

**Asia – Pacific Mercury Monitoring Network
(APMMN)**

**Mercury Wet Deposition Network Field Standard
Operating Procedures**

Version 1.3-, February, 2015

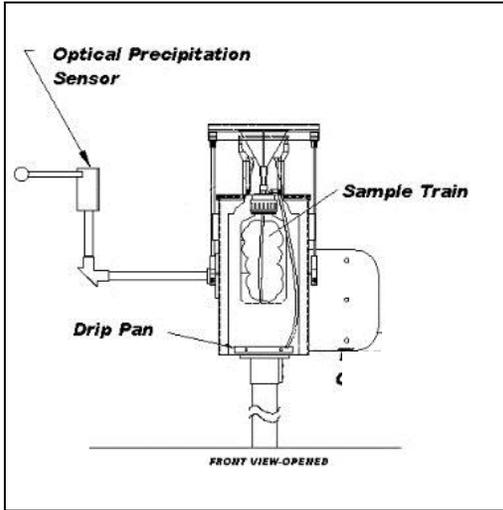
The following Standard Operating Procedure (SOP) is designed to give the new site operator the detail necessary to perform sample retrieval and sample glassware deployment for the Asia – Pacific Mercury Monitoring Network (APMMN). This SOP provides step-by-step instructions on how to:

- Retrieve the previous weeks Mercury (Hg) wet deposition sample
- Complete the Network Observer Form (NOF) for the previous weeks sample
- Deploy the next week's sample
- Start the next weeks NOF
- Report the Precipitation data from a collocated rain gauge.

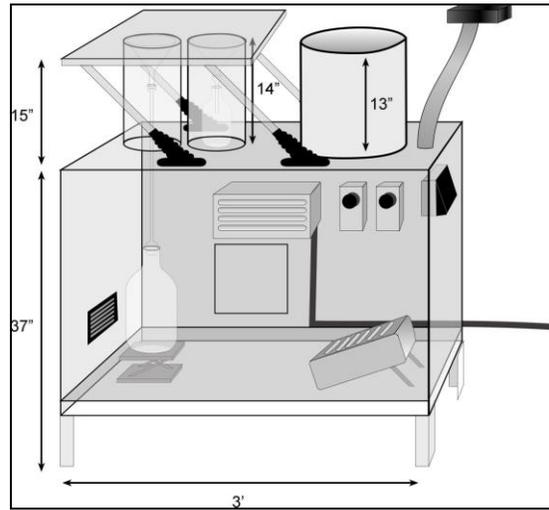
Each site operator should carry out these instructions as they cover critical steps for trace metals clean sample handling outlined in USEPA Method 1669. It is equally critical to follow these instructions consistently each week in order to standardize the sample and glassware handling for each sample.

Note to Operator: There are a number of different collector designs used in this network, and care by the site operator should be used in interpreting this manual, which is written to be used with all collector designs. Notes appear in this manual when different conditions/steps are necessary for individual designs. The collectors being used in this network are shown in Figure 1.

NCON Model MDN 00-125-2



NADP-style Aerochem 301



Japanese-style Model A-TF1



Figure 1. Sampler Designs accepted by the APMMN. Determine the model of sampler that you have at your site, and follow the specific design instructions

When to Change your sample: All network samples are changed on Tuesday mornings, between 8 and 10 AM local time. Please note the exact time on the NOF. If precipitation is occurring during this time and the event is predicted to end within the next day you can wait until Wednesday to change the sample because an 8 day sample is not invalidated. Samples greater than 8 days will be invalidated.

Sample Collection from Previous 7 day sample:

1. **Approach collector from downwind.** Upon arrival at site, before approaching collector, determine which way the wind is blowing. Rule is to always approach the collector facing into the wind.



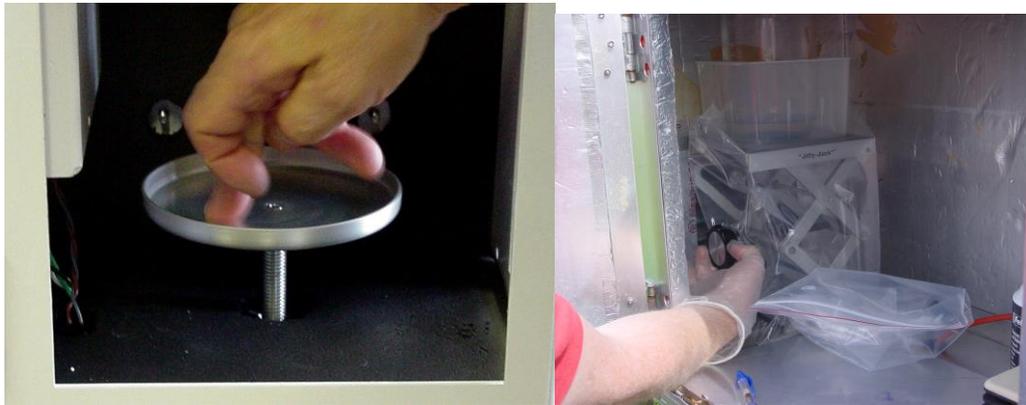
Recording observations of the site.

2. **NOF Section 10: Remarks** - Make observations as to the condition of the collection, site and equipment. Record observations in Section 10: Remarks on NOF. Generally, observations should include the following:
 - Is the lid in the correct position?
 - Is the lid is in the open position, and it is not raining?
 - Is it raining and the lid is not open?
 - Is the lid seal in good condition (no tears, dirt etc)?
 - Is rain sensor free of debris?
 - Is the equipment in good condition?
 - Are there any unusual conditions near the site (fire nearby, construction, heavy snow, etc.)?
 - Does the collector have debris on it (bird feces, dirt, etc.)?
 - Aerochem: is the sensor warm and evaporating water?
 - Is the sampler cycling (opening and closing) constantly?
 - Is the motor box making a “grinding” sound?
 - Is there no water in the sample, even though it has clearly rained?

3. **NOF Section 9: Enclosure Temp** - Open the enclosure door on the collector and record the maximum and minimum temperature in Section 9: Enclosure Temperature on NOF.

If the Max/Min temperature within the collector is out of range (outside of 4.5-38°C), adjust the thermostat to obtain the proper temperature range (increase temp on thermostat if below 4.5°C –decrease cooling fan temperature if above 38°C).

4. After recording Enclosure Temperature, **reset the Max/Min thermometer.** After resetting, the max and min readings should equal the current temperature.
5. **While wearing a new set of gloves, unscrew the base until the bottle is suspended by the funnel and cap, then remove the funnel the compression fitting.**



Lowering jack to gain access to sample bottle (NCON left, Aerochem, right)

6. **Remove the collection cap and retrieve the sample cap from the bag and screw cap onto sample collection bottle.** Ensure the cap is firmly in place
7. **NOF Section 5: Sample Condition** - Make observations of the sample. Identify any debris and check the appropriate box in Section 5: Sample Condition on NOF. 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(s) in section 5, identify the debris further in Section 10: Remarks, providing as much detail as possible. For example: 4 misquotes, 2 large black particles that look like soot; 1 piece of plant matter that 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.



Completing the Network Observer Form.

8. Once all observations of the sample are complete, **place sample bottle in its original sample bottle bag, and seal.**

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

9. **NOF Section 3: Bottle** – Record the sample Off Date and Off Time. Use twenty four hour time (example: 2PM = 1400hrs), from your local time zone.
10. **NOF Section 8: Overflow** - Check the overflow container on sample bottle support for water. If no overflow is present, check the NO box in Section 8: Overflow.

If overflow is present, DO NOT ADD OVERFLOW TO THE SAMPLE, and check the Yes box in Section 8: Overflow. Then pour the water from the overflow container into a graduated cylinder. Measure the overflow and record the volume in milliliters in section 8.

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 the processor determine if this was overflow from the sample bottle.

Cases where there is water in the overflow dish but the sample bottle is not full:
If the sample bottle is not full to the rim, it is likely that precipitation has leaked out of the sample bottle collection assembly. Ensure the sample train was connected properly and the funnel was seated correctly. Document all of your observations in Section 10: Remarks. Try to determine why overflow was present and the bottle was not full, then correct these conditions for the next sample.

Why Discard the overflow found in the dish?

The water in the overflow dish is contaminated. If added to the sample, **this precipitation 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.

11. **Close enclosure door. For the ACM sampler, partially open the lid and remove the old dry side bag from dry side bucket.** Observe the condition of the dry side bag (dirt, bird droppings etc.). Record any observations in Section 10: Remarks. Replace bag by opening the bag and place into bucket. Pull top of bag over edges of bucket and pull bag down so it is firmly in place



- a. Removing old dry-side bag and
- b. Securing new dry-side bag (Aerochem)

12. **Cleaning the Lid Seal:** Partially open the collecting by activating the sensor and disconnecting power when half way open. Take a bottle of distilled water and wet a clean paper towel. Wipe down the lid seal observing the condition of seal and record any observations in Section 10: Remarks. If lid seal is torn, punctured, or looks discolored, please replace if possible.



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

13. **Remove old funnel:** remove the funnel from the top of the collector. Observe condition of funnel (dirt, debris, etc.) and record any observations in Section 10: Remarks. Once removed, place used funnel and cap into plastic bag for transport and cleaning in the lab.



Removing funnel/sample train from chimney. (Aerochem, in picture, Notice how they are being carefully lifted straight up out of the

14. Complete Section 6 part 1 “Site Operations” on the NOE.

If problems are discovered during the weekly check, 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 problem(s) you discovered. **IMPORTANT: Your job does not end here! You must immediately begin to troubleshoot the problem in order to correct any problems found.**

The site operator is responsible for determining when a piece of equipment is not working properly. Your collector must be assessed each Tuesday. If a problem is discovered, the site operator should begin troubleshooting. If the site is down and not working properly, valuable data will be lost which is not recoverable.

- The goal for operating the site is to always have a fully operating collector and rain gauge in order to collect each week’s sample.

15. Cleaning the collector:

- a) Wipe down the top of the lid and surrounding sides.
- b) Wipe down the arms to the lid and boots if present.
- c) Wipe down the top of the collector.
- d) Check the sides of the collector; if needed, wipe down the sides with DI water and paper towel.
- e) Clean any debris off the sensor. Stubborn debris can be removed by using a toothbrush.
- f) Inside the collector, clean the floor so it is dirt and bug free.
- g) Clean any surfaces or areas which have dirt or debris.



Cleaning the lid.



Cleaning the boots



Wiping down chimneys

Deployment of New Sample Gear:

1. **Change your gloves!**
2. **Install new funnel:** Open the bag containing the glass funnel, holding the funnel through the bag away from your body, insert the funnel through the chimney of the collector. (The funnel may require 10 wraps of 1” Teflon tape around the stem **above** the outlet to insure a tight seal into the compression fitting).



Bag covering
new funnel.

3. **Install the sample bottle:** Remove the new sample bottle from the bag and install the new sample collection cap.
4. **Install the overflow container:** Place sample bottle into the overflow container then install the container on the lowered base (Step 5, page 5).
5. **Connect the funnel:** Raise the bottle and connect the funnel to the compression fitting on the sample bottle. Once connected, raise the base to support the bottle. The base should support the bottle but not push the funnel above the edge of the chimney.
6. **Start a new NOF:** Complete Sections 1, 2 and the ON date and time portion of section 3. Place the NOF into a bag (to protect from water damage) and leave in the collector for next week’s sample retrieval (or with supplies).
7. The funnel should now be resting on the lip of the chimney. Carefully remove the plastic bag covering the funnel and connector assembly.

8. **Place the new sample bottle cap** into its plastic bag and store for the next week.
9. **Adjust the height of the sample:** Double check the height of the sample bottle, to ensure the base supports the weight of the bottle and any sample so that it will not pull the funnel from the sample connector assembly. Adjust the funnel height such that it is just barely resting on the chimney cap or the rim of the chimney.
10. **Close the lid:** Restore the power to the unit so the lid closes over the funnel. This may require drying the gridded sensor if equipped.
11. **Transport sample for processing:** Place the bagged sample and accompanying NOF from the previous week into a shipping container for transport to the laboratory for processing.
12. **Close enclosure door and clean up the site** (used gloves, paper towels etc).

Complete the NOF and capture Rain Gauge Data:

Steps to be completed at the Laboratory

1. Remove the 2-Liter bottle from the bag, and weigh the 2 liter sample bottle and cap to the nearest 0.01 gram, and record the weight on the NOF and subtracting the pre-sample weight of the bottle.
2. Calculate the volume of precipitation ($1 \text{ ml H}_2\text{O} = 1 \text{ cm}^3 = 1 \text{ gram}$), and record on the field sheet.
3. Calculate the depth of the precipitation, as determined by the sample volume:
 - area of funnel ($\pi * r^2 = \text{area, in cm}$)
 - volume of sample, in ml
 - depth=volume – area, in cm.
4. Ensure that the NOF is completed. Make a copy for yourself to keep in your site records file.
5. Transfer the sample from the 2-Liter sample bottle to the 150 ml sample bottle
 - a) Retrieve the 2-Liter sample bottle, and one 150ml sample bottle.
 - b) Put on a new pair of nitrile or vinyl gloves.

- c) Invert the 2 Liter sample bottle 3 to 5 times.
- d) Remove the cap from both the 2-Liter and the 150 ml bottle.
- e) Carefully pour 150 ml of the sample into the 150 ml bottle. If there is less than 150 ml in the sample, pour all the sample into the 150 ml bottle.
- f) Replace the tops of both bottles, ensuring tight seal on both bottles.
- g) Label the 150 ml bottle with the sample site name, the sample start date/time, and the sample end date/time.
- h) Place the sample into a sealable plastic bag. Put the sample bag into a second sealable plastic bag (double bag samples).

If not shipping the sample immediately, store the sample in a secure location at room temperature (refrigeration is ok).

- 6. Samples and NOFs can be shipped singly or in bulk. Ship samples at least monthly. Samples do not need to be cooled to ship. Ship the samples to the following address. We suggest using 2 day UPS or FedEx service if possible, but longer times (3-4 days are also acceptable):

Dr. Guey-Rong Sheu
APMMN
Department of Atmospheric Sciences
National Central University
300 Jhong-Da Road
Jhong-Li 320, Taiwan

- 7. Schedule a time to acid-clean all of your glassware, and ensure that you have an adequate supply of gloves, and field sheets for your next sample change-out.

Sample Storage

In order to save funds on shipping samples back and forth, monthly shipping of samples to the laboratory will be chosen by most in the network. For all samples stored, pour all samples into their shipping bottles (per the SOP), double bag the sample, and store under refrigerated conditions of 4°C or lower. Ship samples at least monthly.

Precipitation Data

This network requires that data from an accurate precipitation gage also be collected for determination of precipitation depth. Choose a rain gauge that is onsite (best, within 50 meters of the precipitation collector), or as close as possible to the collector (within 5 kilometers). Without this data, an accurate determination of collector efficiency and mercury wet deposition is not possible.

1. Obtain the daily precipitation information from your precipitation gage.
2. Add the daily sums of precipitation (in millimeters) to the NOF.
3. Calculate the total precipitation for the week, and add this total to the NOF.

Future Site Audits

At some point in the future, the APMMN will begin to do annual site visitation of each site. The purpose of the site visits will be to check the siting criteria for potential problems, assess the working of the sample collector, review training of the site operation for the site supervisor.

At this point, the site visitations are unscheduled, and will begin at some point in the future.

Specific Appendices/Processes

Network Observation Form (NOF)

Changes needed

1. A place to record the size of the funnel and units of size
2. A place within the precipitation record block for recording the units used
3. A place to record the sample volume collected (in laboratory steps)
4. Same for a place to record the sample & sample weight
5. Clear up the HAL NADP USE ONLY block; lets just make it blank and put "lab use only" in the block
6. A place to calculate the precipitation depth from the sample volume (volume
 Add: area of funnel ($\Pi * r^2 =$ area, in cm)
 Add: volume of sample, in ml, where 1 ml=1 cm³
 Add: depth=volume – area, in cm.
7. The box in the upper right corner can be reduced to full weight line, empty weight line and net weight line, and titled "Sample Weight Calculation"

OBSERVER FORM
 Send Completed Form With Each Shipping

<p>1. STATION Name: _____ ID: [][][][]</p>	<p>2. OBSERVER Print name: _____ Initials: [][] [][]</p>	<p>LAB ID _____</p>	<p>Carrier/Tracking # _____</p>																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																									
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Glassware Cleaning Procedure

All components of the sample train (funnel, sample bottle, connection apparatus and parts, sample bottle cap), and sample shipping bottles (and caps) should be thoroughly cleaned using the following method, following from Landis et al., 2002 (see below). All cleaning should be in a negative pressure vent hood in good working order.

Use 10% reagent-grade hydrochloric acid as the rinsing solution for all acid-rinsing steps.

Deionized Water (DIW): should have a minimum resistivity of 18.2 Mohms or greater. If stored, this water should be stored in acid rinsed glassware using appropriate laboratory practice.

Using powder free nitrile laboratory gloves, eye protection, appropriate laboratory safety clothing, and inside of a clean hood:

1. Rinse each component thoroughly three (3) times with fresh rinsing solution.
2. Then rinse thoroughly each component with DIW to ensure no Hydrochloric acid remains.
3. Air dry each component briefly in the vent hood, and store in plastic bags until use.

Reference: Landis, M.S., R. K. Stevens, F. Schaedlich, and E. M. Prestbo, 2002. Development and Characterization of an Annular Denuder Methodology for the Measurement of Divalent Inorganic Reactive Gaseous Mercury in Ambient Air, Environ. Sci. Technol. 2002, 36, 3000-3009.

Adding Pre-charge to the Sample Bottle

Before a new, acid cleaned bottle can be deployed to collect a mercury wet deposition sample, it must have hydrochloric acid (HCL) added to the clean sample container. This HCL “precharge” is added to the sample bottle to adjust the chemistry of the precipitation sample so that the mercury is fixed, and will not be lost to the walls of the bottle. This HCL precharge must be added to all bottles after each bottle cleaning. The steps are:

1. Put on gloves.
2. After acid cleaning the sample bottle, place the clean bottle under the fume hood.
3. Remove the sample bottle cap.
4. Add 10 drops of Hydrochloric Acid (12 Normal).
5. Replace the sample bottle cap, and store the HCL solution bottle.
6. Place a sample label onto the sample bottle.

7. On the label, add a warning that there is acid in the bottle, so that all that handle the bottle will know that it is a dangerous liquid.
8. Also, add to the label the Site Name, the start date of the sample and the end date of the sample, so that this bottle will only be associated with one field sheet.

Sampling Supply List and Part Numbers, etc.

1. Plastic Bags large enough for the 2-L sample bottle, and the funnel.
2. Plastic graduated cylinder 100 ml or 250 ml for measuring overflow.
3. Dry-side plastic bags for buckets
4. Ultra-Pure Deionized Distilled Water (DIW) with a minimum resistivity of 18.2 megaohm or greater, made onsite or supplied from a reputable supplier.
5. “Squeeze bottle” for dispensing onsite and for cleaning the sampler. 500 ml per sample change out should be enough.
6. large “Kimwipes” or alternative for cleaning sampler surfaces.
7. Sample Shipping Bottles (3-5, 150 ml) PETG bottles, used at a rate of 1 bottle per week.
8. Sample collection bottles with caps
Nalgene® Square Media Bottles, PETG, Sterile, Graduated; \$166/12 bottles.
9. Funnel-to-Bottle connection assembly: Teflon Compression Fitting; Stem diameters 3/8” (9.7mm)
10. Five (5) “J” shape ¼ ID Teflon tubing approximately 12 cm (OD to fit compression fitting above).
11. Five (5) borosilicate funnels sized to fit your sampler opening
12. Powderless nitrile or vinyl gloves.
13. Carrying Case to move equipment to the field and retrieve samples
14. 12 Normal trace metal grade hydrochloric acid (quality=<100 ppt mercury or better) purchased from a reliable supplier. Keep 1 liter on hand.
15. “Lab Jack” or other support mechanism to support sample bottle in sampler. Sized appropriately and adjustable for your sampler.
16. overflow container

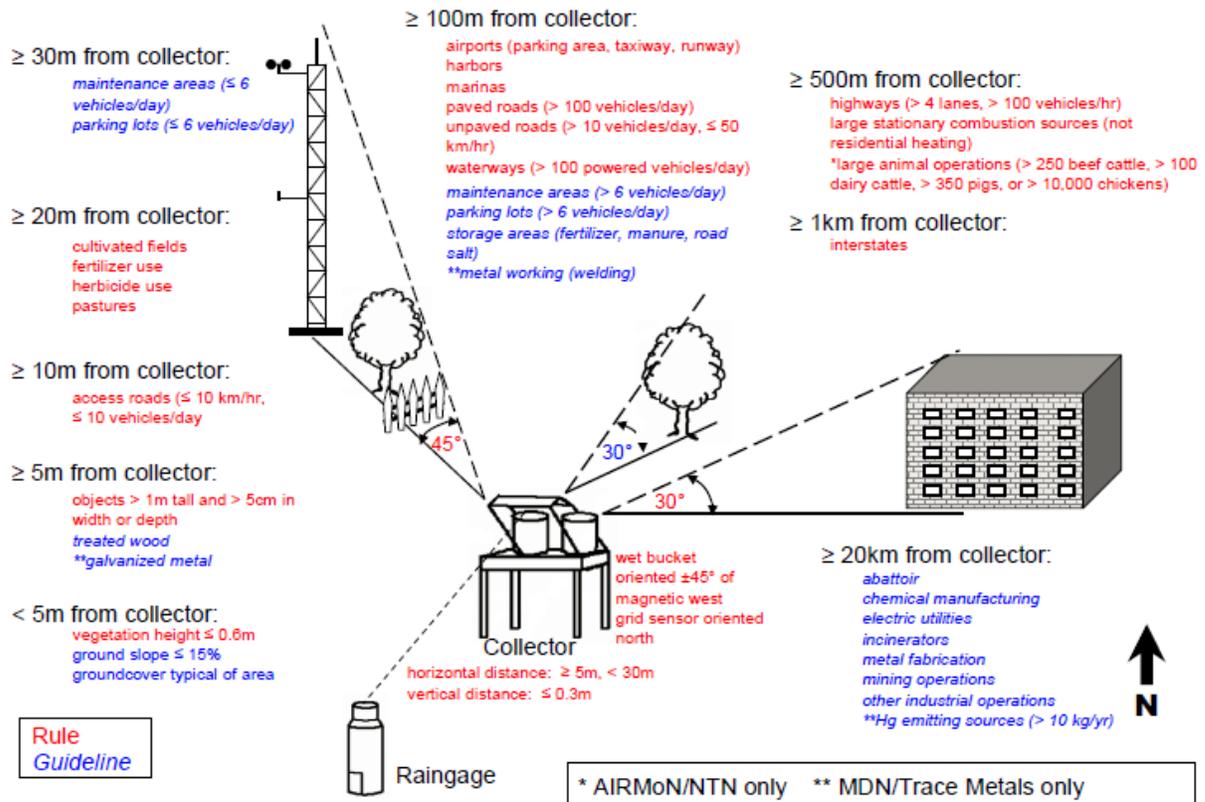
Field Checklist

Step by step outline of weekly field procedures.

1. Observe the condition of the site and the apparent operation of the sampler. Note on the NOF.
2. Open the sampler cabinet, put on a new pair of gloves, detach the sample bottle from the funnel, and close up the sample bottle with its lid.
3. Observe the condition of the sample, note of the NOF, and secure the sampler bottle in its plastic bag.
4. Complete the NOF information and store the NOF and sample for return to the laboratory.
5. Remove the funnel and store in its plastic bag.
6. Clean the entire outside of the sampler, using mercury free water.
7. Retrieve the clean glassware for the next week's sample.
8. Using a new pair of gloves. Place the funnel into the sample chimney.
9. Put the new sample bottle into the sampler, and put the bottle cap into the sealed plastic bag.
10. Connect the sample bottle to the funnel.
11. Adjust the sample funnel height.
12. Close the wet sampler lid.
13. Complete the NOF for the next week's sample.
14. Store plastic bags and cap for the sample, and return all sample supplies.

Siting Diagram

NADP Siting Criteria – Wet Deposition



List of Analytical Flags for assigning to Data.

Table 2. NADP/MDN Notes Codes			
Code	Description	HAL Database condition	Quality Rating Code
e	extended sample	duration of sample exceeds 194 hours	B
d	debris present	At least one of the six debris checkboxes is positive	B
m	missing information	noEventRecorder = true or precip = null or ignoreRG = true or minTemp = null or maxTemp = null	B
z	site operations problem	HMworks = false or minTemp < 32 or maxTemp > 100 or manual	B
h	sample handling problem	bag = true or leak = true or shipping = true	B
b	bulk sample	(manual) bulk = true	C
v	rain gage/sample volume discrepancy	((RG ≥ 0.03 inches) and (bottlecatch < 1.5 ml)) or (bottlecatch/rg < 0.10)	C
u	undefined	(manual) undefined = true	C
f	serious field problem, data integrity compromised	(manual) field protocol = True	C
l	lab error	(manual) labprotocol = true	C
c	contaminated	(manual) contaminated = true	C
p	no precipitation from rain gauge or sample volume	(rgppt is null or ignoreRG = true) AND (bottlecatch is null or ignoreBC)	C
n	no sample	fullmass is null or emptymass is null	--

Quality Rating Codes are defined as:

A=sample is valid, and unflagged.

B=sample is valid, but some issue with the sample was noted.

C=sample is invalid, with a serious problem with the sample noted.

Example of Output Data Format

SiteID	Date On	Date Off	RGPT		SVOL		SUBPPT		HgConc		HgDep		Sample Type	QR	Notes
			mm		ml		mm		ng/L		ng/m ²				
IN34	10/27/2000 18:00	11/01/2000 14:30	0.00		0.00		0.00		--		0.00		D	B	mzh
IN34	11/01/2000 14:30	11/07/2000 14:30	5.84		66.80		5.84		12.84		75.05		W	B	m
IN34	11/07/2000 14:30	11/14/2000 14:30	--		446.80		37.29		4.67		174.48		W	B	dmh
IN34	11/14/2000 14:30	11/21/2000 14:30	--		1.80		0.15		22.03		3.30		W	B	mi
IN34	11/21/2000 14:30	11/28/2000 14:30	7.62		79.80		7.62		4.48		34.17		W	B	m
IN34	11/28/2000 14:30	12/05/2000 18:34	11.62		138.30		11.62		7.06		82.04		W	B	dm
IN34	12/05/2000 18:34	12/12/2000 18:00	30.23		204.90		30.23		5.33		161.28		W	B	mz
IN34	12/12/2000 18:00	12/19/2000 15:00	17.02		366.00		17.02		4.98		84.74		W	B	mh
IN34	12/19/2000 15:00	12/26/2000 15:00	8.38		48.10		8.38		9.89		82.92		W	B	m
IN34	12/26/2000 19:40	01/03/2001 15:00	17.97		44.10		17.97		8.89		159.87		W	B	dmh
IN34	01/03/2001 15:00	01/09/2001 15:00	1.78		122.40		1.78		--		--		W	C	mzf
IN34	01/09/2001 15:00	01/16/2001 14:30	3.11		42.50		3.11		2.44		7.60		W	B	m
IN34	01/16/2001 14:30	01/23/2001 14:30	0.25		0.10		0.25		--		--		W	C	mv
IN34	01/23/2001 14:30	01/30/2001 14:30	16.51		204.90		16.51		9.67		159.68		W	B	dm
IN34	01/30/2001 14:30	02/06/2001 14:00	8.38		38.20		8.38		9.74		81.70		W	B	dmh
IN34	02/06/2001 14:00	02/13/2001 14:00	26.67		323.80		26.67		11.48		306.38		W	B	m
IN34	02/13/2001 14:00	02/20/2001 14:00	3.81		45.80		3.81		10.98		41.84		W	B	m
IN34	02/20/2001 14:00	02/27/2001 14:00	51.31		539.30		51.31		7.04		361.41		W	B	dm