



Chapter A3

Soil sample preparation

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U.S. Geological Survey Open File Report 02-223-A3

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

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Code: Q030

Accepted: 6/25/90

Revised: 3/10/93

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 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 soil sample is disaggregated, if necessary, in the mechanical, ceramic mortar and pestle (soil grinder). The sample is sieved to the required grain size of 100 percent minus 80-mesh (<180 μm) and at least 80 percent minus 100-mesh (<150 μm) using sieves with stainless-steel screens. The sieved fraction is pulverized if further reduction in grain size is required by the subsequent chemical analysis.

Scope

Fifty samples can normally be processed per person day.

Apparatus

- Drying oven
- Nalgene (or similar) trays for air-drying samples
- Aluminum trays for oven-drying samples
- Soil grinder, Nasco-Asplin
- Stainless-steel sieves, with catch pans and lids
- Sieve brush
- Ro-Tap sieve shaker
- Large plastic powder funnel
- Shatter-box, Angstrom or Spex, for pulverizing samples
- Braun vertical pulverizer with ceramic plates
- Sample cartons, 1-oz through 1-pt
- Compressed air source, 40 psi
- Grease gun packed with metal-free grease
- Kimwipes or paper towels
- Quartz sand

Reagents

- Acetone, $\text{C}_3\text{H}_6\text{O}$, laboratory grade

Safety precautions

Eye and ear protection and a dust mask must be worn at all times; it is recommended that a lab coat be worn. Caution must be exercised by the technician while operating grinding equipment. Belts on equipment must be guarded. Keep hands, hair, and clothing away from any moving machinery parts. Remove all jewelry before you begin work. Compressed air (40 psi), used to clean sieves and grinding equipment, presents a safety hazard, particularly to the eyes. A fan or exhaust hood should be used to vent excessive dust. The compressed air stream should never be directed toward the face.

Acetone, used to clean the soil grinder, is extremely flammable, and should be handled accordingly, being kept away from sources of ignition. Avoid breathing acetone fumes by wearing an appropriate respirator and having adequate ventilation. Avoid repeated or prolonged skin contact with acetone. Treatment for acetone exposure is to irrigate eyes with water, wash contaminated areas of body with soap and water, gastric lavage if ingested, followed by saline. See the *CHP* and *MSDS* 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 (bulk).

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.

Procedure

Damp samples are dried overnight in a forced-air drying oven in their original containers or on the nalgene trays upon which they have been spread. To insure proper sample identity is maintained, place the sample bag on the tray with the sample and weight it down with some of the sample. Generally no heat is required, the flow of air in the oven being sufficient to dry the sample. Drying of wet samples is facilitated by setting the oven temperature to 30°C.

If the samples contain aggregates of material following drying they should be disaggregated in the soil grinder.

Remove pebbles and larger rock fragments from the sample by hand. The presence of large pebbles and fragments impedes the operation of the soil grinder and may damage it. Fill the bowl of the soil grinder about halfway. Start the auger and lower it gently onto the sample. If additional fragments or pebbles are revealed, raise the auger well out of the way and turn it off before removing them. When the maximum downward travel of the auger has been reached, maintain this position approximately 30 s, then raise the lever and turn the auger off.

Turn on the switch that activates the chain-driven sieve shaker. Pour the sample slowly onto the 10-mesh screen (2.0 mm). Pour the minus 10-mesh fraction, that is the material that passes through the screen, into the previously labeled sample cartons using a large sample funnel. If there is too much material for the sample carton, split the sample using a Jones Splitter to obtain a split of the sample in an amount to fill the sample carton. Discard the plus 10-mesh fraction, which rolls off into a hopper near the base of the grinder, unless otherwise instructed by the sample submitter. Repeat the soil-grinding process with the remainder, if any, of the sample.

Clean the grinder using a stiff bristle brush and compressed air to rid the sieve screen and the grinder of all dust and soil particles. Wipe the auger bit and bowl with a paper towel or large Kimwipe dampened with acetone.

Caution: Acetone is flammable. Used towels/Kimwipes should be disposed of in fire-proof containers. Plastic or rubber gloves should be worn if working with acetone.

If sieving to a finer grain size is required, pour the sample onto the screen of the proper-sized, clean sieve, with the catch pan of the sieve in place, cover with the lid, and agitate, either by hand or in a Ro-Tap Sieve Shaker, for approximately 2 to 3 min or until no more appreciable gain of finer grained material is realized. Pour the fine fraction from the catch pan into the pre-labeled sample carton using a large, plastic powder funnel. Dispose of the coarse fraction unless otherwise instructed by the submitter. Clean both sides of the sieve screen with a sieve brush and compressed air. Clean the catch pan with an acetone-moistened Kimwipe.

If pulverizing of the sieved sample is required, use the shatter-box to pulverize the sample to 100-mesh (<150 μm) or less or the Braun Pulverizer to grind the sample to approximately 100-mesh.

Pour the sample into the grinding chamber of the shatter-box with the agate puck (and ring, on the Angstrom model) in place in the chamber. Fill the chamber about halfway. Clamp the chamber into place and start the machine. Normal soil grinding to 100-mesh or less is accomplished in approximately 1 to 3 min. Suitability of grind is verified by sieving the sample through a 100-mesh sieve.

Transfer the ground sample to the prelabeled sample carton.

Clean the shatter-box by running quartz sand through it in the same manner as a sample. Use compressed air to rid the chamber of dust and particles and then wipe down with acetone.

For sieved samples that require grinding to only an approximate 100-mesh grain size, pour the sample into a running Braun pulverizer, with previously adjusted, and conditioned grinding plates (see section *Rock sample preparation* for procedure on setting up and operating the pulverizer), with the catch pan in place. Usually, one pass of the soil sample through a properly adjusted pulverizer is adequate. Transfer the ground sample to the previously labeled sample carton. Clean the pulverizer by running approximately 1 tablespoon of sand through it, in the same manner as a sample. Use compressed air to rid the pulverizer of remaining dust and particles.

Equipment maintenance

Lubricate all mechanical equipment 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. Wipe excess grease from the fittings with a Kimwipe or paper towel.

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.