Sample Handling, Acceptance, and Storage

1. Sample Collection

a. Laboratory to maintain all sampling records provided by sampler

2. Sample Containers Provided by Laboratory Include:

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a. Carboys/Sample bottles - a procedure for cleaning carboys must be prepared.   A good cleaning process would include: carboys washed with detergent (non-phosphate), triple-rinsed and final DI rinse.

b. Reused containers for non-trace analyses (assume routine cleaning procedures adequate)

c. Reused container for trace analyses (detergent wash, triple-rinse, acid rinse for metals, solvent rinse for organics).   It is advised that periodic verification of cleanliness be done by running bottle check blanks- if all results <MB for test, cleaning is adequate. If not,verify with other bottles and/or reclean all affected.

d. New containers- can use without cleaning – although periodic bottle blanks are required for HEM analyses.

e. Chemical preservatives- maintain certificates of analysis.

3. Sample Acceptance Policy (Non-SDWA)

a. Samples are considered properly preserved if:

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- ► Wastewater samples meet Table F requirements, NR 219
- ► Groundwater typically mirrors NR 219 requirements
- ► UST aqueous sample pH<2 with 1:1 HCl and ≤ 6°C
- ► UST GRO/PVOC soils Methanol preserved and ≤ 6°C
- ► UST DRO soils ≤ 6°C
- ► Solid samples, in general, require ≤6°C, unless analytical method specifies otherwise, Solid VOCs require methanol preservation

b. Result qualification is required when:

Improperly preserved (e, g., pH>2, temperature >6°C)
Wrong container (e.g., metals in glass bottle, headspace in
VOA vial, etc.)
Hold time is exceeded
Insufficient sample volume to perform analysis
Known sampling errors
Laboratory may reject or require re-sample as alternative to
qualification of sample results

c. If discrepancy between sample collection record and sample receipt the
laboratory must consult with sample collector and/or client to determine if
samples can be analyzed and reported with qualification or re-sampled.

4. SDWA Sample Acceptance Policy

a. Samples must be rejected if:
   ► Hold time is exceeded
   ► Improper preservation
   ► Sample is in wrong container (*e.g., metals in glass, SOC in plastic*)
b. If client directs laboratory to proceed with analysis, all data must be
reported with qualifiers and the client is responsible for consulting with
WDNR to determine what is the proper course of action. All associated
results that are analyzed, instead of rejected, must be reported with a
disclaimer attesting results are not to be used to determine or evaluate
compliance under NR 809.

5. Sample Handling Protocols

a. Unique identification code

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   ► If analyzing own samples daily, can be as simple as Influent/date.
   ► If analyzing own samples, but not daily (i.e. after 2 weeks worth of
samples are accumulated), then record sample name (i.e.
influent/date) on sample containers.
   ► Some examples of possible sample numbering conventions (needed so
that each sample has a unique identifier):
   1) client name/date/matrix
   (Anytown, 6/11/08 Effluent), sequential number for each sample (Anytown's
6/11/08 Effluent for BOD/TSS is 1000, same sample for ammonia is 1001,
total phosphorus is
   1002),
   2) client ID/matrix date (client #10 final effluent from June 11, 2008 could be
10E061108).
b. Unique identification code must be placed on sample container, if sample is
held for analysis (e.g., analyze ammonia and total phosphorus every other
week, each bottle must have an identifier).

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c. Chain of Custody- only required if not doing own analysis (see example chain of custody form)

d. Evidentiary Chain of Custody- only required when performing regulatory investigation or at the request of the client. Highly unusual for routine analysis.

6. Sample Preservation and Hold Times

a. Wastewater preservation and hold times are specified in NR 219, Table F.

b. Groundwater typically defers to method requirements, which are reflected in NR 219

c. SDWA preservation and hold times are specified in NR 809.725, Table F & G.

7. Non-SDWA Thermal Preservation

a. Received on ice (ice cubes still present in sample cooler)

b. Samples not surrounded by ice are properly preserved if:
   - ► Actual sample temperature is determined to be ≤6°C
   - ► Temperature blank is ≤6°C
   - ► Temperature of melt water surrounding samples is ≤6°C

c. Sample not shipped with ice cubes require the actual temperature of a sample, temperature blank or melt water, if present, to verify ≤6°C upon arrival.

8. SDWA Thermal Preservation

a. Section 6.2 of Chapter IV, of SDWA Laboratory Manual states "The laboratory must measure and record the temperature of the sample when it arrives when temperature preservation is required by method." This applies to all analytes except metals, bromate, chloride, fluoride and pH.

b. The use of "blue ice" and other ice packs are strongly discouraged.

9. Sample Receipt Documentation (example Sample Log)

a. Client identity or project

b. Dates of sample collection and laboratory receipt

c. Time of sample collection and laboratory receipt for tests with hold times ≤ 48 hours

d. Unique sample identifier

e. Documentation of sample preservation status or other conditions

f. Unequivocal link between sample collection ID and Laboratory sample #

g. Requested analyses (unless collect and analyze own samples and tests are indicated in a permit or license)

h. Reference to requested test methods, when specified by sampler

10. Sample Storage

a. Procedures to ensure samples maintain integrity, not contaminated, lost, damaged, etc.

b. Any sample requiring thermal preservation at value other than ≤ 6°C shall be stored at ± 2° C of stated temperature

c. 6°C means from above freezing to preferably 6.0°C- if temperature is consistently high, laboratory must adjust to ensure samples are kept from exceeding within rounding

d. Samples are to be kept separate from reagents, standards, and food items in refrigerators. For laboratories with a small number of reagents and standards, these may be kept in a sealed bin within the same compartment, with as much physical space separating the two as possible

e. Sample digests, distillates, extracts, leachates, etc. are to be stored as specified for the sample and test (e.g., metals digestates at room temperature, pH<2, pesticide extracts at ≤6°C)