LIFEINDEXAIR

Technical Guide

Sampling and Chemical Characterization of Particles

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Executive Summary

The Technical Guide is a document of the LIFE Index-Air project, delivered in the context of the Action A2 - Technical Planning, more specifically in Activity A2.1 - Training.

The objective of Action A.2 is to guaranty the quality of all data generated during the project. The success of Actions B is strongly linked with the quality of the experimental data. Possible malfunctioning of the samplers and/or technical problems during the chemical analysis may produce incomplete databases. To minimize these risks and constraints a technical guide was prepared and discussed in a training course organized for technical staff of the project.

This report is divided in two parts: the first one presents the technical guide and the second one presents the report of the training course that was organized to consolidate the technical guide.

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Introduction

The objective of Action A.2 is to guaranty the quality of all data generated during the project. The technical guide presented in this document was developed by IST and NCSR-D to define all the procedures that will be used during the implementation of the Action B.2. After the development of the technical guide, a training course was organized with representatives from all beneficiaries to consolidate the procedures and to train the technical staff.

This report is divided in two parts:

I. Technical guide;
II. Report of the training course that was organized to consolidate the technical procedures and to train the LIFE Index-Air staff.

I. Technical Guide

The technical guide is composed by independent procedures related to each one of the activities to be performed in Action B2. This organization facilitates the consultation of the procedures by the members of the staff responsible by the different activities. Sampling activities will be performed by IST team, while NCSR-D will be in charge with the chemical analysis of the particles. Table 1 present the procedures that are available in this technical guide.

Table 1: Procedures available in the technical guide.

INDEX-1	Procurement and preparation of filters
INDEX-2	Gravimetric analysis of the filters
INDEX-3	Leckel MVS6 operation
INDEX-4	SKC Pump operation
INDEX-5	PM sampling in schools and homes
INDEX-6	PM sampling in mobile environments
INDEX-7	X-ray spectroscopy for the determination of trace elements
INDEX-8	Thermal-optical analysis for the determination of EC/OC
INDEX-9	PAH analysis of the filters

Procedure INDEX-1: Procurement and preparation of filters

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the preparation of Teflon, Quartz and Nuclepore filters to be used in the sampling campaigns that will be performed in 40 homes, 5 schools and 5 vehicles.

This activity will be performed in Campus Tecnológico e Nuclear (CTN) from Instituto Superior Técnico (IST).

2. Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Vânia Martins, Tiago Faria, Marina Almeida-Silva and Isabel Dionísio under the supervision of Marta Almeida.

3. Requirements and conditions of application

3.1 General good practice

- The filters will be prepared in the clean laboratory located in the Physics Department of CTN, which is a class 1000 clean laboratory equipped with a class 100 laminar flow chamber.
- The laboratory should be reserved in advanced by using the Log Sheets located in the antechamber.
- The technicians should use a clean Lab Coat and wear gloves without powder.
- Filters should be handled with care to avoid possible contamination.
- Whenever a filter box is opened, the date of the opening should be recorded in the box and three filters must be removed for blank analysis.

4 Instruments and materials

4.1 Filters specifications

Different samplers and types of filters will be used in order to allow the chemical characterization of elements, EC/OC and PAHs in particles. Table 2 presents the specifications of the filters that will be used in the LIFE Index-Air sampling campaigns to be performed in homes and schools.

Table 2: Specifications of the filters to be used in the sampling campaigns to be performed in homes and schools.

	Indoor (5 Schools and 40 Homes)		Outdoor (5 Schools)	
	Leckel #1	**Leckel #2**	**Leckel #3**	**Leckel #4**
PM2.5-10	**Quartz**	**Nuclepore**	**Quartz**	**Nuclepore**
	25 mm	25 mm	25 mm	25 mm
	Pall Ref. VWR 516-7991	Whatman Pore size 0.4 um Ref. VWR 515-2030	Pall Ref. VWR 516-7991	Whatman Pore size 0.4 um Ref. VWR 515-2030
	225 filters (45MEx5)	225 filters (45MEx5)	225 filters (45MEx5)	225 filters (45MEx5)
PM2.5	**Quartz**	**Teflon**	**Quartz**	**Teflon**
	47 mm	46.2 mm	47 mm	46.2 mm
	Pall Ref. VWR 513-0028	Whatman 2um PTFE PP ring supported filter Ref. VWR 513-0118	Pall Ref. VWR 513-0028	Whatman 2um PTFE PP ring supported filter Ref. VWR 513-0118
	225 filters (45MEx5)	225 filters (45MEx5)	225 filters (45MEx5)	225 filters (45MEx5)
Measurements	Mass	Mass	Mass	Mass
	EC/OC	elements	EC/OC	elements
	PAH		PAH	
Study of the granulometry of the particles				
PEM PM10	PTFE		n/a	
	37 mm			
	SKC Pore size 2 um, Cat no. 225-1709			
	9 filters (3schools+3homes+3outdoor)			
PEM Cascate	PTFE		n/a	
	25 mm			
	SKC Pore size 2 um, Cat no. XXX-XXXX			
	9 filters (3schools+3homes+3outdoor)			
	PTFE		n/a	
	37 mm			
	SKC Pore size 2 um, Cat no. XXX-XXXX			
	36 filters (3schools+3homes+3outdoor)x4fractions			
Measurements	Mass + Elements + PAHs			

Table 3 presents the specifications of the filters that will be used in the LIFE Index-Air sampling campaigns to be performed in transports.

Table 3: Specifications of the filters to be used in the sampling campaigns to be performed in transports.

	Indoor (3 taxis and 2 buses)		Outdoor (3 taxis and 2 buses)	
	PEM #1	**PEM #2**	**PEM #3**	**PEM #4**
	PM10	**PM2.5**	**PM10**	**PM2.5**
Personal Samplers with SKC Pumps	**Teflon**	**Teflon**	**Teflon**	**Teflon**
	37 mm	37 mm	37 mm	37 mm
	SKC Pore size 2 um Cat no. 225-1709	SKC Pore size 2 um Cat no. 225-1709	SKC Pore size 2 um Cat no. 225-1709	SKC Pore size 2 um Cat no. 225-1709
	10 filters	10 filters	10 filters	10 filters
Measurements	Mass	Mass	Mass	Mass
	EC/OC	EC/OC	EC/OC	EC/OC
	Elements	Elements	Elements	Elements
	PAH	PAH	PAH	PAH

In Portugal all the specified filters can be bought to the companies José Manuel Gomes dos Santos, Lda and VWR.

4.2 Filters coding

The identification of samples is performed according with Table 4 and Table 5.

Table 4: Filters coding for Homes and Schools.

		PM2.5-10 (C – Coarse)			PM2.5 (F – Fine)			PM10 (T-Total)		
		Quartz (Q)	Nuclepore (N)	Teflon (T)	Quartz (Q)	Nuclepore (N)	Teflon (T)	Quartz (Q)	Nuclepore (N)	Teflon (T)
									Membrane (M)	
Schools (S&)	Indoor (I)	C-S&-IQ##	C-S&-IN##	n.a.	F-S&-IQ##	n.a.	F-S&-IT##	T-S&-IQ##	T-S&-IM##	
	Outdoor (O)	C-S&-OQ##	C-S&-ON##	n.a.	F-S&-OQ##	n.a.	F-S&-OT##	T-S&-OQ##	T-S&-OM##	
	PERSONAL (P)	n.a.	n.a.	n.a.	n.a.	n.a.	PF -S&%##	n.a.	n.a.	PT-S&%##
	PEM Cascate (CAS)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	CAS-!-S&##
Homes (H&%)	Indoor (I)	C-H&%-IQ##	C-H&%-IN##	n.a.	F-H&%-IQ##	n.a.	F-H&%-IT##	T-H&%-IQ##	T-H&%-IM##	
	Outdoor (O)	C-H&%-OQ##	C-H&%-ON##	n.a.	F-H&%-OQ##	n.a.	F-H&%-OT##	T-H&%-OQ##	T-H&%-OM##	
	PERSONAL (P)	n.a.	n.a.	n.a.	n.a.	n.a.	PF -H&%##	n.a.	n.a.	PT -H&%##
	PEM Cascate (CAS)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	CAS-!-H&%##

(& - ID of the school from A to E; % - ID of the home from 1 to 8 indexed to the ID of the school &; ! - ID of the Cascate cassette from A to E indexed to the ID of the school & and indexed to the ID of the home %).

Table 5: Filters coding for Transports.

		PM2.5 (F – Fine)	PM10 (T-Total)
		Teflon (T)	Teflon (T)
Transports (MT$)	Indoor (I)	F-MT$-IT##	T-MT$-IT##
	Outdoor (O)	F-MT$-OT##	T-MT$-OT##
Transports (MB?)	Indoor (I)	F-MB?-IT##	T-MB?-IT##
	Outdoor (O)	F-MB?-OT##	T-MB?-OT##

($ - ID of the taxi from A to C; ? - ID of the bus from A to B)

4.3 Materials for filters preparation

- 1000 petri-slides
- Laboratory Grade Aluminum Foil 99.0-99.5% in dispenser, 15μm, 120m length and 280mm width
- Apiezon L (tube of 50 g)
- Isooctane
- Beaker
- 200 ml graduated cylinder
- At least 3-digit balance
- Sonicator
- Small paint brush

5. Methodology

Coating the impactor substrate with a thin film of grease reduces the bounce of particles during impaction. In the current study, Apiezon L grease will be used as antibounce coating. In order to obtain a thin film, a coating mixture will be prepared, by dissolving this material in a solvent. Since antibounce coatings contain organic compounds, this procedure will not be applied to the quartz fibre filters which will be analyzed for organic carbon and PAHs. No pre-treatment is needed for the quartz fibre filters.

5.1 Preparation of coating mixture

A mixture of 10% Apiezon L and 90% of isooctane will be prepared. For a mixture of 200 ml, the amount of Apiezon L needed is calculated equal to 15.34 gr. Apiezon L is placed into a pre-weighed beaker and the necessary amount is weighed in the 3-digit balance. 200 ml of isooctane are measured in the graduated cylinder and are added into the beaker. The mixture is sonicated for 10 minutes until homogenization is achieved.

5.2 Preparation of policarbonate nuclepore filters for PM2.5-10 sampling

The 25-mm polycarbonate nuclepore filters which will be used in the 1st stage of the impactor (for the collection of $PM_{2.5-10}$) will be coated with the Apiezon L-Isooctane mixture in order to reduce the bounce of particles during impaction. A thin layer of the antibounce mixture will be applied with a paint brush (Figure 1). This procedure is performed in the fume hood, in order to ensure that the filters are not contaminated during their exposure in the laboratory environment. The filters should remain inside the fume hood for a period of 30 minutes. They are then placed inside the petri dishes and are ready for weighing and deployment in the field. It has to be noted that the application of the antibounce mixture alters the mass of the filters, as well as their chemical composition.

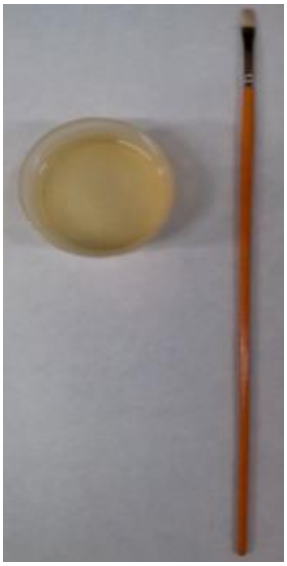

Figure 1: Preparation of policarbonate filters for PM2.5-10 sampling.

Procedure INDEX-2: Gravimetric analysis of the filters

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the gravimetric analysis of Teflon (PTFE), Quartz and Policarbonate Nuclepore filters that will be used in the sampling campaigns to be performed in 40 homes, 5 schools and 5 vehicles.

This activity will be performed in Campus Tecnológico e Nuclear (CTN) from Instituto Superior Técnico (IST).

2. Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Vânia Martins, Tiago Faria, Marina Almeida-Silva and Isabel Dionísio under the supervision of Marta Almeida.

3. Requirements and conditions of application

3.1 General good practice

- The filters will be prepared in the clean laboratory located in the Physics Department of CTN, which is a class 1000 clean laboratory equipped with a class 100 laminar flow chamber.
- The laboratory should be reserved in advanced by using the Log Sheets located in the antechamber.
- The technicians should use a clean Lab Coat and wear gloves without powder.
- Filters should be handled with care to avoid possible contamination and / or loss of material.
- Whenever a filter box is opened, the date of the opening should be recorded in the box and three filters must be removed for blank analysis.
- The identification of samples should be performed according with Table 6 and Table 7.

Table 6: Filters coding for Homes and Schools.

		PM2.5-10 (C – Coarse)		PM2.5 (F – Fine)		PM10 (T-Total)		CASCATE (CAS)	Personal (PF)	Personal (PT)
		Quartz (Q)	Nuclepore (N)	Quartz (Q)	Teflon (T)	Quartz (Q)	Teflon (M)	Teflon (T)	Teflon (T)	Teflon (T)
Schools (S&)	Indoor (I)	C-S&-IQ##	C-S&-IN##	F-S&-IQ##	F-S&-IT##	T-S&-IQ##	T-S&-IM##	CAS-!-S&##	PF -S&%##	PT- S&%##
	Outdoor (O)	C-S&-OQ##	C-S&-ON##	F-S&-OQ##	F-S&-OT##	T-S&-OQ##	T-S&-OM##	n.a.	n.a.	n.a.
Homes (H&%)	Indoor (I)	C-H&%-IQ##	C-H&%-IN##	F-H&%-IQ##	F-H&%-IT##	T-H&%-IQ##	T-H&%-IM##	CAS-!-H&%##	PF - H&%##	PT - H&%##
	Outdoor (O)	C-H&%-OQ##	C-H&%-ON##	F-H&%-OQ##	F-H&%-OT##	T-H&%-OQ##	T-H&%-OM##	n.a.	n.a.	n.a.

- & - refers to the schools ID (ranging from A to E)
- % - ID of the home (ranging from 1 to 8) indexed to the ID of the school &
- filters Membrane (M) are not a type of sampled filters but the mass sum of coarse and fine fractions using membranes filters (Nuclepore + Teflon)
- ! – refers to the sampled fractions using CASCATE device: I – PM10; II – PM10-2.5; III - PM2.5-1.0; IV - PM1.0-0.5; V - PM0.25-0.5; VI - <PM0.25
- ## - sequential numbering of all sampled filters on a specific setting

Table 7: Filters coding for Transports.

			PM2.5 (F – Fine)	PM10 (T-Total)
			Teflon (T)	
Transports	Taxi (MT$)	Indoor (I)	F-MT$-I##	T-MT$-I##
		Outdoor (O)	F-MT$-O##	T-MT$-O##
	BUS (MB?)	Indoor (I)	F-MB?-I##	T-MB?-I##
		Outdoor (O)	F-MB?-O##	T-MB?-O##

- $ - ID of the taxi from A to C
- ? - ID of the bus from A to B

4. Instruments and materials

- Mettler Toledo scale, model UMT5, with reading accuracy of 0.1 µg
- 450 Quartz filters 25 mm
- 450 Quartz filters 47 mm
- 450 Nuclepore filters 25 mm
- 450 Teflon filters 46.2 mm
- 1000 Petri dishes 47 mm
- Laboratory Grade Aluminum Foil 99.0-99.5% in dispenser, 15µm, 120m length and 280 mm width
- Sheet with known weight
- Scalpel
- Plastic tweezers
- Aluminum foil
- Ethyl alcohol

5 Methodology

5.1 Preparation of material

- A scalpel and a plastic tweezers should be previously cleaned with alcohol and dried inside the laminar flow chamber.
- Filters should be weighted on a square of aluminum foil with 5 cm * 5 cm that are cut with a scalpel. The squares of aluminum foil should be cleaned with alcohol and dried inside a petri dish in the laminar flow chamber.

5.2 Filter weighing

- Filters should be weighted before and after sampling.
- By pressing the «On/Off» key switch the balance from standby to the weighing mode. The balance automatically performs a brief self-test and all display segments light up briefly. At the end of the self-test, the balance determines the zero point. This very precise measurement takes several seconds, depending on the stability and acclimatization of the balance.
- Open the door of the balance by pressing the key ◯ and insert a clean and dry aluminum square inside the balance that will work as a plate.

- Set the balance to zero (tare) by pressing the Re-Zero key. The fully automatic door function closes the draft shield. As soon as the warning beep sounds, zeroing of the balance is complete and the fully automatic door function opens the draft shield.
- Load the weighing sample and press the «Print» key. The fully automatic door function closes the draft shield.
- The triangle symbol (print symbol) and the circle symbol of the stability detection (ASD) appear in the display.
- When the symbol of the stability detection (ASD) fades, the warning beep sounds; the triangle symbol also fades and the draft shield opens automatically and the weighing result is automatically printed out.
- The first sample to be weight is a small sheet with known weight for quality control.
- Each sample is weighted three times and the differences between the masses should be lower than 5µg.
- For switching off the balance, lift up the «On/Off» key briefly from below. This closes the draft shield automatically if the fully automatic door function is switched on. Otherwise, close the draft shield manually to prevent the ingress of dust and dirt.

5.3 Filter storage and transport

- Before sampling, store the weighted filters in petri dishes, for Teflon and polycarbonate filters, and Laboratory Grade Aluminum Foil, for quartz filters, identified with the reference of the filter.
- After sampling store the filters in the respective petri dishes or aluminum foil, stabilize the filters during 24h in the laboratory and then proceed with the weighting;
- Store the weighted filters in the respective petri dishes or aluminum foil and place them in the freezer (-10 to -20ºC).
- The filters should be sent to Greece not more than 2 weeks after sampling.
- Ice packs should be used during the transport of filters from Portugal to Greece, in order to avoid losses due to volatilization.

5.4 Records

- All the weights are registered automatically by the printer but the reference of the filter should be written with a pen.

- The sheet coming out of the printer should be stored in the dossier “LIFE Index-Air: Filter weighing”.
- The weights are registered in the excel files “LIFE Index-Air_Filter mass concentrations_Homes.xls”, “LIFE Index-Air_Filter mass concentrations_Schools.xls” and “LIFE Index-Air_Filter mass concentrations_Transports.xls”.

6. Documented information

- The manual of the balance is in dossier Equipment 1 located in storage room;
- LIFE Index-Air_Filter mass concentrations_Homes.xls
- LIFE Index-Air_Filter mass concentrations_Schools.xls
- LIFE Index-Air_Filter mass concentrations_Transports.xls

Procedure INDEX-3: Leckel MVS6 operation

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the sampling procedure using the Leckel MVS6 sampler.

2. Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Tiago Faria and Vânia Martins under the supervision of Marta Almeida.

3. Requirements and conditions of application

3.1. General good practice

- This procedure should be made with gloves and with careful during the handled of filters to avoid possible contamination and / or loss of sample.

4. Instruments and materials

- Leckel MVS6 sampler
- PM10 Head (Figure 2)

Coarse particle collector, developed by NCSR-D, composed by the parts presented in

Figure 2: PM10 lead with coarse particle collector developed by NCSR-D.

- Table 8.
- Gloves
- Filters

Figure 2: PM10 lead with coarse particle collector developed by NCSR-D.

Table 8: Parts of the coarse particle collector

Part	*Photo*	*No.*	
O' ring		4	
25 mm Teflon ring filter holder		4	
upper base		1	
impaction stage - filter holder		1	

4.1. Leckel MVS6 sampler characteristics

This model is equipped with a 6m^3/h-vacuum pump.

Technical data	MVS6
	Uncontrolled approx. 5 m^3/h Controlled 2,3-2,7-3,0-3,5 m^3/h and standard-m^3/h

	Deviation from the set point: < 2%
	Sampling time
	Minimum 1 h – maximum 999 h
	Power supply
	230 V, 50/60 Hz
	Consumption
	MVS6 approx. 300 VA
	Filter diameter
	47 mm and 50 mm
	Diameter of loaded filter surface
	approx. 40 mm
	Dimensions
	Width 310 mm – Height 480 mm – Depth 250 mm
	Weight
	MVS6 approx. 23 kg
	Noise level according to DIN 2058
	<< 35 dBA

Figure 3: Leckel MVS6 characteristics

5. Methodology

5.1 Loading and unloading the filters

Loading the filters

Figure 4: Step L1 - Load the Teflon filter holder of 47 mm diameter and put it in the lower base. Then attach the impaction stage-filter holder on the 47-mm Teflon filter holder.

Figure 5: Step L2 - Place the 25-mm diameter filter on the top of the impaction stage. Be careful to place it exactly at the center. Then press the 25-mm Teflon ring on it.

Figure 6: Step L3 - Attach the upper base of the impaction stage and secure the lower clips. Put the PM10 head on, and secure the upper clips.

Unloading the filters

Figure 7: Step U0 - Initially ensure that the place and conditions for unloading the filters are suitable. A pair of gloves, forceps, two petri dishes, a filter registration protocol and a clear surface for the filter change, are necessary for filter unloading.

Figure 8: Step U1 - Open the upper clips and remove the PM10 head from the filter holders.

Figure 9: Step U2 - Open the lower clips and remove the upper base of the impaction stage. From this point wear gloves for the filter unloading.

Figure 10: Step U3 - Take out the Teflon ring filter holder with the filter. Press gently the Teflon, in a way that the filter can be separated from the ring and then collect the 25-mm filter.

Figure 11: Step U4 - Remove the impaction stage; after that you have access to the 47-mm filter holder.

General guidelines

A. B. C.

Figure 12: A. The set of the NCSR 'Demokritos' coarse particle PM10-2.5 collector, B. spare parts, C. O' ring proper placement at the upper base of the construction.

The construction consists of four parts as seen at Table 8 and Figure 12A above. The main part is the impaction stage – filter holder. This part is designed to collect the particles larger than 2.5 microns (PM10-2.5), for the nominal sampler flow (2.3 LPM). The air passes through its four holes, and the PM2.5 aerosol is sampled at the stage below, onto the 47-mm filter. A Teflon ring is used to hold the 25-mm filter for the collection of coarse particles (PM10-2.5). The upper base is used as inlet for the collection of the PM10-2.5 fraction. The six holes on it should always be clean and open. The upper base has an O' ring that should be always at the correct position (as shown in Fig. 9C) in order to ensure sealing of the sampling system. There are also two O' rings at the Leckel parts, at the head and the lower base, that should be in good condition and placed correctly in order to seal properly. Spare parts are also provided: 5 pieces of O' rings and 5 pieces of 25-mm teflon rings as seen in Table 8 and Figure 12B.

5.2 Programming the sampler

A) SET CLOCK ⬅

1 – Press the grey key.

2 – This menu feature is used only for setting the internal clock's time (HRS/MIN), year (YEAR), and date (DAY/MONTH). The clock has already been correctly set by the manufacturer. For checking the clock see menu feature 2) START BY CLOCK.

Note: If you do not wish to change these settings, choose another menu function by using the white cursor keys.

SETTING THE CLOCK

Press the grey key.

The parameters "HRS/MIN" AND YEAR are automatically set to zero, the parameter "DAY/MONTH" is set to 01.01.

Display Line 2: Set **HRS/MIN** using the white cursor keys.

Press key for a short time:
Display will advance step by step.
Hold down key:
Display will continuously advance, first slowly, the rapidly.
Confirm by pressing the grey key.

Display Line 2: Set **YEAR** using the white cursor keys.

Press key for a short time:
Display will advance step by step.
Hold down key:
Display will continuously advance, first slowly, the rapidly.

Confirm by pressing the grey key.

Display Line 2: Set **DAY/MONTH** using the white cursor keys.

Press key for a short time:
Display will advance step by step.
Hold down key:
Display will continuously advance, first slowly, the rapidly.
Confirm by pressing the grey key.

Display Line 2: **SAVE? NO** ⬅

If you do not wish to save the new settings, just press the grey key. The old settings will remain in the system's memory, the new ones will be deleted. If you would like to save the new settings, press the upper white cursor key.

Display Line 2: **SAVE? YES** ⬅
Confirm by pressing the grey key.

B) Flow rate ⬅

Press the grey key.

Display Line 2: Nm^3/h ? **NO** ⬅
If you don't wish to operate the sampler in Nm^3/h, press the grey key.

Display Line 2: **2.3 m^3/h** ⬅
Select he flow rate by using the white cursor keys.
Very Important: select 2.3 m^3/h

Confirm input of the new (or the unchanged old) flow rate by pressing the grey key.

C) SAMPLING ⬅

Press the grey key.

Display Line 2: Interval ? **NO** ⬅
If you don't wish to operate the sampler in intervals press the grey key.

Display Line 2: **DAY/MONTH:** Set using the white cursor keys.

Press white key for a short time:
Display will advance step by step.

Hold down key:
Display will continuously advance, first slowly, the rapidly.
Confirm the date desired for sampling by pressing the grey key.

Display Line 2: **HOUR/MIN:** Set using the white cursor keys.
Usually the HOUR/MIN is set to 0.00

Press white key for a short time:
Display will advance step by step.
Hold down key:
Display will continuously advance, first slowly, the rapidly.
Confirm the starting time desired for sampling by pressing the grey key.

Display Line 2: **RUN TIME:** Set using the white cursor keys.
Usually the RUN TIME is set to 24h

Press white key for a short time:
Display will advance step by step.
Hold down key:
Display will continuously advance, first slowly, the rapidly.
Confirm the time desired for sampling by pressing the grey key.

Select the option '2) Start by clock' using the white cursor keys.

Press the grey key.

Procedure INDEX-4: SKC Pump operation

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the sampling procedure using the SKC Pump and the PEM for PM10 and PM2.5 and cascade.

2. Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Tiago Faria and Vânia Martins under the supervision of Marta Almeida.

3. Requirements and conditions of application

3.1. General good practice

- This procedure should be made with gloves and with careful during the handled of filters to avoid possible contamination and / or loss of sample.

4. Instruments and materials

- SKC Pump (4)
- PEM PM10 (10LPM 10µm) (2)
- PEM PM2.5 (2)
- PEM Cascate (Sioutas impactor 225-370) (1)
- Gloves
- Filters (PTFE 2 µm with 37 mm and 25 mm)
- Screwdriver
- Flowmeter (MesaLabs Bios Defender 510)

4.1. SKC Pump sampler characteristics

Technical data	
	Sampling time
	Minimum 1 min – maximum 1800 min
	Power supply
	230 V, 50/60 Hz
	Flow Range
	5 to 15 L/min
	Battery Recharge Time
	15 h
	Dimensions
	Width 200 mm – Height 100 mm – Depth 70 mm
	Weight
	approx. 1 kg
	Noise level according to DIN 2058
	52 dBA

Figure 13: SKC Pump characteristics

4.2. PEM PM10 and PM2.5 characteristics

Technical data	
	Dimensions of the filter
	37 mm
	Type of the filter
	2.0 µm PTFE
	Plastic tube
	Always connected with the PEM

Figure 14: PEM characteristics

4.3. PEM with cascade characteristics

Technical data	Dimensions of the filter
	Five impaction stages: — A: PM10-2.5: 25 mm — B: PM2.5-1.0: 25 mm — C: PM1.0-0.5: 25 mm — D: PM0.25-0.5: 25 mm — E: <PM0.25: 37 mm
	Type of the filter
	2.0 µm PTFE
	Plastic tube
	Always connected with the Pump (in order to minimize the any damage with the pump connection)

Figure 15: Cascade impactor characteristics

4.4. Flowmeter characteristics

Technical data	Flow Ranges
	— Low (L) Flow: 5 to 500 mL/min — Medium (M) Flow: 50 - 5,000 mL/min — High (H) Flow: 300 - 30,000 ml/min
	Accuracy
	Volumetric ±1.0%
	Approximate Time per Reading
	1-15 seconds
	Reading Styles
	Single (manual), Continuous or Burst, with averaging function user-selectable from 1 to 100 measurements
	Dimensions
	Width 150 mm – Height 140 mm – Depth 75 mm
	Weight
	approx. 820 g

Figure 16: Flowmeter characteristics

5. Methodology

5.1 Preparation of the filters for PM10 and PM2.5

1 – Prepare the filters according to INDEX-1 procedure;

2 – Put the gloves and clean the PEM with alcohol;

3 – Leave the PEM in a clean place to dry (at least 1 h);

4 – Open the PEM with the screwdriver and put the weighted filter PTFE with 37 mm on the grid;

Figure 17: Preparation of filters for PEM

5 – Close the PEM with the screwdriver, attached the plastic tube and connect it to the SKC pump:

Figure 18: PEM and SKC pump

5.2 Programming Sequences of the SKC pump for PM10 and PM2.5 sampling

1 – Turn on the pump

Press any button

2 – Set up flow rate

Press [▲▼], then *▲▼ *. Flow rate and set flash. Press ▲ or ▼ to change flow rate – **10 L/min**
Press * until END appears then press [▲▼] to save setting and place pump in Hold.

3 – Define the sampling time

Press [▲▼], then *▲▼ *. Press * until ST L/min display then press ▲ to change flashing digit; press * until END appears then press [▲▼] to save new setting.

5.3 Preparation of the filters for Cascade

1 – Prepare the filters according to INDEX-1 procedure;

2 – Put the gloves and clean the PEM Cascate with alcohol;

3 – Leave the PEM Cascate in a clean place to dry (at least 1 h);

4 – Open the PEM Cascate rolling the metal nuts and separating each level (from A [top] to D [bottom]);

Figure 19: Cascade impactor parts 1

5 – Put one weighted filter PTFE with 25 mm in each level hold with a plastic circle and on the bottom of the PEM cascade placed the weighted filter PTFE with 37 mm;

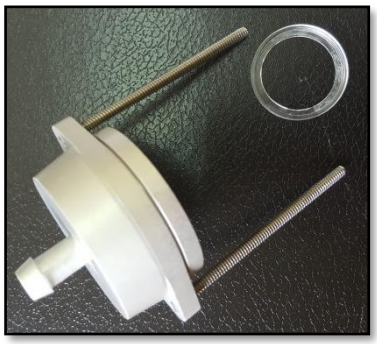

Figure 20: Cascade impactor parts 2

6 – Close the PEM cascade, attach the plastic tube and connect it to the SKC pump.

Figure 21: Cascade impactor and SKC pump

5.4 Programming Sequences of the SKC pump for cascate sampling

1 – Turn on the pump

Press any button

2 – Set up flow rate

Press [▲▼], then *▲▼ *. Flow rate and set flash. Press ▲ or ▼ to change flow rate – **9 L/min**
Press * until END appears then press [▲▼] to save setting and place pump in Hold.

3 – Define the sampling time

Press [▲▼], then *▲▼ *. Press * until ST L/min display then press ▲ to change flashing digit;
press * until END appears then press [▲▼] to save new setting.

5.5 Calibrate SKC pump flow rate with the Sioutas Impactor

1 – Using flexible tubing, connect the outlet of the Sioutas Impactor to the inlet of the SKC pump

2 – Connect the inlet of the Sioutas Impactor to the outlet (suction fitting) of the flowmeter, using a flexible tube

Figure 22: Sioutas Impactor in calibration train with SKC pump and Defender 510 Flowmeter

3 – Turn on the flowmeter and select the measurement type "Single" then press "Enter"

4 – Set up the sample flow in the SKC to 9L/min

Press any button

Press [▲▼], then press the security code*▲▼* in sequence. The flow rate and set will flash.

Set the flow on the pump display by pressing ▲ or ▼ to change flow to the desired rate – **9 L/min**

Press *. Adj will appear

5 – Flowmeter reads

Each time the "Enter" button of flowmeter is pressed, one measurement will be made

6 – Flow adjustment

If the flowmeter reads a higher flow rate than the pump is set for, press ▼ in the pump until they are in agreement. If the flowmeter reads a lower flow rate, press ▲ in the pump until they agree. When pressing ▲ or ▼, the pump display will indicate the adjustment (or correction) made in L/min.

7 – Save settings in the pump

Press * until END appears then press [▲▼] to save flow rate and Adj and exit Setup.

Procedure INDEX-5: PM sampling in schools and homes

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the air quality monitoring campaigns to be performed in 40 homes and 5 schools.

2. Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Tiago Faria, and Vânia Martins under the supervision of Marta Almeida.

3. Instruments and materials

For the sampling campaign performed in each micro-environment the following material should be organized:

Equipment

- 4 Leckel MVS6
- 4 Samplers heads
- 1 DustTrack 8533

Plastic Box with

- Filters: 10 quartz 47mm + 10 PTFE 46.2 mm + 10 quartz 25 mm + 10 nuclepore 25 mm
- Tweezers
- Gloves
- Scissors
- 4 Log sheets: Leckel 1 – Volume; Leckel 2 – Volume; Leckel 3 - Volume and Leckel 4 – Volume;
- 1 Log sheet: ME Characteristics;
- 1 Log sheet: Direct reading equipment;
- 1 Log sheet: ME users diary;
- 1 pen
- 3 Electrical extensions
- 1 timer

4. Methodology

4.1 Selection of the schools and homes

- The sampling campaigns will be performed in 5 schools and 40 homes. 8 homes were selected per school as shown in Figure 23.

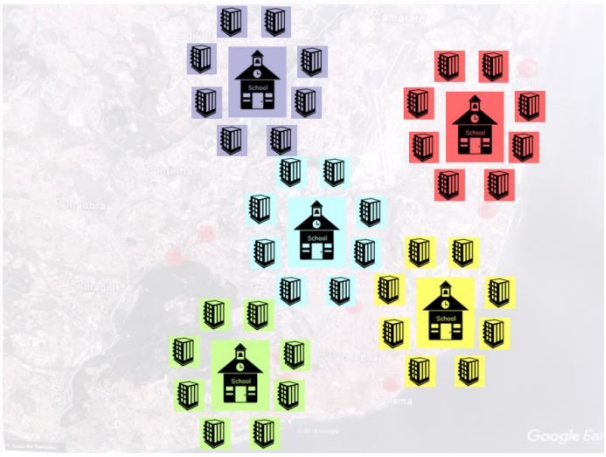

Figure 23: Selection of schools and homes in Lisbon.

- The 5 schools were selected according with:
  - Location (parishes with more children and parishes with more children per m^2);
  - Year of construction;
  - Type of ventilation;
  - Interest of the schools.
- Each school has an ID number presented in **Error! Reference source not found.**.
- The 8 homes per school (total: 40) were selected according with:
  - Location (close to the school);
  - Year of construction;
  - Type of construction;
  - Interest of the parents.

4.2 Sampling periods

- One week sampling campaign will be carried out in each Micro-Environment (5 schools and 40 homes).

Schools

- The sampling will be performed simultaneously indoor (in 1 classroom) and outdoor (in the playground of the school).
- All schools will be monitored during 5 days - from Monday to Friday.
- Sampling will be performed during 7 hours during the occupied time – from 9am to 4pm.

- The samplers should be installed in the school Monday morning. All mornings from Monday to Friday one sampling period will start and timers should be used to finish the sampling period at the end of the day. Friday the equipment should be removed from the school at the end of the day.

Homes

- The sampling will be performed simultaneously indoor (in the living-room) and outdoor (in the playground of the correspondent school).
- All homes will be monitored during 5 days - 4 days during the week and one day during the weekend.
- Sampling will be performed during 11 hours during the occupied time – from 8pm to 7am.
- The samplers should be installed in the homes Monday afternoon. Every day filters should be replaced by new ones and the timers should be used to start and finish a new sampling period. Friday the timer should be used to start the sampling during the weekend. Monday the equipment should be removed from the home.

4.3 Equipment

Schools

- PM2.5 and PM2.5-10 samples will be collected using 4 Leckel MVS6 that will operate with 2.3m^3/h.
- Details on the Leckel MVS6 sampler operation should be consulted in the Procedure INDEX-3.
- Four samplers will work in parallel: 2 installed in the indoor and other 2 installed in the outdoor (Figure 24).
- Two samplers (one installed in the indoor and the other in the outdoor) will work with quartz filters. PM2.5-10 will be sampled in 25 mm filters and PM2.5 will be sampled in 47 mm filters. Before and after sampling these filters will be weighted and stored according with Procedure INDEX-2.
- The other two samplers (one installed in the indoor and the other in the outdoor) will work with Nuclepore and PTFE filters. PM2.5-10 will be sampled in 25 mm Nuclepore fiters and PM2.5 will be sampled in PTFE 47 mm filters. Before and after sampling these filters will be weighted and stored according with Procedure INDEX-2.

- In the indoor of 3 schools one Cascade Impactor Sioutas will collect, in Teflon filters, airborne particles in five size ranges: > 2.5 µm, 1.0 to 2.5 µm, 0.50 to 1.0 µm, 0.25 to 0.50 µm, and < 0.25 µm. Cumulative sampling will be performed during five days only in the occupied period (from 9:00 to 16:00). Details on Cascade Impactor Sioutas operation can be consulted in Procedure INDEX-4.
- In the same 3 schools one DustTrack 8533 will be used to measure real-time mass concentration of PM10 and PM2.5. In each Micro-Environment sampling will be performed during five days. The measuring interval will be 1 minute.
- Indoor instruments will be located in the classroom next to the opposite wall from the blackboard, to avoid direct exposure to chalk or maker's emission, and from the window, to avoid direct outdoor levels interference and disturbances resulting from air currents.
- The outdoor monitoring station will be located in the everyday playground when children usually spent their breaks.
- ***Log sheets Leckel 1 – Volume; Leckel 2 – Volume; Leckel 3 - Volume and Leckel 4 – Volume*** should be used to register the sampling data.
- ***Log sheet – Direct reading equipment*** should be used to register the measuring data.

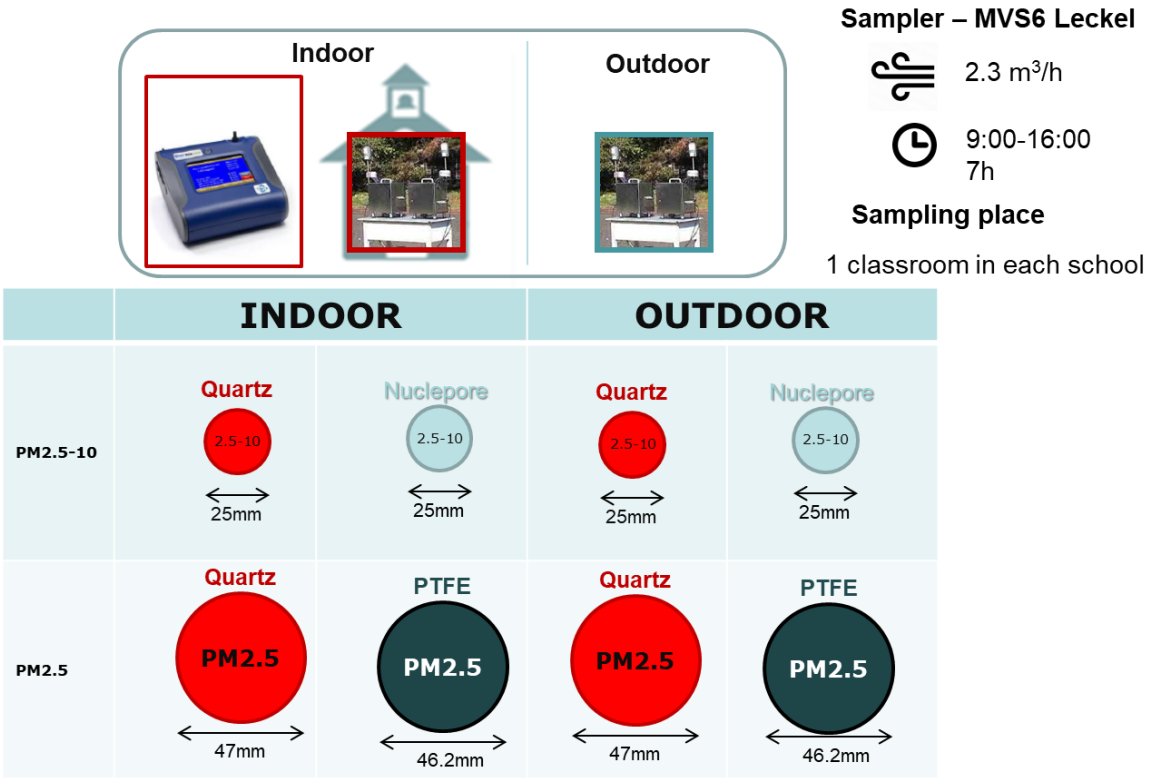

Figure 24: Sampling procedure to be used in schools.

Homes

- PM2.5 and PM2.5-10 samples will be collected using a Leckel MVS6 that will operate with 2.3m^3/h.
- Details on the Leckel MVS6 sampler operation should be consulted in the Procedure INDEX-3.
- Four samplers will work in parallel: 2 installed in the indoor and other 2 installed in the outdoor (Figure 25).
- Due to logistical constrains (power and security), the samplers that will assess the outdoor concentrations of the homes will be installed in the in the playground of the correspondent school.
- Two samplers (one installed in the indoor and the other in the outdoor) will work with quartz filters. PM2.5-10 will be sampled in 25 mm filters and PM2.5 will be sampled in 47 mm filters. Before and after sampling these filters will be weighted and stored according with Procedure INDEX-2.
- The other two samplers (one installed in the indoor and the other in the outdoor) will work with Nuclepore and PTFE filters. PM2.5-10 will be sampled in 25 mm Nuclepore filters and PM2.5 will be sampled in PTFE 47 mm filters. Before and after sampling these filters will be weighted and stored according with Procedure INDEX-2.
- In the indoor of 3 homes one Cascade Impactor Sioutas will collect, in Teflon filters, airborne particles in five size ranges: > 2.5 µm, 1.0 to 2.5 µm, 0.50 to 1.0 µm, 0.25 to 0.50 µm, and < 0.25 µm. Cumulative sampling will be performed during five days only in the occupied period (from 20:00 to 07:00). Details on Cascade Impactor Sioutas operation can be consulted in Procedure INDEX-4.
- In the indoor of the same 3 homes, one DustTrack 8533 will be used to determine real-time mass concentration of PM10 and PM2.5. In each Micro-Environment sampling will be performed during five days.
- Indoor instruments will be located in the living-room next to the opposite wall from a possible fireplace, to avoid direct exposure to pollutant's emission, and from the window, to avoid direct outdoor levels interference and disturbances resulting from air currents.
- ***Log sheets Leckel 1 – Volume; Leckel 2 – Volume; Leckel 3 - Volume and Leckel 4 – Volume*** should be used to register the sampling data.
- ***Log sheet – Direct reading equipment*** should be used to register the measuring data.

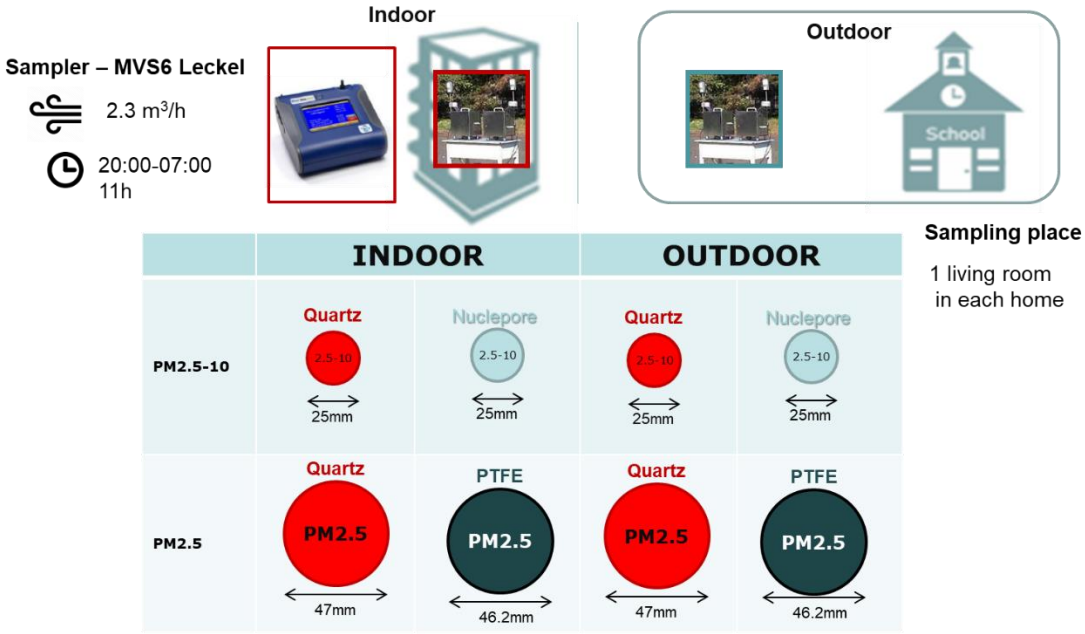

Figure 25: Sampling procedure to be used in homes.

4.3 Additional information to be collected

Information regarding schools and homes characteristics should be collected using the ***Log Sheet ME Characteristics***:

- Building construction year
- Type of ventilation
- Floor
- Area of the room
- Windows material
- Windows orientation (Interior: room windows face to an interior patio, totally surrounded by buildings; Playground: classroom window face a playground which is next to the street; Directly street: room windows face directly to the street)
- Floor material
- Existence of fireplace, candles, incenses, smokers, gas or electric cooker, animals, carpets
- Type of playground (paved vs sand-filled)
- Orientation of the playground (Interior – the playground is completely surrounded by buildings; Street - the playground is partially or totally opened to street)
- Type of the marker used in the blackboard
- Specific sources

Moreover, ME users will be asked to write down, in the ***Log Sheet Diary***, if the windows are open or closed during the measuring periods.

5. Documented information

- Log sheets - Leckel 1 – Volume; Leckel 2 – Volume; Leckel 3 - Volume and Leckel 4 – Volume – Excel files “LIFE Index-Air_Filter mass concentrations_Homes.xls” and “LIFE Index-Air_Filter mass concentrations_Schools.xls”.
- Log sheet – Direct reading equipment – Excel file “Direct reading equipment”
- Log sheet - ME Characteristics – Excel file “ME characteristics”.
- ME Users Diary – Excel file “ME characteristics”.

Procedure INDEX-6: PM sampling in mobile microenvironments

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the sampling campaigns to be performed in 5 vehicles.

2. Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Vânia Martins and Tiago Faria under the supervision of Marta Almeida.

3. Requirements and conditions of application

3.1. General good practice

- The sampling campaign will be performed in mobile micro-environments from Lisbon.
- 2 buses and 3 cars (taxis) will be selected.
- The selected mobile micro-environments should be representative of the transport used by the children in Lisbon.
- The campaign will start in May 2017 and end in June 2017
- The filters will be prepared according the experimental procedures INDEX-1 and INDEX-2.
- The personal pumps (SKC), the GPS (Garmin) and the particle counter must be charged before each utilization.

4. Instruments and materials

- 4 SKC Pump from Leland Legacy SKC battery operated
- 2 Personal Environmental Monitor (PEM) PM10 from Leland Legacy SKC
- 2 Personal Environmental Monitor (PEM) PM2.5 from Leland Legacy SKC
- 4 Plastic tubes
- GPS Garmin, rechargeable batteries operated
- Particle counter
- Miniature black carbon monitor model AE51, from TSI
- Tape
- Plastic straps
- Plastic box (25 x 25 x 10 cm)

5. Methodology

5.1 Sampling periods

The sampling campaign will be performed according the following methodology:

- The sampling period, lasting for 4 weekdays per vehicle, will consist of a daily monitoring (8h/day) from 9:00 to 17:00.
- Particles collected during Monday and Tuesday will be sampled in one filter; and particles collected during Wednesday and Thursday will be sampled in a second filter. This will allow the collection of enough mass for chemical analysis.
- The scheme of the sampling campaign is presented in the follow Table 9.

Table 9: scheme of the sampling campaign in mobile ME

	Monday	Tuesday		Wednesday	Thursday	
Week 1	9:00 – 17:00 Taxi 1	9:00 – 17:00 Taxi 1	Sampling T1	9:00 – 17:00 Taxi 1	9:00 – 17:00 Taxi 1	Sampling T2
Week 2	9:00 – 17:00 Taxi 2	9:00 – 17:00 Taxi 2	Sampling T3	9:00 – 17:00 Taxi 2	9:00 – 17:00 Taxi 2	Sampling T4
Week 3	9:00 – 17:00 Taxi 3	9:00 – 17:00 Taxi 3	Sampling T4	9:00 – 17:00 Taxi 3	9:00 – 17:00 Taxi 3	Sampling T5
Week 4	9:00 – 17:00 Bus 1	9:00 – 17:00 Bus 1	Sampling B1	9:00 – 17:00 Bus 1	9:00 – 17:00 Bus 1	Sampling B2
Week 5	9:00 – 17:00 Bus 2	9:00 – 17:00 Bus 2	Sampling B3	9:00 – 17:00 Bus 2	9:00 – 17:00 Bus 2	Sampling B4

5.2 Equipment

- A set of equipment will be installed inside the vehicles and will measure the indoor air quality of the mobile micro-environments under study.
- Parallel measurements will be performed outdoors.
- Figure 26 presents the equipments that will be used indoors and outdoors the vehicles.

Figure 26: Outline of the sampling campaign in mobile microenvironments

- The SKC pumps will be used to sample PM10 and PM2.5 in PTFE filters with 37 mm of diameter (Figure 27).

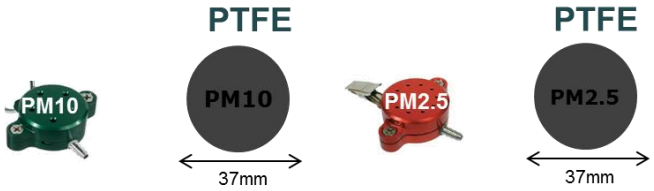

Figure 27: Filters that will be used in the sampling campaign

- For the taxis' sampling campaign the equipments will be distributed throw the front seats:
  - 4 SKC pumps will be placed in the underside of the front seats;
  - 2 PEM (1 PM2.5 and 1 PM10) will be placed near the top of the driver's seat, near the nose;
  - 2 PEM (1 PM2.5 and 1 PM10) will be placed outside the taxi, near the driver's window;
  - The particle counter, GPS and miniature black carbon monitor model AE51 will be placed between both front seats inside of a box.

- For the buses' sampling campaign the equipments will be distributed throw the driver seat:
  - 4 SKC pumps will be placed in the underside of the front seat;
  - 2 PEM (1 PM2.5 and 1 PM10) will be placed near the top of the driver's seat, near the nose;
  - 2 PEM (1 PM2.5 and 1 PM10) will be placed outside the bus, near the driver's window;
  - The particle counter, GPS and miniature black carbon monitor model AE51 will be placed in front of the driver, near the front glass.

6. Documented information

— The manual of SKC pumps is in dossier Equipment 1 located in storage room;

— The manual of the DustTrack is in dossier Equipment 1 located in storage room;

— LIFE Index-Air_Filter mass concentrations_Transports.xls.

Procedure INDEX-7: X-ray spectroscopy for the determination of trace elements

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the determination of trace elements in policarbonate nuclepore filters that will be collected in the sampling campaigns to be performed in 40 homes, 5 schools and 5 vehicles.

This activity will be performed by NCSR "Demokritos" (NCSR-D) at the Environmental Radioactivity Laboratory (ERL).

2. Responsibilities

- This procedure will be implemented by the NCSR-D LIFE Index-Air team.
- The staff involved are Dr. Manousos-Ioannis Manousakas and Dr. Athina-Cerise Kalogridis.

3. Requirements and conditions of application

3.1. General good practice

- The filters will be collected in Lisbon and will be sent to ERL by the Instituto Superior Técnico (IST) in polystyrene petri dishes.
- No pre-treatment of the filters is necessary. Prior to sampling, IST will send to ERL some clean filters from the batch of filters intended for sampling in order to assess the blank levels of the trace elements. This procedure will be repeated whenever a new batch of filters is used. The blank filters should display comparable analyte results with appropriate previous data.
- All filters will be uniquely identified and records will be kept with respect to the manufacturer, purchase date, manufacturer's batch and pack number.
- Filters should be handled with care to avoid possible contamination and / or loss of material.
- Field blanks will be collected throughout the sampling campaigns.
- All samples will be stored by IST in a refrigerator prior to sending to NCSR-D, in order to avoid losses due to volatilization.
- The exposed area of the filter should not come in contact with the petri dishes to avoid loss of sample and alteration of the sample surface.
- If only part of the sample is required for the analysis, the cutting equipment that will be used should be made of a material that leaves no residue when used and does not affect the sample, such as a stainless steel cutter or scalpel. The cutting equipment will be cleaned after every use.

4. Instruments and materials

4.1 Instrumentation

Energy Dispersive X-ray spectrometer (ED-XRF) Laboratory Instrument (Epsilon 5, PANalytical, the Netherlands).

4.2 Materials

- Gases:
    - Helium at least 99,999 % (% by volume)
- Micrommater thin standards on 6.3 µ Mylar (NaCl, MgF_2, GaP, SiO, KCl, CaF_2, V, Fe, Cr, Co, CuSx, Al, Ni, CsBr, RbI, SrF_2, Ge, Ag, Sn, Sb, Pt, AgHg, CdSe, Pb, Au, BaF_2 and Ce) for method calibration
- Thin standards on filter media (KCl, NaCl, Pb, S, Si, AlCe, multi-elemental), UC Davis, for method calibration
- NIST 2783 Air particulate on Filter media standard for method validation
- CRMs 2584 and 2583 standards despersed on filter media for method validation
- Filter cutters
- Stainless steel sample holders
- Aluminum inserts (inner cups) filter 50mm
- Stainless steel sample tray (with eight sample holder positions)
- Stainless steel tweezers for sample handling
- Clean cutting surface (Teflon sheet)
- Acetone, reagent grade
- Filtering paper

5. Methodology

5.1 Theory of operation

PM fiber filters will be analyzed by the X-ray spectrometry (XRF) for the determination of the concentration of trace elements in the sample. The instrument that is used (with Cartesian geometry) offers several advantages when compared with conventional XRF system. Simple ED-XRF spectrometers employ a two-dimensional, or direct excitation, geometry where the X-ray tube irradiating the sample and the detector recording in the spectrum, lie in the same plane. While this offers very efficient sample excitation, the recorded spectrum contains not only the sample spectrum, but also a large amount of the scattered X-ray tube spectrum. This

contributes towards relatively high background levels and negatively influences the detection limits. With a three-dimensional, or Cartesian, geometry the X-ray tube spectrum is eliminated by polarization. The resultant reduction in spectral background makes it possible for much lower detection limits to be achieved. The use of different polarizing targets (secondary targets), placed along the first axis of the optical path employing a three-dimensional geometry, offers further analytical advantages. Whereas some target materials merely scatter the X-ray tube irradiation of the sample, other materials fluoresce, yielding intense, almost monochromatic X-rays that irradiate the sample. By using targets of different materials it is possible to optimize the excitation source specifically for elements of interest. The primary beam from the X-ray tube irradiates a polarizing target placed along the first axis. After scattering at 90° the X-rays travel along the second axis to the sample. The spectrum of the sample is recorded by a detector placed along a third axis. The spectrometer provides selection of 9 secondary targets (Al, CaF_2, Fe, Ge, Zr, KBr, Mo, Al_2O_3, LaB_6) set for optimal measurement of different elemets. Every target is used in each measurement making thus sure that the detection limits are the lowest for every element.

Table 10. Instrumental parameters and measuring time per secondary target.

Secondary Target	kV	mA	Detected elements	Measurement time (sec)
Al	25	24	Na, Mg	800
CaF_2	40	15	Al, Si, P, S, Cl, K	600
Fe	75	8	Ca, Ti, V, Cr	400
Ge	75	8	Mn, Fe, Co, Ni, Cu, Zn	400
Zr	100	6	Br, Rb, Pt, Au, Hg, Pb	400
KBr	100	6	Ga, Ge, As	800
Mo	100	6	Ag, Cd, Cs, Ba, Ce	400
Al_2O_3	100	6	Sn, Sb	400
LaB_6	100	6	Sr	400

5.2 Analytical procedure

During each day of analysis, a standard procedure will be followed, in order to ensure QA/QC of each measurement:

- If the instrument is switched off, both the instrument and the PC are switched on by their master switch.
- The medium in the measuring chamber is set to vacuum or He
- The X-ray tube is switched on using the main key switch
- If the detector is at liquid nitrogen temperature can be switched on, otherwise the instrument is filled with liquid nitrogen and then the detector is switched on
- The sample holders (both outer and inner cups) are cleaned using acetone and filter paper
- Using the tweezers either the NIST 2783 or CRMs 2584 and 2583 standards despersed on filter media standard are loaded in the sample holder
- From the online measurement software the appropriate measuring application (calibration file) is selected
- The position of the samples and the measurement code is given to the software and the measurement is initiated

- After the measurement has finished the results of the standards are compared with previous measurements. If the results are inside the range of two times the standard deviation of the last 20 measurements of the standards (QA/QC chart) then the samples are loaded. If the measurements are outside this range then “Monitor” function of the instrument is initiated and the calibrations curves are corrected.
- Using the tweezers the samples are loaded in the holders and the holders to the trays and the position is recorded to the log book. A blank is loaded for every ten samples.
- The samples are measured using the same procedure as the standards
- Every measurement is repeated three times. If the standard deviation of the successive measurements is higher than 10% the results are discarded and the measurement is repeated.
- After the completion of the measurements the samples are put in their petri dishes and are stored in a refrigerator
- QA/QC requirements are checked every 20 measurements.
- After the end of each analysis day, the instrument should be put in standby mode.
- Detector calibration is performed once every week.

5.3 Data reporting

The elemental concentrations are calculated after sample analysis by running the calculation software. Concentrations are reported in μg/cm^2. The final ambient air concentrations (μg/m^3) will be calculated by multiplying with the total loaded area of the filter (in cm^2) and dividing by the sampled volume (in m^3). The detection limits of the method for some commonly measured elements are presented in Table 11 for Teflon filters.

Table 11: DLs for the measured elements

Element	LOD (ng/m^3)	Element	LOD (ng/m^3)
Na	8.9	Cr	0.2
Mg	9.4	Mn	1.4
Al	3.7	Fe	1.4
Si	16.4	Co	1.4
S	4.2	Ni	0.5
Cl	1.9	Cu	0.5
K	1.4	Zn	3.7
Ca	4.2	Br	2.3
Ti	0.9	Sr	2.3
V	1.4	Pb	1.4

The % uncertainty for each element for every measurement will be reported as well. The primary sources of measurement uncertainty that are taken into account are: Peak area uncertainty. calibration uncertainty. field. sampling and sample deposition error. attenuation of X-ray intensity for light elements and the relative standard deviation of the consecutive measurements (n=3 for each sample). Total uncertainty is calculated as the square root of the combined variances.

5.4 Records

All the raw data files will be stored in the instrument's PC in the "LIFE Index-AIR" folder. The measured elemental concentrations (in $\mu g/cm^2$ and in $\mu g/m^3$) will be recorded along with the calculations performed and the corresponding uncertainties, in the following excel files: "LIFE Index-Air_XRF concentrations_Homes.xls". "LIFE Index-Air_XRF concentrations_Schools.xls" and "LIFE Index-Air_XRF concentrations_Transports.xls".

6. Documented information

- LIFE Index-Air_XRF concentrations_Homes.xls
- LIFE Index-Air_XRF concentrations_Schools.xls
- LIFE Index-Air_XRF concentrations_Transports.xls

Procedure INDEX-8: Thermal-optical analysis for the determination of EC/OC

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the determination of elemental (EC) and organic carbon (OC) by thermal-optical analysis (TOA) of Quartz filters that will be collected in the sampling campaigns to be performed in 40 homes, 5 schools and 5 vehicles.

This activity will be performed by NCSR "Demokritos" (NCSR-D) at the Environmental Radioactivity Laboratory (ERL), Institute of Nuclear & Radiological Sciences & Technology, Energy & Safety (INRASTES).

2. Responsabilities

- This procedure will be implemented by the NCSR-D LIFE Index-Air team.
- The staff involved are Dr. Evangelia Diapouli and Dr. Prodromos Fetfatzis.

3. Requirements and conditions of application

3.1. General good practice

- The quartz filters will be collected in Lisbon and will be sent to ERL by the Instituto Superior Técnico (IST) in Laboratory Grade Aluminum Foil. Quartz fibre filters without binding materials shall be used.
- No pre-treatment of the filters is necessary. Prior to sampling, IST will send to ERL some clean filters from the batch of filters intended for sampling in order to assess the blank levels of EC and OC. This procedure will be repeated whenever a new batch of filters is used. The blank filters should display EC concentrations below the detection limit and OC concentrations on average below 2 µg/cm^2 and with a standard deviation below 1 µg/cm^2.
- All filters will be uniquely identified and records will be kept with respect to the manufacturer, purchase date, manufacturer's batch and pack number.
- Filters should be handled with care to avoid possible contamination and / or loss of material.

- Field blanks will be collected throughout the sampling campaigns. In addition, backup filters will be used during part of the sampling days (once every 10 days) in order to assess the positive artefact caused by the ab(ad)sorption of gaseous species in (on) the filters.

- All samples will be stored by IST in a refrigerator prior to sending to NCSR-D, in order to avoid losses due to volatilization.

4. Instruments and materials

4.1 Instrumentation

Organic Carbon / Elemental Carbon (OCEC) Laboratory Instrument (Model 5L, Sunset Laboratory Inc.)

4.2 Materials

- Gases:
    - Helium at least 99,999 % (% by volume)
    - Helium/oxygen (90:10) mixture with a maximum of impurities of 0.001 % (% by volume)
    - Helium/methane (95:5) with a maximum of impurities of 0.001 % (% by volume).
    - Air ("ultra-zero" or "zero" grade)
    - Hydrogen at least 99,999 % (% by volume)
- Carbon-containing (sucrose) standard solutions, with an accurately determined concentration ranging from 0.4 µg C/µl to 5 µg C/µl
- Precision filter cutter of known area (1x1.5 cm)
- Quartz boat for the filter punch
- Stainless steel tweezers for sample handling
- Silicon covered tweezers
- Clean cutting surface (e.g. aluminium foil (uncoated) or quartz fibre filter)
- Pipette for calibration using standard solutions (10 µl volume).

5. Methodology

5.1 Theory of operation

PM quartz fibre filters will be analysed by the thermo-optical method (TOA) for the determination of elemental (EC) and organic carbon (OC). According to this method, a standard sized punch is cut from the quartz fibre filter sample and is placed in a quartz oven. The oven is then purged with helium, while a stepped temperature ramp increases the oven temperature. Organic compounds and pyrolysis products thermally desorb during this stage and are led into a manganese dioxide (MnO_2) oxidizing oven, where they are quantitatively converted to CO_2 gas. The CO_2 is swept out of the oxidizing oven in the helium stream and is mixed with hydrogen gas. This mixture then flows through a heated nickel catalyst where it is

quantitatively converted to methane. The methane is subsequently measured using a flame ionization detector (FID). After the initial temperature ramp in the quartz sample oven is completed, the oven is cooled and the flow stream is switched to an oxidizing helium/oxygen carrier gas mixture. A second temperature ramp is then initiated in the oxidizing gas stream and any elemental carbon is oxidized off the filter and into the oxidizing oven. The elemental carbon is then detected in the same manner as the organic carbon. The EUSAAR2 thermal protocol will be applied (Cavalli et al., 2010), which consists of 4 temperature steps in the He atmosphere and 4 temperature steps in the He/O_2 atmosphere (Table 12).

Table 12: Temperature steps and step durations for the EUSAAR2 thermal protocol.

Mode	Step	T in °C, duration in s
He	He 1	200, 120
	He 2	300, 150
	He 3	450, 180
	He 4	650, 180
He/O_2	He	No heating, 30
	He/O_2 1	500, 120
	He/O_2 2	550, 120
	He/O_2 3	700, 70
	He/O_2 4	850, 80

In addition to this elemental carbon present in the sample, EC can be formed from some charring of organic carbon as it is pyrolyzed during the initial temperature ramp. This charring of organic carbon results in an artificially low measurement of the organic carbon and a high measurement for the original elemental carbon, if left uncorrected. Charring correction is applied by continuously monitoring the sample's transmittance throughout the heating process by the use of a red light laser. Any charring of the organic carbon results in a decrease in transmittance of the laser. After the initial temperature ramp when the helium is switched to a He/O_2 mixture, all of the elemental carbon is oxidized off and the laser transmittance is returned to the background level. When the resulting FID data are reviewed with an overlay of

the laser absorbance, the point in the second phase oxidizing ramp at which the laser transmittance equals the initial laser transmittance is the split point. Any elemental carbon detected before this point is considered to have been formed pyrolytically by charring of the organic carbon. This carbon is subtracted from the elemental carbon area observed during the oxidizing phase of the analysis and is assigned as organic carbon.

Cavalli F., Viana M., Yttri K.E., Genberg J., Putaud J.-P.: Toward a Standardized Thermal-Optical Protocol for Measuring Atmospheric Organic and Elemental Carbon: The EUSAAR Protocol, Atmos. Meas. Tech., 3, 79-89, 2010.

5.2 Analytical procedure

During each day of analysis, a standard procedure will be followed, in order to ensure QA/QC of the measured elemental and organic carbon concentrations:

- The instrument is put out of standby by pressing the "Out-of-Standby" button in the instrument's analysis software.
- Allow gases to stabilize for 10-15 seconds before igniting the FID.
- Press down on the red ignition button on the front of the FID/Methanator oven unit. Once the flame has been lit, usually signaled by a small pop, use a reflective device to observe water condensation. Then press the "OK" button in the analysis software.
- Normally a burnt punch is left inside the instrument's oven from the last analysis performed. If there is no punch inside the instrument, cut a rectangular punch of 1x1.5 cm from a clean quartz filter and place it inside the oven. The silicon covered tweezers are used to pull out and in the quartz boat where the filter punch is placed. The clean stainless steel tweezers are used for handling the samples.
- Run a clean oven sample by selecting the Total Carbon protocol.
- Without opening the instrument's oven, run an instrument blank sample with the EUSAAR2 protocol. During the analysis, check and record the following parameters: i) pressure (should be in the range 0.5-1.0 psig); ii) laser signal (should be flat during blank analysis and in the range 6000-14000), iii) temperature steps (they should follow the thermal protocol selected); iv) FID signal. After the end of analysis, record the value of FID1_Max (it should be in the range 15000-30000). In addition, run the calculation software and check that the concentration was below 0.2 µg/cm^2.
- Pull out the quartz boat and add with a pipette 10 µl of a sucrose solution of known concentration. Dry the sample by using the relevant function in the analysis software and analyze it with the EUSAAR2 protocol. The concentration should be within ± 5% of the reference value.
- Without opening the instrument's oven, run a CAL GAS sample using the CALGAS protocol. Check if the values of total carbon (TC) and EC/OC are within ± 5% and ± 3% of the reference value, respectively. The reference value has been determined through previous repetitive analyses. It has been calculated as the mean value of 10 consecutive CAL GAS analyses, which should produce results with difference no larger than ± 5%.

- Analyze one punch for a control filter sample. The OC and EC values should be within ± 10% of the reference value. The reference value has been determined through previous repetitive analyses of this control filter. It has been calculated as the mean value of 10 analyzed punches.
- If all the above QA/QC requirements are met, the instrument may be used for the analysis of field samples. A duplicate analysis of one of the samples should be performed after the analysis of 10 consecutive field samples.
- After the end of each analysis day, the instrument should be put in standby mode.

5.3 Data reporting

The elemental and organic carbon concentrations are calculated after sample analysis by running the calculation software. Concentrations are reported in µg/cm^2. The final ambient air concentrations (µg/m^3) will be calculated by multiplying the obtained concentration in µg/cm^2 with the total loaded area of the filter (in cm^2) and dividing by the sampled volume (in m^3). The detection limit of the method is 0.05 µg of carbon/cm^2.

5.4 Records

All the raw data files will be stored in the instrument's PC in the "LIFE Index-AIR" folder. The measured EC/OC concentrations (in µg/cm^2 and in µg/m^3) will be recorded, along with the calculations performed, in the following excel files: "LIFE Index-Air_OC-EC concentrations_Homes.xls", "LIFE Index-Air_OC-EC concentrations_Schools.xls" and "LIFE Index-Air_OC-EC concentrations_Transports.xls".

6. Documented information

- The manual of the (OCEC) Laboratory Instrument is available electronically.
- LIFE Index-Air_OC-EC concentrations_Homes.xls
- LIFE Index-Air_OC-EC concentrations_Schools.xls
- LIFE Index-Air_OC-EC concentrations_Transports.xls

Procedure INDEX-9: PAH analysis of the filters

1. Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the Determination of Polyaromatic Hydrocarbons (PAHs) of Quartz filters that will be used in the sampling campaigns to be performed in 40 homes, 5 schools and 5 vehicles using Gas Chromatography – Mass Spectrometry.

This activity will be performed in the Environmental Research Laboratory (EREL), Institute of Nuclear & Radiological Sciences & Technology, Energy & Safety (INRASTES), National Center for Scientific Research "Demokritos" (NCSRD).

2. Responsabilities

- This procedure will be implemented by the NCSR-D LIFE Index-Air team.
- The staff involved is composed by Dr. Bairachtari Kyriaki, Ms. Dasopoulou Maria and Dr. Maggos Thomas.

3. Requirements and conditions of application

3.1 Normative reference

The methodology of the PAHs analysis is based on the ISO 12884:2000: "*Ambient air-Determination of total (gas and particle-phase) polycyclic aromatic hydrocarbons-Collection on sorbent-backed filters with gas chromatographic/mass spectrometric analysis"*.

4. Apparatus, reagents and materials

Soxhlet exractor system, rotary evaporator, cleanup columns.

Acetone, n-hexane, Diethyl ether, Cyclohexane, Silica gel, Sodium sulfate, Deuterated PAHs standard solution, Standard PAHs solution.

4.1 Instrumentation and conditions

Polycyclic Aromatic Hydrocarbons concentrations will be determined through Gas Chromatography-Mass Spectrometric (GC-MS) analysis. Agilent Technologies 7890A GC System, 5975 C inertXL EI/CI MS Detector, provided with 7683B auto sampler will be used for PAHs determination and quantitation in PM. The gas chromatograph is equipped with split/splitless injector and HP 5MS 60mx0.25mm column with 0.25 μm thickness (Agilent Technologies). Oven temperature program is started from 60°C isothermal for 2 min, then heated up to 80°C at 25°C/min, then heated up to 300°C at 5°C/min and is kept isothermal for 5 min. The heating zones are kept at the following temperatures: injector 285 °C, transfer line 280 °C, ion source 230°C.

5. Methodology

The analytical procedure for the determination of PAHs in atmospheric samples is briefly described below:

After sampling, adsorbents and filters are extracted in a Soxhlet extractor, using cyclohexane for 24 hours at a reflux rate of about 4 cycles per hour. Before the extraction, deuterared PAHs (d8-Nap, d10-A, d10-Phe, d10-Chr, d10-Pyr, d12-B[*ghi*]P and d12-Perylene) are added as internal standards to monitor recovery.

Ambient air extracts are concentrated in a rotary evaporator, loaded onto activated silica gel column chromatography and eluted with n-hexane and n-hexane/dichloromethane (3:2).

PAHs are collected in the second fraction. PAHs fraction is concentrated under a gentle steam of nitrogen and an aliquot is analyzed by GC-MS. Twenty five PAHs are detected (Figure 28), including a group of suspected carcinogens PAHs (SIM and TIC chromatograms).

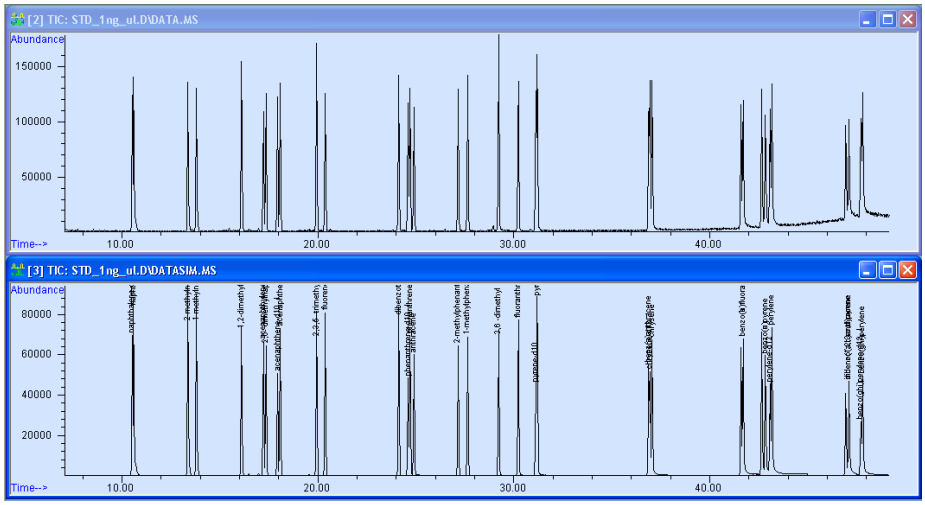

Figure 28: Determination of PAHS in PM samples.

5.1 Analytical procedure

The GC separated PAHs are subsequently analyzed and detected by MS in the Selected Ion Monitoring (SIM) mode. The selected ions (Table 13) are the most intensive and representative in Total Ion Current (TIC) mass spectra of PAHs. Quantitation of PAHs is performed by internal standards methods (deuterated PAHs) with the use of linear calibration graphs in the concentrations range 0.05 to 10 ng/uL.

The detection limits (LOD, LOQ) and the uncertainty (%Uexp; 95% k=2) are presented in Table 13 and Table 14, respectively.

6. Documented information

- LIFE Index-Air_PAH concentrations_Homes.xls
- LIFE Index-Air_PAH concentrations_Schools.xls
- LIFE Index-Air_PAH concentrations_Transports.xls

Table 13: LOD/LOQ of the PAH analysis method

PAHs	LOD	LOQ
	pg/µl	pg/ul
naphthalene	0.56	1.85
2-methylnaphthalene	0.54	1.78
1-methylnaphthalene	0.64	2.11
acenaphthylene	0.24	0.79
1,2-dimethylnaphthalene	0.28	0.92
2,6- dimethylnaphthalene	0.21	0.69
acenaphthene	0.39	1.29
2,3,5- trimethylnaphthalene	0.26	0.86
Fluorene	0.41	1.35
phenanthrene	0.57	1.88
1-methylphenanthrene	0.53	1.75
3,6 -dimethyl phenanthrene	0.43	1.42
anthracene	0.45	1.49
fluoranthrene	0.48	1.58
Pyrene	0.39	1.29
benz(a)anthracene	0.19	0.63
Chrysene	0.30	0.99
benzo(b)fluoranthene	1.00	3.30
benzo(k)fluoranthene	0.78	2.57
benzo(e)pyrene	1.12	3.70
benzo(a)pyrene	0.72	2.38

Perylene	0.59	1.95
indeno(1,2,3-c,d)pyrene	0.91	3.00
dibenzo(a,h)anthracene	0.44	1.45
benzo(ghi)perylene	0.59	1.95

Table 14: % Uncertainty of PAH analysis method

PAHs	**%Uexp (95% k=2)**
naphthalene	11.5
2-methylnaphthalene	22.2
1-methylnaphthalene	18.2
acenaphthylene	6.11
1,2-dimethylnaphthalene	10.2
2,6- dimethylnaphthalene	9.43
acenaphthene	13.0
2,3,5- trimethylnaphthalene	11.2
fluorene	9.19
phenanthrene	14.5
1-methylphenanthrene	7.71
3,6 -dimethyl phenanthrene	12.3
anthracene	6.77
fluoranthrene	10.4
pyrene	6.27
benz(a)anthracene	23.1
chrysene	8.58
benzo(b)fluoranthene	13.9
benzo(k)fluoranthene	26.0
benzo(e)pyrene	14.1
benzo(a)pyrene	11.6

Perylene	15.9
indeno(1,2,3-c,d)pyrene	21.9
dibenzo(a,h)anthracene	16.5
benzo(ghi)perylene	11.3

II. Report of the Training Course

1. Introduction

The present Minutes Report has been generated in order to provide an overview of the Training Course for technical staff, entitled “Sampling and Measurement Procedures on LIFE Index-Air”, with the following institutions: Instituto Superior Técnico (IST) and University of Aveiro (UAVR), both from Portugal; National Centre of Scientific Research "Demokritos" (NCSR-D) and Technical University of Crete (TU-Crete) (both from Greece), and National Institute for Health and Welfare (THL) from Finland.

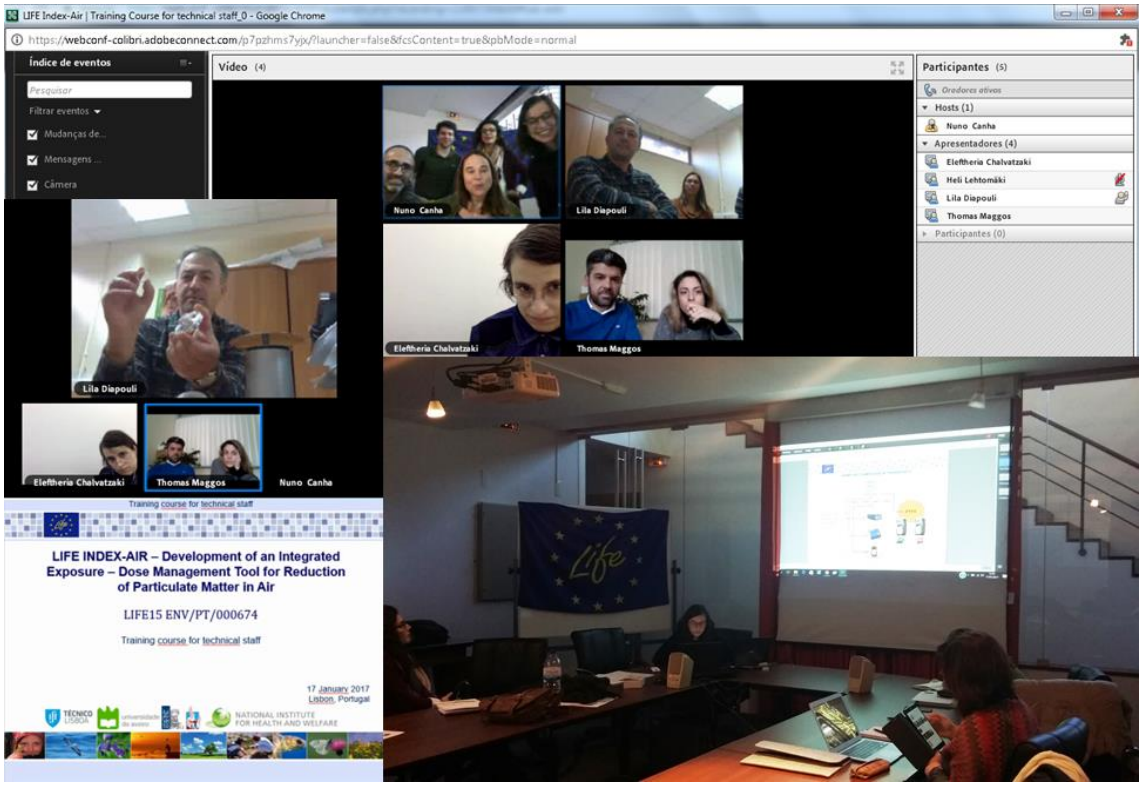

Figure 29: Online course for technical staff “Sampling and Measurement Procedures on LIFE Index-Air”

The training course was hosted by IST and it was held via online, on 17th January 2017. Marta Almeida (IST) was the chair of the meeting and nominated Nuno Canha (IST) as the rapporteur.

2. Objectives

Establishment and staff training of the experimental procedures regarding sampling and chemical characterization of particles to be employed in the LIFE Index-Air is essential in order to optimize and harmonize protocols, along with providing data with excellent quality for further use. Therefore, the aim of this training course was to promote training of the technical staff regarding the sampling and analytical procedures to be used within the scope of LIFE Index-Air.

3. Participating Members

The following members of IST, UAVR, NCSR-D, TU-CRETE and THL participated in the meeting:

- IST – Marta Almeida, Marina Almeida-Silva, Tiago Faria, Isabel Dionísio, Joana Lage, Nuno Canha
- UAVR – Joana Ferreira
- NCSR-D - Evangelia Diapouli, Manousos-Ioannis Manousakas, Thomas Maggos, Konstantinos Eleftheriadis, Kyriaki Bairachtari
- TU-CRETE - Eleftheria Chalvatzaki
- THL - Heli Lehtomäki

The signed list of participants is available in Annex 1.

4. Agenda of the meeting

The working agenda of the training course was the following:

- 12h30* - Welcome
- 12h40 - Interaction with schools: questionnaires and awareness | Marta Almeida (IST)
- 12h50 - Sampling campaigns in schools and homes | Marta Almeida (IST)
- 13h10 - Sampling campaigns in transports | Marina Almeida-Silva (IST)
- 13h40 - Preparation of filters and gravimetric analysis | Nuno Canha (IST)
- 13h55 - Sampling with the MVS6 Sven Leckel (using the device developed by NCSR "Demokritos) | Konstantinos Eleftheriadis (NCSR "Demokritos")
- 14h15 - Break
- 14h30 - Chemical analysis of particles by XRF | Manousos-Ioannis Manousakas (NCSR "Demokritos")
- 14h50 - Chemical analysis of particles by GC-MS | Kyriaki Bairachtari (NCSR "Demokritos")
- 15h10 - Chemical analysis of particles by thermal-optical analysis | Evangelia Diapouli (NCSR "Demokritos")
- 15h30 - Final discussion

*Brussels time

5. Working resume and discussion

The meeting started at 12:30 with the introduction of all participants. Marta Almeida presented the agenda of the meeting and gave a general overview of the training course: objectives, presentations and procedures. The presentations of all participants were supported by PowerPoint files that are in annex 2.

The main outputs from presentations and general discussion were:

- Framework of sampling campaigns
  The sampling campaigns were defined taken into account the sampling environments, type of sampling devices and media filters. Definition of type of media filters for each analytical technique was conducted, along with labelling of samples.

- Adaptation of sampling heads for samplers
  Konstantinos Eleftheriadis presented coarse particle collector to be adapted to the sampling heads of the MVS6 Sven Leckel, developed by NCSR-D. This device will allow sampling PM2.5 and PM10 simultaneously. The design of this new device considered a sampler flow rate of 2.3 L/min.

- Storage and transport of samples
  After collected at Lisbon (Portugal), samples will be analysed in Greece. Weighted filters should be stored in the respective petri dishes or aluminium foil and placed in the freezer (-10 to -20ºC). The filters should be transported, in freeze conditions, to Greece not more than 2 weeks after sampling.

- Analytical techniques for PM characterization
  Except from gravimetric analysis which will be done in Portugal, all samples will be characterized at NCSR "Demokritos" (Greece), using X-ray fluorescence (XRF), gas chromatography–mass spectrometry (GC-MS) and thermal-optical analysis. The fundamentals of each technique were described, along with protocols and its specificities.

Recording video of the Training Course for Technical staff is available on the following link:
https://webconf-colibri.fccn.pt/rec/2285730648

6. Main conclusions of the meeting

The LIFE Index-Air training course held via online with all partners can be summarized as followed:

- PM sampling procedures were discussed and consolidated;
- Analytical techniques for PM characterization were presented;
- Protocols were optimized in order to generate data with the best quality.

7. Approval of the Meeting Minute

According with the LIFE Index-Air Management Guide the minutes shall be considered as accepted if, within 10 calendar days from sending, no Member has sent an objection in writing to the chairperson. The chairperson will send the final version of the minutes by email to all the beneficiaries that were called to the meeting. A copy of the minutes will be archived in the LIFE Index-Air webpage.

ANNEXES

Annex 1: List of Participants

LIFE Index-Air LIFE15 ENV/PT/000674 – Development of an Integrated Exposure – Dose Management Tool for Reduction of Particulate Matter in Air

ATTENDANCE LIST

Title of the meeting: Training Course for Technical Staff

Date: 17 Jan 17 Action no.: A Activity no.: A.2

Start time: 11:30 End time: __:__

Meeting venue (city and country): IST, Lisbon Beneficiary: IST

Organization	Name	E-mail	Signature
IST	Haina Alida Ilha	MARINA@CTN.IST.UTL.PT	[signature]
Univ Aveiro	Joana Ferreira	jferreira@ua.pt	[signature]
IST	Nuno Canha	NUNOCANHA@CTN.TECNICO.ULISBOA.PT	[signature]
IST	Marta Almeida	smarta@ctn.tecnico.ulisboa.pt	[signature]
IST	Joana Lage	joanalage@ctn.tecnico.ulisboa.pt	[signature]
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IST	Tiago Faria	tiagofaria@ctn.tecnico.ulisboa.pt	[signature]
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NCSR Demokritos	Kyriaki Bairachtari	kyriaki@ipta.demokritos.gr	[signature]
NCSR Demokritos	Kostas Eleftheriadis	eleftheri@ipta.demokritos.gr	[signature]
THL	Heli Lehtomäki	heli.lehtomaki@thl.fi	[signature]
TUC	Eleftheria Chalvatzaki	elmhpet@yahoo.gr	E. Xalbatzaki

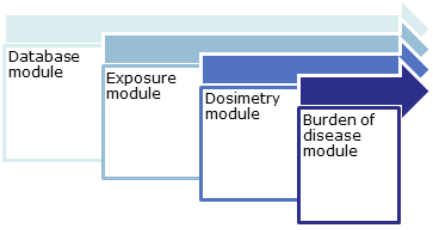
LIFE Index-Air is co-financed by the European Commission

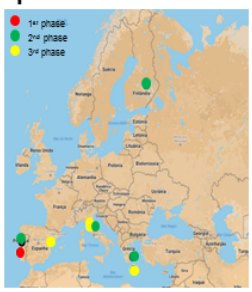
Annex 2: Presentation

Interaction with schools: questionnaires and awareness | Marta Almeida (IST)

OBJECTIVE

LIFE Index-Air project aims to **develop a policy tool** that will support authorities in the **identification of action plans** that effectively **reduce air pollutants concentrations, exposure, dose and health effects**.

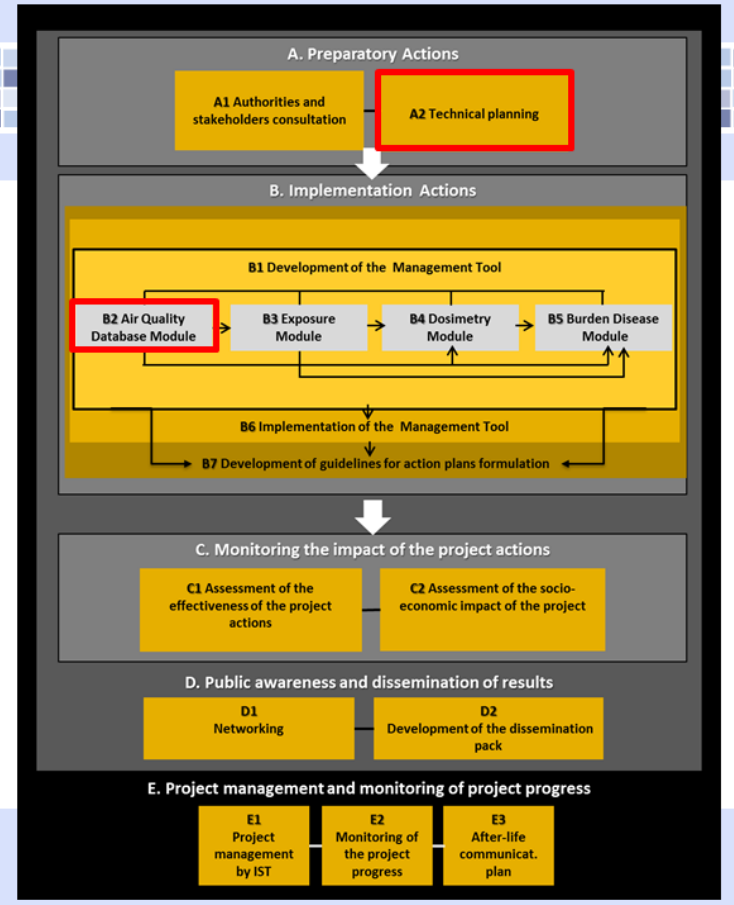




The implementation of the tool in EU cities will demonstrate its suitableness to:

1) calculate population exposure and dose to PM chemical compounds,
2) quantify the health impacts of chemical exposures associated with particles,
3) evaluate the impact of sources on exposure,
4) evaluate exposure reductions apportioned to changes in every accounted source,
5) quantitatively evaluate the impacts of policies on specific human exposure levels as well as plan new ones.

PROJECT STRUCTURE



A2 – Technical Planning IST

Objective:
- to guarantee the quality of all data generated during the project.

A.2.1 Training IST
- Development of a technical guide;
- Organization of 1 training course in Lisbon.

A.2.2 Procurement IST
- Acquisition of equipment and consumables;
- Green procurement wherever possible.

A.2.3 QA/QC IST
- Calibrations
- Inter-comparisons

Deliverables IST
D-A2.1 – Technical guide (15/12/2016)

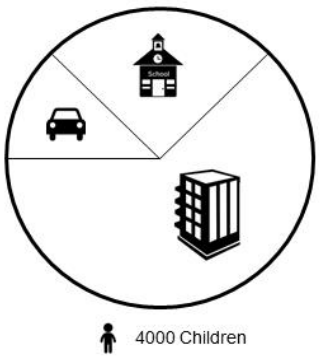
Milestones
M-A2.1 – Organization of a training course for technical staff in Lisbon (15/12/2016) IST
M-A2.2 – Completion of the technical requirements (30/12/2016) IST

Action B

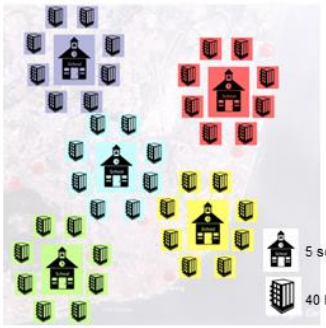
2016	2017	2018	2019	2020

DATABASE MODULE IN LISBON

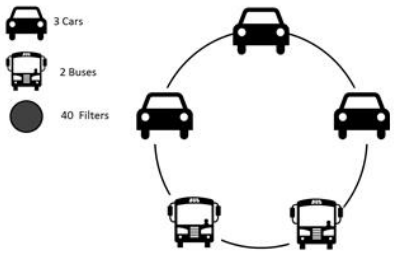
1. Time Activity Pattern



2. Concentrations of PM measured in schools and homes



3. Concentrations of PM measured in transports

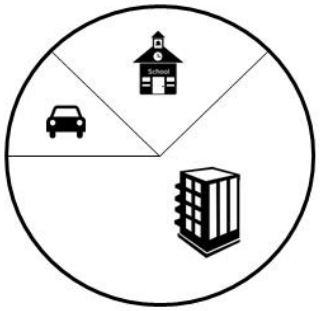


PM2.5 + PM10 + Elements (As + Cd + Ni, Pb) + PAHs (BaP) + EC/OC

DATABASE

DATABASE MODULE

1. Time Activity Pattern



4000 Children

25 Schools

January 2017

25 schools invited and 15 schools on board

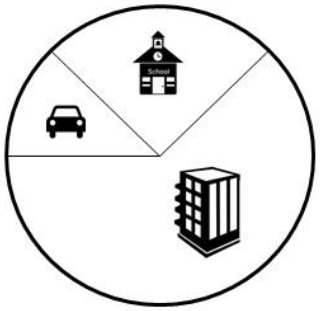
1.1 Invitation to Lisbon schools to participate in the study

Parishes with more children

Parishes with more children per m²

DATABASE MODULE

Time Activity Pattern



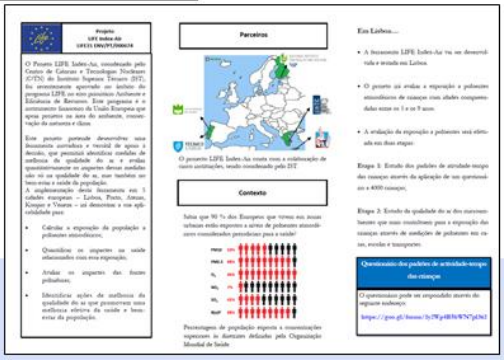
4000 Children

25 Schools

January 2017

Done

1.2 Flyer to be delivered to the schools

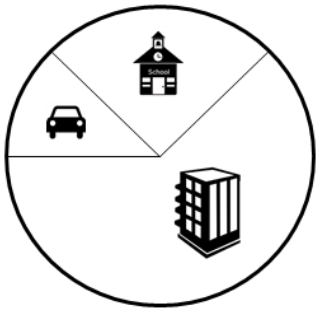


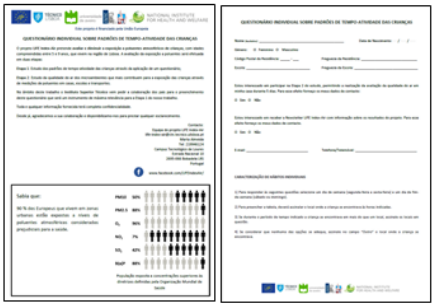
DATABASE MODULE

Time Activity Pattern

1.3 Questionnaire to be delivered to the parents

https://goo.gl/forms/rV4n3lcYU7R0NJDq1





4000 Children

25 Schools

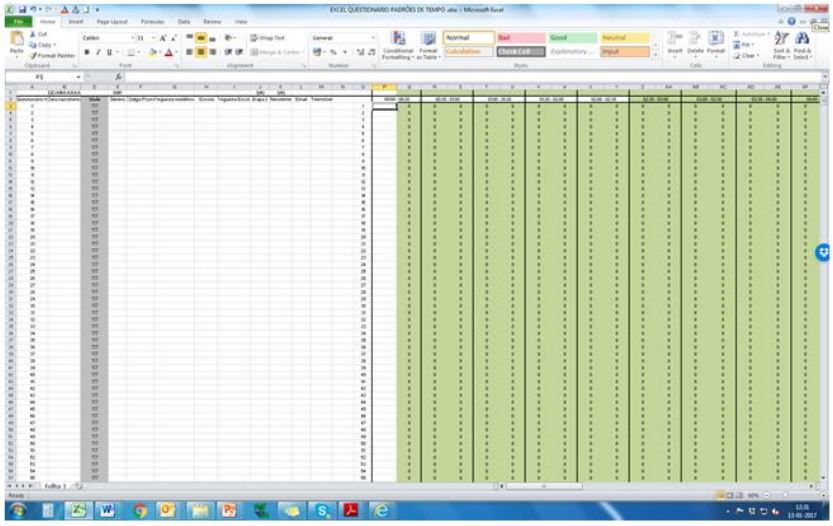
January 2017

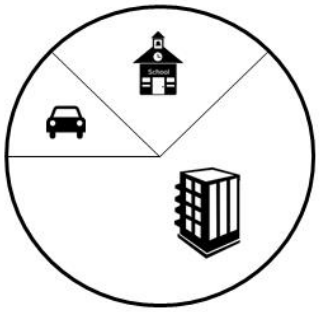
Questionnaire prepared with 2245 in the field

DATABASE MODULE

Time Activity Pattern

1.4 Database to include the data from the questionnaire





4000 Children

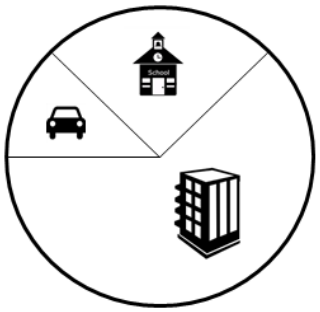
25 Schools

15 February 2017

Database developed

DATABASE MODULE

Time Activity Pattern



4000 Children

25 Schools

January 2017

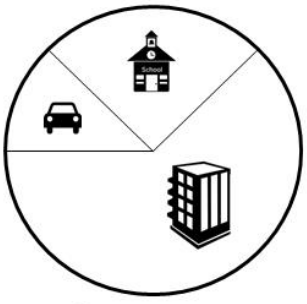
Questionnaire prepared with 2245 in the field

1.5 Awareness activities in the schools

a) Development of a presentation for students;
b) Awareness sessions;
c) Challenge "The air belongs to everyone" in which students should identify a set of behaviors conducive to improving air quality at their school, home or region;
d) The works developed will be presented at the exhibition to be held on June 5, World Environment Day, and the best works will be awarded.

DATABASE MODULE

Time Activity Pattern



4000 Children

25 Schools

February 2017

-

1.6 Measurement of IAQ in all invited schools

a) In the selected schools, measurement of IAQ with portable equipments.

Wolfsense – Graywolf
CO_2, CO, COV

DustTrack
PM1, PM2.5, PM10

Formaldemether
CH_2O

b) One classrooms; 3 hours

c) Report for schools with all results and measures to improve air quality

Sampling campaigns in schools and homes | Marta Almeida (IST)

DATABASE MODULE IN LISBON

1. Time Activity Pattern

2. Concentrations of PM measured in schools and homes

3. Concentrations of PM measured in transports

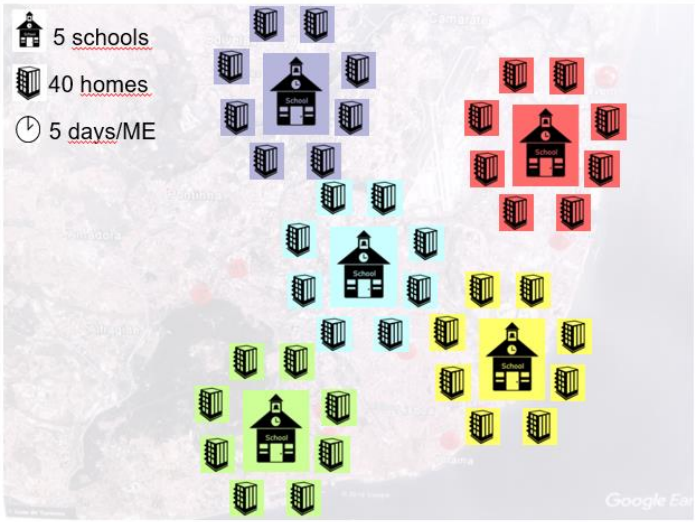
PM2.5 + PM10 + Elements (As + Cd + Ni, Pb) + PAHs (BaP) + EC/OC

DATABASE

DATABASE MODULE

2.2 Pre-selection of homes to perform the LIFE Index-Air sampling campaigns

5 schools
40 homes
5 days/ME

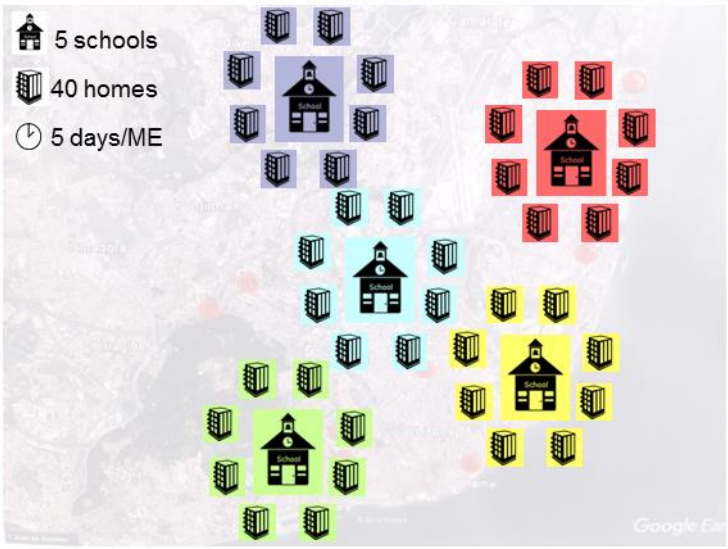


- Motivation of the parents
- Near the school
- Year of construtions

Mar17- Feb18

DATABASE MODULE

2.2 Selection of homes to perform the LIFE Index-Air sampling campaigns



- Motivation of the parents
- Near the school
- Year of construtions

Mar17- Feb18

DATABASE MODULE

2.3 Samplers and materials in schools

Sampler – MVS6 Leckel

2.3 m³/h

9:00-16:00
7h

Sampling place

1 classroom in each school

	INDOOR		OUTDOOR	
PM2.5-10	Quartz 2.5-10 25mm	Nuclepore 2.5-10 25mm	Quartz 2.5-10 25mm	Nuclepore 2.5-10 25mm
PM2.5	Quartz PM2.5 47mm	PTFE PM2.5 46.2mm	Quartz PM2.5 47mm	PTFE PM2.5 46.2mm

DATABASE MODULE

2.3 Samplers and materials in homes

Indoor

Sampler – MVS6 Leckel

2.3 m³/h

20:00-07:00
11h

Outdoor

Sampling place

1 living room
in each home

	INDOOR		OUTDOOR	
PM2.5-10	Quartz 2.5-10 25mm	Nuclepore 2.5-10 25mm	Quartz 2.5-10 25mm	Nuclepore 2.5-10 25mm
PM2.5	Quartz PM2.5 47mm	PTFE PM2.5 46.2mm	Quartz PM2.5 47mm	PTFE PM2.5 46.2mm

Training course for technical staff

LIFE INDEX-AIR – Development of an Integrated Exposure – Dose Management Tool for Reduction of Particulate Matter in Air

LIFE15 ENV/PT/000674

Training course for technical staff

17 January 2017
Lisbon, Portugal

TÉCNICO LISBOA · universidade de aveiro · DEMOKRITOS · NATIONAL INSTITUTE FOR HEALTH AND WELFARE

Sampling campaigns in transports | Marina Almeida-Silva (IST)

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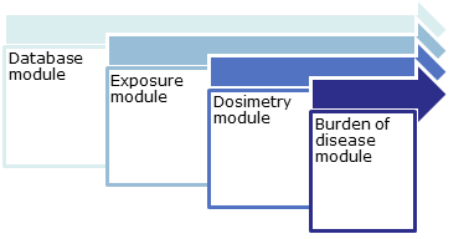

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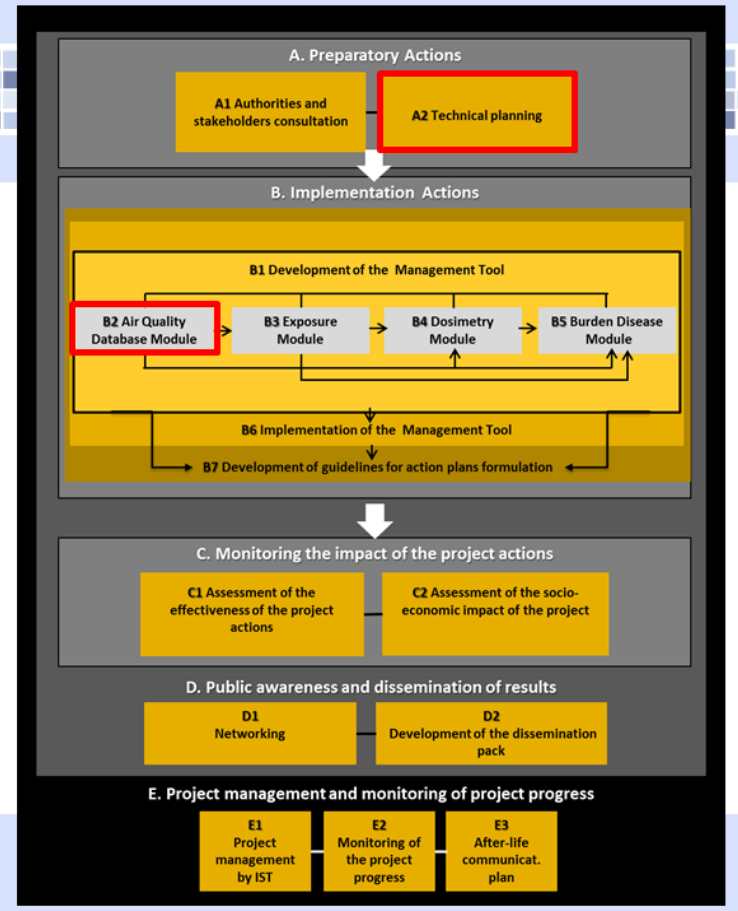
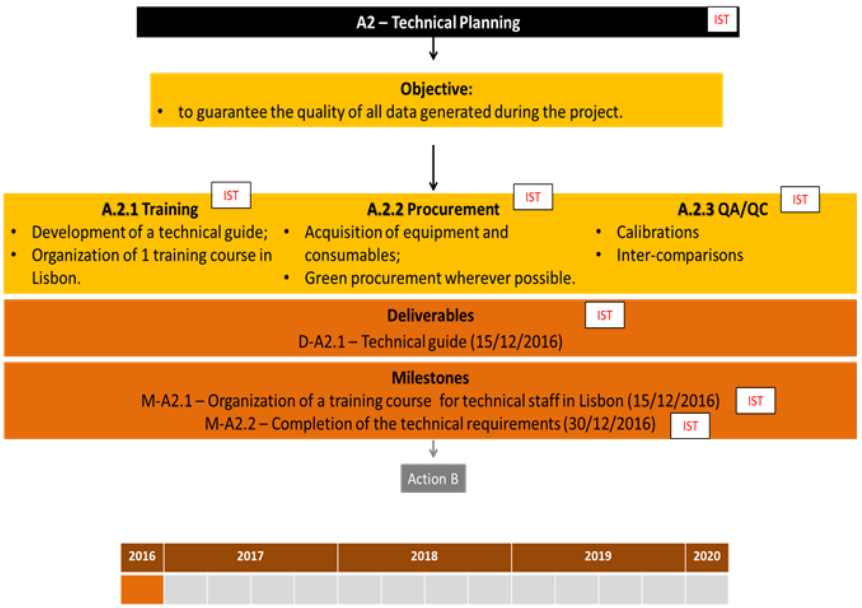
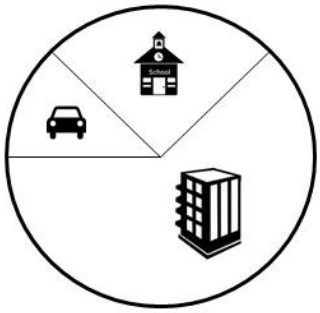




DATABASE MODULE

IN LISBON

1. Time Activity Pattern



4000 Children

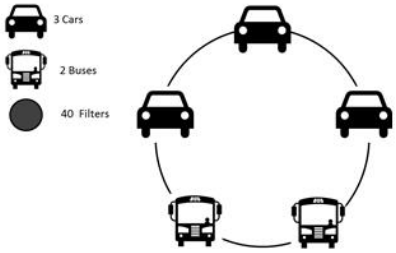
2. Concentrations of PM measured in schools and homes

5 Schools

40 Homes

1800 Filters

3. Concentrations of PM measured in transports



PM2.5 + PM10 + Elements (As + Cd + Ni, Pb) + PAHs (BaP) + EC/OC

DATABASE

5

DATABASE MODULE

IN LISBON

1. Time Activity Pattern

2. Concentrations of PM measured in schools and homes

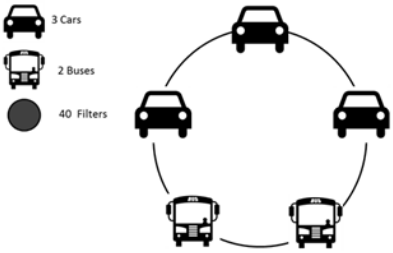
3. Concentrations of PM measured in transports

4000 Children

5 Schools

40 Homes

1800 Filters



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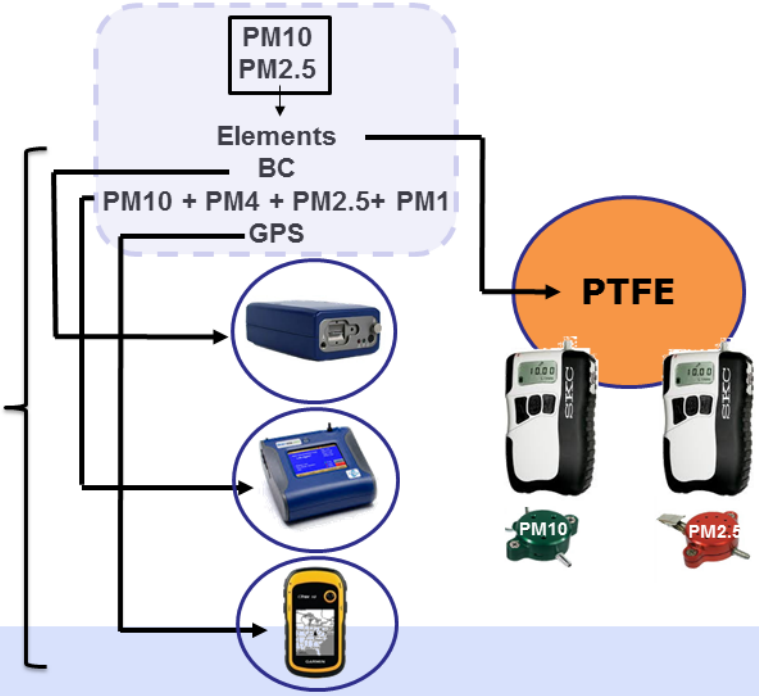
DATABASE

6

SAMPLING CAMPAIGNS IN TRANSPORTS



This was not included in the proposal

SAMPLING CAMPAIGNS IN TRANSPORTS

SAMPLING CAMPAIGNS IN TRANSPORTS

Indoor

Outdoor

SAMPLING CAMPAIGNS IN TRANSPORTS

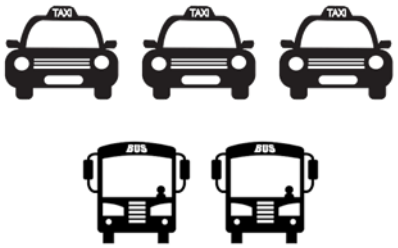
Indoor

Outdoor

9:00 – 17:00

5 weekdays

SAMPLING CAMPAIGNS IN TRANSPORTS



Indoor — 9:00 – 17:00

Outdoor — 5 weekdays

2x each vehicle

SAMPLING CAMPAIGNS IN TRANSPORTS

Indoor — 9:00 – 17:00

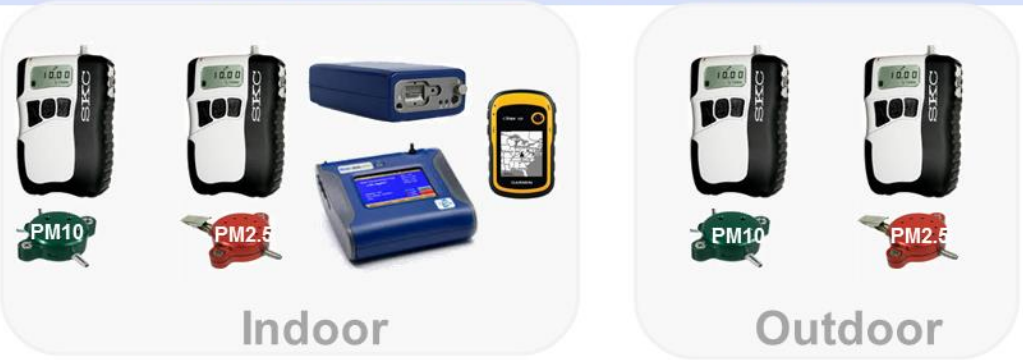
Outdoor — 4 weekdays

2x each vehicle

Indoor

Outdoor

SAMPLING CAMPAIGNS IN TRANSPORTS



SAMPLING CAMPAIGNS IN TRANSPORTS

Car & Bus			
Gravimetry	Mass	All filters	40 filters
XRF	As, Cd, Ni, Pb & others	PTFE	40 filters

5 veh * 4 equip (I/O) * 2 (no. of sampling per vehicle) = 40

9 L/min

8h + 8h / filter
9:00-17:00

PTFE

	Monday	Tuesday	Wednesday	Thursday	Friday
Week 1	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	
	Car 1	Car 1	Car 1	Car 1	
Week 2	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	
	Car 2	Car 2	Car 2	Car 2	
Week 3	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	
	Car 3	Car 3	Car 3	Car 3	
Week 4	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	
	Bus 1	Bus 1	Bus 1	Bus 1	
Week 5	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	9:00 – 17:00	
	Bus 2	Bus 2	Bus 2	Bus 2	

Training course for technical staff

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Preparation of filters and gravimetric analysis | Nuno Canha (IST)

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Preparation of filters and gravimetric analysis

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Goal

GOAL

1) Definition of type of filters to be used in the project and their purpose
2) Preparation of filters and coding
3) Gravimetric analysis, filter storage and transport

This activity will be performed in **Campus Tecnológico e Nuclear** (CTN) from Instituto Superior Técnico (IST), Portugal.

IST LIFE Index-Air team:

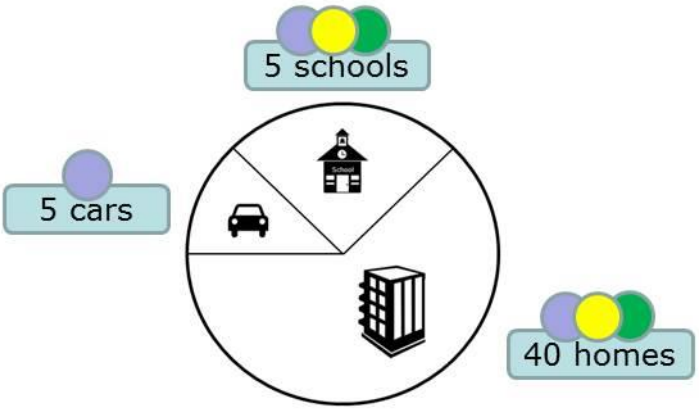
- Isabel Dionísio
- Tiago Faria
- Marina Almeida-Silva
- Marta Almeida (supervision)

Definition of type of filters to be used in the project and their purpose

Teflon

Quartz

Nuclepore



Definition of type of filters to be used in the project and their purpose

Teflon

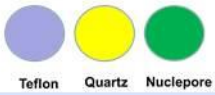
Quartz

Nuclepore

ANALYTICAL TECHNIQUES	TYPE OF FILTERS USED
Mass	
Elements	
EC/OC	
PAH	

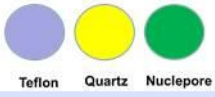
TYPES OF FILTERS USED IN SCHOOLS AND HOMES MONITORING

	Indoor (5 Schools and 40 Homes)		Outdoor (5 Schools and 40Homes)	
	Leckel #1	Leckel #2	Leckel #3	Leckel #4
PM2.5-10	**Quartz**	**Policarbonate Nuclepore**	**Quartz**	**Policarbonate Nuclepore**
	25 mm	25 mm	25 mm	25 mm
	210 filters	210 filters	210 filters	210 filters
PM2.5	**Quartz**	**Teflon**	**Quartz**	**Teflon**
	47 mm	46.2 mm	47 mm	46.2 mm
	210 filters	210 filters	210 filters	210 filters
Measurements	Mass	Mass	Mass	Mass
	EC/OC	Elements	EC/OC	Elements
	PAH		PAH	



TYPES OF FILTERS USED IN TRANSPORTS MONITORING

	3 cars + 2 buses	
ME	Indoor	Outdoor
Equipment	SKC	SKC
PM2.5	**Teflon**	**Teflon**
	37 mm	37 mm
	10 filters	10filters
PM10	**Teflon**	**Teflon**
	37 mm	37 mm
	10 filters	10 filters
Measurements	Mass	Mass
	Elements	Elements



CODING OF FILTERS

GENERAL SETTING

Type of Particles	Type of ME	Location	Type of Filter	Sequencial Number
C – Coarse (PM2.5-10)	**S&** – School	**I** – Indoor	**Q** – Quartz	**##**
F – Fine (PM2.5)	**H&%** – Homes	**O** – Outdoor	**N** – Nucleopore	
T – Total (PM10)	**MT$** – Taxi		**T** – Teflon	
CAS-! – PM Fractions	**MB?** – Bus		**M** - Membrane	

& – refers to the schools ID (ranging from A to E)

% – ID of the home (ranging from 1 to 8) indexed to the ID of the school &

! – sampled fractions using CASCATE device:
I – PM10; II – PM10-2.5; III - PM2.5-1.0; IV - PM1.0-0.5; V - PM0.25-0.5; VI - <PM0.25

$ – ID of the taxi from A to C

? – ID of the bus from A to B

Example of Filter Coding:
- Particles: Fine
- ME: School 1
- Location: Indoor
- Filter 05: Quartz

F-S1-IQ05

CODING OF FILTERS

GENERAL SETTING

		PM2.5-10 (C – Coarse)		PM2.5 (F – Fine)		PM10 (T-Total)		CASCATE (CAS)
		Quartz (Q)	Nuclepore (N)	Quartz (Q)	Teflon (T)	Quartz (Q)	Membrane (M)*	Teflon (T)
Schools (S&)	Indoor (I)	C-S&-IQ##	C-S&-IN##	F-S&-IQ##	F-S&-IT##	T-S&-IQ##	T-S&-IM##	CAS-!-H&%##
	Outdoor (O)	C-S&-OQ##	C-S&-ON##	F-S&-OQ##	F-S&-OT##	T-S&-OQ##	T-S&-OM##	n.a.
Homes (H&%)	Indoor (I)	C-H&%-IQ##	C-H&%-IN##	F-H&%-IQ##	F-H&%-IT##	T-H&%-IQ##	T-H&%-IM##	CAS-!-H&%##
	Outdoor (O)	C-H&%-OQ##	C-H&%-ON##	F-H&%-OQ##	F-H&%-OT##	T-H&%-OQ##	T-H&%-OM##	n.a.

More information on Experimental Procedure | **INDEX-1/2016 - Procurement and preparation of filters**

CODING OF FILTERS

GENERAL SETTING

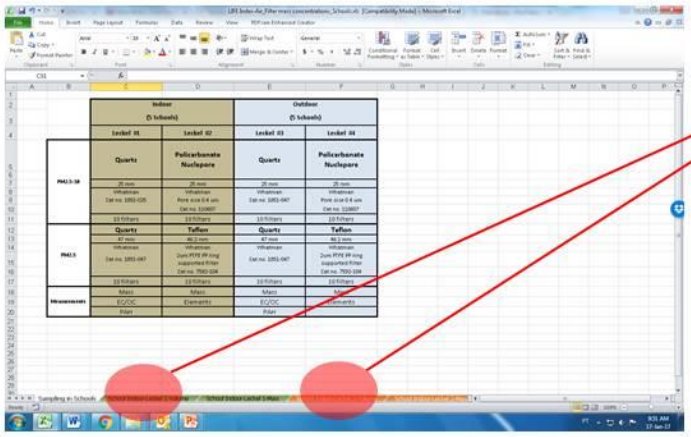
			PM2.5 (F – Fine)	PM10 (T-Total)
			Teflon (T)	
Transports	Taxi (MT$)	Indoor (I)	F-MT$-I##	T-MT$-I##
		Outdoor (O)	F-MT$-O##	T-MT$-O##
	BUS (MB?)	Indoor (I)	F-MB?-I##	T-MB?-I##
		Outdoor (O)	F-MB?-O##	T-MB?-O##

More information on Experimental Procedure | **INDEX-1/2016 - Procurement and preparation of filters**

CODING OF FILTERS - DATABASE

ENTERING DATA

LIFE Index-Air_Filter mass concentrations_Homes.xls
LIFE Index-Air_Filter mass concentrations_Schools.xls



Two sheets for each sampler Leckel # (1-4):

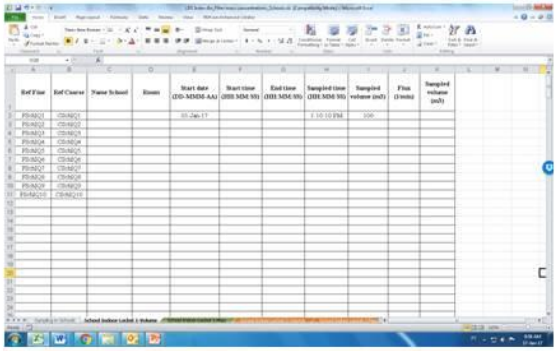
- Volume
- Mass

+
"Resume" sheet
+
"Graphs" sheet

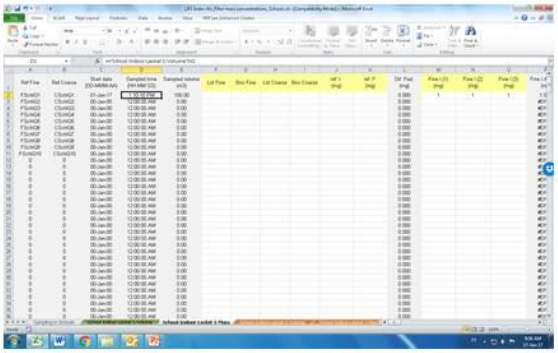
CODING OF FILTERS - DATABASE

ENTERING DATA

LIFE Index-Air_Filter mass concentrations_Homes.xls
LIFE Index-Air_Filter mass concentrations_Schools.xls



Volume

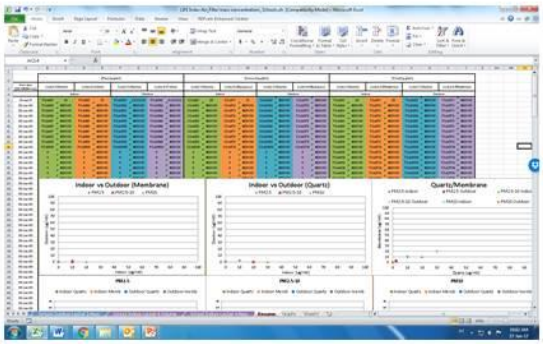


Mass

CODING OF FILTERS - DATABASE

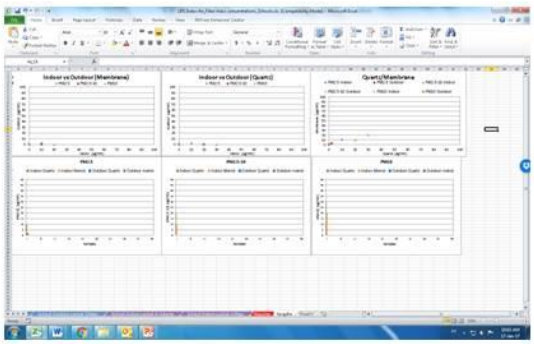
ENTERING DATA

LIFE Index-Air_Filter mass concentrations_Homes.xls
LIFE Index-Air_Filter mass concentrations_Schools.xls



Resume

PM concentrations

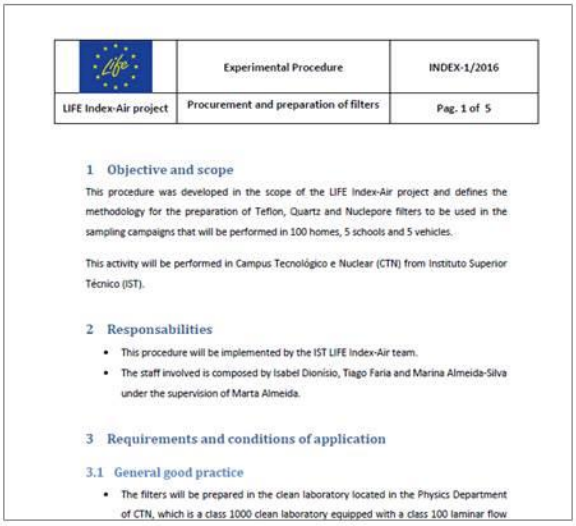


Graphs

I/O Ratios, concentration variability, ratios between matrices

Documentation

Experimental Procedure | **INDEX-1/2016 - Procurement and preparation of filters**



	Experimental Procedure	INDEX-1/2016
LIFE Index-Air project	Procurement and preparation of filters	Pag. 1 of 5

1 Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the preparation of Teflon, Quartz and Nuclepore filters to be used in the sampling campaigns that will be performed in 100 homes, 5 schools and 5 vehicles.

This activity will be performed in Campus Tecnológico e Nuclear (CTN) from Instituto Superior Técnico (IST).

2 Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Isabel Dionísio, Tiago Faria and Marina Almeida-Silva under the supervision of Marta Almeida.

3 Requirements and conditions of application

3.1 General good practice

- The filters will be prepared in the clean laboratory located in the Physics Department of CTN, which is a class 1000 clean laboratory equipped with a class 100 laminar flow

Documentation

Experimental Procedure | **INDEX-2/2016 - Gravimetric analysis of the filters**

	Experimental Procedure	INDEX-2/2016
LIFE Index-Air project	Gravimetric analysis of the filters	Pag. 1 of 4

1 Objective and scope

This procedure was developed in the scope of the LIFE Index-Air project and defines the methodology for the gravimetric analysis of Teflon, Quartz and Nuclepore filters that will be used in the sampling campaigns to be performed in 100 homes, 5 schools and 5 vehicles.

This activity will be performed in Campus Tecnológico e Nuclear (CTN) from Instituto Superior Técnico (IST).

2 Responsabilities

- This procedure will be implemented by the IST LIFE Index-Air team.
- The staff involved is composed by Isabel Dionísio, Tiago Faria and Marina Almeida-Silva under the supervision of Marta Almeida.

3 Requirements and conditions of application

3.1 General good practice

Gravimetric analysis of the filters

Clean laboratory of CTN (IST)
Class 1000 clean laboratory equipped with a class 100 laminar flow chamber.

Mettler Toledo scale, model UMT5, with reading accuracy of 0.1 µg

Stakeholders Consultation - Portugal

LIFE INDEX-AIR – Development of an Integrated Exposure – Dose Management Tool for Reduction of Particulate Matter in Air

LIFE15 ENV/PT/000674

TÉCNICO LISBOA · universidade de aveiro · DEMOKRITOS · NATIONAL INSTITUTE FOR HEALTH AND WELFARE

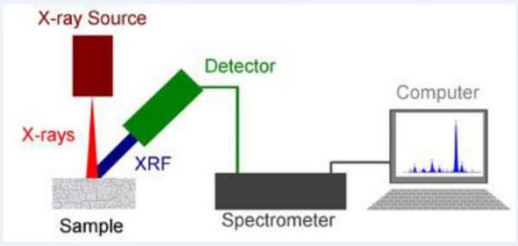
https://www.facebook.com/LIFEIndexAir/

January 2017
Lisbon, Portugal

Chemical analysis of particles by XRF | Manousos-Ioannis Manousakas (NCSR "Demokritos")

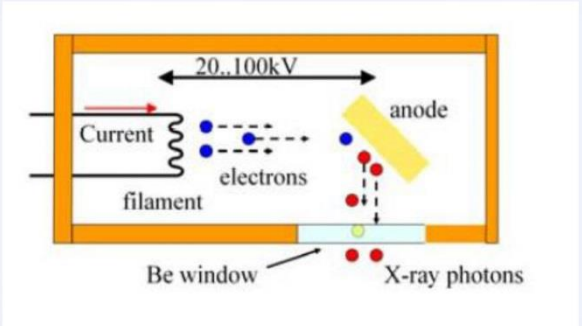
XRF principle

- X-ray fluorescence (XRF) analysis is based on the emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays.

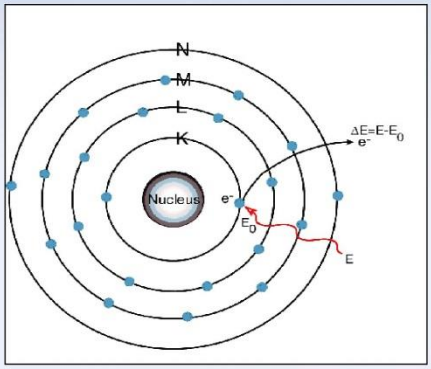


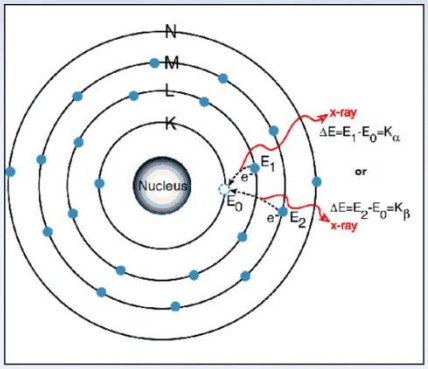
X-ray source

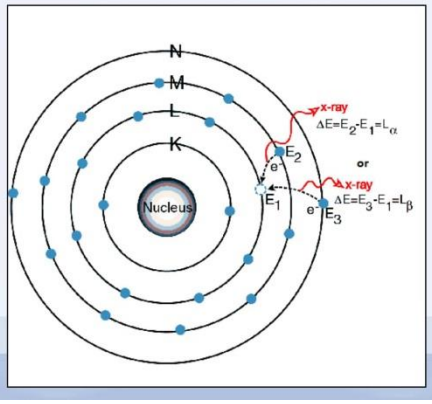
- X-rays originate from the energy loss associated with the interaction of high energy electrons with atoms
  - Continuous radiation (Bremsstrahlung)
  - Characteristic radiation



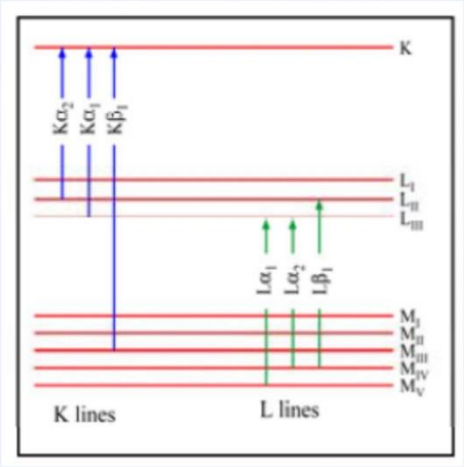
Sample excitation



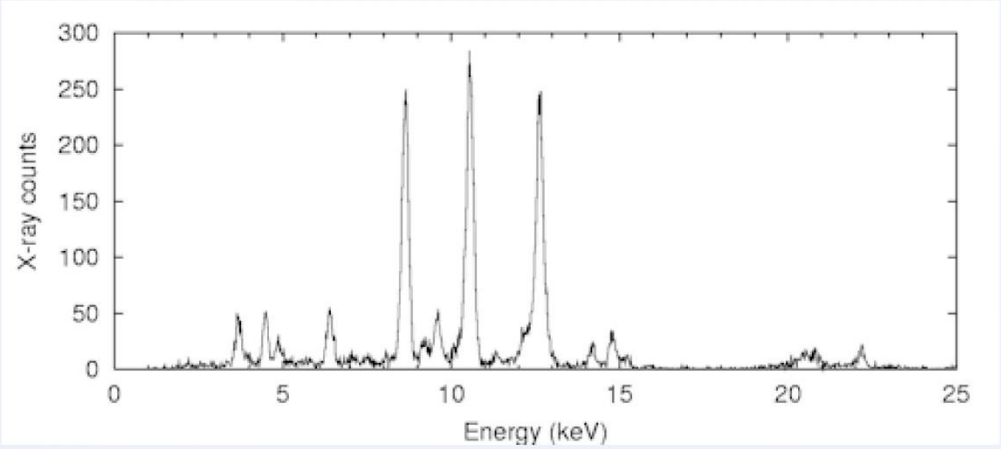




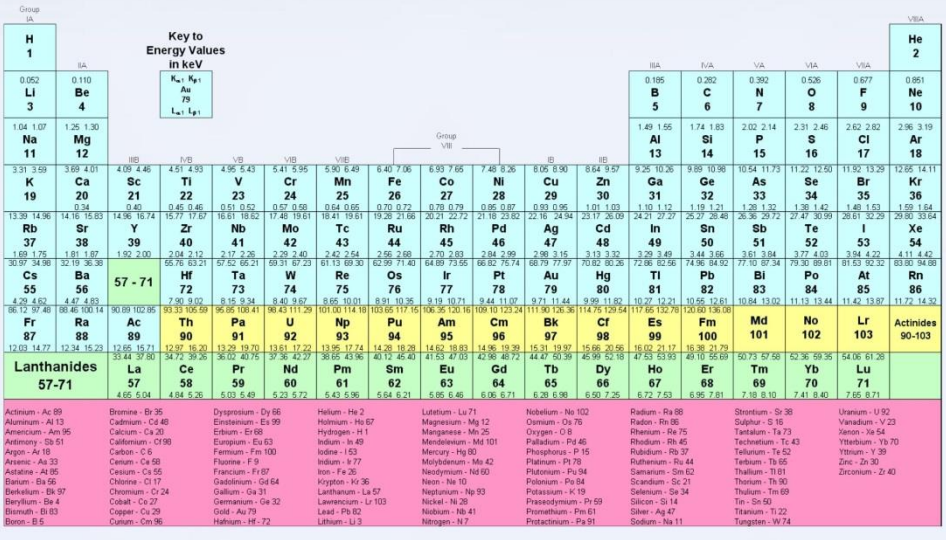
Characteristic lines



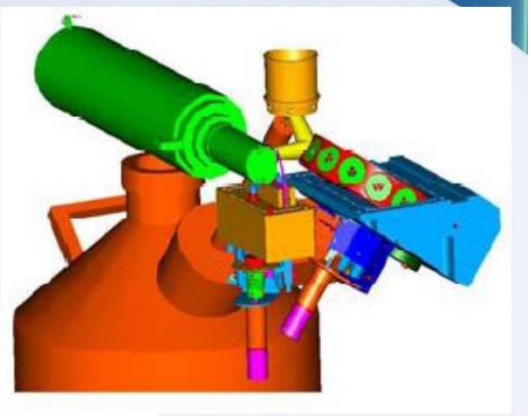
XRF spectra

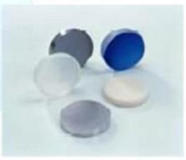


Characteristic X-rays



Epsilon 5 by PANanlytical





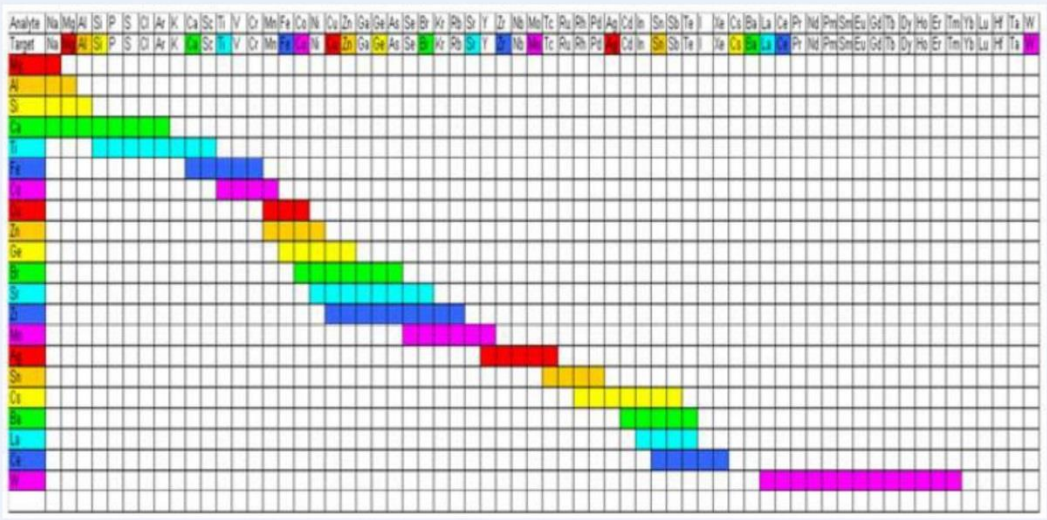
Advantages of Epsilon 5

- The 100 kV tube allows the excitation of K lines up to W. Fluorescence efficiency is higher for K-Lines than for L-Lines
- Not the X-rays from the tube but fluorescence of the target is used to excite the sample. Optimal excitation of the analytes is possible by selecting targets with an energy just above the absorption edge of the analytes. That gives the advantage of optimal and selective excitation
- Because of the 3-D optics the background is extremely low

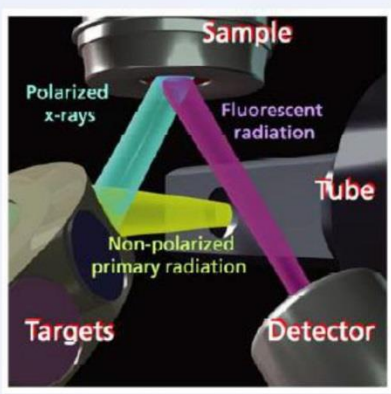
But:

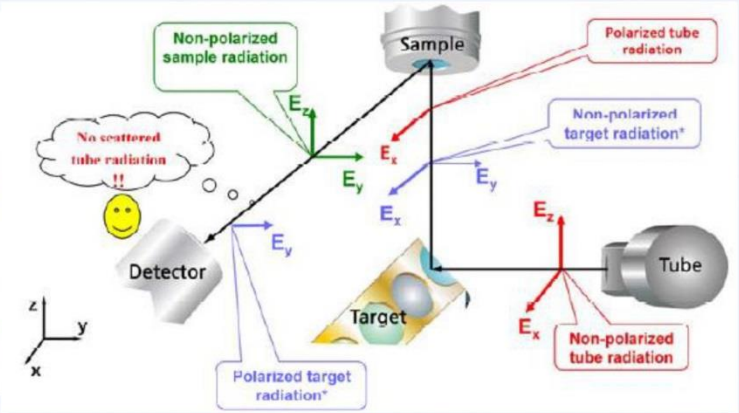
- The advantage of rapid analysis is gone

Target selection

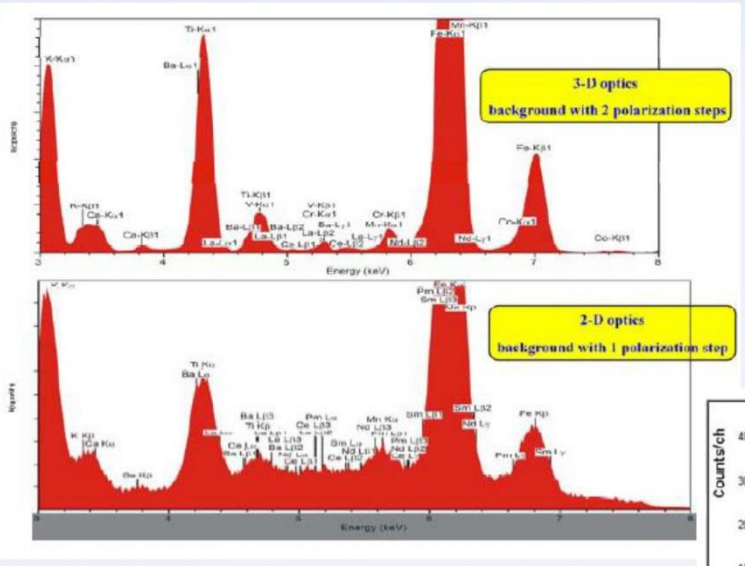


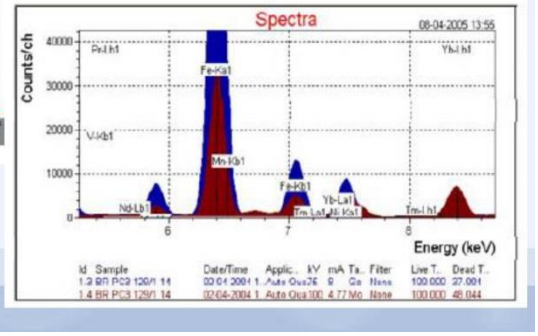
3-D optics





Optimized spectra



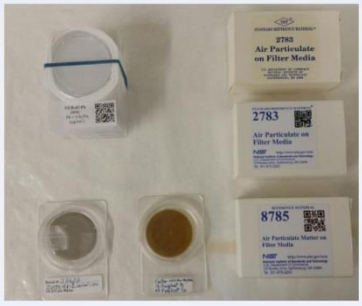


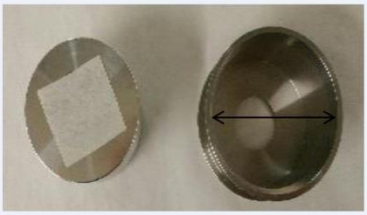
Detection system

- Ge detector
  - Resolution (<140eV)
  - Best efficiency
    (100% over whole range)
  - Escape peaks (at –9.9keV)
  - Use of liquid Nitrogen

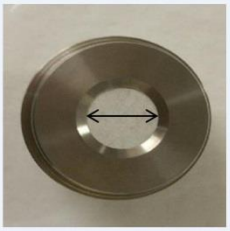
Use gloves and safety glasses

Sample mounting





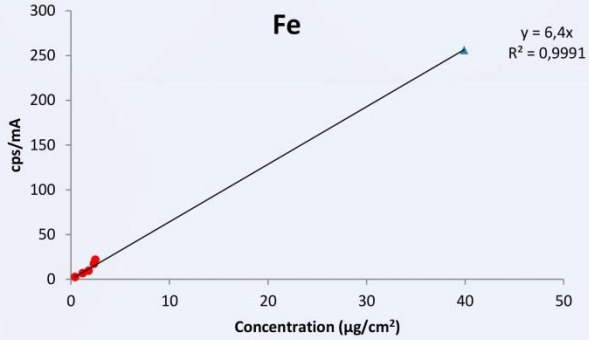
55 mm



24mm

Calibration

- Twenty (20) standards on filter media (UC Davis)
- Thirty two (32) standards on mylar (Micromatter)
- Two (2) NIST Standards (8885, 2783)
- CRMs 2584 and 2583 dispersed on filter media (NCSR Demokritos)



Instrumental parameters

Secondary Target	kV	mA	Detected elements	Measuring time (sec)
Al	25	24	Na, Mg	800
CaF_2	40	15	Al, Si, P, S, Cl, K	600
Fe	75	8	Ca, Ti, V, Cr	400
Ge	75	8	Mn, Fe, Co, Ni, Cu, Zn	400
Zr	100	6	Br, Rb, Pt, Au, Hg, Pb	400
KBr	100	6	Ga, Ge, As	800
Mo	100	6	Ag, Cd, Cs, Ba, Ce	400
Al_2O_3	100	6	Sn, Sb	400
LaB_6	100	6	Sr	400

Detection limits

Element	LOD (ng/m³)	Element	LOD (ng/m³)
Na	8.9	Cr	0.2
Mg	9.4	Mn	1.4
Al	3.7	Fe	1.4
Si	16.4	Co	1.4
S	4.2	Ni	0.5
Cl	1.9	Cu	0.5
K	1.4	Zn	3.7
Ca	4.2	Br	2.3
Ti	0.9	Sr	2.3
V	1.4	Pb	1.4

Detection limits for different filter types

Element	Filter type		
	PTFE	Quartz	Glass Fiber
	ng/m^3		
Ni	0.4	1.7	1.1
As	0.3	0.5	1.9
Pb	3.0	10.5	8.5

Uncertainty estimation

The total uncertainty is estimated by taking into account individual errors for every step of the process:

- Peak overlap (A)
- Calibration (B)
  - ♦ Uncertainty of the standards
  - ♦ Calibration curve error
- Sampling (C)
  - ♦ Flow uncertainty
  - ♦ Deposition area uncertainty
  - ♦ Gravimetric analysis uncertainty
- Sample self absorption (D)
- Quantification uncertainty (E)
- Reproducibility (RSD of three successive measurements) (F)

$$\text{Err} = \sqrt{A^2 + B^2 + C^2 + D^2 + E^2 + F^2}$$

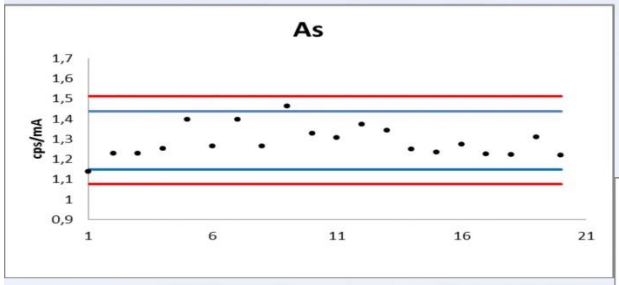
Indicative uncertainties

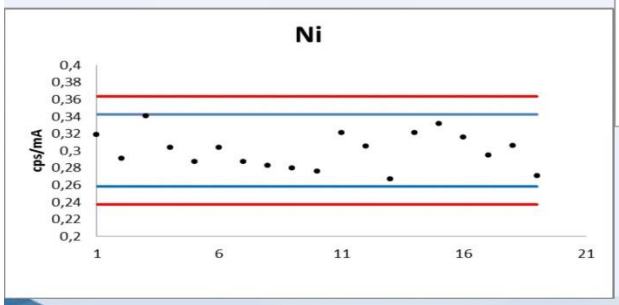
Expanded uncertainties (confidence level 95% κ=2)	
Element	**Uncertainty %**
As	28
Pb	19
Ni	24

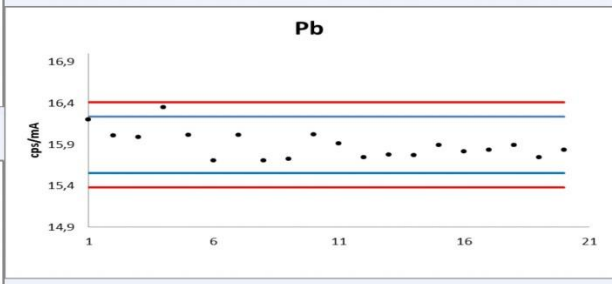
QA/QC

- Before each measurement batch a monitor sample is measured. If the results are inside the range of two times the standard deviation of the last 20 measurements of the standards (QA/QC chart) then the samples are loaded. If the measurements are outside this range then “Monitor” function of the instrument is initiated and the calibrations curves are corrected.
- Every measurement is repeated three times. If the standard deviation of the successive measurements is higher than 10% the results are discarded and the measurement is repeated.
- QA/QC requirements are checked every 20 measurements
- Detector calibration is performed once every week

QA/QC Charts







THANK YOU!

Demokritos
National Centre for Scientific Research

Institute of Nuclear & Radiological
Sciences & Technology, Energy & Safety

Determination of Polycyclic aromatic Hydrocarbons (PAHs) in ambient air using Gas Chromatography – Mass Spectrometry (GC-MS)

Thomas Maggos PhD
Kyriaki Bairachtari PhD

NCSR "Demokritos",I.N.R.A.S.T.E.S, Environmental Research Laboratory,

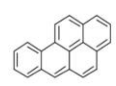
Demokritos
National Centre for Scientific Research

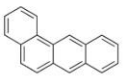
Institute of Nuclear & Radiological
Sciences & Technology, Energy & Safety

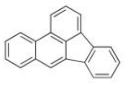
The methodology of the PAHs analysis is based on the **ISO 12884:2000**: "*Ambient air-Determination of total (gas and particle-phase) polycyclic aromatic hydrocarbons-Collection on sorbent-backed filters with gas chromatographic/mass spectrometric analysis*".

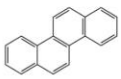
25 PAHs could be detected in the gas & particle phase.

Among them B[a]A, B[b]F, B[k]F, B[a]P, Chr, are considered to be probable human carcinogens, whereas other PAHs such as AcPy, Ant, Flut, fluorene, are not classified as promoters of the same health risk.


benzo[*a*]pyrene


benz[*a*]anthracene


benzo[*b*]fluoranthene


chrysene

Storage after the sampling

According to the **International Standard** the filters should be **"***refrigerated and protected from light for transport to the laboratory. Store the samples at 4 oC or below and for no longer than two weeks prior the extraction***"**

Expose to high temperatures or light may cause PAH degradation and volatilization (transport refrigerator, ice packs)

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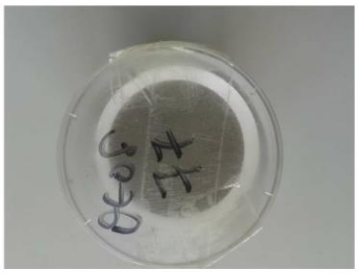
Analytical procedure

Sample extraction

After sampling, filters are extracted in a Soxhlet extractor, using cyclohexane for 24 hours at a reflux rate of about 4 cycles per hour.



Monitoring the recovery
Before the extraction, deuterared PAHs (d8-Nap, d10-A, d10-Phe, d10-Chr, d10-Pyr, d12-B[*ghi*]P and d12- Perylene) are added as internal standards on the filter to monitor recovery.

Sample concentration
The extracts are concentrated in a vacuum rotary evaporator to about 1-2 ml

Sample clean up
The sample extract is loaded onto activated silica gel column chromatography and eluted with dichloromethane in n-hexane (3:2 volume fraction).

Further concentration
PAHs fraction is collected and concentrated (to about 200ul) under a gentle steam of nitrogen and an aliquot is analyzed by by GC/MS.

Instrumentation and conditions

Polycyclic Aromatic Hydrocarbons concentrations are determined through gas chromatography-mass spectrometric analysis. Agilent Technologies 7890A GC System, 5975 C inertXL EI/CI MS Detector, provided with 7683B autosampler is used for PAHs determination and quantitation in air samples.

•The gas chromatograph is equipped with split/splitless injector and HP 5MS 60mx0.25mm column with 0.25 μm thickness (Agilent Technologies).

•Oven temperature program started from 60°C isothemal for 2 min, then heated up to 80°C at 25°C/min, then heated up to 300°C at 5°C/min and kept isothermal for 5 min.

•The heating zones were kept at the following temperatures: injector 285 °C, transfer line 280 °C, ion source 230°C.

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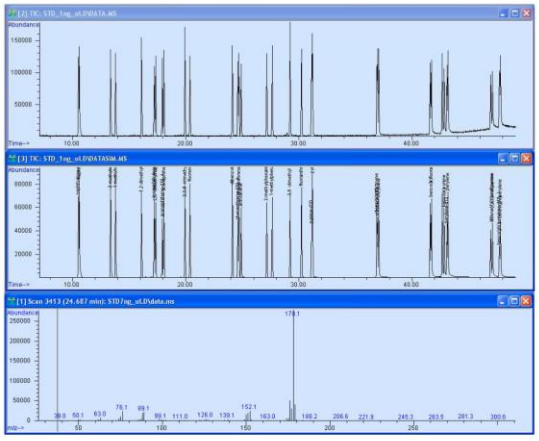
Analytical procedure (3)

•The GC separated PAHs are subsequently analyzed and detected by MS operated in the Selected Ion Monitoring (SIM) mode. The selected ions (Table 1) are the most intensive and representative in Total Ion Current (TIC) mass spectra of PAHs. Quantitation of PAHs is performed by internal standards methods (deuterated PAHs) with the use of linear calibration graphs in the concentrations range from 0.05 to 10 ng/uL.



PAH	Primary Ion	Secondary Ions	
Naphthalene- d8	136	137	134
Naphthalene	128	127	-
2-Methylnaphthalene	142	141	115
1-Methylnaphthalene	142	141	115
1,2-Dimethylnaphthalene	156	141	-
Acenaphthylene	152	151	-
2,6-Dimethylnaphthalene	156	141	-
Acenaphthene-d10	164	162	-
Acenaphthene	153	154	-
2,3,5-Trimethylnaphthalene	170	155	-
Fluorene	166	165	-
Phenanthrene-d10	188	189	-
Phenanthrene	178	176	-
Anthracene	178	176	-
2-Methylphenanthrene	192	191	-
1- Methylphenanthrene	192	191	-
3,6-Dimethylphenanthrene	206	191	-
Fluoranthrene	202	200	-
Pyrene-d10	212	213	-
Pyrene	202	200	-
Benzo[a]pyrene	228	226	-
Chrysene-d12	240	236	-
Chrysene	228	226	-
Benzo[b]fluoranthrene	252	250	253
Benzo[k]fluoranthrene	252	253	250
Benzo[e]pyrene	252	250	253
benzo[a]pyrene	252	250	253
Perylene-d12	264	260	265
Perylene	252	250	253
Indeno[1,2,3,-c,d]pyrene	276	277	-
Dibenz[a,h]anthracene	278	276	279
Benzo[g,h,i]perylene-d12	288	144	-
Benzo[g,h,i]perylene	276	277	-

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PAH	Uexp 95% k=2	LOD (pg/µl)	LOQ (pg/µl)
naphthalene	11.5	0.56	1.85
2-methylnaphthalene	22.2	0.54	1.78
1-methylnaphthalene	18.2	0.64	2.11
acenaphthylene	6.11	0.24	0.79
1,2-dimethylnaphthalene	10.2	0.28	0.92
2,6- dimethylnaphthalene	9.43	0.21	0.69
acenaphthene	13.0	0.39	1.29
2,3,5- trimethylnaphthalene	11.2	0.26	0.86
Fluorene	9.19	0.41	1.35
phenanthrene	14.5	0.57	1.88
1-methylphenanthrene	7.71	0.53	1.75
3,6 -dimethyl phenanthrene	12.3	0.43	1.42
anthracene	6.77	0.45	1.49
fluoranthrene	10.4	0.48	1.58
Pyrene	6.27	0.39	1.29
benz(a)anthracene	23.1	0.19	0.63
Chrysene	8.58	0.3	0.99
benzo(b)fluoranthene	13.9	1	3.3
benzo(k)fluoranthene	26	0.78	2.57
benzo(e)pyrene	14.1	1.12	3.7
benzo(a)pyrene	11.6	0.72	2.38
Perylene	15.9	0.59	1.95
indeno(1,2,3-c,d)pyrene	21.9	0.91	3
dibenzo(a,h)anthracene	16.5	0.44	1.45
benzo(ghi)perylene	11.3	0.59	1.95

- % Uexp: 6.11 – 26.0
- LOQ: 0.69 – 3.3

LIFE Index Air

Sampling and Measurement Procedures on LIFE Index-Air Training Course for technical staff

Chemical analysis of particles by thermal-optical analysis

Evangelia Diapouli, NCSR "Demokritos"

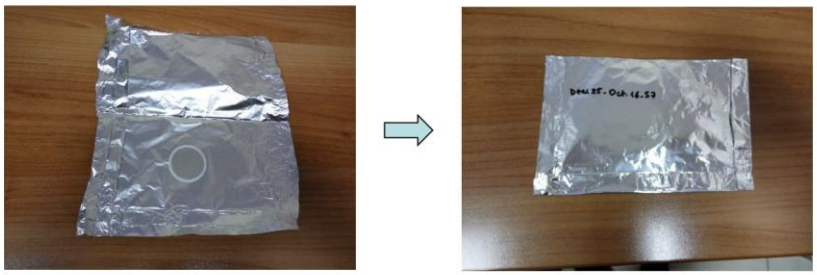
Online Course - 17 January 2017

Objective

- Determination of elemental (EC) and organic carbon (OC) by thermal-optical analysis (TOA) of Quartz filters that will be collected in the sampling campaigns to be performed in 40 homes, 5 schools and 5 vehicles.
- This activity will be performed by NCSR "Demokritos" (NCSR-D) at the Environmental Radioactivity Laboratory (ERL), Institute of Nuclear & Radiological Sciences & Technology, Energy & Safety (INRASTES).

Requirements and good practices

- The quartz filters will be collected in Lisbon and will be sent to ERL by IST in Laboratory Grade Aluminum Foil.
- Quartz fibre filters without binding materials shall be used.



Requirements and good practices

- No pre-treatment of the filters is necessary. Prior to sampling, IST will send to ERL some clean filters from the batch of filters intended for sampling in order to assess the blank levels of EC and OC.
- This procedure will be repeated whenever a new batch of filters is used.
- The blank filters should display EC concentrations below the detection limit and OC concentrations on average below 2 $\mu g/cm^2$ and with a standard deviation below 1 $\mu g/cm^2$.

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Requirements and good practices

- All filters will be uniquely identified and records will be kept with respect to the manufacturer, purchase date, manufacturer's batch and pack number.
- Filters should be handled with care to avoid possible contamination and / or loss of material.
- All samples will be stored by IST in a refrigerator prior to sending to NCSR-D, in order to avoid losses due to volatilization.

Requirements and good practices

- Field blanks will be collected throughout the sampling campaigns.
- In addition, backup filters will be used during part of the sampling days (once every 10 days) in order to assess the positive artefact caused by the ab(ad)sorption of gaseous species in (on) the filters.

Theory of operation

The sample is put into an oven which is purged with helium, while a stepped temperature ramp increases the temperature.

⇩

Organic compounds and pyrolysis products thermally desorb and are led into a MnO_2 oxidizing oven, where they are quantitatively converted to CO_2 gas.

⇩

The CO_2 is swept out of the oxidizing oven in the helium stream and is mixed with hydrogen gas. This mixture then flows through a heated nickel catalyst where it is quantitatively converted to methane. The methane is subsequently measured using a flame ionization detector (FID).

Theory of operation

At a second stage, the oven is cooled and the flow stream is switched to an oxidizing helium/oxygen carrier gas mixture.

⇩

A second temperature ramp is then initiated in the oxidizing gas stream and any elemental carbon is oxidized off the filter and into the oxidizing oven.

⇩

The elemental carbon is then detected in the same manner as the organic carbon.

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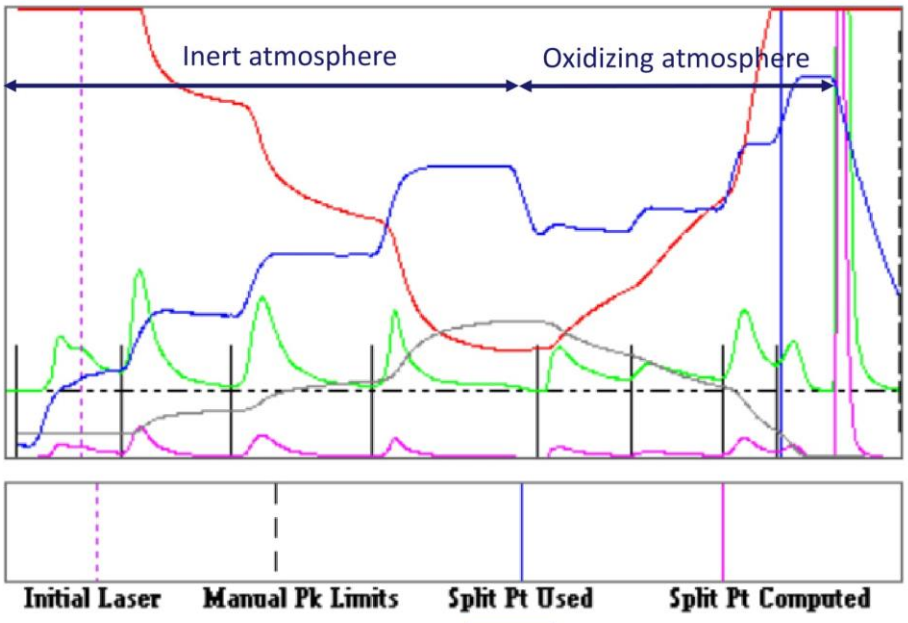
Theory of operation

In addition to this elemental carbon present in the sample, EC can be formed from some charring of organic carbon as it is pyrolyzed during the initial temperature ramp.

This charring of organic carbon results in an artificially low measurement of the organic carbon and a high measurement for the original elemental carbon, if left uncorrected.

Charring correction is applied by continuously monitoring the sample's transmittance throughout the heating process by the use of a red light laser.

Theory of operation



Instrumentation

Organic Carbon / Elemental Carbon (OCEC) Laboratory Instrument (Model 5L, Sunset Laboratory Inc.)

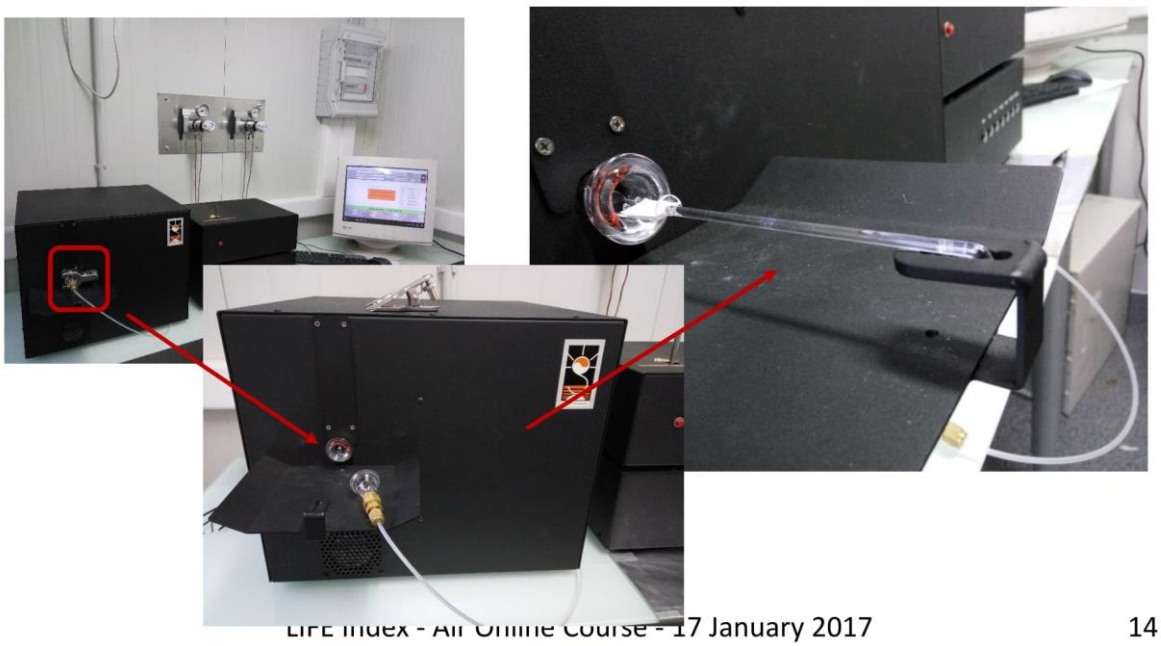
EUSAAR2 Thermal protocol

Mode	Step	T in ° C, duration in s
He	He 1	200, 120
	He 2	300, 150
	He 3	450, 180
	He 4	650, 180
He/O_2	He	No heating, 30
	He/O_2 1	500, 120
	He/O_2 2	550, 120
	He/O_2 3	700, 70
	He/O_2 4	850, 80

Analytical procedure

- Clean ovem
- Instrument blank (below 0.2 µg/cm^2)
- Sucrose standard (± 5% of reference value)
- Cal gas (to check the performance of the detector)
- Control filter (± 10% of reference value)
- If all the above QA/QC requirements are met, the instrument is used for the analysis of field samples.

Analytical procedure



Data reporting

- The elemental and organic carbon concentrations are calculated after sample analysis by running the calculation software.
- Concentrations are reported in µg/cm^2.
- The final ambient air concentrations (µg/m^3) will be calculated by multiplying the obtained concentration in µg/cm^2 with the total loaded area of the filter (in cm^2) and dividing by the sampled volume (in m^3).
- The detection limit of the method is 0.05 µg of carbon/cm^2.