
# Chapter A2

## Stream-sediment sample preparation

*By Thomas R. Peacock, Cliff D. Taylor, and Peter M. Theodorakos*

**U.S. Geological Survey Open File Report 02-223-A2**

**Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey**

# Contents

# Stream-sediment sample preparation

*By Thomas R. Peacock, Cliff D. Taylor, and Peter M. Theodorakos*

Code: Q020

Accepted: 6/25/90
Revised: 11/18/93

## Principle

Most samples of naturally occurring material require some kind of physical preparation prior to chemical analysis. Samples require preparation to effect one or more of the following: (1) reduce the sample to a size that is more conveniently transported; (2) increase the sample surface area to enhance the efficiency of subsequent chemical attack; (3) homogenize the sample to ensure that a subsample is representative of the entire sample; and (4) separate the sample into components based on mineralogy, grain size, or other physical and morphological criteria. Sample preparation is an important step in the analytical process. Without careful preparation, and attention to inter-sample contamination, the worth of the subsequent analyses is significantly diminished.

The dry stream-sediment samples are disaggregated by hand, as necessary, and as much organic material as possible is removed. The samples are sieved to pass an 80-mesh screen (<180 μm) or the particle size specified on the RFA. The sieved fraction is generally ground in a mechanical pulverizer, placed in a 3-oz container and mixed to ensure homogeneity.

## Scope

Approximately 50 samples can be processed per day.

## Apparatus and materials

- Ro-Tap table top-mounted sieve shaker
- Sieves with stainless steel screens, with pans and cover
- Sample cartons, 3-oz
- Sieve brush
- Large sample funnel, plastic
- Compressed-air source, 40 psi
- Braun vertical pulverizer with ceramic plates
- Jones splitter with catch pans
- Grease gun for lubricating equipment
- Kimwipes or paper towels
- Assortment of tools for equipment maintenance

## Safety precautions

Eye and ear protection and a dust mask must be worn and it is recommended that a lab coat be worn. Caution must be exercised in operating equipment, particularly the Braun pulverizer, which has the potential of inflicting serious injury if not properly, and carefully used. Belts on equipment must be guarded and power should be turned off prior to dislodging any jammed material from the equipment with a push stick. Keep hands, hair, and clothing away from any moving machinery parts. Remove all jewelry before you begin work. Compressed air, used to clean the sieves and equipment, presents a safety hazard, especially to the eyes. The compressed-air stream should never be directed toward the face. A fan or exhaust hood should be used to vent dust. See the *CHP* for further information.

## Preliminary procedure

Check the Request for Analysis form (RFA) for notes on mineralogy of samples, requests for preparation that varies from standard procedure, and disposition of excess sample.

Verify that the number of samples received and the field numbers on the sample collection/transport bags correspond to the number of samples and field numbers listed on the RFA. If they do not correspond, contact Sample Control.

Properly label the correct number of sample cartons with the laboratory number assigned to each sample. Label both the container tops and sides using permanent ink-markers, or premade labels. Affix premade labels to the tops and sides of the cartons with clean transparent tape.

Place the labeled sample containers in a cardboard tray labeled with the required information: (1) assigned job number, (2) submitter's last name, and (3) number of samples in the job.

If the samples are wet or damp, place them in a drying oven and dry at 60°C until they are thoroughly dry.

## Procedure

Disaggregate the samples, if necessary, by pounding the sample bag with a hammer or mallet on a contaminate free surface that is cleaned between samples.

Arrange five sieves of the specified mesh size on a counter top, placing a sieve pan under the sieve at the bottom of the stack, and sieve separator pans under the other four. If a series of sized fractions of the samples is requested, the sieves are stacked with the largest mesh sieve at the top and progressively smaller mesh sizes to the bottom. The sieve of smallest mesh size is placed at the bottom.

Open the sample bags and pour five samples into the sieves in a sequential order. Remove as much organic material, larger pebbles, and rock fragments as possible, and further disaggregate any small clumps, still present, by hand. The sieves should not be overfilled, but loaded loosely enough so that the material can move freely in them. Samples of large volume should be divided among as many sieves as needed or, if they contain abundant fine material, split with a Jones splitter to a volume that can be suitably placed in one sieve. Excess sample should be saved or discarded, whichever is noted on the RFA.

Place a sieve cover on the sequentially arranged stack of sieves. Place the heavy metal Ro-Tap cover over the top of the stack and lift the stack into the frame of the Ro-Tap. Adjust the Ro-Tap for the height of the stack by loosening the friction lock bolts on either side of the base plate and sliding the stack up or down on the vertical rails as needed. The stack is positioned correctly when the metal cover plate is up against the metal tongs of the top bracket. Flip down the front half of the hinged top bracket and make sure the automatic hammer is in the down position.

Turn on the Ro-Tap sieve shaker and run long enough to allow thorough sieving of the samples. The length of time required depends on sample composition, average grain size, sieve mesh size, and the volume of sample in the sieve. Sieving the samples for 3 minutes is generally adequate.

During the interval that the sieve shaker is running, clean a second set of five sieves in preparation for loading with the next five samples. The sieves should be thoroughly brushed on both sides of the screen with a sieve brush. All particles should be blown from the sieves with compressed air. If the sieves become particularly dirty, or if the grains become trapped in the screens, they can be placed in an ultrasonic bath for more vigorous cleaning.

When sieving is complete and the sieve shaker has been turned off, remove the stack of sieves. Each sample should be removed from the stack in proper sequence. The coarse fraction of each sample is saved in the sample submittal bag or discarded, whichever is noted on the RFA. The fine fraction of each sample is poured into its corresponding, appropriately labeled, sample carton using a large plastic sample funnel. Care must be taken to maintain the proper order in which the samples were placed into the shaker. As added assurance for maintaining correct sample identity, a small piece of paper with the sample's laboratory number written on it may be placed in the sieve with the sample prior to stacking the sieves.

After the samples have been sieved, they must ordinarily be pulverized to the grain size required for the chemical analysis, and then mixed in a tube-type mixing machine. The sample is considered acceptable if 100 percent passes an 80-mesh screen (<180 μm) and at least 80 percent passes a 100-mesh screen (<150 μm). Refer to the section of this manual entitled *Physical preparation of rock samples* for the details regarding the pulverizing and mixing procedure. The nature and small grain size of the sediment samples facilitates pulverizing and pulverizer cleanup. Pour each pulverized sample back into its carton from the pulverizer catch pan with the aid of the sample funnel. Blow away remaining particles from the pulverizer, pan, and counter top. Run approximately 1 oz of quartz sand through the pulverizer, discard the pulverized sand, and again blow away remaining particles. The equipment should now be suitably clean for introduction of the next sample.

## Equipment maintenance

All mechanical equipment should be lubricated at least once each week or every 250 samples. Use a grease gun containing metal-free grease (i.e. free of elements of interest in analysis) and make certain the lubricant is injected into all of the grease fittings. Do not over-lubricate and wipe excess grease from the fittings with a Kimwipe or paper towel.

Check and make sure all nuts and bolts are securely tightened, prior to turning on any equipment. Check moving parts, including pulverizer belt and grinding plates, for wear. Replace worn parts.