

KTL/ULTRA/EU Environment and Climate contract No ENV4-CT97-0568

Determination of absorption coefficient using reflectometric method

Identification code: SOP ULTRA /KTL-L - 1.0		APPROVALS			
Full SOP Working SOP # pages _____		Coordinator: ___/ ___/ ___ _____			
Issue Date: ___/ ___ . _____		PIC: ___/ ___/ ___ _____			
Revision No: Revision date: ___/ ___ . _____ Revision description:		Coordinator: PIC:			
Revision No: Revision date: ___/ ___ . _____ Revision description:		Coordinator: PIC:			
Revision No: Revision date: ___/ ___ . _____ Revision description:		Coordinator: PIC:			
Distributed to:	Name of recipient:	Original date	Rev. 1. date	Rev. 2. date	Rev. 3. date
KTL Kuopio					
University of Wageningen					
GSF Oberschleissheim					
ECN Petten					
University of Kuopio					
University of Helsinki					

Determination of absorption coefficient using reflectometric method

1.0 Purpose and applicability

The purpose of this SOP is to establish a uniform method for measurement of absorption coefficients (a) of PM_{2.5} filters sampled during the ULTRA and EXPOLIS projects.

This SOP covers the following steps concerning the measurement of a from 37 mm and 47 mm Teflon filters:

- Preparation and calibration of the Smoke Stain Reflectometer instrument prior to and during the measurements
- Method of measurement and QA
- Data recording and handling

2.0 Definitions

- SSR: Smoke Stain Reflectometer
- Mask: a round plate in which the measuring head is located during measurements
- White standard: white area in the standard plate
- Grey standard: grey area in the standard plate
- Control filter: a clean, non-exposed filter; must be similar to those used in sampling (taken from the same lot/batch of filters as the sampling filters)
- Field blank: a control filter, not exposed to sampling air flow but otherwise handled like a regular sample filter

3.0 References

The development of this SOP is based on SOP "Preparation of standard operating procedures" SOP ULTRA/KTL-G-1.(*).

International Standard ISO 9835: Ambient air - Determination of a black smoke index (1993).

"Measurement of PM_{2.5} in ambient air with the Harvard Impactor", SOP ULTRA/UoW-F-2.(*

*) This statement refers to the latest SOP revision available. Make sure that you know and have it.

4.0 Discussion

In order to get comparable and reliable results all PM_{2.5} filters have to be handled very carefully and uniformly. Necessary precautions need to be adopted to prevent any contamination.

This SOP can be adopted also to determine absorption coefficients for PM₁₀ and PM₁ filters.

5.0 Responsibilities

5.1 The coordinator and the Principal Investigators= Committee (PIC) of ULTRA study have reviewed and approved this SOP and are responsible for its contents. Local PI=s will provide the field workers with the up to date version of this SOP and collect the old versions to the coordinating center.

5.2 If the procedures of this SOP are changed, the change has to be documented and the SOP changed, reviewed and approved by the local principal investigator(s). (Figure 1. \cong Local and temporal deviation from or local change of the SOP \cong).

5.3 Field workers are obliged to work according to this SOP and not to change the procedures without the consent of the local PI. All temporary changes in the applying this SOP have to be carefully documented (who changed, when, why, what changes, possible impacts) and approved by the local PI. (Figure 1. \cong Local and temporal deviation from or local change of the SOP \cong). (SOP ULTRA/KTL-G-1.).

5.4 This SOP was drafted by Marko Vallius, M.Sc., at the ULTRA center Kuopio.

6.0 Equipment and materials

a) Equipment

- Smoke Stain Reflectometer: Diffusion Systems Ltd. Model 43 (M43D) or other comparable instrument
- Standard plate (White/Grey): supplied with the instrument
- Pair of tweezers

b) Materials

- Five (5) control filters
- PM sample filters
- PM field blank filters

7.0 Procedures

7.1 Preparation

7.1.1 Preparation of the SSR instrument

Prepare the SSR for measurement as follows (linearity check):

- Clean the measuring head, mask and standard plate with pure C₂H₅OH (or other suitable solvent) using a non-lint cloth
- Switch on the SSR-instrument and let it warm for at least 15 minutes
- Before attaching the measuring head, adjust the reading to 0.0 by using the *zero* knob in the front panel of the SSR
- Insert the measuring head tightly in the mask
- Attach measuring head to SSR central unit
- Locate the measuring head over the white standard and adjust the reflectance reading to 100.0 by using *coarse* and *fine* knobs in the front panel

- Move the measuring head over the grey standard; the reading should be within the limits given for the standard plate in the manufacturers manual.

7.1.2 Calibration

- If linearity check gives acceptable reflectance values (limits given above), place one of the five control filters (taken from the same lot/batch of filters as the sampling filters) centrally over the white standard, measure reflectance from the center of the filter and adjust the reading to 100.0 ; record this comparison reflectance reading in data form, repeat measurement four (4) times using different location of measuring head for each measurement (5-point method; figure 2) and record readings in the data form
- Without readjusting the reflectance reading, measure reflectance for the other four control filters using the 5-point method and record readings
- Calculate arithmetic mean of reflectance values for each control filter; the filter having the \cong median mean \cong of reflectance values is selected for the *primary control filter* (see figure 3), which is used for recalibration of the SSR during the measurement of sample filters
- If the five values measured from the primary control filter have standard deviation larger than 0.5 units, disqualify the filter, pick a new clean filter from the batch and redo the selection process until a suitable primary control filter is found.
- Recalibrate SSR to 100.0 using the selected primary control filter; use the midpoint of the filter when adjusting the reading to 100.0
- Repeat calibration using the primary control filter after every series of twenty-five (25) sample filters. Record the reflectance reading of the control filter in the data form before readjusting the reading to 100.0

7.2 Measurement procedure

7.2.1 Measurement of reflectance

- Calibrate the SSR according to paragraph 7.1.2
- Clean the measuring head, mask, standard plate and tweezers with pure C_2H_5OH using a non-lint cloth
- Make sure that the measuring head is tightly attached to the mask
- Remove a sample filter from Petri dish using tweezers and locate it centrally on the white standard
- Locate the measuring head with utmost caution over the sample filter and record the reflectance reading
- Make four (4) additional measurements per sample filter using the 5-point method and record reflectances
- Clean mask, standard plate and tweezers after every series of twenty-five (25) sample filters simultaneously with recalibration

7.2.2 Calculation of absorption coefficient (a)

This instruction is based on the \cong International Standard ISO 9835: Ambient air - Determination of a black smoke index \cong (1993).

Absorption coefficient a for sample filters is calculated using equation

$$a = (A / 2V) H \ln (R_F / R_S)$$

where

- R_S is the reflectance of the sample filter as percentage of R_0
 R_0 is the reflectance of the clean control filter (100.0 by definition)
- R_F is the average reflectance of the field blank filters
- V is the volume sampled, in cubic metres, m^3
- A is the area of the stain on the filter ($780 A \cdot 10^{-6} m^2$)

$A = \pi (d/2)^2$, where d is the inner diameter of the filter's poly support ring. For Andersen 37 mm $2 \mu m$ pore size Teflon filters (part nr SA240PR100) the value of A is $780 A \cdot 10^{-6} m^2$.

Sample volume V is calculated as the product of mean flow rate and sampling time, or directly from an integrating sample volume meter; for details see "SOP ULTRA/UoW-F-2."

Report the absorption coefficient to the first decimal place and in exponential form ($X A \cdot 10^5$).

7.3 Quality assurance

- At the end of each measurement session, measure reflectances again for at least 10 % of the filters. If the (average) reflectance of the duplicates deviates more than $\nabla 3 \%$ from the original results, all the filters measured during the corresponding session need to be measured again.
- All field blanks are analyzed according to this SOP
- Make the reflectance measurements in as dark a room as possible to eliminate the effects of sun and other light sources on the measurements
- Do not point the measuring head towards any light source (doing so might damage the instrument !)
- To prevent contamination of the filters during measurements make sure that the instruments and the working environment are clean
- Do not use "wavy" or "curved" filters for PM sampling. Curved filters will inconvenience the measurement of reflectance and should be rejected prior to sampling of $PM_{2.5}$.
- After the ULTRA2 field work 20 exposed, 5 unexposed and 5 control filters will be circulated in all centers where measurements of absorption coefficient have been made.

8.0 Data records

- Following data are recorded from the absorption coefficient measurements in the *Data form* (figure 4) and computer files:
 - * date and place of measurements
 - * measurement personnel identification data
 - * Relative humidity in the measurements location
 - * filter lot/batch No. (printed in the filter package)
 - * instrument data (type and model No.)
 - * filter identification codes

- * reflectance readings from all control filters and all calibrations (specify filter type in the “filter code” -column of the data form)
- * reflectance readings and average readings from the sample filters and field blanks
- * sample volume for each sample (exposed) filter
- * calculated absorption coefficient $a_{0.2000}$,

for all measured sample filters and field blanks

- Deviations from and changes of this SOP are recorded on the sheet \cong Local and temporal deviations from or local change of the SOP (figure 1)

9.0 Sample archiving

Sample filters are handled and stored according to SOP ULTRA/UoW-F-2.

10.0 Implementation and application

This SOP will be distributed by KTL ULTRA center to all ULTRA centers and KTL EXPOLIS center by mail or telefax. Reception of a new SOP or revision should always be confirmed to KTL ULTRA center.

11.0 Attachments

Figure 1. Local and temporal deviation from or local change of the SOP

Figure 2. 5-point method for measuring reflectance of filters

Figure 3. Principle of selecting the primary control filter for calibration of the SSR

Figure 4. Black Smoke data form

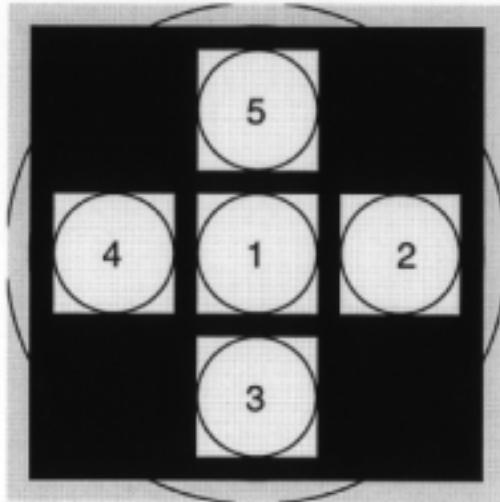
Figure 5. SOP confirmation sheet

Figure 1. Local and temporal deviation from or local change of the SOP.

Identification code:	Center: _____
Deviation Change No: ___ pages ___	Approval by Principal Investigator
Begin date: ___/___/___ End date: ___/___/___	Date and Signature: ___/___/___ _____
Original text(s); full paragraph, page No:	Changed text(s), full paragraph:

Figure 2. 5-point method for measuring reflectance of filters

The measurements of reflectance are made from five different points of each filter as indicated in the figure below.



Numbered areas (1 to 5) indicate the spots where the reflectometer light beam should approximately hit during reflectance measurement.

NOTE : Be careful not to measure reflectance from the edges of the filters , i.e. from the poly support rings !

Figure 3. Principle of selecting the primary control filter for calibration of the Smoke Stain Reflectometer

The primary control filter is selected from the original five control filters. Selection is based on reflectance measurements as shown in the example below:

	Filter 1	Filter 2	Filter 3	Filter 4	Filter 5
measurement 1	adjust 100.0	100.1	99.7	100.1	100.4
measurement 2	100.1	100.1	99.9	100.3	100.2
measurement 3	100.1	100.1	100.1	100.2	100.4
measurement 4	99.9	100.2	100.0	99.9	100.3
measurement 5	100.1	100.0	100.0	100.4	100.1
average reflectance	100.03	100.10	99.94	100.18	100.28

In this case the primary control filter would be FILTER 2, since it has the \cong median average \cong reflectance among the five filters and standard deviation less than 0.5 units.

--	--	--	--	--	--	--	--	--	--

Figure 5.

SOP CONFIRMATION SHEET

Determination of absorption coefficient using reflectometric method

This SOP has been received by Principal Investigator of

Research center _____ Date ___ / ___ / _____

Signature of PI: _____

INSTRUCTIONS :**0) Keep this sheet attached to the original copy of the corresponding SOP**

- 1) When copying the SOP, mark the date of copying for each copy, number each copy
- 2) When delivering the SOP copy, take the signature and mark the date
- 3) When delivering a new revision to this SOP, collect previous SOP copies away and confirm with signature and mark the date
- 4) After each change fax this sheet to coordinator

Copy	Date of the copy	Delivered to Signature	Date of delivery	Received back PI signature	Received back Date
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Coordinator fax : + 358 - 17 B 201 265