

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

Measurement of PM_{2.5} in ambient air with the Harvard Impactor

Identification code: SOP ULTRA /UoW-F-2.0		APPROVALS			
<input type="checkbox"/> Full SOP <input type="checkbox"/> 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
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<input type="checkbox"/> University of Wageningen					
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MEASUREMENT OF PM_{2.5} IN AMBIENT AIR WITH THE HARVARD IMPACTOR

1. Purpose and applicability

This SOP contains the protocol for performing measurements of PM_{2.5} in outdoor air for the EU-multicenter study ULTRA 2.

The principle of the method is that air is drawn by a pump through a size selective inlet (Harvard impactor) and next a filter on which airborne particles are collected quantitatively. The impactor is designed to sample particles of 2.5 µm with an efficiency of 50% at a flow rate of 10 l/min (larger particles less efficiently, smaller particles more efficiently). The collected fraction is denoted as PM_{2.5}. By weighing the filter before and after sampling the particle mass of the sample can be determined. Sample volume is determined by measurement of the sample flow before and after each sampling period and by recording of elapsed sample time.

2. Definitions

PIC: Principal Investigators Committee

PM_{2.5}: particle fraction with a 50% aerodynamic cutoff diameter of 2.5 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 PM_{2.5} in ambient air using the Harvard Impactor. Protocol for the CESAR study, University of Wageningen, the Netherlands.

Hosiokangas J. Measurement of PM₁₀ and PM_{2.5} in ambient air using the Harvard Impactor. Protocol for the 'Three cities study', KTL, Kuopio Finland.

"Preparation of standard operating procedures", SOP ULTRA/KTL-G-1.(*

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

4. Discussion

This protocol is based upon the protocols used for the CESAR and Three Cities Studies. The filter weighing and conditioning criteria are based upon the 1997 EPA requirements.

5. 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. "Local and temporal deviation from or local change of the SOP").

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. "Local and temporal deviation from or local change of the SOP"). (SOP ULTRA/KTL-G-1.).

5.4 This SOP was drafted by Gerard Hoek, Ph.D., at the University of Wageningen, the Netherlands.

6. Equipment and materials

6.1 Equipment

1. Impactor complete (impactor base, impactor body, impactor plate, nozzle and sampling head)
2. Spare nozzle
3. Spare impaction plates
4. Calibration adaptor
5. Spare O-rings
6. Pump unit
7. Micro balance for filter weighing

The impactors have been obtained from Air Diagnostics and Engineering, Inc. (Naples, Maine, USA). The impactor consists of the impactor base and body, a rubber ring to prevent leaks, the nozzle providing the size selective sampling, an impactor plate collecting the coarse particles and a sampling inlet. A calibrator adapter (a head with an 8 mm external diameter outlet plastic tube to which a rotameter can be connected) can be exchanged for the inlet to measure the flow rate before and after sampling. Between the impactor base and body the filter cassette is inserted. The filter cassette consists of two Andersen filter holder rings. In the filter cassette the filter is inserted above the drain disk which serves to support the filter.

Sampling can be performed with different pump units, therefore no exact guidelines are given. The pump unit should contain as a minimum a large pump capable of sampling considerably more than 10 l/min, a flow regulator and an elapsed time indicator. For the remainder of the protocol we assume that a constant flow of 10 l/min is maintained with a critical orifice. The elapsed time indicator records the time that the pump has been running.

The pump unit can be placed outdoors or inside a building, but the impactor inlet needs to be outside.

6.2 Materials

8. Filter holders (Andersen plastic filterholder rings, 37 mm part no SAFH240P; own filter holder for German center)
9. Drain disks (37 mm PE drain disk filter supports; partnr SN230800, Costar Europe; Millipore AP1003700 for German center)
10. Filters (37 mm 2 µm pore size Andersen Teflon filters with poly support ring, part no. SA240PR100)
11. Rotameters for measuring flows of 10 l/min or gasmeter to obtain integrated volume
12. Equipment to install the impactors in the field
13. (If air conditioned weighing-room is not available) Desiccator to provide a constant relative humidity at between 30 and 40%.
15. Petri dishes (diameter at least 55 mm) or other equipment to transport and store filters
16. Silicone oil (grade 316 or other but not less viscous to coat the impaction plates with a drop)
17. Flat pointed tweezers (to insert filter in the filter holder in the laboratory)
18. Refrigerator at 4 °C or less.
19. Bracelet used by computer engineers to prevent static electricity
20. Freezer

6.3 Paper materials

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

7. Procedures

7.1 Preparations in the laboratory

7.1.1 Check of critical orifice, external rotameter, elapsed time indicator and filters

Before using the pump units the critical orifice should be checked to ensure that the appropriate flow (10 l/min) is delivered. This can be done by measuring the flow through the critical orifice connected to the pump unit with a soap film meter or another flow measurement device. Compare elapsed times of the different pump units by running the pump units simultaneously. Elapsed times should not differ more than 5 minutes for a 24-hour period.

Document consistency of rotameters by regular comparison with the same internal standard. Record the readings in a table. The comparison has to be performed at least before the study period and after the study period. The internal standard can be a soap film meter, a dry gas meter or a rotameter that remains in the laboratory all the time. During a site visit to all study centers a comparison will be made with one certified standard flow measurement device (if available).

Check a new lot of filters carefully before using them. If several filters show deficiencies, send the filters back to the manufacturer. Specifically, the filters should be flat (no curvature) and should not contain small holes.

7.1.2 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. 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 below 40% but preferably in the range of 30-40% (which is the EPA criterion). During one test day it should be controlled within +/- 5%.

An air-conditioned weighing and filter storage room is preferred. If an air-conditioned weighing and storage 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. 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 a salt in the weighing chamber of the analytical balance is kept.

7.1.3 Weighing of clean filters

Filters shall be weighed no more than 30 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. Document room temperature, relative humidity and air pressure from external measurement equipment.
2. 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; five in Germany). Rezero after each filter (or external mass piece) weighing.
3. 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.
4. Weigh two (the same) 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 10 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.

5. 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 10 µg of the target weight. The target weight is the weight predicted on the basis of previous measurements of the filters, using the same procedure as for the blank filters.

6. Weigh two other blank and exposed filters at the end of a weighing session to document that the weighing conditions are still appropriate.

NOTE 1: the exposed check 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.

NOTE 3: 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.

Filter handling should be done with flat pointed tweezers, without touching the filter sampled area. Touch only the support ring. 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 you accidentally bang the balance or the weighing table, new checks of the balance must be carried out.

7.1.4 Filling the filter cassette with the drain disk and Teflon filter.

Use clean (ethanol) flat pointed tweezers to insert drain disk and Teflon filter in the filter holder rings. First, put the drain disk in one ring, next put the Teflon filter with the **support ring on the upside** on top of the drain disk. Finally put the second ring on top of the Teflon filter and clamp. The Teflon filter and drain disk are fixed now. The filter cassette can be transferred to the field in a petri dish with the sample ID. The upside ring must be marked, to ensure that the cassette is inserted correctly in the impactor in the field.

7.1.5 Cleaning and oiling of impactor plates

Use subsequently a hot soap solution, pure water and ethanol to clean impactor plates that have been used in the field (and new ones). Dry the plates with the porous side down on clean

tissue paper. The purpose of cleaning the impactor plate is to remove particles from the oil. It is not a problem if some oil remains on the plate.

Put one drop of the appropriate grade (grade 316) of silicone oil on the porous side of the impaction plate. The plate absorbs oil, so it may not be visible after a while. Don't add more oil until it is visible, as this may result in spilling of oil in the impactor during sampling. Store the oiled impactor plate in a clean petri dish for transport to the field.

7.1.6 Sample identification

The central laboratory should mark the petri dishes with an ID number that is meaningful and easy to understand in the field. A system could be:

wppyyzz,

with:

w = code for country (for example F for Finland)

p = pollutant indicator (P2 for PM2.5)

yy = day ('06')

zz = month ('01')

7.2 Field procedures

7.2.1 Installing equipment for the first time

1. Install the complete impactors such that the inlet is 2 ± 0.2 meter above the surface on which the sampler is placed. There should be at least 1 meter between the PM2.5 inlet and another low volume pump unit and at least two meters from a High Volume sampler inlet.
2. The inlet of the impactor should be placed upside down (inlet down, impactor base up). It has been the experience of the Harvard School of Public Health (George Allen) that this is necessary for outdoor sampling to prevent condensation in the impactor affecting the filter.
3. Connect the impactor base to the pump unit inlet.

7.2.2 Preparing the impactor unit for sampling

4. Insert subsequently a rubber ring on the impactor base, a filter cassette with filter and drain disk. The Teflon filter should be on the UPSIDE, that is to the impactor body side (facing the inlet and thus the incoming airflow)
5. Clamp the impactor body on the impactor base
6. Gently push the first part of the nozzle on the body, making a twisting movement. It is important not to push too rough, since this will damage the O-rings!
7. Place an oiled clean impaction plate in the nozzle. A clean plate should be used for each sampling period. The oiled part should face the incoming air.
8. Gently push the second part of the nozzle on the body, making a twisting movement. It is important not to push too rough, since this will damage the O-rings!
9. Gently push the calibrator adaptor on the nozzle, making a twisting movement. It is important not to push too rough, since this will damage the O-rings! When the impactor base has been connected to the pump unit and the pump turned on and warmed up for at least 3 minutes, flow rate can be measured. After measuring the flow rate, remove the calibrator adaptor gently, making a twisting movement

10. Gently push the sampling head on the nozzle, making a twisting movement. It is important not to push too rough, since this will damage the O-rings! The impactor is ready for sampling now.

7.2.3 Sampling flow

Sample flow is measured at the inlet of the impactor. Read the flow at the middle of the ball of the rotameter when it is stable. Rotameters should be kept in an exact vertical position when the flow reading is taken.

The sample flow will remain constant provided that the pressure drop over the filter due to sampled particles and humidity is not too large. Constant flow is important for two reasons. First, the sample flow (before and after sampling) is used in the calculation of sample volume. Second, particles larger than 2.5 μm will be sampled if the flow rate drops appreciably. Sample flow should at the start of sampling be 10 l/min (± 0.5 l/min). If the flow rate is lower than 9.5 l/min, a leak has probably occurred in the impactor system (possibly by a damaged O-ring). In case of a start flow below 9.5 L/min, flow should be measured at the pump inlet. If this flow is 10 l/min, a leak is present. If this flow is also low, the orifice is probably dirty (or an internal leak has developed). If so, the orifice should be taken from the pump unit and replaced by a spare orifice and cleaned in the laboratory. Re-install the orifice so that the thinner part of the orifice is facing the impactor (compare the other orifices in the pump units).

A flow rate after a sampling period lower than 9.5 l/min may occur due to filter clogging or due to a dirty orifice. When the sampled filter has been exchanged for a new clean filter and start flow is measured, you will be able to differentiate between the two causes.

If the end flow is frequently less than 9 l/min, use an external timer to turn the sampler ON and OFF during for example half an hour each hour. In this way a representative sample can be taken of a day while limiting the amount of collected particles. Note this carefully on the field form (Figure 2).

7.3 Treatment of samples in the laboratory after sampling

After sampling filters should be collected from the field and stored in the refrigerator at 4 °C or less within 48 hours. Alternatively the filter can be placed in the conditioning room directly. This is to limit weight losses due to volatilization of (among others) ammonium nitrate from Teflon filters which have been documented to occur in one week. Transport from a Teflon filter (in the filter cassette with the sampled side up) from the laboratory to the field and back should be done with plastic petri dishes. In the laboratory the Teflon filter is removed from the cassette. Filter handling should be done with flat pointed tweezers, without touching the sampled area. Only touch the support ring on the filter. The filter is next transferred to a numbered petri dish (sampled side UP). The drain disk and filter holder rings can be re-used. Filters can be stored in the refrigerator for a maximum period such that the time between retrieval from the field and the weighing does not exceed 30 days.

The weighing and conditioning procedures are the same as specified in section 7.1.1. After weighing the filter should be stored in the same plastic petri dish for later analyses, at -20 °C or lower.

7.4 Sample change instructions

This is a summary of this section documenting step by step how samples should be changed in the field. First, the steps for starting a new sample are given. Next, the procedures for dealing with an old sample are given. In the discussion it is assumed that you turn on the sampler and return the next day to change filters. Slight modifications are necessary if timers are used.

The laboratory should provide the following items:

- marked filter cassettes (containing filter plus drain disk) in petri dishes with sample identification
- petri dishes with oiled impactor plates
- field forms with instructions for sampling date
- rotameter for 10 l/min

7.4.1 Installing a new filter

1. Take a filter cassette containing a pre-weighed filter and a drain disk from a numbered petri dish and insert it on the impactor base. Record the ID number of the filter on the field form. Be sure to install the cassette with the Teflon side facing the impactor body (that is facing the inlet and thus the incoming airflow)
2. Install the impactor body, the oiled impactor plate, nozzle and calibrator adaptor on the impactor base.
3. Set the elapsed time indicator to zero
4. Turn the pump unit on. Have the pump warm up for at least 3 minutes
5. Measure the flow rate by connecting a calibrated rotameter to the calibrator adaptor. Read the rotameter at the middle of the ball if it is stable. The flow rate should be 10 l/min (± 0.5 l/min). Record the measured value on the field form. If the flow at the calibrator adaptor is below 9.5 l/min step 6 should be conducted (section 3.2)
6. *(only if the flow is below 9.5 l/min)* Remove the impactor base from the pump unit and measure the flow at the PM2.5 inlet of the pump unit. This flow should be equal to the flow measured above at the calibrator adaptor (a 0.5 l/min difference is acceptable). Record the measured value on the field form. If the flow is higher, there probably is a leak in the impactor. In that case, the impactor should be dismantled, checked and reassembled. Reconnect the impactor base to the pump unit.
7. Remove the calibrator adaptor and connect the sampling head to the nozzle. Be sure to position the impactor UPSIDE DOWN (inlet down, base up)
8. Record start time (local time, hh:mm) and start date

Collecting a sampled filter

1. Inspect the equipment and record irregularities on the field form (such as pump not running anymore, sampling lines not connected to pump, construction work near site)
2. Remove the sampling head and instead install the calibrator adaptor on the nozzle. Measure the flow rate using a calibrated rotameter. The flow rate should be 10 l/min (± 0.5 l/min). If the flow rate is below 9 l/min, use the external timer next time to limit the amount of collected particles. Alternatively, the orifice could be dirty and should be cleaned. When you measure the flow rate with a new filter installed, you will know which is the case. Record

the measured value on the field form.

3. Turn the pump off using the ON/OFF button
4. Read and record the elapsed time indicator and record the end time (local time, hh:mm) and end date
5. Remove the filter cassette containing the filter and drain disk from the impactor base. Store in the petri dish with the ID number of the filter (written on the field form).
6. Next, remove the impactor plate and take it back to the laboratory for cleaning. The order of points 5. and 6. is important as some oil may be spilled on the filter when removing the impactor plate.

7.5 Quality control procedures

7.5.1 Internal quality control

1. 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.
2. Document consistency of rotameters by regular comparison with the same internal standard. The internal standard can be a soap film meter, a dry gas meter or a rotameter that remains in the laboratory all the time. Record the readings in a table. The comparison has to be performed before and after the study period.
3. Weigh four blank Teflon filters each day that filters are weighed, see section 7.1.3. Record the weights in a control chart.
4. Weigh four exposed Teflon filters each day that filters are weighed, see section 7.1.3. Record the measured weights in a control chart.
5. Document weighing conditions in the laboratory (Temperature, Relative Humidity and ambient pressure) on a control chart.
6. Collect at least 10 field blanks during the study period. Take filter to the field, load the sampler with the filter, remove immediately (*leave in the field some days if timers are used*) and take filter to the lab. The blanks are used to determine the **detection limit and average field blank**. Collect field blanks equally spread over the entire study period.
7. Conduct measurements with two collocated samplers on the study for at least 10 days. This is to document precision of the measurements, which is critical information if one wants to analyze (true) variations in time of particle concentrations. Preferably these measurements should be spread equally over the study period. If this is not feasible, perform the collocated measurements before or after the study period.

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.

7.5.2 External quality control

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

During a site visit to all study centers a comparison will be made of the used rotameters with one certified standard flow measurement device.

7.6 Calculations

The PM_{2.5} concentration is calculated as :

$$C = M/V$$

C = concentration of PM_{2.5} (µg/m³)
M = collected particle mass (µg)
V = sample volume (m³)

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 weight change of field blank filters (µg)

Filter weights (W2 and W1) are adjusted for temperature, relative humidity and barometric pressure during the weighing session using the procedures developed in the EXPOLIS study and the manufacturers manual for the specific balance.

Sample volume is calculated as the product of the mean flow rate and the sampling time :

$$V = ((A*F1+A*F2)/2)*T/1000$$

A = calibration factor of rotameter used for taking flow reading
F1 = adjusted rotameter reading before sampling (l/min)
F2 = adjusted rotameter reading after sampling (l/min)
T = sampling time (minutes)
1000 = transformation of liter to m³

Rotameter readings are adjusted for the factor calculated from the comparison with an external, certified flow measurement device.

Given the precision of concentration determination, only one decimal will be given when individual concentration data are presented.

Concentration data will not be accepted if:

- The elapsed time is less than 16 hours (67% of time)
- Start flow < 9.5 l/min or > 10.5 l/min
- End flow < 8 l/min
- Weighing of check filters or mass pieces unacceptable (section 7.1.3)

8. Data records

- control charts weighing conditions
- control chart external mass pieces
- control chart check filters
- calibration table rotameter
- filter weighing sheets
- field forms containing sampling characteristics
- spread sheet containing all information for a specific sample
- UoW will provide Excel spreadsheets for the first three charts.

9. Sample archiving

After analysis of the filters by the local laboratory (weighing and reflectance measurement), filters are stored in clean petri-dishes in a freezer at -20 C or lower until they are sent to the laboratory for elemental composition determination.

10. Implementation and application

NA

11. Attachments

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

Figure 2. Field form PM2.5 Impactor.

Figure 3. 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. Field form PM2.5 Impactor.

FIELD FORM PM2.5 IMPACTOR

UNIT NUMBER			
NUMBER FILTER			
INSTALLATION DATE			
START DATE			
START TIME			
ELAPSED TIME INDICATOR START			
NUMBER ROTAMETER			
START ROTAMETER READING UPSTREAM			
ROTAMETER READING DOWNSTREAM ¹			
COLLECTION DATE			
END DATE			
END TIME			
NUMBER ROTAMETER			
END ROTAMETER READING UPSTREAM			
ELAPSED TIME INDICATOR END			
IRREGULARITIES			

¹ to be measured if flow rate below 9.5 l/min

Figure 3.

SOP CONFIRMATION SHEET

Measurement of PM_{2.5} in ambient air with the Harvard Impactor

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					
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10					

Coordinator fax : + 358 - 17 - 201 265