Designation: D5435 – 13

Standard Test Method for Diagnostic Soil Test for Plant Growth and Food Chain Protection[1]

This standard is issued under the fixed designation D5435; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 standard. No other units of measurement are included in this standard.

1.3 All measured and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.4 *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
D6026 Practice for Using Significant Digits in Geotechnical Data
E617 Specification for Laboratory Weights and Precision Mass Standards

3. Terminology

3.1 For definitions of common terms used in this standard, refer to Terminology D653.

4. Summary of Test Method

4.1 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.

5. Significance and Use

5.1 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 (pi 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.

NOTE 1—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

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[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.

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

and objective testing/sampling/inspection and the like. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Interferences

6.1 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.

6.2 The cross contamination from glassware is a serious problem. All glassware is soaked in 1 + 1 HNO_3 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.

6.3 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.

6.4 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.

6.5 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.

6.6 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**)[3] or the isobutyl alcohol extraction method as proposed for water soluble *P* have been used with limited success on these samples.

7. Apparatus

7.1 *2 mm (No. 10 Sieve),* constructed from polyethylene.

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

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

7.4 *Filter Funnels,* 11 cm.

7.5 *Filter Paper,* [4] 15 cm in diameter.

7.6 *Sample Storage Bottles,* 50 mL polyethylene.

7.7 *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.

7.8 *Precise Automatic Diluter,* for use with soil test solutions and standards 1:50 in $SrCl_2$ solution for Mg and Ca determinations by flame AA or ICP.

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

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

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

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

7.13 *50-mL Test Tubes, 15-mL Volumetric Pipettes, and 1-mL Measuring Pipettes,* for use in *P* determinations.

7.14 *50-mL Erlenmeyer Flasks, 10-mL Volumetric Pipettes, and 1-mL Measuring Pipettes,* for *S* determination.

8. Reagents and Materials

8.1 *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 $MgCl_2 \cdot 6H_2O$
2.00 M $CaCl_2$	294.04 g of $CaCl_2 \cdot 2H_2O$
0.010 M KH_2PO_4	1.3609 g of KH_2PO_4
1000 mg/1 s	5.435 g of K_2SO_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.

8.2 *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 1:1 concentrated HNO_3-water mixture and diluting to 1 L with pure water.

8.3 *DTPA,* diethylenetriaminepentaacetic acid.

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

8.5 *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

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[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[4] 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.

100 mL and check for Na and other contaminants. A cation exchange resin can be used to remove the Na.

8.6 *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.

8.7 *DTPA Soil Test 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 % Superfloc-127 solution	100.00
Preservative solution	10.00

8.7.1 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.

8.8 *$SrCl_2$ Diluting Solution*— Dissolve 161.2 g of $SrCl_2 \cdot 6H_2O$ in pure water and dilute to 1 L. Dilute 40.0 mL of this solution to 1 L with pure water.

8.9 *Ammonium Molybdate Reagent*—Dissolve 4.17 g of $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ 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.

8.10 *Stannous Chloride Reagent*—Dissolve 5 g of $SnCl_2 \cdot 2H_2O$ 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.

8.11 *Acid Seed Reagent (6 N HCl containing 20 mg/1 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.

8.12 *$BaCl_2 \cdot 2H_2O$ Crystals,* 20 to 60 mesh material is required.

8.13 *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

8.14 *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 solution and dilute to 1 L with pure water. Store in the refrigerator.

8.15 *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.

8.16 *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.

8.17 *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

TABLE 1 Working Standards for Atomic Absorption Spectrophotometer

Working Standard Number	Amount of Combined Stock Standard per 500 mL of Working Standard
1	50.0
2	40.0
3	30.0
4	20.0
5	12.50
6	8.00
7	6.00
8	4.00
9	2.00
10	1.00

Elemental Concentrations of Working Standards

Element	Unit	Standard Number				
		1	2	3	4	5
Na	10^{-4} *M*	10.0	8.0	6.0	4.0	2.5
K	10^{-4} *M*	10.0	8.0	6.0	4.0	2.5
Mg	10^{-4} *M*	40.0	32.0	24.0	16.0	10.0
Ca	10^{-4} *M*	200.0	160.0	120.0	80.0	50.0
Al	mg/L	8.0	6.4	4.8	3.2	2.0
Mn	mg/L	8.0	6.4	4.8	3.2	2.0
Fe	mg/L	8.0	6.4	4.8	3.2	2.0
Ni	mg/L	2.0	1.6	1.2	0.8	0.5
Cu	mg/L	2.0	1.6	1.2	0.8	0.5
Zn	mg/L	3.0	2.4	1.8	1.2	0.75
Pb	mg/L	2.0	1.6	1.2	0.8	0.5
Cd	mg/L	2.0	1.6	1.2	0.8	0.5

Element	Unit	Standard Number				
		6	7	8	9	10
Na	10^{-4} *M*	1.6	1.2	0.8	0.4	0.2
K	10^{-4} *M*	1.6	1.2	0.8	0.4	0.2
Mg	10^{-4} *M*	6.4	4.8	3.2	1.6	0.8
Ca	10^{-4} *M*	32.0	24.0	16.0	8.0	4.0
Al	mg/L	1.28	0.96	0.64	0.32	0.16
Mn	mg/L	1.28	0.96	0.64	0.32	0.16
Fe	mg/L	1.28	0.96	0.64	0.32	0.16
Ni	mg/L	0.32	0.24	0.16	0.08	0.04
Cu	mg/L	0.32	0.24	0.16	0.08	0.04
Zn	mg/L	0.48	0.36	0.24	0.12	0.06
Pb	mg/L	0.32	0.24	0.16	0.08	0.04
Cd	mg/L	0.32	0.24	0.16	0.08	0.04

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

9. Sample Preparation

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

9.2 *Synthetic Soil Mixtures*—For mixtures of fly-ash, spoils, compost, etc. to make a synthetic soil, the materials should be prepared as soil and the mixture should be wetted, air-dried, and mixed at least two and preferably three times before it is tested. Otherwise, the system will be changing and the test solution cannot reach equilibrium.

10. Calibration

10.1 *Working Standards*—Chose working standards from those described above. Dilute samples with element concentrations above the range of the instrument or standards with solution containing 100 mL of compensating solution per litre.

10.2 *Calibration*—Use the working standards to prepare the necessary calibration curves for calculating the element concentrations in the samples. The number of standards used will depend on the linear range for each instrumental method. Pay careful attention to the instrument manufacturer's instruction in this regard.

11. Procedure

11.1 *Blank Preparation*—Prepare a blank soil test solution by pipetting 40.0 mL of pure water (RO + deionized or equivalent), 5.0 mL of soil test solution, and 5 mL of 0.0275 *M* TEA into a 125-mL Erlenmeyer flask and mix. Check the pH of this solution. It should be 7.3 ± 0.05; if not, adjust the amount of 0.0275 *M* TEA and pure water used to obtain a pH of 7.3 ± 0.05 in a final volume of 50.0 mL of soil test solution. Greater precision can be achieved by working with 1-L volumes for 20 samples at a time.

11.2 *Soil Test Equilibration*—Weigh 5.000 g of soil (< 2 mm) into a 125-mL Erlenmeyer flask. Add to the flask the amount of pure water needed to obtain a pH of 7.3 ± 0.05 and a final volume of 50.0 mL with no soil present as determined in the blank preparation step (see 11.1). Add 5.0 mL of soil test solution to the flask. Add to the flask the amount of 0.0275 *M* TEA needed to obtain a pH of 7.3 ± 0.05 and a final volume of 50 mL with no soil present as determined in the blank preparation step (see 11.1). Stopper the blank and sample flasks with plastic stoppers and place on a rotating shaker for 1 h at 150 oscillations per minute. Allow the flasks to stand for an additional 21 to 23 h and filter the samples through filter paper.[4]

11.3 *pH*—Determine the pH of an aliquot of sample using a pH meter and glass and reference electrodes standardized against pH 4.01 and 7.0 buffers.

11.4 *Elemental Analysis*—The actual analysis of sample will vary with instrumentation. Na and K may be analyzed by flame emission spectroscopy; for Ca and Mg by Flame AA, the test solution and standards may need to be diluted 1:50 in the $SrCl_2$ diluting solution using a precise automatic diluter. Both Ca and Mg may be determined by Flame AA. All other elements may be determined by Flame or Flameless AA, or both, depending on the concentrations in solution as well as by ICP.

11.5 *Na and K*—These two elements are easily determined in the blank and soil test solution samples by flame emission spectroscopy.

11.6 *Mg and Ca*—Dilute the standards and blank and soil test solutions samples 1:50 in the $SrCl_2$ diluting solution using a precise automatic diluter. Determine the concentrations of these elements in the blank and samples by atomic absorption spectrophotometry or by ICP.

11.7 *Al, Mn, Fe, Ni, Cu, Zn, and Cd*— Determine the concentration of these elements in the blank and soil test solution samples by atomic absorption spectrophotometry.

11.8 *P*—Add 0.5 mL of the ammonium molybdate reagent to 15.00 mL each of blank and soil test solution samples and each *P* standard, and mix. Add 0.3 mL of the stannous chloride reagent, mix, and read the absorbencies on a colorimeter at 660-mu using a cell with a 2-cm light path after 6 but before 11 min.

11.9 *Sulfate, S*—Add 1 mL of acid seed reagent to 10.00 mL of blank and sample soil test solutions and each *S* standard, mix, and add 0.5 g of $BaCl_2{\cdot}2H_2O$ crystals (20 to 60 mesh). Let stand for exactly 1 min, mix until the crystals are dissolved, and read the absorbencies on a colorimeter at 420 mu between 2 and 8 min using a cell with a 2-cm light path.

12. Interpretation of Results

12.1 Table 2 shows interpretation offered with respect to plant growth and food-chain protection.

12.2 The above interpretative data represent "small exchange" calibration values from the existing data for over 10 000 samples. Critical levels for the quantity values presented above, based on field response data, varies with crops and local field conditions. The low levels are not usually deficient and high levels are not usually toxic.

13. Effects of Storage

13.1 Soil samples may be stored in an air-dry condition for prolonged periods with no effect on test levels of the different elements. However, maintaining samples in a wet condition at room temperature renders the results for Mn, Fe, Cu, P and possibly other elements unreliable. Differences between moist and air-dried samples have been found, especially for Mn and Al.

TABLE 2 Interpretation of Results

Element[A]	Low	Normal	High
P 10^{-4} *M*	1	1 to 2.5	2.5 to 5
K10^{-4} *M*	1.5	1.5 to 4	4 to 8
Mg 10^{-4} *M*	5	5 to 16	16 to 20
Ca 10^{-4} *M*	40	40 to 60	60 to 120
Al mg/kg	5	5 to 12.5	12.5 to 20
Mn mg/kg	20	20 to 50	50 to 100
Fe mg/kg	10	10 to 50	50 to 75
Cu mg/kg	2	2 to 10	10 to 30
Zn mg/kg	2	2 to 40	40 to 70
Pb mg/kg	2	2 to 10	10 to 30
Cd mg/kg	0.1	0.1 to 0.85	0.85 to 1.6
Na mg/kg	50	50 to 100	100 to 150
S mg/kg	20	20 to 50	50 to 100
Ni mg/kg	0.5	0.5 to 5	5 to 15
Mo mg/kg	0.04	0.04 to 0.5	0.5 to 5

[A] P, K, Ca, and Mg are expressed as concentrations in the equilibrating solution and all others are amounts desorbed or solubilized from the soil (solution concentrations × 10).

14. Report: Test Data Sheet(s)/Form(s)

14.1 Record as a minimum the following general information:

14.1.1 Sample/specimen identifying information, such as Project No., Boring No., Sample No., Depth, etc.

14.1.2 Any special selection and preparation process, such as removal of gravel or other materials.

14.1.3 Technician name, and date.

14.1.4 Report the quantities (Q) and intensities (I) of each test element.

15. Precision and Bias

15.1 *Precision*—The within laboratory standard deviations and maximum allowable differences are as follows:

Element	Standard Deviation	Maximum Difference
K	0.088	0.23
Ca	1.691	4.40
Mg	0.661	1.72
Mn	2.184	5.68
Fe	0.176	0.458
Cu	0.016	0.042
Zn	0.042	0.109
Na	0.266	0.692
Al	0.096	0.250
Ni	0.021	0.055
Pb	0.039	0.101
Cd	0.042	0.109

15.2 *Bias*—There is no acceptable reference value for this test method; therefore, bias cannot be determined.

16. Keywords

16.1 diagnostic soil test; food chain; plant

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SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D5435 – 03 (2008)) that may impact the use of this standard. (Approved Nov. 1, 2013.)

(1) Revised Sections 1, 2, 7, 10, and 12.
(2) Added Sections 3 and 14.
(3) Modified Note 1 in accordance with D18 policy.
(4) Deleted Calculation Section.