



Chapter C

Dry moist samples to constant weight under ambient conditions, calculate % moisture

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Principle

Percent moisture in soils, sediments and rocks is determined after drying the sample at room temperature in a forced air oven for a period of seven days. The objective of this method is to determine the amount of saturated water in the sample that is not chemically bonded to the matrix material. Determination of the percent moisture allows for the normalization of analytical data to a dry weight basis.

Interferences

There are three general types of interferences that are of concern to this method. The first is the presence of non-aqueous (organic) compounds that volatilize during the drying stage. Weight loss due to the presence of these organic compounds will lead to a positive bias in estimating percent moisture. The second type of interference is due to the presence of hygroscopic or air sensitive compounds that retain water under standard temperature and pressures or undergo weight gain due to air oxidation. The third type of interference is due to a weight loss from the physical departure of biological organisms from the sample during the drying process.

Scope

An operator can analyze approximately 100 samples per drying interval (7 days), including blanks, duplicate, and spiked (H_2O) reference samples. A lower reporting limit of 0.5% is possible using a sample weight of 50g. This method is applicable to soils and sediments that are saturated with water.

Apparatus

Two balances are used for this method. The first a 250 g capacity 3 place, top loading balance for samples <120g, and the second a 2500g capacity 2 place, top loading balance for samples >120 g. A 4 place top loading analytical balance with a 120g capacity is the minimum requirement for this procedure. It is recommended that all balances used in this procedure be enclosed in a protective area to minimize the affect of air currents on balance measurement. The samples are run in the sample containers in which they were shipped.

Forced air drying oven.

Reagents

Deionized water to test precision of method.

Safety precautions

Normal laboratory safety procedures should be followed. Analyst should minimize respiration of dried airborne material due to potential inorganic and biological hazards .

Procedure

1. A calibrated balance is selected which has the appropriate capacity for the expected range of sample mass. Two calibration masses are selected (50g, 200g) which approximate the lower and upper sample masses. The mass of each calibration standard is recorded in a lab book, along with the date and operator name each time the balance is used.
2. The sample is weighed as received using a balance with appropriate capacity. The original sample weight is recorded in a lab book and is also written on the container cap and side.
3. The sample is transported to the drying oven where the cap is removed and placed beneath the open container.
4. After three days, the bottle is removed from the oven, capped, and shaken to redistribute moist sample material from the container bottom. The cap is removed placed beneath the container and the pair placed back in the oven.
5. Sample is dried for an additional four days. During that time the sample is capped, shaken, and observed. After that time the sample is removed from the oven and the contents examined for obvious signs of moisture. If the sample is considered "dry" the container is resealed and transported to the lab and reweighed
6. The sample is dried for another day.
7. Sample mass is determined using the same analytical balance employed in steps 1 and 5 . If the difference of the sample weights between this weight and the weight in step 5 is less than 1% relative the "dry" weight is recorded in a lab book and on the cap and side of the container.
8. Since the "as received" wet sample cannot be easily transferred to a clean container without loosing sample, the mass of the empty sample container should be determined by transferring the dry and easily poured sample contents into a clean bottle. The sample container is then washed, dried, and reweighed to determine bottle mass. If the same type of bottle is used for all samples, then random samples (2) representing different bottle types (size, shape, density) are selected and their contents transferred to a temporary individual container. The original containers are then washed, dried, and weighed to determine bottle mass. The average mass is calculated and used in subsequent calculations.

Calculations

Calculations for percent moisture are carried out using a template created using spreadsheet software. Information on wet weight, dry weight, bottle mass and percent moisture is transferred from lab notebooks to computer and calculations performed.

Percent moisture is determined using equation 1.

$$\text{Eqn 1. \% moisture} = \frac{\text{Wet mass, g} - \text{Dry mass, g}}{\text{Wet mass, g} - \text{Bottle mass, g}} \times 100$$

Assignment of uncertainty

Analytical results are presented below for percent moisture determined using selected reference materials, and quartz sand. At the present time geochemical reference materials are not available which provide certified, recommended, or information values for percent moisture. Table 1 presents information on percent moisture using quartz sand spiked with variable amounts of deionized water. The sample percent moisture levels represents levels commonly encountered in natural samples. The calculated percent recovery values were based on the comparison between the mass of water added versus mass of water lost during drying.

Table 1. —Percent moisture determinations in quartz sand spiked with deionized water

<i>Test No.</i>	<i>Sand, g</i>	<i>H2O added, g</i>	<i>n</i>	<i>Theoretical % Moisture.</i>	<i>Observed % Moisture</i>	<i>% recovery</i>
1	50.	9.907	3	16.54	16.37	99.0
2	50.	19.885	3	28.45	28.31	99.5
4	100.	49.750	2	33.22	33.15	99.8
3	50.	49.750	3	49.87	49.87	100.0

An estimation of the determination limit for the percent moisture method was calculated using samples of USGS reference material BSK-1, GXR-2 and quartz sand. BSK-1 is a sediment material collected from the Kestersen National Wildlife refuge in central California, and GXR-2 is a surface soil collected from Park City, Utah. The samples were dried at 110 °C for two hours and then allowed to cool in a desiccator filled with Drierite. Aliquots of the dried material were transferred into pre-weighed glass bottles and aliquots of deionized water added. Sample mass was obtained before and after adding deionized water. Samples were then dried using standard method guidelines.

Table 2. —Determination limit calculations for percent moisture

<i>Sample ID</i>	<i>Description</i>	<i>n</i>	<i>Theoretical % Moisture</i>	<i>Observed % Moisture</i>	<i>% Recovery</i>
Sand	silica sand	2	0.038	0.37	974
GXR-2	soil	2	0.091	1.194	1312
Sand	silica sand	2	0.200	0.211	105
BSK-1	sediment	2	0.491	0.480	97.7
Sand	silica sand	2	2.218	2.156	97.5
GXR-2	soil	2	3.529	3.211	97.7
BSK-1	sediment	2	6.174	5.982	97.9

Results in table 2 indicate that percent moisture determinations are accurate and precise down to a value of approximately 0.2%. Below this moisture level, significant uncertainty is encountered due in large part to the variation in mass measurements over several days. Based on these results a determination limit of 0.5% is considered appropriate.