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Standard Test Methods for Use of Hyperspectral Sensors for Soil Nutrient Analysis of Ground Based Samples[1]

This standard is issued under the fixed designation D8438/D8438M; 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 describes procedures for sampling and testing of soils obtained from ground-based samples using diffuse reflectance spectrometry using handheld portable spectrometers measuring spectra in visible and near infrared (vis-NR) and mid-infrared (MIR) range. The sensor can measure moisture content, PH, organic matter, Cation Exchange Capacity (CEC) as well as macro and micro elemental nutrients in parts per million (PPM) or percentage, including but not limited to nitrogen, phosphorous, potassium, zinc, iron, boron, sulfur, calcium, magnesium, and manganese.

1.2 There are two methods that can be used to perform the test.

1.2.1 *Method A*—The analysis is performed in the laboratory on the sample after the sample has been oven dried and sieved.

1.2.2 *Method B*—The analysis is performed in the field on a moist sample after homogenization. After post-processing of multiple reflectance site data using methods A and B, the moisture content can be measured, and the spectral signature is normalized for moisture content.

1.3 The limitation of this method is that the results of an individual test for elemental analysis would not be the same as exacting reference values from traditional wet chemical lab analysis used by soil scientists. Results of wet chemistry tests or tests from soil science libraries may be used to calibrate a specific site model comprised of many individual tests. Spectral data for organics has shown to be as accurate as conventional methods such as Test Methods D2974.

1.4 For soil nutrient analysis the sample is not finely ground as in typical qualitative spectral analysis as outlined in standard Practice E1252. The spectrometer is checked periodically during testing using procedures in accordance with Guide E1866 performance testing.

1.5 Moisture content is a preferred term in agricultural applications. For this standard, gravimetric water content may be measured in accordance with Test Methods D2216 when drying samples and used to calibrate the site model, but the overall results of spectral analysis are more qualitative, and the term Moisture Content is used in this standard.

1.6 *Units*—The values stated in either SI units or inch-pound units [given in brackets] are to be regarded separately as standard. Wavelengths are stated only in nanometers, nm. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.7 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026. The procedures used to specify how data is collected, recorded or calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.7.1 Spectral data is acquired by electrical data acquisition systems and therefore numeric data is carried through recording and into databases without rounding of numeric data.

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

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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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 D18.01 on Surface and Subsurface Investigation.

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids
D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass
D2974 Test Methods for Determining the Water (Moisture) Content, Ash Content, and Organic Material of Peat and Other Organic Soils
D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
D4643 Test Method for Determination of Water Content of Soil and Rock by Microwave Oven Heating
D4700 Guide for Soil Sampling from the Vadose Zone
D6026 Practice for Using Significant Digits and Data Records in Geotechnical Data
D6907 Practice for Sampling Soils and Contaminated Media with Hand-Operated Bucket Augers
E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
E1252 Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis
E1866 Guide for Establishing Spectrophotometer Performance Tests

3. Terminology

3.1 *Definitions*—Terminology in accordance with Terminology D653 and shall be used where applicable.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *macro-nutrient, n—in agronomy*, an element required in large amounts for plant growth and development (for example, nitrogen, phosphorous and potassium).

3.2.2 *micro-nutrient, n—in agronomy*, an element required in smaller amounts for plant growth and development (for example, sulfur, calcium, magnesium, manganese, zinc, copper, boron, iron, sodium).

3.2.3 *Power Spectral Density (PSD), n—in spectral analysis*, the energy variation that takes place within a vibrational signal, measured as frequency per unit of mass.

3.3 *Acronyms:*

3.3.1 *Vis-NIR*—near infrared light spectrum from 350 to 2500 nm.

3.3.2 *MIR*—mid infrared light spectrum from 2500 to 4000 nm.

4. Summary of Test Methods

4.1 The test methods involve obtaining a sample that is homogenized to eliminate the effect of stratification.

4.1.1 Method A the sample is air dried or dried in accordance with Test Methods D2216 or Test Method D4643 then homogenized through sieving prior to scanning.

4.1.2 Method B the samples are only homogenized via mixing and stirring.

4.2 The samples are then scanned with a hyperspectral sensor to collect the spectral signature in the soil sample.

4.3 A reflectance and adsorption record for each test is obtained for each sample and all data are combined into a site database for post processing modeling calibration.

5. Significance and Use

5.1 Spectral analysis of soils for agricultural use is being used worldwide to obtain rapid data on soil nutrients. for the purpose of agricultural management including fertilizer application and other amendments such as pH adjustment, organic supplements, etc. Satellite, aerial, and ground-based sampling methods are being used. This test method applies to ground-based, terrestrial field applications where samples are taken from the ground, generally in the root zone. Use of these rapid remote sensing techniques allow for more detailed and economic data acquisition than older cumbersome sampling and wet chemistry testing methods used in the past by soil scientists for soil nutrient evaluations.

5.2 This test method describes procedures for sampling and testing of field soils using diffuse reflectance spectrometry using handheld portable spectrometers measuring spectra in visible and near infrared (vis-NR) using dried sieved or wet samples. There is a worldwide effort to collect spectral databases of soils. The procedures specified here follow procedures as outlined in the United Nations Food and Agricultural Organization (FAO) primer on Vis-NIR and MIR spectroscopy of soils (**1**)[3]. Other organizations such as IEEE are actively working on additional guidance documents that will be incorporated in future revisions of this test method.

5.2.1 This standard describes the procedures (Section 12) for using hyperspectral sensor data to measure moisture content as a percentage, pH, Organic Matter (OM) as a percentage, Cation Exchange Capacity (CEC) measured in 10 cmol c /kg could hold 10 cmol of Na + cations (with 1 unit of charge per cation) per kilogram of soil, but only 5 cmol Ca 2+ (2 units of charge per cation), as well as micro and macro nutrients in soils measured in PPM (parts per million)or a percentage, including, but not limited to nitrogen, phosphorous, potassium, boron, zinc, iron, sulfur, calcium, magnesium, and manganese.

5.2.2 Research has shown that the Vis-NIR data for OM content is as accurate as other tests such as the burn off test in Test Methods D2974(**2**). Analysis of natural moisture samples using method B can provide faster testing and better estimates of OM are normalization for moisture (**3**). Wet sampling allows for many more samples to be rapidly scanned in the field and therefore more samples and more detailed coverage of the site.

5.3 This standard does not address sensors that measure in the mid infrared range, MIR, are more expensive and there is less spectral data available. MIR spectral analysis is performed on dried samples that are finely grinded (**4**). MIR modeling

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

requires a high level of calibration against recognized laboratory procedures and physical properties.

5.4 Spectral data can differ from older reference tests typically based on wet chemistry methods such as pore fluid extractions such as those outlined in soil survey manuals (**5**). These old methods require extensive labor costs and long turnaround times. However, soil scientists are accumulating large databases of spectral libraries which have been checked and calibrated with baseline chemical data. The soil survey manual (**5**) also has early (2014) procedures for Vis-NIR testing methods on dry specimens.

5.5 The accuracy of the measurement is determined by the accuracy of the calibration of the baseline measurements that are calibrated by chemical processing. On critical/new projects the sampling plan may include samples for wet chemistry testing to help calibrate the site model. The large amount of data that is collected at a site is combined into a site-specific database which is subject to complex model training to optimize the dataset. This standard will not provide detailed guidance on modeling and the FAO document (**1**) provides a good overview of the current procedures for dataset modeling. Dataset modeling requires adjustments for texture, water content, and geology and generally is linked to other appropriate spectral libraries available from many sources (**6**).

5.5.1 Horizon and Soil taxonomic order as auxiliary variables improve prediction accuracy of models. Regional, local, and past site-specific data, and taxonomic historic data base libraries may be used to help calibrate a site model.

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 and objective testing/sampling/inspection/etc. 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. Apparatus

6.1 *Hyperspectral Vis-NIR Sensor*—Any spectral sensor that meets the requirements of Table 1 can be used. There are a variety of desktop systems that can meet these specifications but a sensor with contact probe with a minimum field of view of 10 mm [0.4 in.] is recommended for field testing. The sensor houses and A/D converter, computer, lamp, and fiber optic cable (Note 2).

6.1.1 The sensor must have a minimum operating range of 350 nm to 2500 nm.

6.1.2 *Analysis Software*—The computer software shall allow acquired sequences to be archived and retrieved for evaluation and allow real time display of the IR spectra signature. The software should allow viewing of the reflectance signature for specified wavelengths (350-2500 nm). Additional processing operations on each raw image sequence (for example, averaging, subtraction, noise reduction derivatives) may be performed to improve detectability of subsurface elements and nutrients.

6.1.3 Lab benchtop devices should be equipped with mounting specimen holders, or pucks specifically designed for soil analysis. These are typically borosilicate petri dishes.

TABLE 1 Specifications for Vis-NIR Spectrometer

Wavelength Range	350-2500 nm
Resolution	3 nm @ 700 nm 6 nm @ 1400/2100 nm
Scanning Time	100 milliseconds
Signal-to-Noise Ratio	
Visible Near Inferred	9000:1 @ 700 nm
Short Wave Inferred 1	9000:1 @ 1400 nm
Short Wave Inferred 2	4000:1 @ 2100 nm
Photometric Noise	
Visible Near Infrared	4.8×10^{-5} AU or 48 µAU@ 700 nm
Short Wave Infrared 1	4.8×10^{-5} AU or 48 µAU@ 1400 nm
Short Wave Infrared	2 1.1×10^{-4} AU or 110 µAU@ 2100 nm
Visible Near Infrared Detector	(350-1000 nm) 512 element silicon array (1001-1800 nm) and (1801-2500 nm)
Short Wave Infrared 1 and 2 Detectors	Graded Index InGaAs Photodiode TE Cooled

NOTE 2—An ASD LabSpec Spectroradiometer is a commonly used device. For MIR analysis A Bruker FTIR is a commonly used device (**1**,**5**). Lower cost spectrometers with limited wavelength ranges are available but may not meet the specifications of Table 1 and may result in inaccurate estimation of soil properties. Each spectrometer comes with its own computer and data reduction programs and calibration models (**7**).

6.2 *White Reference Tile*—Compressed Polytetrafluoroethylene (PTFE) White reference tile to baseline the sample measurement. The tile should be enclosed in a protective container when not in use, and protective measures should be taken during cleaning to prevent personnel from direct exposure to Perflourinated compounds (PFAs).

6.2.1 The reference panel should be large enough to cover the entire field of view of the contact probe. No exact measurement can be stated since equipment may vary.

6.3 *Wavelength Reference Calibration Puck or Tape*—Manufacturer provided reference spectra check standard. 10mm Mylar calibration sheet is used.

6.4 *A Light Source,* which could be a 50 Watt integrated light source or a lamp with a single-ended quartz halogen filament capsule 4.25V, 4.5-Watt Halogen. Preferred light source is Tungsten Quartz Halogen b/1500rs.

6.5 *Nonporous Container and Nonporous Tool* to stir and homogenize sample. Stainless steel bowl and mixing tool such as a spoon, pestle, or 2 mm hand sieve are recommended. to reduce crop residue, debris, and gravel from being included in sample.

6.6 *Lab or Hand Sieve*—Specification E11 2mm (No. 10) sieve.

6.7 *Sample Bags or Containers to be Used for Method A*—Paper bags, plastic jars, zip-top bags.

6.8 *Sample Container for Use in Method B to Scan the Sample*—The tall walled container must hold the sample and minimize exposure to outside light when scanning. White HDPE plastic jars and other tall wall containers 50 to 150 mm [2 to 6 in.] diameter, this should be a nonporous container to reduce contaminants similar to a bucket, cup, HDPE jars can also be used. A muglight is a closed system which prevents external light from entering the field of view and reduces the loss of light from the halogen lamp. It is important that external light is controlled during the scanning process to produce replicable results.

6.9 *Soil Sampler Probe* to extract soil core with a minimum diameter of 2 cm [1 in.]. The most commonly used sampler is the hand operated soil step sample tube or hammered Veihmeyer agricultural samplers (Guide D4700). Hand bucket augers (Practice D6907) can be used. Bucket augers are slower to operate, less accurate in depth, but do mix and remold the soil during sampling.

6.10 *Drying Oven for Method A*—A thermostatically controlled drying oven, preferably of the forced-draft type, capable of maintaining a uniform temperature of 110°C ± 5°C throughout (see Test Methods D2216).

6.11 *Standard Soil Lab Grinder for Method A*—(110V/60Hz or 220V/50Hz) with a recommended 2mm (No. 10) perforated stainless steel plate.

6.12 *Wet/dry 600 Grit Sandpaper.*

6.13 *Portable pH and Moisture Sensors,* if required in the sampling plan.

7. Safety Hazards

7.1 PTFE when aerosolized can be hazardous, therefore the reference tile must be cleaned under running water with wet sandpaper to prevent aerosolized particles from entering the air.

8. Sampling and Test Specimens

8.1 *Sampling Plan*—All sampling and testing should be performed in accordance with a specific site sampling plan. To develop a site-specific sampling plan the following steps are taken.

8.1.1 Collect all known information about the site including but not limited to review of existing soil surveys, existing previous soil testing, aerial photography to delineate geologic features, soil pedology and taxonomy of the site.

8.1.2 Perform a site reconnaissance and look at site features such as accessibility and any limiting features.

8.1.3 Layout a sampling grid. Due to the economic and fast testing, finer grids can be used using spectral analysis. The grid may be supplemented with additional samples in areas of interest such as geologic features. Grids can range from 0.5 to 10 Hectares [1 acre to 20 acres] depending by region, size, and crop

8.1.3.1 Each regional government may have its own nutrient management requirements for minimum sample spacings, but these can easily be exceeded using spectral analysis. Local, regional, and country regulations may specify spacings and time interval requirements for sampling.

8.1.4 The sampling plan must outline specific sampling procedures to be performed. This will include.

8.1.4.1 Sampling and processing methods to obtain test specimens, including methods for sample mixing, reduction, drying, sieving, etc.

8.1.4.2 Preservation, transport, and processing samples for dry sampling (Method A).

8.1.4.3 Field testing of samples (Method B) including frequency of reference check testing.

8.1.4.4 Delineation of samples for any validation reference (wet chemistry, moisture, pH, etc.) testing based on quality control requirements.

8.1.4.5 Equipment reference check testing and cleaning requirements. Manufacturer's operations manuals should be followed. Wet scanning will require regular cleaning of the light receptor lenses.

8.1.4.6 Sample labeling, transport, and retention instructions.

8.2 Core samples are typically taken at depths intervals of 0-15 or 0-20 cm [0-6 to 0-8 in.] for most analytes. It is recommended that for nitrogen tests the depth should be greater, varying by region from 30 to 45 cm [12 to 18 in.] and even deeper based on crop type and root zones. The depth data, geolocation data, and date and time are recorded in accordance with Guide D4700 or Practice D6907 as required in the sampling plan. The core sample must be homogenized to eliminate the effects of the stratification of elements at different soil depths. For Method A this will be in accordance with sampling plan standard soil lab soil preparation methods, while for Method B the sample will be homogenized in a nonporous container using a nonporous solid tool (such as a clean, non-rusty spoon).

8.3 For Method A, a test specimen of only a 3 to 5 grams of soil is required for the actual bench test. The sample for the test depth in the field is a homogenized mixture of soil of 100 to 200 grams and bagged for laboratory processing. The sample can be air dried or oven dried (Test Methods D2216) depending on the sample plan requirements. Air drying is easy for sands silts and loamy soils, but heavy clay soils may require days to dry. Oven drying using Test Methods D2216 is generally done overnight and is safe to preserve organics and mineralogy. The sample is then sieved through a 2 mm sieve, and a split or grab sample of screened material is used for the bench testing.

8.4 In the case of Method B, the sample will be scanned in the field. About 100 to 200 grams at natural moisture after homogenization is required to enable the contact probe field of view to be only on the test specimen without a background material. The sampling plan may require the sample to be retained and bagged for dry lab testing or reference testing.

9. Preparation of Apparatus

9.1 If no power source is available at the test location, make sure that the batteries of the hyperspectral sensor and computer are fully charged upon arrival at the test location. The hyperspectral sensor should be turned on according to manufacturer's instructions and the sampling plan, typically for a minimum of 1 hour prior to starting scan analysis to allow internal system to warm up for accurate calibration.

9.2 Clean the white reference panel with wet/dry 600 grain sandpaper and water until water beads off surface.

9.3 Place probe on the white reference panel and obtain a "white reference" reading. This reading must be taken every