RUPIOH/EU Environment and Climate contract QLRT-2001-00452

DETERMINATION OF ABSORPTION COEFFICIENT USING REFLECTOMETRIC METHOD

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1.0 PURPOSE AND APPLICABILITY

The purpose of this SOP is to establish a uniform method for measurement of absorption coefficients (*a*) of $PM_{2.5}$ and (small fraction of) PM10 filters sampled during the RUPIOH project. This SOP covers the measurement of *a* from 37 mm Teflon filters.

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

This SOP is based on the SOP for the Ultra study, http://www.ktl.fi/ultra.

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

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 PM_{10} filters.

5.0 RESPONSIBILITIES

1 The Coordinator and PI's are responsible for final review and approval of this SOP.
2 The local Principal Investigator is responsible that new versions of this SOP are available for every member of the project team and that older SOP versions are collected and destroyed.
3 Members of the project team are responsible for working according to this SOP and reporting of local and temporal deviations and local changes of this SOP

6.0 EQUIPMENT AND MATERIALS

6.1 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

6.2 Materials

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

6.3 Paper materials

- Laboratory forms to record reflectance readings

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 C_2H_5OH (or other suitable solvent) using a non-lint cloth
- Attach the measuring head
- Switch on the SSR-instrument and let it warm for at least 30 minutes
- Remove 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 Selection of primary control filter
This is performed once at the start of the project.
- 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

"median mean" 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.

This primary control is used to set the reflectometer to 100.00 in subsequent sessions.

7.2 Measurement procedure

7.2.1 Measurement of reflectance

- Prepare the SSR according to paragraph 7.1.2
- Recalibrate SSR to 100.0 using the selected primary control filter; use the midpoint of the filter when adjusting the reading to 100.0
- 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 additional measurements per sample filter using the 5-point method and record reflectances
- Repeat calibration using the primary control filter after every series of 25 sample filters. Record the reflectance reading of the control filter in the data form before readjusting the reading to 100.0. The reflectance should be between 98 and 102. If not, the samples should be re-measured.
- Clean mask, standard plate and tweezers after every series of 25 sample filters simultaneously with recalibration
- 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 ± 3 % from the original results, all the filters measured during the corresponding session need to be measured again.
- All boxes of filters should be numbered (rank number). If you open a new box of filters, carefully record the lot number and check the reflectance of blank filters to make sure they agree within two units of the primary control filter. If not, separate average field blanks need to be calculated.

7.2.2 Calculation of absorption coefficient (*a*)

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

Absorption coefficient *a* for sample filters is calculated using equation

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

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 as percentage of R_0
- V is the volume sampled, in cubic metres, m^3
- A is the area of the stain on the filter ($780 * 10^{-6}$ m^2)

R_F is the overall average reflectance of the field blank filters, but if filters are from different lots systematic differences may occur. Then the average field blank per lot is used. It is very important to record (during pre-weighing when filters are taken from the box) what filters belong to which lot.

$A = \pi (d/2)^2$, where d is the inner diameter of the filter's poly support ring. For Andersen 37 mm 2 μm pore size Teflon filters (part nr SA240PR100) the value of A is 780 * 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 RUPIOH 2.0.

Report the absorption coefficient to the first decimal place and in exponential form (X * 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 ± 3 % from the original results, all the filters measured during the corresponding session need to be measured again.
- At least 18 field blanks are analyzed
- 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 $PM_{2.5}$.
-10 control filters will be circulated in all centers where measurements of absorption coefficient have been made.

8.0 DATA RECORDS

- reflectance readings from all control filters (selection of the primary control filter and regular check of the primary control filter with the other four control filters)
- Following data are recorded from the absorption coefficient measurements in the *Data form* (figure 3):
  * date and place of measurements
  * measurement personnel identification data
  * filter lot/batch No. (printed in the filter package)
  * instrument data (type and model No.)
  * filter identification codes
  * reflectance readings from the sample filters and field blanks

9.0 SAMPLE ARCHIVING

Sample filters are handled and stored according to SOP RUPIOH 2.0 and 3.0.

10.0 IMPLEMENTATION AND APPLICATION

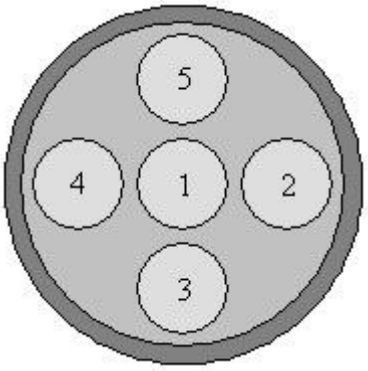
This SOP will be distributed by IRAS to all RUPIOH centers by mail or telefax. Reception of a new SOP or revision should always be confirmed to IRAS.

11.0 ATTACHMENTS

Figure 1 5-point method for measuring reflectance of filters
Figure 2 Principle of selecting the primary control filter for calibration of the SSR
Figure 3 Reflectance reading laboratory form

Figure 1. 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 2. 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 ”median average” reflectance among the five filters and standard deviation less than 0.5 units.

Figure 3. Reflectance readings laboratory form.

Project	RUPIOH
Instrument	
Date	
Name technician	

Sample code*	Reflectance reading				
	1	2	3	4	5
Grey tile					
Control filter*					

* after 25 filters, primary control filter should be measured
** at the end 10% of the samples have to be remeasured