

84-024 EXTRACTABLE TRACE ELEMENTS**1. Application**

- 1.1 These two methods provide a fairly rapid procedure for determining trace elements in soils. It has been recognized that contamination can be minimized prior to analysis by grinding with mortar and pestle and sieving through nylon screens. Both of the outlined procedures have been used to assess metal solubilities and contamination in soils. 0.05M EDTA has been used to assess copper availability for regulating sludge application to soils. Similarly, 0.005M DTPA has been used to assess the solubilities in soils of both nutrient and non-nutrient metals. Evidence indicates that the DTPA-extractable metals are generally related to plant availabilities.

2. Apparatus

- 2.1 125 mL Erlenmeyer flasks.
- 2.2 Parafilm.
- 2.3 Filter funnels.
- 2.4 Filter paper (Whatman #42).
- 2.5 Repipet dispensing bottle (accuracy 1%, reproducibility 0.1%).
- 2.6 Reciprocating shaker (176 oscillations per minute).
- 2.7 Atomic absorption spectrophotometer (Model 1200 Varian techtron).
- 2.8 Test tubes (50 mL of a type suitable for high speed centrifugation).

3. Reagents

- 3.1 DTPA (diethylenetriaminepentaacetic acid) extracting solution: The DTPA extraction solution is prepared to contain 0.005M DTPA, 0.01M CaCl_2 , 0.1M TEA and is adjusted to pH 7.30. To prepare 4 liters of this solution dissolve 59.68 g of reagent grade TEA (triethanolamine), 7.868 g of DTPA (diethylenetriamenepentaacetic acid), and 5.88 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in approximately 200 mL of dionized water. Allow sufficient time for the DTPA to dissolve, and dilute to approximately 3.5 liters. Adjust the pH to 7.30 ± 0.05 with 1N HCl, dilute to 4 liters and mix well. This solution is stable for several months.
- 3.2 EDTA (disodium ethylenediaminetetraacetic acid) extracting solution: The EDTA extracting solution is prepared to contain 0.05M disodium EDTA, 0.01M

CaCl₂, and 0.1M TEA. To prepare 20 liters of this solution dissolve 372.2 g of disodium EDTA, 29.4 g of CaCl₂ and add 268 mL of TEA (triethanolamine) and make the volume to 20 liters. Adjust pH to 7.0 with NaOH or HCl.

3.3 Certified atomic absorption standards +1%.

4. Procedure

4.1 DTPA extractable trace elements.

- 4.1.1 Weigh 20 g of air dry soil ground to pass a 2 mm sieve (nylon) into a 125 mL Erlenmeyer flask.
- 4.1.2 Add 40 mL of DTPA extracting solution.
- 4.1.3 Cover each flask with stretchable parafilm and secure uprights on a reciprocating shaker.
- 4.1.4 Shake at a speed of about 176 cycles/min for 2 hours.
- 4.1.5 Filter by gravity through Whatman #42 filter paper. Analyse the filtrates for Zn, Cu, Ni, Pb and Cd by atomic absorption.

NOTE: a) Make the appropriate standards in DTPA extracting solution.
b) It is preferable to keep DTPA soil extracts refrigerated and analysis completed within several days. DTPA soil extracts will develop a precipitate within 2 or 3 days if left standing at room temperature.

4.2 EDTA extractable trace elements.

- 4.2.1 Weigh 10 g of air dry soil ground 2 pass a 2 mm sieve (nylon) into a 50 mL plastic test tube.
- 4.2.2 Add 20 mL of EDTA extracting solution, stopper and shake for 30 minutes.
- 4.2.3 Centrifuge at 20,000 G for 10 minutes. If necessary filter through Whatman #42 filter paper to remove any floating particles.
- 4.2.4 Save the filtrate for the determination of Cu, Mn, Zn, Pb, Fe, Ni, Co, Cd, Cr by atomic absorption.

NOTE: a) Make up appropriate standards in EDTA extracting solution.

5. Calculations

5.1 $\mu\text{g/g}$ element in soil = $\mu\text{g/mL}$ in extract x 2

6. Precision

6.1 Insufficient data available.

7. References

- 7.1 Lindsay, W.L. and Norwell, W.A. 1969. Equilibrium relationships of Zn^{2+} , Fe^{3+} , Ca^{2+} and H^+ with EDTA and DTPA in soils. Soil Sci. Soc. Am. Proc. 33, 62-68.
- 7.2 Webber, M.D. and Corneau, D.G.M. Metal Extractability from sludge-soil mixtures. Int. Conf. on Heavy Metals in the Environment Proc., Vol. 1 205-225.

Notes