use electrothermal AAS (with graphite furnace) for the determination of lead. This is, however, only needed if accurate determinations at low levels is necessary.

8.4 Calculations

The concentration of lead in air is determined by the formula

$$CPb = \frac{Ce \cdot Ev}{Av}$$

where

CPb	concentration of lead in µg Pb m^3
Ce	concentration determined in the extract from the filter in µg Pb ml^{-1}
Ev	volume of extract from the filter in ml
Av	air volume in m^3

8.5 Reports

The data are filled into the report form designed for the reporting of lead for the project.

Procedure for sampling and analysis of dust fallout from the air

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Procedure for sampling and analysis of dust fallout from the air

1. Introduction

This procedure is designed for the sampling of dust fallout from the atmosphere using conventional dustfall buckets.

2. Principle

Dust fallout is atmospheric particles which sediments from the atmosphere by gravitational forces. Also particles which are washed out by precipitation is collected by the sampler and operationally included into the dustfall fraction.

The dust fallout is collected by a dust fall collector bucket ("dust bucket") with a horizontal surface. Normally a small amount of water and an antifreeze agent is added to the dust bucket to prevent resuspension of collected particles. This may not be useful in a hot climate as this wetting agent will evaporate quickly.

The most important fraction is called waterinsoluble dustfall. This fraction is determined by washing out the particles collected in the bucket by water and collect the particles on a filter. The amount of particles collected on the filter is determined gravimetrically by the weight difference between unexposed and exposed filter.

Part of the collected particles may dissolve during the washout and be transferred to the water phase. This fraction is called watersoluble dustfall. This fraction is determined by evaporation of a fixed amount of water (used for the washing out of the bucket). This fraction is also determined gravimetrically as the weight of solid residue left after the evaporation.

Both the waterinsoluble and watersoluble fraction of the dustfall is determined in accordance with ISO/DIS 4222.2.

3. Sampling

3.1 Sampler

The dustfall collector is a standard NILU SF-1 dustfall collector with a diameter of 20 cm so that the horizontal upper area for collection of dust of 0.0314 m^2.

The sampler is equipped with a stand with a birdring so that a sampling height of about 2 m above the ground may be achieved.

3.2 Cleaning of the sampler

Before exposure the sampler must be cleaned with a suitable laboratory detergent so that all particles are removed, after which it is rinsed with a suitable solution of water as specified below:

If only the weight of dustfall is to be collected, the sampler may be cleaned with only tap water.

If chemical compounds such as major ions shall be determined in the dustfall, the sampler has to be thoroughly rinsed with deionized water.

If chemical compounds such as trace metals shall be determined in the dustfall, the sampler has to be thoroughly rinsed with a dilute solution of an acid such as nitric acid. In such cases the metal ring on the top of the sampler has to be removed, otherwise it will be attacked by the acid and contaminate the sampler.

3.3 Sampling solution

During the exposure the sampler shall be filled with a collection solution containing the following compounds:

470 ml of deionised water
30 ml of ethylenglycol monomethyl ether

This solution is filled into a bottle of 500 ml size which shall be shipped out together with the sampler.

The purpose of this solution is to prevent freezeing, resuspension of collected particles and the growth of microorganisms in the sampler.

In a hot climate with little or no rain this solution may rapidly evaporate from the sampler. In such cases the sampling solution may be omitted, as the solution has no effect on the sampling conditions.

3.4 Sampling conditions

At the site the stand is placed in a suitable position on the ground so that a sampling height of 2 m is achieved. Make sure that the stand is thoroughly fixed to the ground.

Remove the lid of the sampler. If desired the sampling solution is added to the sampler. Place the bucket sampler in the stand.

After exposure remove the sampler from the stand, place the lid on the top and transport the sampler in a horizontal position back to the laboratory for analysis. It is important that the sampler is transported in such a way that collected particles are not lost.

4. Analysis

4.1 Gravimetric equipment

For the weighing of the collected dust a microbalance with an accuracy of at least 0.1 mg should be used. The balance should be placed in a room with controlled temperature and humidity. A temperature of 21 +/- 3 °C is suitable. The relative humidity should be less than 50% and controlled to +/- 5 %.

The most important parameter to control is the relative humidity. If these criteria are not met in the weighing room, the filters should be conditioned in a dessiccator before and after weighing. The dessiccator must have a suitable drying agent in the bottom which must be properly conditoned so that it is active. In such cases the filters must be weighed immediately after transfer from the dessiccator to the balance.

4.2 Filtration equipment

For this parameter a filtration equipment is needed. A Buchner funnel with a diameter of 9 cm is suitable. Other dimensions may be used if desirable. 9 cm diameter provides a rapid filtration and the risk to need many filters for one determination is minimised. The funnel must be equipped with a vacuum receiving bottle with a volume large enough to keep the rinsing solution produced from one sampler bucket.

Above the opening of the funnel is placed a coarse net, which is intended to remove coarse fallout components such as blades, insect etc. These are not intended to be included into the waterinsoluble dust fallout fraction.

The filtration equipment must be cleaned according to the parameters desired. The same criteria with the respect to cleaning must be followed as specified in chapter 3.2. for the cleaning of the equipment.

Different types of filters may be used. Recommended types are:

Schleicher and Schüll 582.2 Weissband paperfilter (Schleicher and Schüll, Germany)
Whatman GF/C glassmicrofiber filter (Whatman Ltd, Maidstone, Kent, UK)

For gravimetric analysis it is recommended to use the glass microfiber filter as these are less affected by moisture. The dimension of the filter must suit for the Buchner filtration funnel used.

4.3 Determination of waterinsoluble dustfall

Dry the filters at 110 degrees C for 2 hours. Condition the filters used in the weighing room or in the dessiccator for 24 hours. Weigh the filter before exposure to the nearest 0.1 mg and fill in the net weight in the dustfall worksheet form.

Mount the filter in the Buchner funnel, and mount the funnel into the filtration equipment stand. Place the net over the opening of the funnel. If there is aqueous solution in the dustfall collector, swirl the collector so that the collected dustfall is suspended in solution. If there is no solution in the dustfall collector, add 500 ml of demineralized water. Swirl to suspend the particles.

If it is difficult to suspend all particles in the bucket, use a plastic tool to loosen the particles and bring them into suspension.

Add the suspension to the filtration funnel and evacuate to filtrate. If it is difficult to transfer all particles, flush the bucket with a small amount of demineralised water and transfer to the filtration funnel.

If it is so much particles in the bucket that the first filter becomes blocked, use a new filter and mark the filters with the corresponding number 1, 2, 3, etc. in the dustfall worksheet form.

After exposure the filters may be placed on a glass petridish or watchglass plate and dried at 110 degree C for 2 hours. They may now be placed in a plastic petridish and conditioned in a the weighing room or in a dessiccator for 24 hours. Then the filters are weighed and the numbers are filled into the dustfall worksheet form.

If watersoluble dustfall is not ordered, the filtrate collected in the filtration stand may be discarded.

4.4 Determination of watersoluble dustfall

Record the total volume of filtrate used for the filtration of waterinsoluble dustfall, and fill it into the watersoluble dustfall worksheet form.

For the gravimetric determination of watersoluble dustfall a beaker with a size of minimum 100 ml is needed. Dry the beaker at 110 °C for 2 hours, cool in a dessiccator for at least 2 hours and weigh to the nearest 0.1 mg. Fill in the number in the watersoluble dustfall worksheet form.

Fill the beaker with accurately 100 ml of filtrate and dry it on a hotplate at 95 °C until the solution have evaporated completely. Then dry the beaker at 110 °C for 2 hours and cool in a dessiccator for at least 2 hours. Weigh the beaker and fill into the watersoluble dustfall worksheet form.

5. Calculations

The upper diameter of the NILU SF-1 dustfall collector is 0.20 m, making the upper area 0.0314 m^2. The amount of waterinsoluble and watersoluble dustfall is normalised to 30 days according to recommendations given by ISO/DIS 4222.2, so that the unit reported is mg/m^2 x 30 days.

The amount of waterinsoluble dustfall is calculated by the expression:

$$Wisd = \frac{(Wa - Wn) \cdot 30}{0.0314 \cdot t}$$

where

Wisd	weight of waterinnsoluble dustfall in the unit mg/m^2 x 30 days
Wa	weight of filter with dustfall collected in the unit mg
Wn	net weight of the filter before the dustfall is collected in the unit mg
t	exposure time in the unit days (24h)
0.0314	horizontal area of the sampler in the unit m^2
30	factor used for normalisation to 30 days

Correspondingly the amount of watersoluble dustfall is calculated by the expression:

$$Wwsd = \frac{(Wba - Wbn) \cdot Sv \cdot 30}{0.0314 \cdot 100 \cdot t}$$

where

Wwsd	weight of watersoluble dustfall in the unit mg/m^2 x 30 days
Wba	weight of beaker with dry residue after evaporation in the unit mg
Wbn	net weight of the beaker before solution is added in the unit mg
Sv	total volume of solution in the filtration collector flask in the unit ml
t	exposure time in the unit days (24h)
100	volume of subsample taken for evaporation in the unit ml
0.0314	horizontal area of the sampler in the unit m^2
30	factor for normalisation to 30 days

6. References

ISO/DIS 4222.2, Draft for international standard for the collection of dustfall.

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		Date: 01.11.98
Printed date: 04.11.98		Issue no: 001

EIMP - Air quality
QA-QC-procedures

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

EIMP - Air quality QA-QC-procedures	Page 2 of 4 pages
Printed date: 04.11.98	Date: 01.11.98 Issue no: 001

Table of contents

EIMP Air quality
QA-QC-procedures

1. Introduction

This procedure describes preparation of quality control samples intended for daily use in the control of ion chromatography analysis of sulphate ($SO_4^{=}$) and nitrite (NO_2^{-}). These analysis are used for the determination of SO_2 and NO_2 in air, using the filterpack, the glass sinter sampler as well as the passive sampler. Both SO_2 and NO_2 in air are converted to sulphate ($SO_4^{=}$) and nitrite (NO_2^{-}) during sampling and sample preparation, so that the use of sulphate ($SO_4^{=}$) and nitrite (NO_2^{-}) solutions are adequate in the control of the analysis of these compounds in air.

Two QC samples are prepared, one high and one low, to cover the concentration range of the samples. The samples are analysed in duplicate so that both X-charts and R-charts may be prepared.

2. QA-QC samples

2.1 Stock solutions

Two stock solutions of 1000 µg/ml of both compound separately, as specified in Table 1, must be prepared. The stock solutions should be prepared from high purity quality salts of $NaNO_2$ and Na_2SO_4. The salts used should be dried at 105 °C and cooled in a dessiccator before they are weighed. The stock solutions should be stored in either PP or glass bottles with tight caps. The stock solution for $SO_4^{=}$ is stable for at least one year when stored in a refrigerator at 4 °C. The stock solution for NO_2^{-} is stable for six months when stored in a refrigerator at 4 °C.

Table 1.
Preparation of stock solutions for quality assurance of ion chromatographic analysis of sulphate ($SO_4^{=}$) and nitrite (NO_2^{-}).

Compound	Salt recommended	Amount of salt to get 1000 µg/ml	Assigned concentration
NO_2 (NO_2^{-})	$NaNO_2$	1,500g	1000 µg NO_2^{-}/ml
SO_2 ($SO_4^{=}$)	Na_2SO_4	1,479g	1000 µg $SO_4^{=}$/ml

2.2 QC samples

Two QC samples are prepared at a concentration of 10.00 µg/ml and 1.000 µg/ml of both $SO_4^{=}$ and NO_2^{-} in the same solution.

2.2.1 QC sample High -10.00 µg/ml

The sample is prepared by adding 10.00 ml of both the stock solutions to a 1000 ml volumetric flask, so that a mixed sample containing 10.00 µg/ml og both $SO_4^{=}$ and NO_2^{-} is achieved.

2.2.2 QC sample Low - 1.000 µg/ml

The sample is prepared by adding 1.000 ml of both the stock solutions to a 1000 ml volumetric flask, so that a mixed sample containing 1.000 µg/ml of both $SO_4^{=}$ and NO_2^{-} is achieved.

2.2.3 Storage

After dilution to the volume, the samples should be transferred to PP or glass storage bottles with a tight cap and stored tightly capped at 4°C. When treated this way the samples are stable for 1 month. Care should be taken to NO_2^{-}, as this may degrade during storage. If problems should occur especially for the QC sample Low, the storage time should be reevaluated.

3. QA-QC procedures

The QC sample High and Low should be analysed by ion chromatography each time SO_2 or NO_2 samples are analysed. A warning and action limit of +/- 5 % and +/- 10 %, respectively, should be observed.

The samples must be analysed in duplicate to achieve data for the production of R-charts.

The data shall be presented by the software package named "Quality". This software package have procedures for the preparation of both X-charts and R-charts. Once the data have been punched into the software package, X- and R-charts may be produced.

3.1 X-charts

A X-chart is a concentration versus time plot of the QC-samples. The charts should be plotted with the warning and action limits marked on the plot.

3.2 R-charts

The R-chart is a plot of the difference between the two parallels analysed for each of the QC sample high and low. The charts should be plotted with the warning and action limits marked on the plot.

3.3 Actions to be taken

After 20 data sets have been collected, the warning and action limits are evaluated based on the quality achieved. Appropriate action is discussed with the EIMP air quality chemical expert.

If results of 1 analyses is outside the action limit, or 2 subsequent analysis are outside the warning limit, actions must bee taken to evaluate possible errors in the calibration procedure of the ion chromatograph.

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, Morten C Andersen, Ulla Lund, Anwar**
From	**Oddvar Røyset**
Subject	**Equipment needs at CEHM**
Date:	**02.11.98**

Equipment needs at CEHM of Cairo University

During my work 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**
Autosampler for the Dionex DX 100 Ion Chromatograph (recommended the Gilson 222 type or equivalents)	High	ca 75000
Water treatment system for production of pure water	High	ca 50000
New microbalance for TSP with larger weighing chamber to fit for 10"x8" highvolum filters	High	ca 25000
Laboratory shaking machine for the extraction of NO2 tubes	High	13000-15000
Computer	High	7000
Desiccator	High	1300
Filtration equipment for dustfall	High	3000
Volumetric flasks of 1000 ml, 10 units		1500

It was also noted by dr. Ahmed Soliman Abd Ellah, that the laboratory would need 1 - 2 chemists more when the full EIMP program for SO2 and NO2 including about 20 stations was implemented.

Comments

Autosampler.

The ion chromatographs were not equipped with autosamplers. This is necessary for the routine analysis of samples for the EIMP project. When the project is running as planned about 200 samples has to be analysed each week. It is not possible to analyse more than about 4 samples per hour. To carry out this number of samples it is necessary to run the instrument during the evening and nights. This can only be achieved by the use of an autosampler. It is recommended to buy one autosampler in the beginning and install it on the DX 100 IC instrument. It is recommended to use a Gilson 222 sampler, not the type delivered from Dionex.

Water purification system
The reverse osmosis and deionisation system in the CEHM-had very low capacity (only 2 litres per hour). To clean the air samplers, much larger capacity is needed. It is recommended to install a reverse osmosis/electrolytic purification system (preferably also equipped with UV-desinfection) such as those produced by Millipore, which also is eqquipped with a storage tank for pure water. As it is now, equipment have to be cleaned in tap water. This is not acceptable. The equipment recommended is the Millipore Elix 5 L/h reverse osmosis/electrodeionisation unit equipped with 60 l storage tank, UV desinfection, recirculating pump and preferably a pressure delivery pump. The price of this equipment should be between DKK 40000 - 50000.

New microbalance
The microbalances had weighing chamber which were too small to fit the highvolume filters used (200x250 mm). It is recommended to get a new microbalance with a larger weighing chamber to fit the filters. However, if a suitable stand may be produced, a new balance may not be needed.

Laboratory shaker
The NO2 samplers need to be shaken during extraction. As at least 50 samples must be processed each week, a professional system is needed. A low cost laboratory shaking machine is recommended. Such are available at a price of about US$ 2000-2500, DKK 13000 - 15000.

Computer
The laboratory needs access to a separate computer for storage of the data for SO2, NO2, TSP etc. It is important to have access to graphical presentation procedures for a rapid quality control of the data.

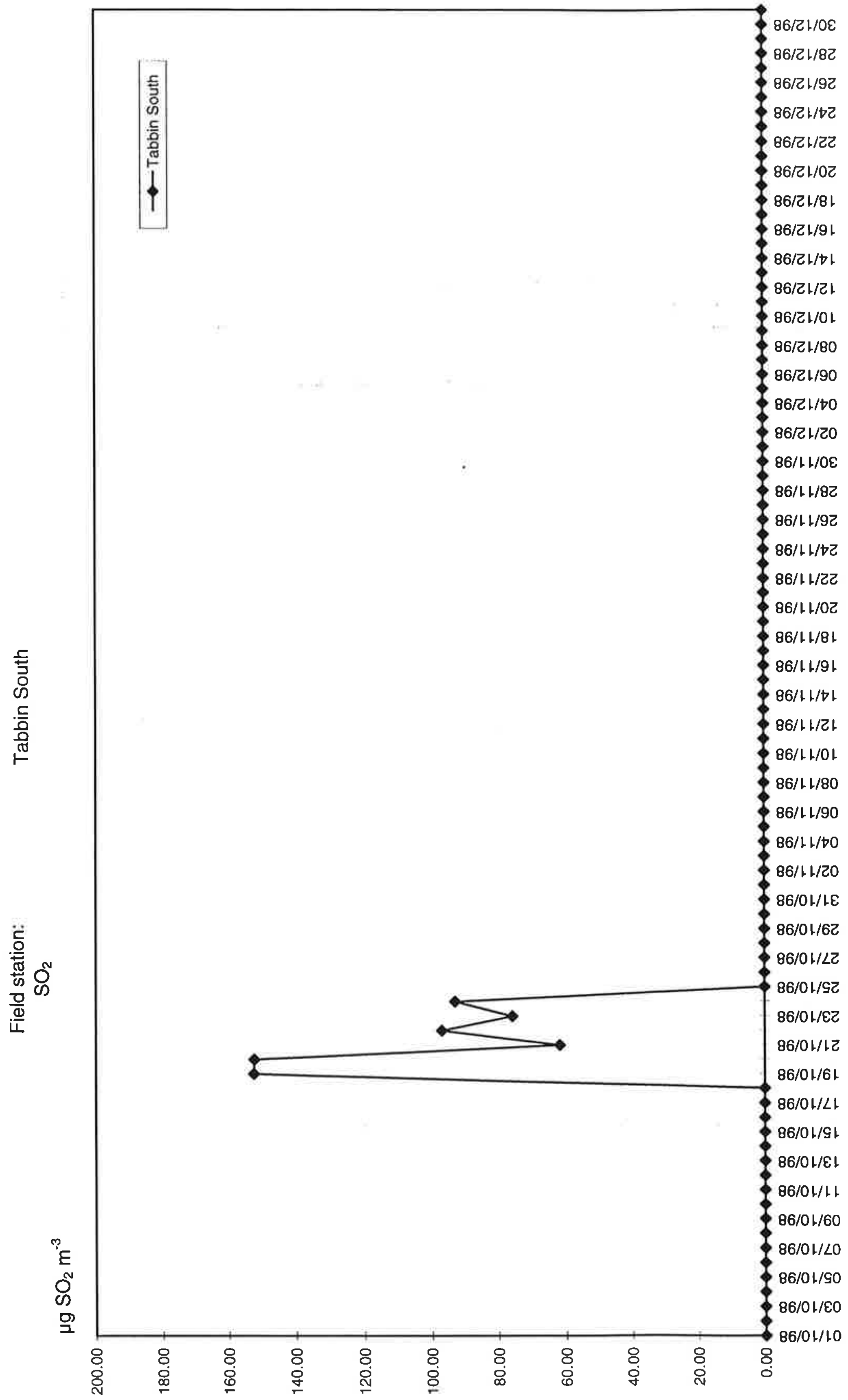
Filtration equipment for dustfall
It is necessary to have a filtration equipment for the gravimetric determination of dustfall from air. The equipment should contain a 5 L receiving vacuum bottle and a 90 mm Buchner funnel.

Desiccator
This is needed for the storage of impregnated filters.

Volumetric flasks of 1000 ml, 10 units
The laboratory lacked volumetric 1000 ml flasks. This is needed for the preparation of standard solutions and sample extraction solutions.

Appendix E

Measurements at Nasr City and Tabbin South


Field station:
SO$_2$
Tabbin South
µg SO$_2$ m^{-3}
200.00
180.00
160.00
140.00
120.00
100.00
80.00
60.00
40.00
20.00
0.00
01/10/98
03/10/98
05/10/98
07/10/98
09/10/98
11/10/98
13/10/98
15/10/98
17/10/98
19/10/98
21/10/98
23/10/98
25/10/98
27/10/98
29/10/98
31/10/98
02/11/98
04/11/98
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12/11/98
14/11/98
16/11/98
18/11/98
20/11/98
22/11/98
24/11/98
26/11/98
28/11/98
30/11/98
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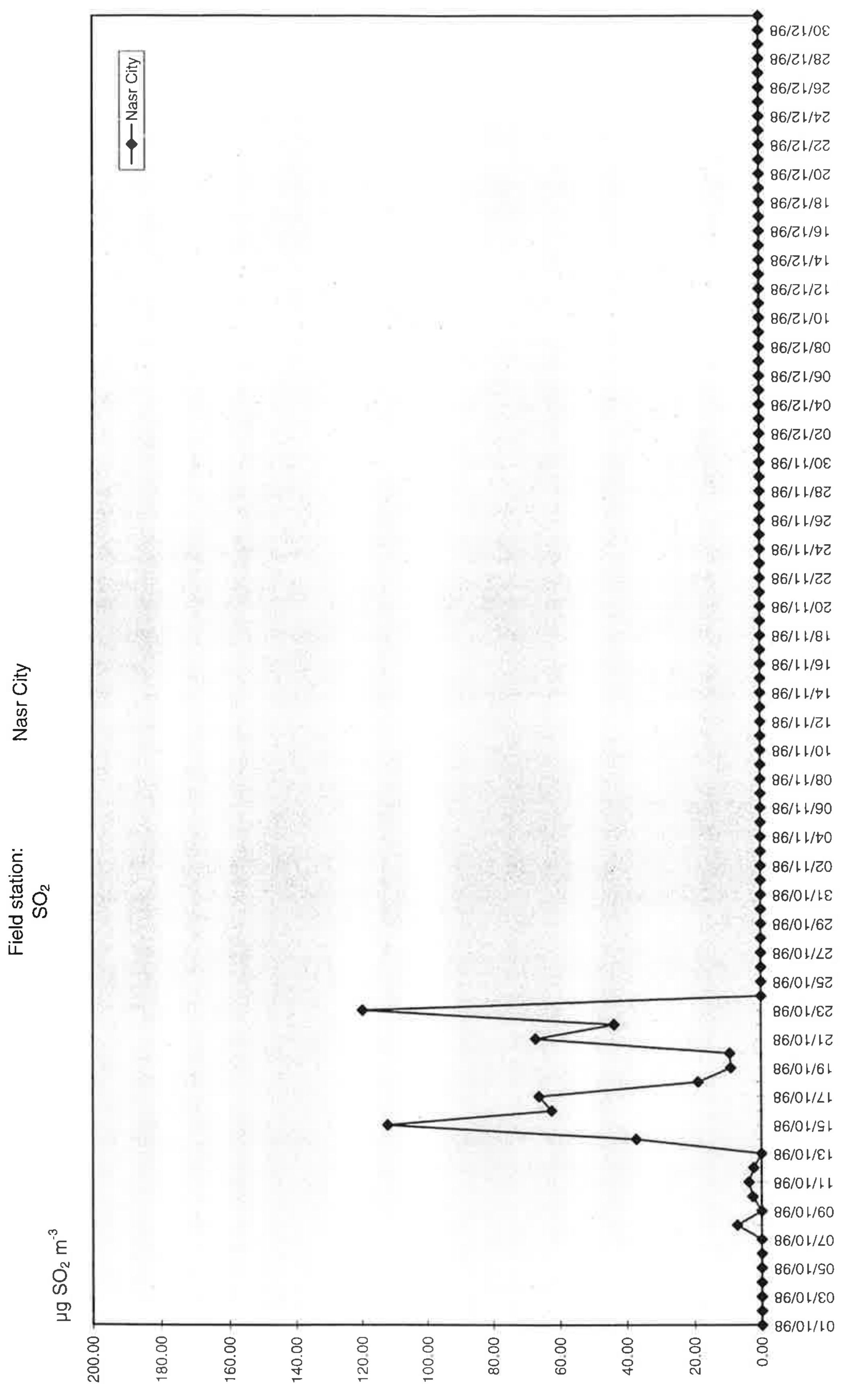
28/12/98
30/12/98
Tabbin South


Field station: Nasr City
SO$_2$
µg SO$_2$ m^{-3}
0.00
20.00
40.00
60.00
80.00
100.00
120.00
140.00
160.00
180.00
200.00
01/10/98
03/10/98
05/10/98
07/10/98
09/10/98
11/10/98
13/10/98
15/10/98
17/10/98
19/10/98
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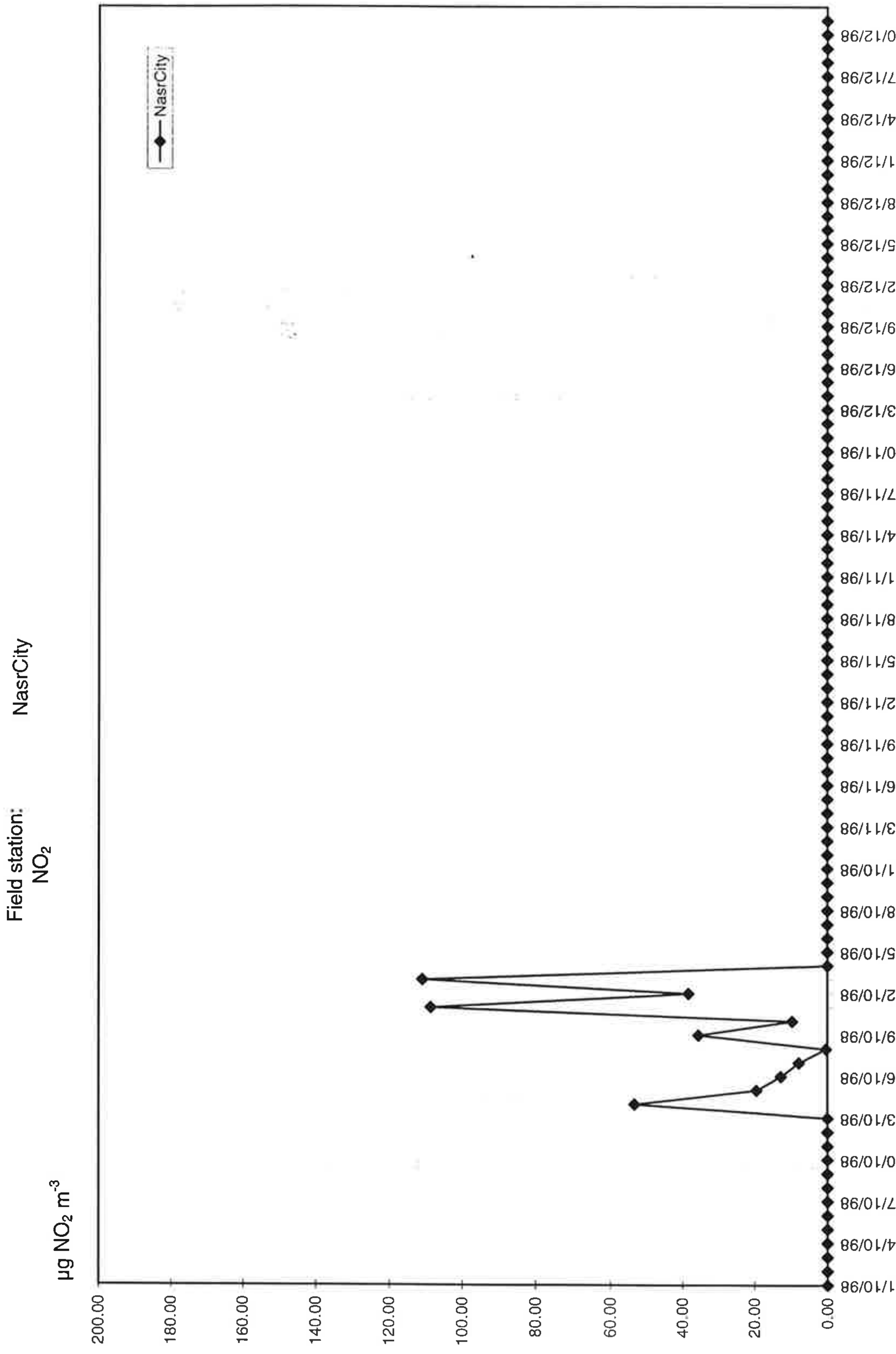
26/12/98
28/12/98
30/12/98
Nasr City


Field station: NasrCity
NO_2
µg NO_2 m^{-3}
200.00
180.00
160.00
140.00
120.00
100.00
80.00
60.00
40.00
20.00
0.00
01/10/98
04/10/98
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27/12/98
30/12/98
NasrCity

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

Mission 10 Report

PROJECT LEADER
Bjarne Sivertsen

NILU PROJECT NO.
O-96013

AUTHOR(S)
Oddvar Røyset and Bjarne Sivertsen

CLASSIFICATION *
A

CONTRACT REF.

REPORT PREPARED FOR:
COWI/EIMP
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ABSTRACT
The tenth mission to Egypt on the DANIDA EIMP programme included training in chemical analysis methods for NO_2, SO_2, TSP, PM_{10} and dustfall for the CEHM-laboratory at Cairo University, Giza.

NORWEGIAN TITLE
Overvåkingsprogram for luftkvalitet i Egypt

KEYWORDS

Air Quality	Chemical Analysis	Training

ABSTRACT (in Norwegian)

** Classification*	*A*	*Unclassified (can be ordered from NILU)*
	B	*Restricted distribution*
	C	*Classified (not to be distributed)*