

## Protocol for Acquiring Waters for Mercury and Methylmercury Analysis for the REMAP Assessment of Mercury in Vermont and New Hampshire Lakes

### General Sampling Procedures and Order of Operations:

The sampling station is located in the field using non-differential GPS, to correspond with the lakes' deep hole. For lakes on which gasoline powered craft are required for sampling, the engine is shut off downwind of the station, and staff rows the craft into place. The boat is secured by anchor, and adequate scope let out to avoid contamination of the hypolimnetic zone of interest by the anchor or sediment drift. The ideal craft for performing these procedures consists of two non-metal canoes, attached together into a 'pontoon craft' by means of 2 wooden boards (ie. 2x4's).

For all lakes, collection of parameters requiring clean handling precedes collection of other parameters in like moieties. The order of collection and handling is as follows:

Arrange sampling equipment → Don sampling attire and gloves → Surface grab for aqueous methylmercury and total mercury sample → Hypolimnetic teflon Kemmerer grab for aqueous methylmercury and total mercury sample → Remove clean attire and gloves → Hydrolab® profile and Secchi measurement → Collect then handle other water chemistry parameters using standard Kemmerer sampler → Dirty hands collects sediments → 'Clean hands' handles extruded sediment.

### Acquisition of Water for Mercury and Methylmercury Analysis by CVAFS

A surface grab for aqueous mercury samples is collected at each study lake. In addition, for those lakes which stratify, a sample is acquired from one meter above the sediment water interface, using an all-teflon Kemmerer sampler which is cleaned to conform to EPA Method 1631 specifications. The sampling depth is determined by on-board SONAR, and not by means of a depth tape or any other device which contacts the lake water and sediment in the vicinity of the sampling point. Techniques for the collection of aqueous mercury samples will conform to the EPA method 1669 "Clean hands'-dirty hands' techniques. In brief, sampling staff must wear clean new gloves, and may wear windsuits. "Clean hands" wears shoulder-length gloves, which may be covered by a second pair of regular-length gloves to provide for dexterity. Glove are new from the box at the time they are put on. In the event of contact with any metal surface, or any obviously dirty non-metal surface, gloves must be replaced with a new pair.

Aqueous mercury samples are stored double-bagged in Method 1631-specification cleaned teflon bottles, in a separate cooler, referred to as the 'clean cooler.' Samples are preserved in situ with 3.6ml concentrated trace-metals grade HNO<sub>3</sub>, using a new pipet tip rinsed twice in mercury-clean 10% HCl, and once in the trace metal grade HNO<sub>3</sub>.

This procedure describes collection of epilimnetic and hypolimnetic water samples. When using the teflon Kemmerer sampler, it is highly recommended that 'dirty hands' wear a new tyvek windsuit, which greatly facilitates handling the sampler. In addition, it is recommended that for such a collection, both 'clean' and 'dirty hands' wear at minimum two layers of gloves, to permit efficient stripping one pair in the event of glove contamination. Gloves and other tools must be kept in a clean, sealable plastic box such as a new cooler. This is referred to as the 'clean box.' Finally, each

member of the sampling team must read and understand Section 4 of EPA Method 1669 (Philosophy of Contamination Control) prior to initiation of sampling.

While awaiting shipment to the analytical facility, all preserved samples must be stored in a designated clean cooler or non-metallic box. It is preferable that the samples be stored away from indoor laboratory area which is not class-100 clean, and/or where mercury may have been released due to the breakage of fluorescent bulbs, thermometers, or mercury-bearing wastes or reagents.

*Procedures:*

Epilimnetic Grab Sampling

- Waterproof sample labels are prepared using waterproof ink.
- 'Dirty hands' opens the 'clean box,' gloves, and dons a tyvek suit.
- 'Dirty hands' removes shoulder gloves, and assists 'Clean hands' in donning shoulder-gloves and shorter gloves if necessary. From this point forward, 'Clean hands' handles nothing but the sample bottle, or the inner ziplock bag which contains the sample bottle.
- 'Dirty hands' opens the 'clean cooler,' and removes 1 1000ml double bagged bottle. 'Dirty hands' opens the outer bag.
- 'Clean hands' reaches into the outer bag, opens the inner bag, removes the bottle, and folds the inner bag over.
- 'Dirty hands' seals the outer bag, and replaces it into the 'clean cooler.'
- 'Dirty hands' removes the autopipet from the clean cooler, and affixes a new pipet tip.
- 'Dirty hands' rinses the pipet tip 2X in reagent-water dilute 10% HCl, and 1X in HNO<sub>3</sub>. Rinsates are evacuated into a waste-acid container.
- 'Clean hands' opens the sample bottle, evacuates the contents, and closes the bottle.
- 'Clean hands' submerses the bottle to a 0.5 meter minimum depth, opens the bottle, and fills it 1/3rd full. The bottle is then surfaced, shaken, opened, and the rinsate evacuated away from the immediate sampling point. The bottle is resealed. This is repeated 2X.
- 'Clean hands' re-submerses the bottle, and allows the bottle to fill entirely. The bottle is recapped underwater.
- 'Clean hands' surfaces the bottle, and opens the cap slightly.
- 'Dirty hands' draws 3.6 ml trace-metal grade HNO<sub>3</sub>, and pipets this into the sample bottle. 'Clean hands' then **tightly** caps the bottle.
- 'Dirty hands' opens the clean cooler, withdraws, then opens the outer bag.
- 'Clean hands' unfolds the inner bag, replaces the bottle, and seals the inner bag. 'Dirty hands' then seals the outer bag, affixes the label, and replaces the double-bagged sample in the clean cooler.

Hypolimnetic Kemmerer Sampling

- 'Dirty hands' un-bags the double-bagged teflon Kemmerer, affixes the line, and rinses the sampler 3x in lake water by submersing the sampler, forcefully retrieving it, and allowing it to drip off. The sampler is then lowered 2 meters below the boat, and tied off.

- 'Dirty hands' opens the 'clean cooler,' and removes a 500ml double bagged bottle. 'Dirty hands' opens the outer bag.
- 'Clean hands' reaches into the outer bag, opens the inner bag, removes the bottle, and folds the inner bag over.
- 'Dirty hands' seals the outer bag, and replaces it into the 'clean cooler.'
- 'Dirty hands' lowers the Kemmerer sampler to 1 meter from the sediment-water interface, and trips the closure mechanism with the non-metallic messenger. The sampler is retrieved.
- 'Clean hands' opens and evacuates the bottle. 'Dirty hands' directs the sample stream from the Kemmerer sampler to fill the bottle 1/3. 'Clean hands' caps the bottle, shakes vigorously, and evacuates the rinsate. This is repeated 2X.
- 'Dirty hands' directs the sample stream to fill the bottle entirely. 'Clean hands' caps the bottle, and 'Dirty hands' re-submerses and ties off the Kemmerer sampler at 2 meters of depth.
- 'Dirty hands' draws 1.8 ml trace-metal grade HNO<sub>3</sub> and pipets this into the sample bottle which was opened by 'Clean hands'. 'Clean hands' **tightly** caps the bottle.
- 'Dirty hands' opens the clean cooler, and withdraws then opens the outer bag.
- 'Clean hands' unfolds the inner bag, replaces the bottle, and seals the inner bag. 'Dirty hands' then seals the outer bottle, affixes the label, and replaces the bottle in the clean cooler.
- 'Dirty hands' bags the Kemmerer sampler with new bags, using "Clean hands" assistance.