



Designation: D5435 – 03(Reapproved 2008)

Standard Test Method for Diagnostic Soil Test for Plant Growth and Food Chain Protection¹

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1. Scope*

1.1 This test method covers the determination of quantity (Q) and intensity (I) results for several elements in soils, spoils, fly-ash, and other soil substitutes to ascertain their suitability for the growth of vegetation and possible adverse effects of metals on the food chain.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

E617 Specification for Laboratory Weights and Precision Mass Standards

2.2 EPA Standard:

U.S. EPA Method 3050 A³

3. Summary of Test Method

3.1 A representative sample material to be tested is air dried, sieved to pass a 2-mm screen, equilibrated for 16 to 20 h with the soil test solution containing the optimum activities (I) of

H_3O^+ , Ca^{++} , Mg^{++} , K^+ and DTPA (diethylenetriaminepentaacetate) to render a small exchange of metals from the solid phase to the solution phase. This test method is calibrated with respect to pH, K, Ca, Mg, $H/(Ca + Mg)^{1/2}$, Mg/K, Ca/Mg, Mn, Fe, Cu, Zn, Al, Pb, Ni, and Cd. In addition to the exchangeable or labile amounts (Q), or both, of the same elements, comparable levels of the negative logarithm values analogous to pH may be calculated using the ligand constants for the respective elements.

4. Significance and Use

4.1 The bioavailability of chemical elements is poorly related to the chemical composition of soils and plant growth media containing a mineral or any type of adsorbed phase. The chemical potential (π for element, i,) is an intensity parameter (I), and the sorbed amount in equilibrium with the soil solution is a measure of the quantity (Q). These parameters for each element (essential or toxic) should be measured in the presence of other elements at or near the desired intensity. This test method is the only method that generates these results simultaneously for several elements. The computer software allows these values to be related to the total sorbed quantities of the different elements. For many substrates, it has been found that the theory for the method holds to the degree that vegetation has been established on many non-soil substrates and soil-water-food chain problems have been evaluated by this test method. This test method has been used on many sites in Pennsylvania and other locations to monitor the effect of sewage sludge applications on land as a source of essential elements for plants with no harmful effects on the food chain. It has also been used to evaluate synthetic soils produced from fly-ash alone or as a component of coal refuse for the establishment of vegetation on mine spoils, coal refuse piles, and abandoned mine lands.

4.2 The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice **D3740** are generally considered capable of competent and objective testing/sampling/inspection and the like. Users of this standard are cautioned that compliance with Practice **D3740** does not in

¹ This test method is under the jurisdiction of ASTM Committee **D18** on Soil and Rock and is the direct responsibility of Subcommittee **D18.22** on Soil as a Medium for Plant Growth.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

*A Summary of Changes section appears at the end of this standard

itself assure reliable results. Reliable results depend on many factors; Practice **D3740** provides a means of evaluating some of those factors.

5. Interferences

5.1 Quality control in reagent preparation is more difficult than for methods that involve fewer elements and compounds. External contamination is a problem to be anticipated. Rubber is a source of Zn and Cd contamination. Rubber gloves used to remove glassware from an acid bath must be removed for subsequent rinse procedures. The blank solution should be tested for Na and other elements. Often the superfloc contains Na which should be removed by use of a cation exchange resin. In addition the filter paper should be checked for Na.

5.2 The cross contamination from glassware is a serious problem. All glassware is soaked in 1 + 1 HNO₃ for 2 h; rinsed with distilled water three times followed with rinsing in distilled, deionized water three times. Remove rubber gloves after first rinsing with distilled water to prevent contamination with Zn.

5.3 The testing solution should be prepared fresh every week. Limited studies indicate that the relative amounts of the different elements desorbed from soils by DTPA change as the prepared solution ages.

5.4 The effect of Ca and other ions on the atomic absorption determination of Cd is significant and requires that an adjustment be made. Background correction will eliminate this source of error.

5.5 The TEA (triethanolamine) is used to prevent excessive dissolution of trace metals from high p-H soils. The buffer capacity is not sufficient, however, to prevent a reflection of soil acidity on trace element availability in soil.

5.6 For some soils the colorimetric determination of phosphorus is not adequate because of the development of turbidity with stannous chloride used as the reducing agent or when *P* is very near the detection limit of 1 µm/L. Other reducing agents as proposed by Watanabe and Olsen (**1**)⁴ or the isobutyl alcohol extraction method as proposed for water soluble *P* (see section 12.9) have been used with limited success on these samples.

6. Apparatus

6.1 *No. 10 Sieve*, (U.S. Sieve Series Standard 2 mm) constructed from polyethylene.

6.2 *Erlenmeyer Flasks*, 125-mL capacity with polyethylene-covered rubber stoppers or equivalent.

6.3 *Rotating Shaker*, 150 rpm for 125-mL flasks.

6.4 *Filter Funnels*, 11 cm.

6.5 *Filter Paper*,⁵ 15 cm in diameter.

6.6 *Sample Storage Bottles*, 50 mL polyethylene.

6.7 *pH Meter*, with reference and glass electrodes, that is accurate to ±0.02-pH units when standardized against pH 4.01 and pH 7.00 buffers.

6.8 *Precise Automatic Diluter*, for use with soil test solutions and standards 1:50 in SrCl₂ solution for Mg and Ca determinations by flame AA or ICP.

6.9 *Automatic Diluter*, accurate, calibrated, for diluting soil test solutions that are above the range of the working standards.

6.10 *Atomic Absorption or ICP Spectrometer*, equipped with background correction.

6.11 *Colorimeter*, with 2-cm light-path cells or autoanalyzer for colorimetric analyses.

6.12 *Analytical and Top-Loading Balances*, accuracy to be verified using Specification **E617** weights.

6.13 *50-mL Test Tubes, 15-mL Volumetric Pipettes, and 1-mL Measuring Pipettes*, for use in *P* determinations.

6.14 *50-mL Erlenmeyer Flasks, 10-mL Volumetric Pipettes, and 1-mL Measuring Pipettes*, for *S* determination.

6.15 *Various Beakers*, Erlenmeyer flasks, graduated cylinders, volumetric pipettes, flasks, storage bottles, magnetic heating and stirring unit, refrigerator, and other equipment as required for preparing and storing stock solutions, soil test solutions, reagents, and standards.

6.16 *Computer Facilities*—Computer software that is compatible with IBM-PC and mainframe computers is available to produce the chemical potentials (negative logarithm of the activity of respective ions in soil or substrate solutions) is available. The software is beneficial in producing output that provides decision support for users of this test method.

7. Reagents and Materials

7.1 *Stock Solutions*—Prepare the following stock solutions by dissolving the indicated amount of the pure salt in pure water and diluting to 1 L:

Stock Solution	Amount of Salt per Litre of Solution
0.10 M NaCl	5.84 g of NaCl
0.25 M KCl	18.64 g of KCl
1.00 M MgCl ₂	203.31 g of MgCl ₂ ·6H ₂ O
2.00 M CaCl ₂	294.04 g of CaCl ₂ ·2H ₂ O
0.010 M KH ₂ PO ₄	1.3609 g of KH ₂ PO ₄
1000 mg/l S	5.435 g of K ₂ SO ₄

The use of the above reagents to prepare stock solutions enables a more constant pH to be achieved when TEA is added than the use of compounds considered to provide standards of more accurate composition. Since the chloride salts are not suitable for primary standards, they should be compared with reliable, primary standards and the volumes used are adjusted to meet the required final concentrations.

7.2 *Primary Standards*—Use commercial primary standards for AA or ICP applications, or both, or prepare 1000-mg/L stock solutions of Al, Zn, and Cd by dissolving 1.000 g of the pure metal in 50 mL of 1:1 concentrated HCl-water mixture and diluting to 1 L with pure water. Prepare 1000 mg/L stock solutions of Mn, Fe, Ni, and Cu by dissolving 1.000 g of the pure metal in 50 mL of 1:1 concentrated HCl-water mixture and diluting to 1 L with pure water.

⁴ The boldface numbers in parentheses refer to a list of references at the end of this test method.

⁵ The sole source of supply of the apparatus known to the committee at this time is S & S Number 402S. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

pure metal in 1:1 concentrated HNO_3 -water mixture and diluting to 1 L with pure water.

7.3 *DTPA*, diethylenetriaminepentaacetic acid.

7.4 *TEA* (0.0275 M *TEA*)—Dissolve 4.101 g of *TEA* in pure water and dilute to 1 L.

7.5 *Superfloc-127 Solution*, 1 % —To 700 mL of pure water in a 1-L beaker on a magnetic stirring unit, slowly add 10.0 g of *Superfloc-127*, a small portion at a time, with continuous stirring and gentle heating. Cover the beaker with a watch glass and allow this solution to stir overnight. Transfer this solution quantitatively to a 1-L volumetric flask and dilute to 1 L. Store this solution in the refrigerator. Dilute 10 mL of the solution to 100 mL and check for Na and other contaminants. A cation exchange resin can be used to remove the Na.

7.6 *Preservative Solution*—Dissolve 0.1 g of phenylmercuric acetate in 20 mL of dioxane and dilute to 100 mL with pure water. This solution will be cloudy. Store in the refrigerator.

7.7 *Baker Soil Test*⁶ *Solution*—Dissolve 1.5734 g of *DTPA* in 300 mL of pure water with gentle heating. Transfer this solution quantitatively to a 1-L volumetric flask. Add to this the indicated amounts of the following stock solutions:

Stock Solution	Amount of Each Stock Solution per Litre of Soil Test Solution, mL
0.25 M KCl	10.00
1.00 M MgCl_2	10.00
2.00 M CaCl_2	25.00
1 % <i>Superfloc-127</i> solution	100.00
<i>Preservative solution</i>	10.00

7.7.1 The solution will turn cloudy upon adding the preservative solution. Vigorous mixing will turn the solution clear. Dilute the flask contents to 1 L with pure water and store the soil test solution in the refrigerator. Prepare a fresh test solution weekly.

7.8 *SrCl₂ Diluting Solution*—Dissolve 161.2 g of $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ in pure water and dilute to 1 L. Dilute 40.0 mL of this solution to 1 L with pure water.

7.9 *Ammonium Molybdate Reagent*—Dissolve 4.17 g of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ in 33 mL of pure water. Add to this 47 mL concentrated H_2SO_4 . When cool, add 87 mL of pure water. Store in a dark-colored glass bottle in the refrigerator.

7.10 *Stannous Chloride Reagent*—Dissolve 5 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 12.5 mL of concentrated HCl. Store in a dark-colored glass bottle in the refrigerator. Dilute 1 mL of this solution to 75 mL with pure water for use on a daily basis.

7.11 *Acid Seed Reagent* (6 N HCl containing 20 mg/l S as K_2SO_4)—Dilute 40.0 mL of the 1000-mg/L S standard to 1 L with pure water. Mix 50.0 mL of this solution with 50.0 mL concentrated HCl to obtain the acid seed reagent.

7.12 *BaCl₂ · 2H₂O Crystals*, 20 to 60 mesh material is required.

7.13 *Stock Standard Solution*—Prepare a combined stock standard by pipetting the indicated amount of each stock solution listed as follows into a 1-L volumetric flask and diluting to volume with pure water:

Stock Solution	Amount of Stock Solution per Litre of Combined Stock Standard, mL	Element Concentration in Combined Stock Standard
0.10 M NaCl	100.0	10×10^{-3} M
0.25 M KCl	40.0	10×10^{-3} M
1.00 M MgCl_2	40.0	40×10^{-3} M
2.00 M CaCl_2	100.0	200×10^{-3} M
1000 mg/L Al	80.0	80 mg/L
1000 mg/L Mn	80.0	80 mg/L
1000 mg/L Fe	80.0	80 mg/L
1000 mg/L Ni	20.0	20 mg/L
1000 mg/L Cu	20.0	20 mg/L
1000 mg/L Zn	30.0	30 mg/L
1000 mg/L Pb	20.0	20 mg/L
1000 mg/L Cd	20.0	20 mg/L

7.14 *Compensating Solution*—Dissolve 1.573 g of *DTPA* in 300 mL of pure water with gentle heating. Transfer this solution quantitatively to a 1-L volumetric flask. Add to this 10.0 mL of the preservative solution and 100.0 mL of the 1 % *Superfloc-127*⁶ solution and dilute to 1 L with pure water. Store in the refrigerator.

7.15 *Working Standards for Atomic Absorption Spectrophotometer* —Prepare ten flame atomic absorption standards by pipetting the indicated amount of combined stock standard for each working standard listed in Table 1 into a 500- mL volumetric flask, adding 50.0 mL of compensating solution and 5.0 mL of concentrated HNO_3 , and diluting to volume with pure water.

7.16 *Phosphorus Working Standards*—Dilute 100.00 mL of the 0.010 M KH_2PO_4 stock solution to 1 L with pure water to give a solution 0.001 M P. Prepare phosphorus working standards in the range 0 to 20×10^{-6} M P by diluting aliquots of the 0.001 M from standard to 100 mL with pure water. Prepare these standards for use on a daily basis. Five or six standards should be sufficient to prepare a calibration curve.

7.17 *Sulfur Working Standards*—To 100.0 mL of the 1000 mg/L sulfur standard, add 100.0 mL of the soil test solution and dilute to 1 L with pure water to yield a 100 mg/L sulfur standard. Prepare the working sulfur standards by pipetting the indicated amounts of 100 mg/L sulfur standard and soil test solution for each sulfur standard listed as follows into a 100-mL volumetric flask and diluting to volume with pure water. Prepare these standards for use on a daily basis:

Standard Number	Amount of 100 mg/L S Standard per 100 mL of Working S Standard, mL	Amount of Soil Test Solution per 100 mL of Working S Standard, mL	S Concentration in Working S Standard, mg/L
1	0.00	10.00	0
2	2.00	9.80	2
3	5.00	9.50	5
4	10.00	9.00	10
5	20.00	8.00	20
6	30.00	7.00	30
7	40.00	6.00	40

8. Sample Preparation

8.1 *Sample Preparation*—Air dry and crush the soil or soil substitute sample to pass a 2-mm sieve

⁶ Baker Soil Test is a registered trademark of Land Management Decisions to protect the name and enable the software to be maintained and updated as required to provide “users” of franchised laboratories with maximum benefits from this test method.