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16. Abstract The Texas Department of Transportation has been having problems with organic matter in soils that they stabilize for use as subgrade layers in road construction. The organic matter reduces the effectiveness of common soil additives (lime/cement) in stabilization projects. The researchers developed a technique using UV-Vis spectroscopy to measure the harmful organic matter in another project (0-5540). This project consisted of purchasing three UV-Vis instruments, equipping them with software to measure the organic matter and doing two trainings with the Texas Department of Transportation. Following the trainings, four laboratories analyzed 20 natural soil samples and three laboratory standards to determine repeatability and reproducibility between the laboratories. Researchers also continued testing real project soils to see what mitigation techniques researchers could use. Researchers determined that three replicates need to be run to achieve 95 percent confidence that the measured value is the true value. Researchers determined that soils with organic matter below 1.5 percent can be safely treated, and soils with an organic matter to Eades & Grim optimum lime (OM:EG) ratio less than 0.5 have the greatest potential for mitigation with additional lime application. Additionally, calcium chloride added to the soil with the lime improved the formation of pozzolanic reaction products and strengths of some soils. This work illustrates the complex nature of organic interactions with soil stabilizers and the many questions left unresolved.			
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**TEST PROCEDURE FOR DETERMINING ORGANIC MATTER
CONTENT IN SOILS – UV-VIS METHOD**

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and the
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Test Procedure for

DETERMINING ORGANIC CARBON CONTENT IN SOILS — UV-VIS METHOD



TxDOT Designation: Tex-???-E

Effective Date: DRAFT

1. SCOPE

- 1.1 This method uses the UV-Vis absorption properties of soil extracts to estimate total soil organic carbon (SOC) content. The method was tested for a wide variety of soils with SOC ranging between 0 and 5 percent.
 - 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.
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2. DEFINITIONS

- 2.1 *Cuvette*—A small tube of circular or square cross section, sealed at one end, made of plastic, glass, or fused quartz and designed to hold samples for spectroscopic analyses.
 - 2.2 *Filtrate*—Material (soil and water) that has passed through a filter.
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3. APPARATUS

- 3.1 *Computer and the following accessories:*
 - BP2 battery pack
 - SL1 tungsten halogen light source
 - Power regulator
 - AC power supply
 - Green wave spectrometer (UVNb-50), 200-1050 nm wavelength range
 - Fiber optic cable
 - Green USB cable
 - 16V adapter cable
 - 3.2 *Weighing paper.*
 - 3.3 *Auger sampler.*
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- 3.4 *Core sampler*, 2 in. diameter × 4 ft. long.
 - 3.5 *Balance/Scale*, minimum capacity of 1200 g, calibrated to weigh to nearest 0.01 g.
 - 3.6 *Sieves*, U.S. Standard No. 4 (4.75 mm) and No. 40 (425 μm).
 - 3.7 *Mortar and pestle*.
 - 3.8 *Crusher*.
 - 3.9 *Cuvettes*, 1 cm UV-Vis.
 - 3.10 *Centrifuge tubes* (Polypro), 50 ml.
 - 3.11 *Syringe* (BD), 10 ml.
 - 3.12 *Graduated cylinder TD (ex)* (glass or plastic), 10 ml.
 - 3.13 *filter holder with filter*, Holder, VWR Cat No. 28144-109; Filter, Fisher Cat. No. 09-719-2D (0.45 μm), 25 mm.
 - 3.14 *Kimwipes* or equivalent lint-free wipe (4.5 X 8.4 in).
 - 3.15 *Scoop*, 0.1 ml volume.
 - 3.16 *Wash bottle*, 16 oz. (500 ml), for 1 N hydrochloric acid and sodium pyrophosphate solutions.
 - 3.17 *1 L volumetric flask*, 500 ml volumetric flask.
 - 3.18 *Latex gloves*.
 - 3.19 *Sample splitter*.
 - 3.20 *Burrell wrist action shaker* (optional).
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4. MATERIALS

- 4.1 *Distilled or deionized water*.
- 4.2 *Na pyrophosphate decahydrate*, $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$
- 4.3 *Hydrochloric acid*, HCl.
- 4.4 *Sodium hydroxide*, NaOH.
- 4.5 *Standards of known SOC content*, At least two standards (one soil with SOC <1% and the other with SOC around 2%). Pat Harris at Texas Transportation Institute and Claudia Izzo at the Texas Department of Transportation have three soils available as standards.

PART I—FIELD ESTIMATION OF SOIL ORGANIC MATTER

5. SCOPE

- 5.1 The following procedure describes preparing soil samples for estimating organic carbon content in the field using a fixed-volume scoop. For best results, samples must be relatively dry to the touch so they can pass through a #40 sieve.

6. PROCEDURES

6.1 *Preparing reagents:*

- 6.1.1 Place 500 ml of deionized water into 1 L volumetric flask.
- 6.1.2 Add 10 g of NaOH and 44.6 g of Na pyrophosphate and stir until dissolved.
- 6.1.3 Add additional deionized water to make 1 L of solution; cap flask.
- 6.1.4 To prepare 1 N hydrochloric acid solution, add 250 ml of deionized water to 500 ml volumetric flask.
- 6.1.5 Add 41.43 ml of 37% HCl to the deionized water and stir.
- 6.1.6 Add additional deionized water to make 500 ml of solution; cap flask.

6.2 *Preparing sample:*

- 6.2.1 Sample soil within the depth of proposed stabilization with a hand auger at the sampling frequency required by the guide schedule. Collect soil samples where there is an obvious change in soil type (plasticity) or color.
- 6.2.2 Obtain a 300 g representative sample.
- 6.2.3 Pulverize the 300 g to pass the No. 40 (425 μ m) sieve.
- 6.2.4 Split the sample and obtain ~15 g of representative sample. Fill three 0.1 ml scoop samples from this split for more accuracy. This means split the material to obtain three samples of 0.1 ml each and run each sample through the UV-Vis test.
- 6.2.5 Fill the 0.1 ml scoop to the top. This should be done three times putting each scoop of sample in separate 50 ml (1.7 oz) polypropylene centrifuge tubes (to provide triplicate samples for analysis).
- 6.2.6 Place 0.1 ml of each standard material (from Section 4.5) in a separate 50 ml (1.7 oz) polypropylene centrifuge tube. At least two different standard materials should be prepared.

- 6.2.7 Label a 50 ml (1.7 oz) polypropylene centrifuge tube as a blank to be used later.
- 6.3 *Extracting organic matter:*
- 6.3.1 Add 5 ml of 1 N HCl to each of the three replicates, the two standard samples and the polypropylene centrifuge tube labeled as Blank (no soil in the Blank).
- 6.3.2 Vigorously shake the centrifuge tubes of soil/HCl solution by hand for 10 sec. at 1 min. intervals for a total of 5 min.
- 6.3.3 Add 20 ml of Na pyrophosphate solution to each of the three replicates, the two standard samples, and the polypropylene centrifuge tube labeled as Blank.
- 6.3.4 Vigorously shake the centrifuge tubes of soil/HCl and Na pyrophosphate solution by hand for 10 sec. at 1 min. intervals for a total of 5 min. (there should be 25 ml of solution in each centrifuge tube).
- 6.3.5 Add approximately 10 ml of the liquid to a 10 ml syringe and attach a 0.45 µm polycarbonate syringe filter.
- 6.3.6 Place the filter opening above a clean 1 cm methacrylate cuvette and gently depress the syringe plunger to force the extract through the filter and into the cuvette. **Important:** When filtering, gently depress the syringe plunger to dispose of ~1 ml of solution in a waste container. Use the rest of the solution in the syringe to fill the cuvette.
Note: Bubbles and particulates will result in measurement errors, so be careful to ensure that the extract in the cuvette is free of bubbles and particulates. Treat the Blank as the other samples, it should be filtered as well.
- 6.3.7 Wipe the outside of the cuvette clean with a Kimwipe® or equivalent delicate task wipe to remove dirt, fingerprints, or anything else that will obstruct a light beam from passing through the cuvette and filtrate. The sample is now ready to place in the UV-Vis instrument for determining the OM content of the soil.
Note: The cuvettes are disposable. Use a new cuvette with each sample but make sure that they are clean when using them; packing Styrofoam will adhere to the sides of the cuvette.
- 6.4 *Stellarnet UV-Vis spectrometer setup:*
- 6.4.1 Connect the BP2 battery to the power regulator by plugging the 16 volt cable in the left-hand female receptacle labeled **OUT** on the battery. Ensure that the switch on the battery pack is set to **16V**.
- 6.4.2 Next, connect the UV-Vis tungsten halogen light source to the power regulator using the 12 volt cable. Make sure you use the cable labeled 12 volts when you connect it to the light source.
- 6.4.3 Now connect the green wave spectrometer to the black cuvette holder attached to the front of the tungsten halogen light source via the fiber optic cable. Make sure that the fiber optic cable is connected properly; there is an arrow on the cable that points to the green wave spectrometer when the cable is properly connected.

- 6.4.4 Finally, connect the green wave spectrometer to the Hp Mini notebook computer with the green USB cable.
Note: Turn the computer on before connecting the USB cable to the computer.

6.5 *Measuring soil organic carbon:*

- 6.5.1 Double-click the **Spectrawiz Excel** icon on the desktop to open the macro for measuring organic matter. Click on the “Organic Carbon” spreadsheet. At this point you are ready to enter your sample labels or “Sample ID.”
Note: Sample IDs should always start in row 2 of column A.
- 6.5.2 The toolbar of the workbook should display two new control buttons “Step 1: Analysis Setup” and “Step 2: Sample Analysis.” Click on the “Step 1: Analysis Setup” control button, and it will guide the user through important steps in a checklist that should be performed before sample analysis. It is important that the user go over the checklist thoroughly. Click “Continue” when you finish the checklist. At this point the program will check the sample table.
Note: If there are no “Sample ID” values in row 2 of column A of the “Organic Carbon” spreadsheet, a message will be displayed for the user to “Please enter sample IDs.” If no messages are displayed after clicking “Continue,” you are ready to move on to sample analysis.
- 6.5.3 Click on the “Step 2: Sample Analysis” button. The program will again check to make sure the instrument and sample table are ready to go. If everything is in place, a dialog box will appear with instructions for collecting the dark spectrum. The shutter button is at the back of the light source and is released when it is fully extended. Release the red shutter button on the back of the light source (fully extended), then click the **OK** button in the open spreadsheet, which will collect a dark spectrum.
- 6.5.4 After the dark spectrum is collected, instructions for collecting the “reference” spectrum will be displayed in a dialog box. Insert the reagent blank in the cuvette holder and depress the shutter button on the back of the sample holder, then click the **OK** button in the open spreadsheet to collect a reference spectrum.
Note: Prior to placing any cuvette into the cuvette holder, be sure to clean the cuvette with a Kimwipe® or comparable lab wipe to remove any residue that may interfere with the beam.
- 6.5.5 After collecting the dark and reference spectra, samples are ready to be analyzed; follow the onscreen instructions displayed in the “Step 2: Sample Analysis” spreadsheet
- 6.5.6 After analyzing the last sample in the sample table, the user can choose to save the data. If “yes” is chosen, the data will be saved as a text file in the “SOCdata” folder on the desktop, using the specified filename. **DO NOT SAVE OVER THE PROGRAM.**

- The percent organic carbon will be listed on the spreadsheet when you are finished. The standard materials should have concentrations of: Austin $1.2 \pm 0.24\%$, Beaumont $1.5 \pm 0.30\%$, and SH6 $0.46 \pm 0.09\%$. These values are for the laboratory test, so the field test standard values may be less precise than the laboratory test.

7. TEST REPORT

- 7.1 Report average organic carbon concentration in percent to one decimal place. If the organic carbon is present in concentrations larger than one percent, then samples should be tested in the laboratory using Part II of this test method.

PART II—LABORATORY TEST

8. SCOPE

- 8.1 The following procedure describes preparing soil samples for estimating organic carbon content in the laboratory using an analytical balance.

9. PROCEDURES

9.1 *Preparing reagents:*

- 9.1.1 Place 500 ml of deionized water into 1 L volumetric flask.
- 9.1.2 Add 10 g of NaOH and 44.6 g of Na pyrophosphate and stir until dissolved.
- 9.1.3 Add additional deionized water to make 1 L of solution; cap flask.
- 9.1.4 To prepare 1 N hydrochloric acid solution, add 250 ml of deionized water to 500 ml volumetric flask.
- 9.1.5 Add 41.43 ml of 37% HCl to the deionized water and stir.
- 9.1.6 Add additional deionized water to make 500 ml of solution; cap flask.

9.2 *Preparing sample:*

- 9.2.1 Sample soil within the depth of proposed stabilization with a hand auger at the sampling frequency required by the guide schedule. Collect soil samples where there is an obvious change in soil type (plasticity) or color.
- 9.2.2 Obtain a 300 g representative sample.
- 9.2.3 Air-dry the sample to constant weight. Do **NOT** oven-dry samples.
- 9.2.4 Pulverize the 300 g to pass the No. 40 (425 μ m) sieve.
- 9.2.5 Split the sample and obtain ~15 g of representative sample. Weigh three 0.1 g samples from this split for more accuracy. This means split the material to obtain three samples of 0.1 g each and run each sample through the UV-Vis test.
- 9.2.6 Weigh the sample to 0.1 g \pm 0.01 g.

- 9.2.7 Weigh 0.1 g of each standard material (from Section 4.5) in a separate 50 ml (1.7 oz) polypropylene centrifuge tube. At least two different standard materials should be prepared.
- 9.2.8 Label a 50 ml (1.7 oz) polypropylene centrifuge tube as a blank to be used later.
- 9.3 *Extracting organic matter:*
- 9.3.1 Add 5 ml of 1 N HCl to each of the three replicates, the two standard samples, and the polypropylene centrifuge tube labeled as Blank (no soil in the Blank).
- 9.3.2 Vigorously shake the centrifuge tubes of soil/HCl solution by hand or place on a mechanical shaker for 10 sec. at 1 min. intervals for a total of 5 min.
- 9.3.3 Add 20 ml of Na pyrophosphate solution to each of the three replicates, the two standard samples, and the polypropylene centrifuge tube labeled as Blank.
- 9.3.4 Vigorously shake the centrifuge tubes of soil/HCl and Na pyrophosphate solution by hand or place on a mechanical shaker for 10 sec. at 1 min. intervals for a total of 5 min. (there should be 25 ml of solution in each centrifuge tube).
- 9.3.5 Add approximately 10 ml of the liquid to a 10 ml syringe and attach a 0.45 µm polycarbonate syringe filter.
- 9.3.6 Place the filter opening above a clean 1 cm methacrylate cuvette and gently depress the syringe plunger to force the extract through the filter and into the cuvette. **Important**—When filtering, gently depress the syringe plunger to dispose of ~1 ml of solution in a waste container. Use the rest of the solution in the syringe to fill the cuvette.
Note: Bubbles and particulates will result in measurement errors, so be careful to ensure that the extract in the cuvette is free of bubbles and particulates. Treat the Blank as the other samples, it should be filtered as well.
- 9.3.7 Wipe the outside of the cuvette clean with a Kimwipe® or equivalent delicate task wipe to remove dirt, fingerprints, or anything else that will obstruct a light beam from passing through the cuvette and filtrate. The sample is now ready to place in the UV-Vis instrument for determining the OM content of the soil.
Note: The cuvettes are disposable. Use a new cuvette with each sample but make sure that they are clean when using them; packing Styrofoam will adhere to the sides of the cuvette.
- 9.4 *Stellarnet UV-Vis spectrometer setup:*
- 9.4.1 Connect the BP1 battery to the power regulator by plugging the 16 volt cable in the left-hand female receptacle labeled **OUT** on the battery. Ensure that the switch on the battery pack is set to **16V**. You can connect the 110 V AC power supply to the BP1 battery pack for indoor laboratory use in the plug labeled **IN** on the battery.

- 9.4.2 Next, connect the UV-Vis tungsten halogen light source to the power regulator using the 12 volt cable. Make sure you use the cable labeled 12 volts when you connect it to the light source.
- 9.4.3 Now connect the green wave spectrometer to the black cuvette holder attached to the front of the tungsten halogen light source via the fiber optic cable. Make sure that the fiber optic cable is connected properly; there is an arrow on the cable that points to the green wave spectrometer when the cable is properly connected.
- 9.4.4 Finally, connect the green wave spectrometer to the Hp Mini notebook computer with the green USB cable.
Note: Turn the computer on before connecting the USB cable to the computer.
- 9.5 *Measuring soil organic carbon:*
- 9.5.1 Double-click the **Spectrawiz Excel** icon on the desktop to open the macro for measuring organic matter. Click on the “Organic Carbon” spreadsheet. At this point you are ready to enter your sample labels or “Sample ID.”
Note: Sample IDs should always start in row 2 of column A.
- 9.5.2 The toolbar of the workbook should display two new control buttons “Step 1: Analysis Setup” and “Step 2: Sample Analysis.” Click on the “Step 1: Analysis Setup” control button, and it will guide the user through important steps in a checklist that should be performed before sample analysis. It is important that the user go over the checklist thoroughly. Click “Continue” when you finish the checklist. At this point the program will check the sample table.
Note: If there are no “Sample ID” values in row 2 of column A of the “Organic Carbon” spreadsheet, a message will be displayed for the user to “Please enter sample IDs.” If no messages are displayed after clicking “Continue,” you are ready to move on to sample analysis.
- 9.5.3 Click on the “Step 2: Sample Analysis” button. The program will again check to make sure the instrument and sample table are ready to go. If everything is in place, a dialog box will appear with instructions for collecting the dark spectrum. The shutter button is at the back of the light source and is released when it is fully extended. Release the red shutter button on the back of the light source (fully extended), then click the **OK** button in the open spreadsheet, which will collect a dark spectrum.
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Note: Prior to placing any cuvette into the cuvette holder, be sure to clean the cuvette with a Kimwipe® or comparable lab wipe to remove any residue that may interfere with the beam.
- 9.5.5 After collecting the dark and reference spectra, samples are ready to be analyzed; follow the onscreen instructions displayed in the “Step 2: Sample Analysis” spreadsheet.

9.5.6 After analyzing the last sample in the sample table, the user can choose to save the data. If “yes” is chosen, the data will be saved as a text file in the “SOCdata” folder on the desktop, using the specified filename. **DO NOT SAVE OVER THE PROGRAM.**

- The percent organic matter will be listed on the spreadsheet when you are finished. The standard materials should have concentrations of: Austin $1.2 \pm 0.24\%$, Beaumont $1.5 \pm 0.30\%$, and SH6 $0.46 \pm 0.09\%$.

10. TEST REPORT

10.1 Subtract 0.4 percent from the result to produce an estimate of the true organic carbon percentage. Report average organic carbon concentration in percent to the nearest tenth. This value will probably decrease as the skill of the analyst improves.

11. DATA INTERPRETATION

11.1 If the measured organic carbon is less than 1 percent, then there is no problem using calcium-based additives to stabilize the soil. If the soil pH is greater than 8, then higher percentages of organic carbon (2–3 percent by wt.) may be safely treated with calcium-based additives. Additional testing will be required with soils above 2 percent organic carbon as measured with this test method. Additional testing is required to confirm this observation.

12. PRECISION AND BIAS

12.1 *Precision*—Make three replicate tests and average for a maximum error of 0.20 percent at the 95 percent confidence level.

12.2 *Bias*—The UV-Vis method using the measurement spoon on average is unbiased but does not track 1:1 with the known organic matter content reference values. The UV-Vis method with the spoon overestimates organic matter content at low true values, and underestimates at higher true values. The UV-Vis method using the analytical balance is biased by 0.4 percent. Reduce the result by 0.4 percent to produce an estimate of the true organic matter percentage. These numbers will change as the skill of the analyst improves with repetition.

