STANDARD OPERATING PROCEDURE No. 47

SNOW AND RAINFALL COLLECTION FOR ISOTOPIC ANALYSIS

REVISION LOG		
Revision Number	Description	Date
47.0	Original SOP	1/29/04
47.1	Objectives, contingency, revised sample collection & analysis	2/10/04
47.2	Corrections from S. Earman, NM Tech	2/12/04
47.3	Corrections by PJP	5/19/2004
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47v5	Additions by JMS	8/31/04
47v6	Edits LMK	7/3/06
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47v7	Editorial by SKA	10/24/08

1.0 PURPOSE AND SCOPE

This Standard Operating Procedure describes the method for collecting rainfall and snow in order to determine the stable isotopic composition, especially deuterium and ^{18}O, and the quantity of snow melt and rain fall intercepted during a given sampling period. The isotopic composition of meteoric waters can then be compared with the isotopic composition of water from the saturated and vadose zones as well as with the composition in water vapor. The second objective of this SOP is to quantify the mass of snow melt and rain fall collected during a given period to provide an upper constraint on the amount of water available for infiltration processes at that location.

This SOP only covers the collection of the precipitation waters, not their analysis. The reader is referred to SOP 25 for stable isotope analysis methods.

2.0 RESPONSIBILITIES AND QUALIFICATIONS

The Team Leader and Characterization Team will have the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate staff to implement this SOP and for ensuring that the procedures are followed accurately.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP and to receive specific training regarding these procedures, if necessary.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this SOP to the Team Leader.

3.0 DATA QUALITY OBJECTIVES

This SOP addresses objectives 1 and 7 in the data quality objectives outlined by Virginia McLemore for the "Geological and Hydrological Characterization at the Molycorp Questa Mine, Taos County, New Mexico".

- Determine how the hydrogeochemistry and water balance dynamics influence rock pile weathering and stability.
- Determine if pyrite oxidation, moisture content, and microbe populations affect rock pile weathering and stability.

4.0 RELATED STANDARD OPERATING PROCEDURES

The procedures set forth in this SOP are relevant to activities described in the following SOPs:

- SOP 1 Data management (including verification and validation)
- SOP 2 Sample management (including chain of custody)
- SOP 10 Meteorological station maintenance
- SOP 25 Stable isotope analyses
- SOP 36 Sample preservation, storage, custody, shipping

The procedures set forth in this SOP also are intended for use with the project sampling plans.

5.0 EQUIPMENT LIST

The following materials are required for collecting liquid-phase water samples for quantifying precipitation and analyzing stable isotope concentrations (field):

- Spare precipitation collector
- Spare 2-3 ft length of drain tube, 3/8 inch ID and 1/2 inch OD
- Two spare small hose clamps

- Clean, leak-proof HDPE 4-liter container and lid/cap, with a known empty weight (without lid) and containing a pre-weighed quantity of mineral oil sufficient to create a 2-cm thick layer across the container's entire width (weight without lid).
- Spare leak-proof caps for storage containers
- Box or cooler to store and transport the container in an undisturbed state
- Indelible marker
- Precipitation field sample forms (attached as Appendix I) and chain of custody forms
- Phillips head screw driver (to remove and refasten the cover to the storage box)
- Extra hardware cloth screen as replacement (if needed)
- Two extra wallboard screws 1.5 inch (if needed)
- Pick and shovel to uncover storage box
- Plastic bag to cover collector while the cover material is being replaced (prevents rock pile material from entering the collector)
- Rebar driver for installing rebar supports for the precipitation collector
- Rope or flagging for the rebar to mark out the site

The volume of the storage container must be larger than the maximum amount of water expected to be collected during the sampling period. The maximum amount of water to be collected varies with season and with the length of the sampling period. The collector has a cross-sectional area for collection equal to 177 cm^2. A single rain storm can supply as much as 5 cm of water during the monsoon season, yielding a single storm volume of 885 cm^3, or nearly one liter. Therefore, the minimum volume for a 30-day sampling period during the monsoon season is roughly four liters (one gallon). The minimum volume for a 30-day sampling period during the snow season should be one to two liters (0.25 to 0.5 gallon).

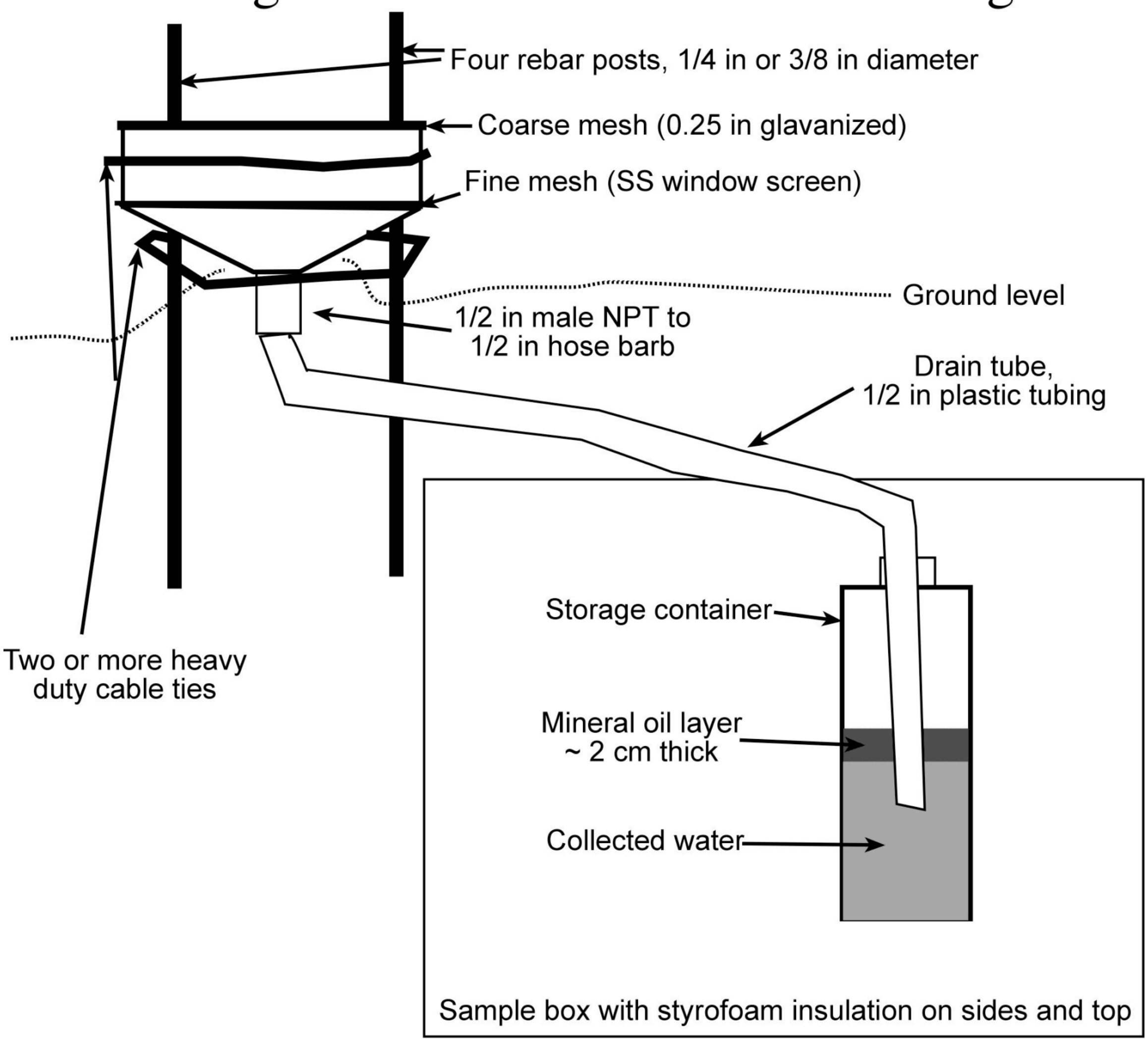
The following materials are required for preparing liquid-phase water samples for stable isotope analysis and quantifying precipitation (laboratory):

- Scale, accurate to at least 0.1 g
- Two clean, dry, Qorpak 60 mL sample bottles
- One clean, dry, Nalgene or HDPE bottle with leakproof cap, with sufficient volume to hold as much of the collected water as possible.
- Clean, dry separatory funnel or Ziploc freezer bag, 1 gallon size
- Scissors (if using the Ziploc instead of the funnel)
- indelible marker
- Clean, leak-proof , glass or HDPE one to four liter container, with a known empty (dry) weight
- Mineral oil
- Laboratory tape or water-proof labels and indelible marker pen
- Alconox (or similar cleaner)

6.0 COLLECTOR CONSTRUCTION, INSTALLATION, AND MAINTENANCE

One collector will be used to collect both snow melt and rain fall at each site. The collector design, which is adapted from designs recently employed for isotope research in New Mexico and the American Southwest [Johnson et al., 2002; Earman et al., 2003], focuses on collecting snow melt for isotopic analysis under conditions as close as possible to *in situ* snow pack conditions at the ground surface. The design is shown in Figure 1. The sampling schedule will determine whether the bulk of the collected water is from snow melt or from rain fall. Storage containers may be sampled and replaced as often as every month, but no less often than twice a year (i.e., just prior to the first expected snows and at the end of the snow season).

Figure 1: Snow/rain collector design



Sketch not to scale.

The basic design is a vertical standpipe connected via a hose pipe to an underground storage container. A layer of mineral oil, at least 2 cm thick, is added to the storage container to prevent evaporative loss. The mineral oil also intercepts and traps nearly all the debris which might enter the storage container. The topmost coarse mesh prevents insects and debris from entering or blocking the drain tube. The finer window screen mesh retains the snow within the standpipe until it melts and proceeds downward to the storage container.

Installation begins with selecting the location for the collector (Figure 1) and for the storage box, which can be constructed of plywood or pressed board and insulated with Styrofoam slabs. Excavate a hole for the storage box, which should be placed deep enough to prevent the box from moving or tipping. Based on our observations this winter, the top of the box should be set at a minimum depth of 10 cm and covered with material from the rock pile. The collector should be installed at a higher elevation than the top of the storage box and stabilized with upright rods (e.g., rebar) and heavy-duty cable ties. Drive the rebar into the ground adjacent to the storage box using a rebar driver. The top of the collector should protrude 10-15 cm above ground level. Completely cover the top of the precipitation collector with a plastic bag or other material to prevent rock pile material from entering the collector while placing the rock pile cover material on top of the storage box and around the base of the precipitation collector. Try and cover as much of the drain tube as possible. Remove the plastic bag or other material covering the top of the collector once the cover material is in place.

The drain tube is crucial to collecting samples properly. It should be affixed to the collector bottom (a 0.5 inch male NPT to 0.5 inch hose barb connector) using a hose clamp. The drain tube should be cut just long enough to prevent any kinking or similar obstruction to rapid water flow. The tube should be protected from kinking, pinching, or ripping by covering it with finer-grained material from the rock pile or shielding it with PVC pipe or other similar material. The drain tube should extend at least 10-15 cm into the storage container to prevent an inadvertent disconnect between the collector and the storage container.

The area surrounding the collector and storage box should be demarcated with posts and rope or flagging to prevent disturbance of the site by vehicles or workers on foot. In locations where it is likely that rock pile material could easily enter the collector, a vertical shield or screen should be installed to divert rock pile material around the collector.

Collectors should be routinely inspected and maintained. Inspections should be conducted monthly during the monsoon season (July – October) and at least every two to three months during the winter season (November–May). If the collector has become filled with sediment or other material, the storage container should be removed and both the collector and drain tube cleaned of all foreign material. The storage container should then be replaced with a new prepared container (see section 6). Field personnel should also ensure that the collector faces upwards, the storage box is adequately covered, and that the site flagging is in good repair and sufficient to alert other workers to the site's presence. All inspection observations (including the nature of any foreign materials found within the collectors) and repairs should be noted in the field report. The hardware cloth screen covering the top of the collector that prevents most debris from entering the collector should be replaced if it is broken or missing.

Field personnel should also note the presence of an ice blockage within the collector or the drain tube, any melt water trapped above the blockage, and the presence or absence of icy patches on the rock pile surface in the vicinity of the collector. Freeze-thaw cycles can alter the isotopic composition of the water if it is not protected by a mineral oil layer. Such alteration is expected within the collector above the window mesh and is the reason for adding the window mesh to the collector design. Alteration of snow within the collector should be analogous to the alteration of the snow on the rock pile, so it is possible that ice formation within the collector is desirable if it also occurs on the rock pile surface.

7.0 PROCEDURES

7.1 Container prep

1. The container should be labeled with a container ID (not a sample ID) on the bottom of the container using indelible marker. If there is no container ID on the bottom, use the indelible marker to write a new ID (can be arbitrary but must not conflict with those already in use). Check the database for conflicts with container IDs already in use.
2. Weigh each labeled, empty, dry container without cap to within 0.1 g and record the weight and container ID.
3. Add enough clean mineral oil to form a 2-cm thick layer within the container. Record the weight of the container and oil to within 0.1 g.
4. Cap the container.
5. Store the bottle until it must be transported safely to the site for installation at the collector.

7.2 Container replacement in the field

1. Note the date, time, and weather conditions in the field activity log.
2. Note whether the precipitation collector contains any sediment or other foreign matter on the precipitation sample form. Cover the top of the collector with a plastic bag or cloth to prevent rock pile material from entering during excavation.
3. Remove the cover material from the storage box to expose the entire top of the box. Work carefully so that the precipitation collector and drain tube are not damaged. Set the cover material aside so that it can be reused to cover the box.
4. Loosen the cover by removing the screws using a Phillips head screwdriver.
5. Examine the drain tube for trapped foreign matter or water and for kinks, tears, or other damage. Record your observations on the precipitation sample form.
6. Label the replacement storage container on the side wall with the appropriate sample ID. Strike-out all other sample IDs. Record the container ID and sample ID on the precipitation sample form.
7. Remove the cap and drain tube from the storage container in the sample box. If the tube holds a significant amount of water from a kink or other obstruction, disconnect the drain tube so that water within the tube does not enter the storage container.
8. Securely cap the used storage container.

9. Securely connect the cap with the drain tube onto the new storage container and place the container inside the storage box. Adjust the drain tube so that the possibility of kinking is minimized by pushing the drain tube as far as possible into the new storage container. Refasten the cover with at least two screws.
10. Replace the cover material so that as much of the storage box as possible is covered by 5-10 cm of cover material. Ensure that the drain tube is covered by finer-grained material, rather than cobbles, so that flow is not obstructed by pinching.
11. Remove the bag or cloth cover from the top of the precipitation collector.
12. Examine the condition of the sample within the used storage container, and record the color, presence and amount of sediments, and estimate the volume of the water on the precipitation sample form. Store the container for safe transport back to the lab.
13. Complete the precipitation sample and chain of custody forms.

7.3 Laboratory preparation of samples for analysis

1. Weigh the container with water and oil to the nearest 0.1 g. Record the weight. Calculate and record the weight of water within the container by subtracting the previously recorded weight of the container and mineral oil from the weight of the container, water, and mineral oil.
2. If some, or all, of the sample is frozen, allow it to completely melt by equilibration to room temperature. DO NOT apply any direct heat.
3. Select two Qorpak bottles and an HDPE or Nalgene bottle to hold the stable isotope sub-sample, archive sub-sample, and water chemistry sub-sample.
4. Label the bottles using lab tape with sample IDs and the appropriate postfix code for each sub-sample: stable isotope sub-sample has postfix –7, archive sub-sample has postfix –10, and the water chemistry sub-sample has postfix –11. Thus, sample XXX-YYY-ZZZZ will have its stable isotope sub-sample labeled as XXX-YYY-ZZZZ-7 and its water chemistry sub-sample labeled as XXX-YYY-ZZZZ-11.
5. Carefully decant the oil and water into a clean, dry, separatory funnel. If the storage container contains sediment, prevent the sediment from flowing into the separatory funnel by decanting only the sediment-free water. If a separatory funnel is not available, a clean, dry, one-gallon Ziploc freezer bag can be used instead of the funnel.
6. Allow the decanted oil and water mixture to separate.
7. Carefully fill each of the two Qorpak bottles with sample water, ensuring that as little oil as possible enters the Qorpak bottles. If a Ziploc bag is used, cut one bottom corner of the Ziploc bag with the scissors to allow the water to flow into the Qorpak bottles, retaining any oil present in the bag..
8. Carefully cap the Qorpak sample bottles with Poly-Seal lids. Cap the water chemistry sub-sample.
9. Measure the pH and conductivity of the water chemistry sub-sample following the Bureau of Geology's standard procedures. Record the results in a lab notebook and on the sample forms.
10. Safely transport the stable isotope sub-sample to Dr. Andrew Campbell's stable isotope lab for analysis, following all chain of custody procedures. Safely store the archive and water chemistry sub-samples until the results from the stable isotope

analysis have been evaluated. The water chemistry sub-samples can then be destroyed.

11. Clean the used storage containers with Alconox (or similar cleaner) and clean water, then allow the container to dry entirely. Dispose of the used mineral oil and Ziploc bag, if used, following NM Tech procedures.

8.0 QUALITY ASSURANCE/QUALITY CONTROL

Laboratory scales should be calibrated using standard weights prior to measuring. Ensure that the oil and water have completely separated before filling the Qorpak bottles. Use extra care to ensure that the oil and water remain separated while filling the Qorpak bottles. Minimize sample handling and record all losses of sample mass.

Effective decontamination is achieved by using clean, dry storage containers.

9.0 DOCUMENTATION

The precipitation sample form is shown in Appendix I.

10.0 REFERENCES

Earman, S., Campbell, A. R., and Phillips, F. M., Stable Isotopes in Snow: Implications for the Design of Precipitation Collectors and Studies of Groundwater Recharge, Eos Trans. AGU, 84(46), Fall Meet. Suppl., Abstract H21D-0890, 2003

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Scholl, M., 2003, Precipitation isotope collector designs, http://water.usgs.gov/nrp/proj.bib/hawaii/precip_methods.htm

General references:

Claassen, H. C., and Downey, J. S., 1995. A model for deuterium and oxygen 18 isotope changes during evergreen interception of snowfall, Water Resources Research, v. 31, no. 3, pp. 601-618.

Cooper, L. W., 1998. Isotopic Fractionation in Snow Cover, in Kendall, C., and McDonnell, J., eds., Isotope Tracers in Catchment Hydrology, Elsevier, Amsterdam.

Friedman, I., Benson, C., and Gleason, J., 1991. Isotopic changes during snow metamorphism, in Taylor, H. P., O'Niel, J. R., and Kaplan, I. R., eds., Stable Isotope Geochemistry: A Tribute to Samuel Epstein, The Geochemical Society, San Antonio, TX.

12. APPENDIX I: Precipitation Sample Form

precip_field_sample_form

PRECIPTATION FIELD SAMPLES

SAMPLE INSTALLATION

Field id SSS-JMS-0004 **Storage cont id** C **Feature id** 18

Date installed 2/7/2004 **Installed by** JMS **UTM easting** 454302 **UTM northing** 4060227

UTM zone 13 **Elevation** 8708 **Method of obtaining elevation** HHGPSoff

Waypoint **Location assurance** HHGPSoff **Depth start** 0

Point of location field location **Sample location**

Location description of sample west of hot spot, north of lysimeter, base of rock pile slope

Media water **Type of sample** routine sample

weather conditions cold, sun behind rock piles

Method of sample collector **Sample preservation** mineral oil

Type original design, 1 **Diameter** 6

SOP number 47 **Deviation SOP** tied collector to rebar

Reason for sampling stable isotopes

Comments 1 sample collected for moisture content (SSS-JMS-0003), ice at 60 cm, VTM filled out paperwork

PREINSTALLATION

wgt empty 96.28 **wgt bottle oil** 277.14

SAMPLE COLLECTION

Date collected 3/26/2004 **Collected by** JMS **no_days in place** 19

Laboratory id NM1 **Date weighed** 3/30/2004 **wgt bottle oil water** 626.67

wgt water 349.53 **eq inches water** **Mass** **pH** 5.8

Conductivity 104.7 **Alkalinity** **Temperature** **color** clear

sampling comments 0.33 liter, condensation on bottom of lid

SAMPLE ANALYSES

delta O -18.6 **delta D** -147.3

Fieldphotos

Photo_number: Photographer:

Image_type: Field Date: Feature_id:

Location: Direction:

Keywords:

Caption:

Comments:

Link: Digital Slide Photograph

CameraType Pixels 0

Record: 1 of 1

sample_anal_request subform

thin_section [ ] thin_section_comp [ ] bulk_density [ ] bulk_density_comp [ ]

Weathering_cells [ ] samples_from_Kim [ ] samples_from_Kim_after [ ]

petrographic [ ] petrographic_comp [ ] clay_min [ ] clay_min_comp [ ]

Alteration [ ] carbonates [ ] paste_pH [ ] paste_pH_com [ ] MC [ ] MC_comp [ ]

mineralogy [ ] pyrite [ ] paste_conductivity [ ] paste_conductivity_com [ ]

Crushed [ ] pyrite_reserve [ ] part_size [ ] part_size_comp [ ]

DI_leach [ ] DI_leach_comp [ ] part_size_chem [ ] part_size_chem_comp [ ]

reflect_spect [ ] microbes [ ] XRD [ ] XRD_comp [ ] Ar_Ar [ ] Ar_Ar_comp [ ]

probe [ ] probe_comp [ ] XRF [ ] XRF_comp [ ] ICP [ ] ICP_comp [ ]

stable_isotopes [x] stable_isotopes_comp [ ] pore_water [ ] pore_water_comp [ ]

Cations_anions [ ] pH_cond_TDS [ ] shear_box [ ]

Comments:

Record: 1 of 1