WEIGHING OF TEFLON FILTERS TO DETERMINE PARTICLE MASS CONCENTRATIONS

1 Purpose and applicability

This SOP contains the protocol for weighing Teflon filters to calculate PM2.5 and PM10 concentrations in indoor and outdoor air for the EU-multicenter study RUPIOH.
The principle of the method is that by weighing the filter before and after sampling the particle mass of the sample can be determined.

2 Definitions

PM2.5: particle fraction with a 50% aerodynamic cutoff diameter of 2.5 μm
PM10: particle fraction with a 50% aerodynamic cutoff diameter of 10 μm
SOP: standard operating procedure

3 References

Chow J. Measurement methods to determine compliance with ambient air quality standards for suspended particles. J Air Waste Manage Assoc 1995;45:320-82.

Hoek G, Meliefste K, Oldenwening M. Measurement of PM10 and PM2.5 in ambient air using the Harvard Impactor. Protocol for the CESAR study, University of Wageningen, the Netherlands.

Hosiokangas J. Measurement of PM10 and PM2.5 in ambient air using the Harvard Impactor. Protocol for the 'Three cities study', KTL, Kuopio Finland.

4 Discussion

This protocol is based upon the protocols used for the EU studies CESAR, Three Cities Studies, Ultra-2 and TRAPCA. The filter weighing and conditioning criteria are based upon the 1997 EPA requirements.

5 Responsibilities

1 The Coordinator is 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 Equipment and materials

6.1 Equipment

1 Micro balance for filter weighing

6.2 Materials

2 Filters (37 mm 2 μm pore size Andersen Teflon filters with poly support ring, part no. SA240PR100
3 (If air conditioned weighing-room is not available) Desiccator to provide a constant relative humidity at between 30 and 40%, using NaBr0 + 2 aq.
4 Petri dishes (diameter at least 55 mm) or other equipment to transport and store filters
5 Flat pointed tweezers (to insert filter in the filter holder in the laboratory)
6 Refrigerator at 4 °C or less.
7 Bracelet used by computer engineers to prevent static electricity

6.3 Paper materials

6 Field forms to record data in the field
7 Laboratory forms to record weighing conditions, checks of external mass pieces, checks of control filters, rotameter calibration

7 Procedures

Filter handling should be done with flat pointed tweezers, without touching the sampled area of the filter. Touch only the support ring.

7.1 Conditioning of filters

Both before and after sampling the filter should be equilibrated at a constant relative humidity and temperature during at least 24 hours and not more than 48 hours. Based upon 1997 EPA requirements, the mean room temperature during a test day should be between 20 and 23°C. During one test day the temperature should be controlled within 2°C. Mean relative humidity during a test day should be in the range of 30-40%. If static charge problems are controlled well, relative humidity is allowed to be below 30%. During one test day it should be controlled within +/- 5%.

A temperature and relative humidity controlled weighing and filter storage room is preferred. If such a room is not available, a desiccator can be used containing a box with a saturated solution of a salt resulting in the required relative humidity. To achieve a relative humidity between 30 and 40% the desiccator could be filled with a saturated solution of $CaCl_2$.The petri dish should be open when used in the desiccator. The desiccator should be placed in the same room as the analytical balance to minimize the time that filters are exposed to another relative humidity. For the same reason, a small beakerglass with a saturated solution of $CaCl_2$ in the weighing chamber of the analytical balance is kept.

7.2 Weighing of filters

Filters shall be weighed no more than 60 days before the sampling period.
The microbalance must be suitable for weighing the type and size of the filters used. A reading precision of 1 μg is necessary. The balance must be calibrated at installation and recalibrated as specified by the manufacturer, but no less than once per year. Each center will use different balances, therefore no exact specifications are given here.

Before each filter weighing session, a series of checks should be conducted. If these checks are not satisfactory (after corrective actions), no weighing of filters can be performed.

1. Perform an internal calibration of the balance to make sure it is working properly
2. Document room temperature, relative humidity and barometric pressure from external measurement equipment. Barometric pressure can be obtained from appropriate routine meteorological stations
3. Use the most sensitive range of the microbalance. Conduct zeroing and calibration checks of the balance as specified in the manufacturers manual. Take readings only if the stability indicator is on and the indicated weight does not change for some seconds (20 in the Netherlands). Rezero after each filter (or external mass piece) weighing.
4. Weigh external mass pieces with weights close to the filter weight before each weighing session. The weight should be within 5 μg from the target weight (= average of the previous 10 measurements)
5. Weigh two blank Teflon filters. These two filters should remain in the laboratory in a temperature and humidity controlled place. The weight of both filters should be within 30 μg of the target weight. The target weight is the weight predicted on the basis of previous measurements of the filters. Before the start of the study, the filters have to be measured on at least five separate days. If check filters (blank and exposed) are used for a long period, the weight increases with time. Therefore the predicted weight is calculated as a moving average of the previous 10 weighing days.
6. Weigh two exposed (aged, see note 1) Teflon filters. These two filters should remain in the laboratory in a temperature and humidity controlled place (such as a desiccator). The weight of both filters should be within 30 μg of the target weight. The target weight is the average weight of the previous 10 measurements of the filters, using the same procedure as for the blank filters.
7. Weigh the same two blank and two exposed filters at the end of a weighing session to document that the weighing conditions are still appropriate.

NOTE 1: the exposed control filters should be aged, that is all the volatile components (such as ammonium nitrate) must have evaporated already. Keep the exposed check filters for 24 hour at 60 ± 5 °C.

NOTE 2: Use an effective technique to minimize static electricity problems. An electrostatic charge will prevent a microbalance from operating properly. To reduce static charge within the balance, it may be necessary to place a radioactive ionizing unit (i.e. Po-210 or Am-241) in the weighing chamber. It may also be necessary to pass the filters over an ionizing unit before they are weighed.

NOTE3: Magnetic fields (i.e. from a personal computer) may also disturb weighing, the use of a 'μ-metal' can be considered to limit this.

If all checks are satisfactory, filters can be weighed. To limit random errors and transcription errors, filters are weighed two times. The same filter should not be remeasured immediately but after at least a set of 10 filters have been measured. If the weight differs more than 5 μg, the two measurements are both discarded and a new set of two measurements is conducted until the two measurements agree within 5 μg. The average of the readings will be used in further calculations. If you accidentally bang the balance or the weighing table, new checks of the balance must be carried out.

If a desiccator is used, filters should be weighed within 1 minute after taking them out of the desiccator, in order to limit changes of relative humidity to affect filter weight. The lid of the desiccator should be closed every time after a filter is taken from the desiccator. If desiccators are used, after a filter has been measured once, store it in a second desiccator with the same relative humidity

7.3 Treatment of samples in the laboratory after weighing

After weighing the filter should be stored in the same plastic petri dish for later analyses, at 4 °C or lower.

7.4 Quality control procedures

7.4.1 Internal quality control

1. Document weighing conditions in the laboratory (Temperature, Relative Humidity and barometric pressure) on a control chart.
2. Document accuracy of microbalance with certified mass pieces. This should be conducted prior to each session of weighing filters, see section 7.1.3. Record the measured weight in a control chart
3. Weigh two blank Teflon filters each day that filters are weighed prior and post each session, see section 7.2. Record the weights in a control chart.
4. Weigh two exposed Teflon filters each day that filters are weighed prior and post each session, see section 7.2. Record the measured weights in a control chart.

NOTE Measures 3 and 4 are very useful in addition to measure 1, because environmental influences such as humidity may (and will!!) affect the weight of filters differently than a metal mass piece. Handle the filters with great care!

It is recommended to document all the control procedures in Microsoft Excel spreadsheets rather than on paper.

7.4.2 External quality control

The coordinating center will distribute six exposed and four unexposed filters to the centers after he field study to determine differences in the weighing procedure.

7.5 CALCULATIONS

Collected particle mass is calculated as the difference in weight of the filter before and after sampling. The average field blank is subtracted from this value:

$M = W2 - W1 - B$

W1 = adjusted filter weight before sampling (μg)
W2 = adjusted filter weight after sampling (μg)
B = mean adjusted filter weight change of field blank filters (μg)

Filter weights (W2 and W1) are the average of the two weight readings of the filter during each weighing session. Filter weights are adjusted for the deviation of the control filter weights on a weighing day from the nominal value. For blank filters, the average deviation of the two blank Teflon filters from the nominal value of the two blank filters is subtracted. For exposed filters, the average deviation of the two exposed Teflon filters from the nominal value of the two exposed filters is subtracted. The nominal value is defined as the average of the previous 10 weighign sessions.

8 Data records

- control charts weighing conditions
- control chart external mass pieces
- control chart control filters
- filter weighing sheets

9 Sample archiving

After analysis of the filters by the local laboratory (weighing and reflectance measurement), filters are stored in petri-dishes in a freezer at 4 ^{o}C or lower.

10 Implementation and application

NA