

HACH SOIL MANUAL

Calcium Sulfate Extraction for Soil Used for: Nitrate-Nitrogen

The Calcium Sulfate Extraction is used to extract nitrate-nitrogen from soils and can be used for all classes of soils.

CALCIUM SULFATE EXTRACTION PROCEDURE

1. Using a 5-gram soil scoop, measure 2 scoops of the prepared soil sample into a round sample bottle.
2. Using the 0.1 gram plastic spoon, add 1 level spoonful of Calcium Sulfate to the round sample bottle containing the sample.
3. Using a 25-mL graduated cylinder, accurately measure 20 mL of dionized water into the cylinder and transfer it to the round mixing bottle.
4. Repeat steps 1-3 for each soil sample undergoing extraction.
5. Cap and shake each bottle vigorously for 1 minute.
6. Using a plastic funnel and filter paper, filter the contents of the bottle into another round sample bottle.
7. Analyze this extract for nitrate-nitrogen within 2 hours. If this is not possible, then the extract may be refrigerated for 24 hours before the analysis.

Nitrate-Nitrogen in Soil (0-60 ppm). Cadmium Reduction Method

PROCEDURE:

1. Obtain a calcium sulfate extract for the soil sample by performing the Calcium Sulfate Extraction procedure.
2. Label 1 Color Viewing Tube "S" for sample and another Color Viewing Tube "B" for blank. Thoroughly rinse both Color Viewing Tubes with deionized water.
3. Add a small amount of the sample extract (about 1/4" deep) to the Color Viewing Tube marked "S". Cap the tube with a rubber stopper and shake it for a few seconds. Discard this solution.
4. Add the extract to both tubes until the meniscus is even with the 5-mL mark (bottom of frosted area).
5. Add the contents of one NitraVer 5 Powder Pillow to the tube marked "S". Cap and shake the tube vigorously for exactly one minute. *Note: This kit contains a 15 mg/L nitrate-nitrogen standard so the analyst can learn proper shaking technique and use of the color wheel. It is suggested that the analyst practice with the standard until repeated results are within 1 mg/L of each other. The standard should read near 15 mg/L. If the experimental concentration is less than 14 mg/L or greater than 17 mg/L, repeat the procedure until satisfactory results are obtained.*
6. Immediately place tubes "S" and "B" into the comparator with tube "B" in the outside hole and tube "S" in the inside hole.
7. Five minutes after completing Step 6, hold the Color Comparator up to a light

source. Rotate the disc until the color in the window for tube “B” matches the color in the window for tube “S”. Record the value in the scale window.

Take two more readings of the sample, rotating the color disc between each reading. Complete all three readings within 10 minutes of completing Step 6.

Note: Readings before 5 minutes or after 10 minutes will result in an inaccurate value.

Note: It is very important the standard and the sample are viewed under the same lighting conditions.

8. Take the average of the three readings and multiply it by 2 for the available nitrate-nitrogen value in the soil.
9. When analysis is complete, rinse the labware with deionized water. Shake dry. Store the color disc in the plastic bag provided.

Mehlich 2 Extraction for Soil Used for: Calcium + Magnesium, Phosphorus, Potassium

The Mehlich 2 Soil Extractant is used for extracting calcium and magnesium, phosphorus, and potassium from soils. It is suitable for most classes of soils. With highly calcareous soils, calcium and magnesium results will be artificially high.

PREPARING THE MEHLICH 2 EXTRACTANT

Prepare the Mehlich 2 Soil Extractant as follows:

1. Measure 20 ml of the Mehlich 2 Soil Extractant Concentrate into a 25-ml graduated cylinder.
2. Transfer the extractant concentrate to one of the flip-top dispensing bottles provided.
3. Add deionized water to the dispensing bottle until the volume reaches the bottom of the neck. Invert several times to mix.
4. Using the laboratory pen, label the bottle with the extractant name to avoid confusion when working with other extractants. The Mehlich 2 Soil Extractant is now ready for use.

MEHLICH 2 EXTRACTION PROCEDURE

1. Using a 2-gram scoop, measure 1 scoop of the prepared soil sample into a round sample bottle.
2. Using a 25-mL graduated cylinder, accurately measure 20 mL of the diluted Mehlich 2 Soil Extractant into the cylinder, then transfer it to the round sample bottle.
3. Repeat steps 1-2 for each soil sample undergoing extraction.
4. Cap and shake each bottle 5 times.
5. Using a plastic funnel and filter paper, filter the contents of the bottle into another round sample bottle.
6. Use this filtered extract for calcium + magnesium, phosphorus, and potassium analysis. *Note: The filtered Mehlich 2 Soil Extract is suitable for approximately 24 hours. If stored longer, refrigerate to prevent microbial growth.*

Note: The volume of extract obtained from the filtration should be adequate for the analysis of calcium + magnesium, phosphorus and potassium

Phosphorus in Soil (0-130 mg/L or 0-130 ppm). PhosVer 3 (Ascorbic Acid) Method

PROCEDURE:

1. Obtain a Mehlich 2 extract for the soil sample by performing the Mehlich 2 Extraction.
2. Using the 2.5 mL dropper, add 2.5 mL of the filtered sample extract prepared in Step 1 to a 25 mL graduated cylinder. Dilute to the 25 mL mark with deionized water, stopper, and invert to mix.
3. Label 1 Color Viewing Tube "S" for sample and another Color Viewing Tube "B: for blank. Thoroughly rinse both Color Viewing Tubes with deionized water. Shake the tubes to remove the remaining rinse water.
4. Add a small amount of the diluted extract (about ¼" deep) to the Color Viewing Tube marked "S". Cap the tube with a rubber stopper and shake it for a few seconds. Discard this solution.
5. Add the diluted Mehlich 2 Extract to both tubes until the meniscus is even with the 5 mL mark on the tubes (bottom of frosted area).
6. Add the contents of one PhosVer 3 Powder Pillow to the "S" tube. Cap and shake tube vigorously for one minute.
7. Immediately place tubes "S" and "B" into the comparator with tube "B" in the outside hole and tube "S" in the inside hole.
8. Three minutes after completing step 6, hold the Color Comparator up top a light source. Rotate the disc until the color in the window for tube "B" matches the color in the window for tube "S". Record this value in the window. Take two more readings for the sample, rotating the color disc between each reading. Complete all three readings within 10 minutes of completing Step 7. *Note: Readings before 3 minutes or after 10 minutes will result in an inaccurate value.*
Note: It is very important the standard and the sample are viewed under the same lighting conditions.
9. Take the average of the three readings and multiply this average by 3.3 for the available phosphate-phosphorus value in the soil.
10. When the analysis is complete, rinse the color viewing tubes with deionized water and store the color disc in the plastic bag provided.

Potassium (Exchangeable) in Soil (0-250 mg/L or 0-250 ppm). Turbidimetric Tetraphenylborate Method

1. Obtain a Mehlich 2 extract for the soil sample by performing the Mehlich 2 Extraction.
2. Using the 1 mL dropper, add 3.0 mL of the sample extract from Step 1 to a 25 mL cylinder.
3. Add deionized water to the 21 mL mark on the cylinder. Firmly cap the

- cylinder with a #3 rubber stopper and invert to mix.
4. Add one Potassium 2 Reagent Powder Pillow and 3 mL of Alkaline EDTA Solution to the cylinder. Cap the cylinder and invert several times to mix. Allow the solution to stand for at least 3 minutes.
 5. Add the contents of one Potassium 3 Reagent Powder Pillow. Firmly cap and shake the cylinder vigorously for 10 seconds. Allow the solution to stand for at least 3 minutes but no longer than 10 minutes. A white turbidity will develop.
 6. While looking straight down into the cylinder, slowly insert the Potassium Dip Stick vertically into the solution until the black dot is no longer visible from above the cylinder.
 7. Hold the dipstick in that position and rotate the cylinder so you can see the scale on the dipstick. Look across the surface of the sample to the scale on the dipstick. Record the number (in mm) on the dipstick scale where the surface of the sample meets the dipstick scale.
 8. Repeat Steps 6-7 two more times recording the value each time. Take the average of the three readings. Refer to the potassium conversion table on the following page to determine the level of potassium in the soil.
 9. When analysis is complete, rinse the labware with deionized water.

POTASSIUM CONVERSION TABLE

Dipstick Reading mm	mg/L (ppm) Potassium	hg/Ha Potassium	meq/100 g Potassium
80	87	194	0.22
75	94	210	0.24
70	101	225	0.26
65	109	243	0.28
60	118	263	0.30
55	129	281	0.33
50	143	319	0.37
45	159	355	0.41
40	180	401	0.46
35	207	462	0.53
30	243	542	0.62
25	294	6546	0.75

Aqueous Extraction Method for Soil Used for: Salinity and pH

Use the Aqueous Extraction method to prepare the soil sample for salinity and pH measurements. The soil suspension does not need to be filtered to determine pH and salinity— measurements can be made directly in the suspension.

AQUEOUS EXTRACTION PROCEDURE

1. Use a 5 gram scoop, measure 4 scoops of the prepared soil into a 50 mL plastic beaker.

2. Using a 25 mL graduated cylinder, accurately measure 20 mL of dionized water and transfer it to the 50 mL beaker.
3. Repeat steps 1-2 for each soil sample undergoing extraction. *Note: To prevent confusion when working with several samples, label each beaker with the sample or test name.*
4. Using the spatula, stir the contents of the beaker for 1 minute at 10-minute intervals over a 30 minute period.
Note: Rinse the spatula with deionized water before stirring each sample.
5. After 30 minutes, use the prepared sample for salinity and pH determination.
Note: Because conductivity is affected by minute amounts of potassium chloride resulting from pH determination, measure the salinity of the sample before measuring pH.

pH in Soil. Electrode Method

PREPARATION

Calibrate the Pocket Pal pH Tester according to the directions below.

1. Prepare a pH 7.00 buffer solution by adding the contents of one pH 7.00 Buffer Powder Pillow to a 50-ml beaker containing 50 mL of water. Swirl the beaker to dissolve the contents.
2. Slide the on/off switch located on top of the Pocket Pal pH Tester to on.
3. Remove the protective cap from the bottom of the instrument and immerse the electrode tip one inch below the surface of the solution. If necessary, calibrate the instrument by adjusting the calibration screw near the pocket clip with the trim tool (screwdriver) provided to obtain a reading of 7.0.

PROCEDURE

1. Obtain an aqueous extract for the soil sample by performing the Aqueous Extraction.
2. Immerse the tip of the calibrated Pocket Pal pH Tester one inch below the surface of the aqueous sample extract and stir gently until the soil is completely suspended.
3. Record the stabilized pH reading to the nearest 0.1 pH unit.
4. Rinse the pH electrode with deionized water. Remove the excess by wiping the tip with a tissue before continuing to the next sample.

Once the pH of each sample has been determined, rinse the electrode with deionized water. Slide the on/off switch of the Pocket Pal pH Tester to off and cover the electrode with the protective cap before storing.

Salinity (Conductivity) in Soil. Electrode Method

PREPARATION

1. Add 50 mL of the Saturated Calcium Sulfate Extraction Solution used for the Calcium Sulfate Extraction procedure in Section 3 to a 50-mL plastic beaker.
2. Slide the on/off switch on top of the Pocket Pal Conductivity Tester to on.

3. Remove the protective cap from the bottom of the instrument. Immerse the electrode tip one inch below the surface of the calcium sulfate extraction solution. If necessary, calibrate the instrument by adjusting the calibration screw near the pocket clip with the trim tool (screwdriver) provided. Adjust the value to 2200 μS . At 2200 μS , the display will read 22 with a small 100 in the upper left hand corner.

PROCEDURE

1. Obtain an aqueous extract for the soil sample by performing the Aqueous Extraction 3.
2. Immerse the tip of the calibrated Pocket Pal Conductivity Tester one inch below the surface of the sample extract prepared in Step 1 and stir gently until the soil is completely suspended. *Note: The range of the Pocket Pal Conductivity Tester is 100-19,900 μS . To obtain the proper value in $\mu\text{S}/\text{cm}$, multiply the large reading on Pocket Pal Conductivity Tester by the small value (100) in the upper left hand corner of the display. This gives the conductivity value in $\mu\text{S}/\text{cm}$. However, the conductivity of soils should be expressed in mS/cm . Simply divide the large-sized reading by 10 to determine conductivity in mS/cm .*
3. When the digital display stabilizes, record the value. Divide the value in the display by 10 to determine the mS/cm of conductivity. *Note: If the larger display is 199 or greater, the concentration of salt is greater than the capacity of the meter. Dilute the suspension by adding an additional 20 mL of deionized water. Multiplying the final answer by two. If this dilution results in a display reading that is still above capacity, additional 20 mL volumes should be added until a suitable reading is obtained. For every 20 mL added, add two to the multiplication factor (if a total 40 mL is added, multiply by 2; if a total 60 mL is added, multiply by 3 and so on).*
4. Rinse the electrode with deionized water, and remove the excess by wiping the tip with a tissue before continuing to the next sample. *Note: To maintain or improve performance, periodically clean the stainless steel electrode by rinsing it in alcohol.*
5. Once the conductivity of each sample has been determined, rinse the electrode with deionized water. Slide the on/off switch to off and cover the electrode with the protective cap before storing.