

MDN Project Specific SOP FGS-MDN-01

Original Written May 1999 By Bob Brunette

Revised March 17, 2003 by Kirsi Longley/Bob Brunette

MDN Glass Sample Train Collection and Deployment-Trace Metal Clean Sample Handling

The following Standard Operating Procedure (SOP) is designed to give the new site operator the detail necessary to perform sample retrieval and sample glassware deployment for the Mercury Deposition Network (MDN). Although this SOP may seem lengthy, it provides step-by-step instructions on how to:

- Retrieve the previous weeks Hg Wet Deposition sample
- Complete the MDN Observer Form for the Previous Weeks sample
- Deploy the next week's sample glassware
- Start the next weeks MDN Observer From
- Retrieve last weeks Belfort Rain Gauge chart
- Deploy the next week's Belfort Rain Gauge chart

Each site operator should carry out these instructions as they cover critical steps for trace metals clean sample handling. It is equally critical to follow these instructions consistently each week in order to standardize the sample handling and glassware handling for each sample. Not following these procedures could result in contaminating samples and/or systematic or random error in this process. This SOP is also designed as a review and refresher to veteran site operators.

Sample Collection From Previous 7 day sample:

1. <u>Approach Aerochemetrics (ACM) collector from downwind</u>. Upon arrival at site, before approaching ACM collector, determine which way the wind is blowing. Rule of thumb is to always approach the collector facing into the wind.



Recording observations of the site.

- <u>MOF Section 10: Remarks</u> Make observations as to the condition of the collection site and equipment. Record observations in Section 10: Remarks on MDN Observer Form (MOF). In general, observations should include the following:
 - Is it raining during sample change? If yes, is the lid open and resting on the dry side bag?
 - If it is not raining, is the lid covering the funnel? If yes, is the lid seal firmly in place over funnel?
 - Is the lid seal in good condition (no tares, dirt etc)
 - Is rain sensor free of debris?
 - Is the equipment generally in good condition?
 - Are there any unusual conditions near the site (fire near by, construction, heavy snow, etc.)
 - Does the collector have debris on it (bird poop, dirt, etc)?
- 3. <u>MOF Section 9: Enclosure Temp</u> Open the enclosure door on the ACM collector and record the Max/Min temperature in Section 9: Enclosure Temperature of MOF.

If the Max/Min temperature within the collector is out of range (outside of 40-100F), adjust the thermostat to obtain the proper temperature range (increase temp on thermostat if below 40F -Decrease Cooling fan temperature if above 100F).

4. After recording Enclosure Temperature on the MOF<u>, reset the Max/Min</u> <u>thermometer</u> by turning the button on bottom of thermometer clockwise. The max and min indicator needles should both arrest to the same position.



Lowering jack to gain access to sample bottle.

- 5. <u>Lower the sample bottle jack</u> until the bottom of the thistle tube clears the mouth of the sample bottle.
- 6. While wearing a new set of gloves, retrieve the sample bottle cap from zip lock bag, and screw cap onto sample bottle. Ensure the cap is firmly in place. Once cap is in place, remove the sample bottle from bottle jack.
- MOF Section 5: Sample Condition Make observations of wet deposition (rain) in sample bottle. Identify any debris in sample and check the appropriate box in Section 5: Sample Condition of MOF. If any of the following debris is present in the sample, check yes next to the appropriate category in section 5.
 - Bird droppings
 - Cloudy or discolored
 - Soot/Ash/Dirt Particles
 - Insects/Animal matter
 - Leaves/Twigs/Pollen/Plant Matter

-After checking the appropriate box in section 5, identify the debris further in Section 10: Remarks, in as much detail as possible. For example: 4 misquotes, 2 large black particles...looks like soot particles; 1 piece of plant matter...looks like a pine needle; etc....

-If debris is present but does not fit any of the categories above, describe the debris in your own words and record this in section 10.

- If there is no debris present, check the NO box for each category of Section 5: Sample Condition.

 Once all observations of sample are complete, <u>place sample bottle in its original</u> <u>sample bottle bag, and zip lock the bag closed.</u> Place the sample bottle bag in the back of enclosure.

This is an important step as the Ziplock bag protects the outside of the sample bottle from collecting dirt and particles. Dirt and other particles typically have mercury adsorbed onto their surfaces. If this material was to accidentally get into the sample bottle, it could significantly contaminate the sample.

Although the bottles are wiped clean when they are received at Frontier, our goal is also to minimize bringing outside sources of particles into Frontier's clean rooms.

If your sample is received at the HAL with the Ziplock bag open, the sample will be flagged via a special code. This will be reflected in the Quality Rating Code that is assigned to each MDN sample.



Completing MOF.

- 9. <u>MOF Section 3: Bottle Record the sample Off Date and Off Time</u>. For time please use military hours (example: 2pm = 1400hrs). Also use the standard time for your region (i.e. Pacific Standard Time).
- MOF Section 3: Bottle Check the sample bottle ID recorded in Section 3: Bottle of MOF vs the bottle ID inscribed on the bottle. This is critical to ensure that the sample is processed properly upon arrival at the HAL.
- 11. <u>MOF Section 8: Overflow</u> Check the overflow container on sample bottle jack for overflow. If no overflow is present, check the No box in Section 8: Overflow.

If overflow is present, check the Yes box in Section 8: Overflow. Pour the water from the overflow container into a plastic graduated cylinder. Measure the overflow and note the volume in milliliters and record this in the appropriate place in section 8.

Important Notes On Overflow:

Water found in the Overflow Dish should coincide with a full sample bottle:

If overflow is present, the sample bottle should be full to the top. Note if sample bottle is full in Section 10: Remarks. This will help those processing the sample to see if in fact this was an overflow from the sample bottle. If the sample bottle is not full, check to ensure that the sample glassware was connected and that there was not cracks in the glassware where water could have escaped the sample train and entered the sample overflow dish. If the sample train was not intact, it is important to document all of your observations in Section 10: Remarks.

<u>Cases where there is water in the overflow dish however the sample bottle is not full:</u> In some rare cases, the overflow dish could have some water in it but this water is not considered to be overflow. If the sample bottle is not full to the rim, it is likely that rain has fallen through the gap between the funnel and the rim of the chimney and collected in the overflow dish. This again is in rare cases where high winds have driven the rain sideways and into this gap described above. In this case, simply make a note of this on the MOF, pour the water out of the overflow dish and dry the dish out with a paper towel.

Why discard the overflow found in the dish?

The water that has entered the overflow dish is contaminated from contact with the dish and is not part of the sample. Please do not try to add the overflow into the sample bottle as this will contaminate and disqualify your sample. Once you have measured and recorded the volume of the overflow, pour it out and dry the inside of the overflow container with a paper towel.

12. <u>Close enclosure door. Remove old dry side bag from dry side bucket</u>. Observe the condition of the dry side bag (dirt, bird droppings etc). Record any observations in Section 10: Remarks.



Removing old dry-side bag.



Securing new dry-side bag.

13. <u>While wearing clean gloves</u>, retrieve the new dry side bag from the cooler that contains the clean glassware. Open the bag and place into bucket. Pull top of bag over edges of bucket and pull bag down so it is firmly in place.

Special Note:

Once you have pulled down the dry side bag over the edges of the dry side bucket, you have exposed a clean surface for the lid seal to rest upon. Avoid touching the edge of the dry side in order to preserve this as a clean surface.

The function of the dry side bag is to ensure the lid seal is free of dust and particles. When the collector opens during a rain event, the lid seal moves from covering the funnel and comes to rest on the dry side bucket, which is covered with the dry side bag. The dry side bag and bucket protects the location of the lid seal that comes in contact with the glass funnel. It therefore is critical to ensure that the lid seal stays particulate free so it will not introduce contaminates into the glass funnel.

Cleaning the Lid Seal:

14. Wearing clean gloves, take the bottle of distilled water from enclosure and wet a clean paper towel.



Cleaning lid seal as it cycles to cover the dry-side bag.

15. Holding the wet paper towel in one hand, apply a few drops of water to rain sensor. <u>As collector lid cycles to cover dry side, wipe the lid seal with the wet paper</u> <u>towel</u>. While wiping down the lid seal, observe the condition of seal and record any observations in Section 10: Remarks. If lid seal is torn, punctured, or looks discolored, call Kirsi Longley at Frontier Geosciences Inc (877-622-6960) to receive a new lid seal.

Removing old sample train:

16. <u>Remove the glass funnel-thistle tube train from the Wet side of collector by</u> <u>carefully lifting this unit straight up</u>. Observe the condition of thistle tube and funnel (dirt, debris, etc) and record any observations in section 10: Remarks.



Removing funnelthistle tube train from chimney. (Notice how they are being carefully lifted straight up out of the collector.)

17. <u>Separate the used funnel and thistle tube by removing the blue clip.</u> Retrieve the plastic bag from enclosure (placed there from last week). Place the used thistle tube in the original plastic bag. Keep all used glassware outside of enclosure. Be sure not

to mix the used glassware from the previous week with the new glassware for the week to come. Always keep the new glassware in the shipping cooler until needed.

18. <u>Complete Section 6 part 1:</u> "Site Operations" on the MOF.

The following questions in this section are designed to ensure that the site operator has checked that the ACM collector, Belfort and Event Recorder are working properly. This section is a prompt to make sure that the site operator understands that this needs to be done each week the sample is picked up.

- (1) Sensor heater and motor box are operating properly. Lid is in the correct position.
- (2) Rain Gauge operated properly during the week.
- (3) Event Recorder worked properly and indicates the collector lid opened and closed promptly after each precipitation event.



Feeling if the sensor is heating properly.

If problems are discovered during the weekly check of the MDN equipment, the site operator should check the "No" box adjacent to the piece of equipment they are having problems with. You should note in Section 10: Remarks, the problems you a discovering. IMPORTANT: Your job does not end here! You must <u>immediately</u> begin to troubleshoot the problem in order to correct any problems found.

Problems with Equipment:

Aerochem Rain Collector: If the lid is in the open position, and it is not raining, this is a definite indication that something is wrong with your collector. Further, if it is raining and your collector is not opening to collect rain, your collector needs immediate attention. Obtain your NADP/MDN Site Operations Manual and turn to Appendix A: "Aerochem Metrics Rain Collector". Page A13 of this appendix has a thorough and complete Troubleshooting guide, which describes a number of "symptoms" and then details how to diagnose the problem. When you feel you have identified the problem, you must call Kirsi Longley or Bob Brunette at the HAL in order to request replacement parts.

Belfort Rain Gauge and Event Recorder: An indication that your Belfort is not working properly could be a number of things:

- Belfort accumulation trace did not advance from Tuesday to Tuesday
- Belfort accumulation pen does not coincide with the Event Recorder
- Event Recorder pen is not deflecting up when there is rain accumulation and dropping back down when rain stops and
- Pens not tracing on chart paper.

If these or other issues are discovered, the site operator should immediately refer to NADP/MDN Site Operations Manual and turn to Appendix B: Belfort Recording Rain Gauge. Page B13 of this appendix has a complete Troubleshooting guide for the Belfort Rain Gauge.

Things to remember if your collector or rain gauge is not working properly:

- (1) The site operator is principally responsible for maintaining and operating the site.
- (2) The site operator is principally responsible for determining when a piece of equipment is not working properly. The ACM and Belfort must be assessed each Tuesday to ensure they are working properly.
- (3) If a problem is discovered, it is principally the site operator's responsibility to correct the problem immediately. This means referring to the Troubleshooting guides above to help in correcting the problem.
- (4) If the problem cannot be corrected or needs a replacement part, <u>the operator</u> <u>must call the HAL immediately.</u> It is not sufficient for the site operator to <u>note the problem on the MOF in order to communicate a problem exists.</u> It is not up to the HAL to discover that you have a problem through reviewing your MOFs. The HAL reviews your MOFs days after you did or did not discover a problem. Unless the HAL receives a call directly, the problem will persist for weeks before it is discovered.
- (5) If the site is down and not working properly, valuable data is lost which is not recoverable.
- (6) The goal for operating the site is to always have a fully operating collector and rain gauge in order to catch each week's sample.
- (7) In order to minimize down time due to collector malfunction etc, the site operator must do the following:
 - (a) Attempt to diagnose the problem.
 - (b) If need be, refer to the NAD/MDN Site Operations Manual and the Troubleshooting guides therein.
 - (c) Call the HAL the day the problem was discovered with your diagnosis.
 - (d) Kirsi Longley or Bob Brunette will discuss the problem over the phone and verify your diagnosis.
 - (e) The HAL will send replacement parts if needed and direct the site operator what the next course of action should be.

19. <u>Cleaning The Equipment:</u>

Special Cleaning Note:

At this point in the process, it is now the best time to perform routine "house cleaning" of the MDN ACM. This is the point where you have taken out, capped and bagged the previous week's sample. Further, the new set of glassware for next week's sample is still sitting in the cooler, bagged and protected. This means that your cleaning activities can in no way contaminate the previous week's sample or next weeks glassware. Therefore this is the best opportunity to thoroughly clean the following items. I recommend to use paper towels and water or "409" glass cleaner:

- a) Wipe down the top of the lid and surrounding sides.
- b) Wipe down the 4 "arms" of MDN lid.
- c) Clean off the rubber boots at the base of each arm.
- d) Wipe down the top of the ACM in general.
- e) Check the sides of the enclosure. On occasion, wipe the sides down with water and a cloth.
- f) Clean any debris off the sensor using 409 and distilled water. Stubborn debris can be removed by scrubbing sensor grid using a toothbrush.
- g) Inside the enclosure, clean the floor of the collector so it is dirt and bug free.
- h) Clean any other surfaces or areas, which you feel, are beginning to build up dirt and other debris.



Cleaning the lid.



Cleaning the boots.



Wiping down chimneys.

Deployment of New Sample Gear:

- 20. <u>Wearing clean gloves, remove new sample bottle from sample cooler</u>. Obtain a new MOF and record the Sample Bottle ID number in section 3 of MOF.
- Record the date and time the new sample glassware is deployed.
- Complete Sections 1&2 of MOF and place MOF into a zip lock to protect from water damage.

Place bagged MOF into collector enclosure for next week's sample retrieval.

21. <u>Remove new sample bottle from zip-lock bag and place into overflow container on sample bottle jack. Leave the bottle cap in place.</u>



Placing new sample bottle in overflow dish.

- 22. <u>Remove the new thistle tube in the plastic bag from shipping cooler.</u> Leave the <u>Ziplock bag on the thistle tube until instructed to remove during this process</u>! It will act as a barrier to protect the thistle tube while you are deploying your glassware.
- 23. <u>Place the plastic bag (containing the thistle tube) under one arm with the Ziplock end</u> (the end that seals) facing away from you.
- 24. With the thistle tube under your arm, open the Ziplock end of the plastic bag with your free hands.
- 25. Still holding the plastic bag under one arm, retrieve the new glass funnel. Leave it in the Ziplock bag until directed to remove! It will protect the funnel while you are handling the glassware.
- 26. Open the Ziplock bag containing the glass funnel, holding the bag away from your body. Hold the glass funnel through the bag.



Connecting funnel and thistle tube using clean sample handling.

- 27. Holding the glass funnel through the Ziplock bag, insert the "male end" of the funnel's ground glass joint into the "female end" of the thistle tube.
- 28. Once connected, slowly remove the thistle tube from the plastic bag via the glass funnel.
- 29. <u>Place the blue clip over the ground glass joint</u> to secure connection. **Do not twist or try to friction fit this ground glass joint connection**. The clip provides plenty of support for this connection.
- 30. Holding the entire unit by the funnel (**still holding funnel through Ziplock bag**), approach collector.
- 31. Insert the now connected funnel-thistle apparatus (both still with the Ziplock bags on) tube down into the 1st chimney of the collector. Be sure to take care and not scrape the bottom of thistle tube down the inside of the chimney. However, if this were to happen, the Ziplock bag will protect the glassware.



Bag covering new funnel.

- 32. <u>The funnel should now be resting on the lip of the chimney</u>. <u>Carefully remove the</u> <u>Ziplock bag covering the funnel AND the Ziplock bag on the thistle tube</u>.
- 33. Store the plastic bags in the enclosure for next week.
- 34. <u>Raise the sample bottle on the bottle jack up to just below the thistle tube, allowing</u> room to remove the bottle cap.
- 35. <u>Before this next step Change Your Gloves then remove the bottle cap and place</u> <u>the bottle cap in the bottle bag inside enclosure.</u> Seal the Ziplock bag that now contains the sample bottle cap. This is an important step! During the next 7 days, the Ziplock bag will protect the cap from potential contaminants.
- 36. <u>Adjust the height of the sample bottle to meet the thistle tube-funnel.</u> Ensure that the weight of the glass funnel and thistle tube is primary supported by the mouth of the sample bottle. This is done by adjusting the bottle jack upwards such that the bottle, being raised, begins to raise the thistle-funnel upwards. You can observe the funnel raising above the rim of the chimney cap or the aluminum chimney. Adjust the funnel height such that it is just barely resting on the chimney cap or the rim of the chimney.
- 37. From inside enclosure, look to make sure the sample bottle, thistle tube, and funnel are connected.



Notice the thistle tube bulb resting securely on top of bottle mouth.

38. Stand on the North side of the MDN ACM (remember that each MDN ACM rain sensor is pointed magnetic North). Facing away from the sample chimneys with your back to the open funnel, cup the rain sensor on the right side with one hand and blow off any remaining water. The lid should cycle back and come to rest on top of the funnel. Check to make sure the lid seal makes a good seal with the top of the glass funnel. Make any necessary adjustments to the height of the bottle-thistle tube-funnel.

Special Note: Regards Blowing Water Off The Rain Sensor

The step described above is very important. Your sample funnel is now open and exposed. If the operator was to blow the water off the sensor toward the open funnel, there is great potential for contamination of the sample to occur. If the operator has fillings in their teeth, it is likely that their saliva has detectable concentrations of mercury. Dental fillings are mercury amalgams, and therefore have a significant amount of mercury in them. Further, there is potential to blow debris/particles toward the open funnel which could also cause contamination. Always blow the sensor off with your back towards the open funnel. (See picture on next page).



Notice he is facing away from the exposed new sample train.

- 39. <u>Place the sample from the previous week, used thistle tube, and MOF from previous week into shipping cooler.</u>
- 40. Close enclosure door and clean up site (used gloves, paper towels etc)

Changing Chart Recorder Paper In Belfort Rain Gauge:

The following are instructions on how to complete the previous week's rain gauge chart, deploy the new chart for next week and complete your MDN MOF.

- 1. Unlock Rain gauge door and lock in the open position.
- 2. **Defining The End Point (stop point) of the Belfort Precipitation Record:** Make a vertical "tick mark" on chart paper with the rain gauge pen where it currently rests, to signify the end of the sample period. This can be done by gently moving the pen reservoir tip up and down on the chart paper with your finger. This will generate a vertical "cross hair". Also, move the chart drum with your hand forward and back to create the vertical "cross hair". You have now clearly identified the stop point of the precipitation record.
- 3. Event Recorder Operation Verification: Check to ensure that the Event Recorder was working when you had the MDN ACM Collector open earlier in this procedure (when removing and deploying your glassware). You should see on the chart paper an ER pen spike at the end of measuring period. The ER pen should have moved to the open or "Up" position during this time. It should have then returned to the closed or "Baseline" position signifying the ACM lid was closed over the wet side of the collector.
- 4. <u>Move both the rain gauge pen and event recorder pen back off chart paper</u> by moving pivot bracket out away from the chart drum.
- 5. Unscrew the nut on top of the chart recorder drum.
- 6. <u>Remove the chart recorder drum from the clock timer</u> (pull straight up on drum from inside of rain gauge housing). Rotate drum from inside the gauge housing and pull the drum outside of the Belfort access door.
- If your rain gauge clock operates on batteries, ignore this step and go to 8. Rewind clock 10 times gently or until you begin to feel resistance. <u>Do not overwind clock -</u> <u>this will damage the timing unit.</u>
- 8. <u>Remove the chart recorder paper (from previous week)</u> from chart recorder drum by pulling out clip.
- 9. <u>Record sample end date and time</u> on chart recorder in the place provided.

- **10.** <u>Verify Belfort Clock Timer Is Operating Properly:</u> Examine the rain gauge chart to ensure that the precipitation recorded ended on the current day and time. If the day and time are not accurate, insure that the Belfort clock is being wound sufficiently. If you are sure the clock was wound sufficiently, call the HAL for a replacement clock immediately. If your clock operates on batteries, change the batteries and label the new batteries with the date they were started. This will help you determine how long your batteries should last.
- 11. <u>Empty Rain Gauge Bucket:</u> Check top of rain gauge and observe level of water in rain gauge catch bucket. Remove top housing of rain gauge collector and dump out the catch bucket until approx one inch of water is left standing in bucket.



Removing the Belfort bucket.

- 12. Place chart recorder paper from previous week in sample shipping cooler.
- 13. Obtain a new sheet of chart recorder paper and record start date and start time on chart paper.
- 14. Facing chart recorder paper, fold right hand edge behind the chart paper.
- 15. Place the left hand of chart paper on drum and line up left edge with the clip notch on the cart paper drum.
- 16. Wrap the rest of the chart paper around the drum.
- 17. Place the chart paper clip down the fold of the chart paper. The bottom of clip should fit into hole on bottom edge of the chart drum. The top of clip should fit into the top notch on edge of chart drum. <u>Make sure the chart paper is flat and smooth around the chart drum to ensure pen can move flat over surface</u>.
- 18. Replace chart drum with new chart paper on top of timer.

- 19. Screw the top nut on top of chart drum until snug.
- 20. Lower the rain gauge pen and event recorder pen back to the chart recorder paper by moving pen pivot bracket back toward chart drum.
- 21. <u>Check both pen reservoirs to ensure they are full of ink.</u> If not, add ink to the reservoir. At close inspection, the ink reservoirs should have a crown of ink, almost brimming, to ensure that there is plenty of ink for the week's trace.
- 22. Line up rain gauge pen (lower pen) with the corresponding time and day on chart paper and let pen rest on this position. THE RAIN GAUGE PEN DOES NOT HAVE TO START AT ZERO ON THE RAIN GAUGE CHART PAPER. As long as the start and end points are clearly marked, then we can calculate the daily precipitation totals.



Cross hairs.



Filling pen reservoir with ink.

- 23. <u>Ink a "cross hair" on the start point of the new week's rain gauge trace</u>. Move the tip of the reservoir up and down **gently** with your finger to produce the vertical "cross hair". Move the chart drum forward and back to signify the horizontal "cross hair. Ensure that you have returned the pen to the starting point (current date and time). You now have established a well-defined start point for your rain gauge chart. Further, this helps to get you pen "inking" on the chart paper.
- 24. Close and lock rain gauge door.

Complete the MDN MOF and Interpret Rain Gauge Chart:

25. Return with shipping cooler to your office or some place comfortable to finish filling out the MOF.

- 26. Interpret Rain Gauge Chart: Complete section 7: Precipitation Record of MOF. This is simply done by transferring the information from the rain gauge chart into section 7 of the MOF. Calculate the daily precipitation values for each day of last week's sample using the rain gauge chart (see page 25 of the NADP/MDN Site Operations Manual and the MDN Rain Gauge Interpretation SOP for instructions). The daily total rain volumes, recorded in inches, must be recorded on the MOF in Section 7: Precipitation Record.
- 27. <u>Supplies:</u> Field Forms, Gloves, Rain Gauge Charts, Rain Gauge Ink, MDN ACM Air Filters, Lid Seal Pads. <u>Check to see if you need any supplies as listed in section 11 of MOF</u>. Circle whatever supplies you need in the appropriate section of the MOF. By circling an item in Section 11 of the MOF, this tells our shipping department to ship the needed supplies with next weeks glassware.

Important Note Regards To Supplies:

Be sure to factor in at least 2 weeks from the time you order supplies and the time you will receive them. In other words, please do not wait to order supplies after you have used the last of your supplies. For example, when you have 3 rain gauge charts and 3 MOFs left, this would be a good time to order new ones. Again, please allow for at least 2 weeks before you receive replacement supplies.

- 28. Place completed MOF and Rain Gauge chart in shipping cooler with sample. Secure sample shipping cooler lid with shipping tape by circling the entire cooler with tape.
- 29. Ship the cooler to the following address within 48 hours of collecting sample. We suggest using 2 day UPS/FedEx service if possible:

Attention: Kirsi Longley

 (phone:
 206-622-6960)

 (toll free:
 877 622 6960)

 (fax:
 206 622 6870)

 (Email:
 kirsil@frontiergeosciences.com)

MDN Site Liaison Frontier Geosciences Inc 414 Pontius Ave. N, Suite D Seattle, WA. 98109

If you have any questions in regards to the procedure outlined above, please call Kirsi Longley at the number above.