California Department of Pesticide Regulation Environmental Monitoring Branch 1001 I Street, P.O. Box 4015 Sacramento CA 95812-4015 SOP Number: METH017.00 Previous SOP: None

Page 1 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

KEY WORDS

Soil water characteristic curves, pore-size distribution, pressure plate extractor, soil water content, matric potential, disturbed soil samples

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Segawa (2003) defines and describes the Environmental Monitoring Branch organization and personnel, such as management, senior scientist, quality assurance officer, and project leader.

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SOP Number: METH017.00

Previous SOP: None

Page 2 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

1.0 INTRODUCTION

1.1 Purpose

Soil water characteristic curve, also referred to as a retention curve, shows the relationship between soil water content and soil matric potential. It is a fundamental part of the characterization of soil hydraulic properties. It depends on soil texture and soil structure (Klute, et al., 1986). The soil water characteristic is a basic soil property that is required for the study of plant-available water, infiltration, drainage, hydraulic conductivity, irrigation, water stress on plants, and solute movement in subsurface soil (Kern, 1995). One method to determine soil water characteristic curves in the laboratory is by using a pressure plate extractor with various soil suctions applied and by measuring the changes in soil water content. This standard operating procedure (SOP) outlines how to determine the 15 bar equivalent moisture content of the soil water characteristic curve. The user will also need to refer to the SOP by Fong and Tuli (2018) to determine the bulk density needed to calculate the equilibrium moisture content at 15 bar on a volume basis. The equilibrium moisture content conditions at 15 bar pressure in agricultural fields are uncommon, but knowing the water content at 15 bar (which is called the permanent wilting point) provides useful information about the range of available water capacity. The available water capacity represents the amount of water that the soil can hold and use for plant root uptake during plant growth (Hanks, 1992).

1.2 Definitions

Deionized (DI) water is water that has had the ions removed. Deionization does not significantly remove uncharged organic molecules, viruses, or bacteria except by incidental trapping.

Permanent wilting point is the amount of water at which plant roots cannot extract water and will lead to permanent wilting.

Undisturbed soil sample is a soil sample that has not undergone any physical processing since collection and is preserved in its natural structure.

Disturbed soil sample is a soil sample that has been crushed and sieved through a 2 mm opening sieve.

2.0 APPLICATION

The 15 Bar Ceramic Pressure Plate Extractor provides a sturdy, positive sealing pressure vessel (SoilMoisture Equiment Corp., 2015). The soil water content at the permanent wilting point will vary with soil texture and structure. This SOP is an experimental

SOP Number: METH017.00 Previous SOP: None

Page 3 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

procedure that continues the SOP method of Fong and Tuli (2018) to create a complete soil water characteristic curve from saturation to permanent wilting point.

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- 3.1 15 Bar Ceramic Pressure Plate Extractor
- 3.2 15 bar ceramic plates with neoprene diaphragm
- **3.3** Rubber soil sample retaining rings
- **3.4** Outflow tubes
- **3.5** (1) 500 mL beaker
- **3.6** (3) 50 mL flasks
- **3.7** Parafilm
- 3.8 Masking tape
- **3.9** Analytical balance (accurate to 0.01 g)
- **3.10** Regulator, gauges
- **3.11** Compressed nitrogen gas (N₂)
- **3.12** Saturated disturbed soil sample(s)
- **3.13** Wash bottles filled with DI water
- **3.14** Lab spatula
- **3.15** 25 cc Scooper
- **3.16** Plastic storage bin with lid
- **3.17** Aluminum cans (250 mL)
- **3.18** 6x9 inch Sealable nylon bags
- **3.19** Kimwipes
- **3.20** Drying oven
- **3.21** Oven mitts
- **3.22** Dessicator
- 3.23 (1) White sheet of paper
- **3.24** Pen
- **3.25** Permanent marker (black)
- 3.26 Scissors

4.0 PROCEDURES

4.1 Soil Preparation

4.1.1 The undisturbed soil sample rings used in Fong and Tuli (2018) will be processed and used as disturbed soil samples. Thus, prepare sealable nylon bags labeled with the study number, location, sample number, and collection date (Figure 1).

Page 4 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

- 4.1.2 Crush the soil ring sample and sieve it through a 2 mm opening sieve according to the SOP method by Lyon (2009).
- 4.1.3 Transfer the crushed and sieved soil into the nylon bag labeled with the corresponding sample number.



Figure 1. Crushed soil in labeled nylon bags.

4.2 Preparing the ceramic plates

- 4.2.1 Place clean and dry 15 bar ceramic plates into the empty plastic container. The amount of 15 bar ceramic plates used in the experiment will depend on the number of soil samples available. The maximum number of soil samples placed on a plate is around 12 and a total of three 15 bar ceramic plates can be placed into the Ceramic Pressure Plate Extractor. The 15 bar ceramic plate is sealed on one side by a thin neoprene diaphragm. An internal screen keeps the diaphragm from being in close contact with the ceramic plate and provides a passage for the flow of water (SoilMoisture Equipment Corp., 2015)
- 4.2.2 Add enough DI water to the plastic storage bin to completely submerge the three ceramic plates; then cover the bin with its lid. Keep the ceramic plates submerged for 24 hours to allow them to reach complete saturation (Figure 2).

Page 5 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

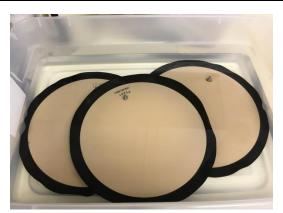


Figure 2. Three ceramic plates submerged in DI water for 24 hours.

4.3 Setting up the ceramic plates

4.3.1 Remove the wing nuts and pull back the extractor lid to open the extractor (Figure 3).

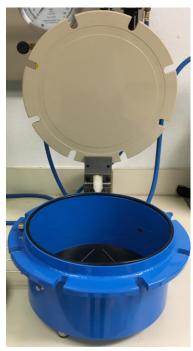


Figure 3. Open pressure extractor vessel.

4.3.2 Place the first saturated ceramic plate on the lowest triangular support to prevent the lower ceramic plate from breaking under differential pressure between the top and bottom of the ceramic plate (Figure 4).

Page 6 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

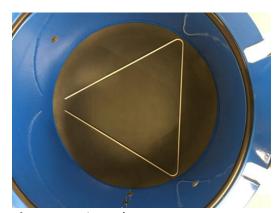


Figure 4. Triangular support.

4.3.3 Connect the outflow tube on the ceramic plate to the outflow port on the side of the extractor (Figure 5). If necessary, rotate the plate so that the outflow tube is horizontally stretched as to not interfere with the placement of the next ceramic plate above.

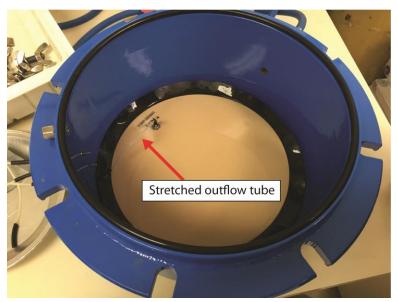


Figure 5. Outflow tube, stretched.

4.3.4 Place objects, such as rubber or pvc tubes and wood blocks, between the extractor wall and neoprene diaphragm to position the neoprene diaphragm upwards to hold water on the ceramic plate surface (Figure 6A and B).

Page 7 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

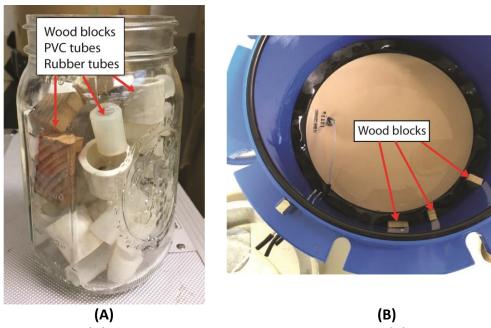


Figure 6. (A) Wood blocks, pvc tubes, and rubber tubes **(B)** Ceramic plate after DI water application with wood blocks.

- 4.3.5 Apply additional DI water onto the ceramic plate. The standing water level should be 0.5 mm in height (Figure 6).
- 4.3.6 Place three plastic spacers (as shown below) in the middle of the plate to separate the ceramic plates (Figure 7).

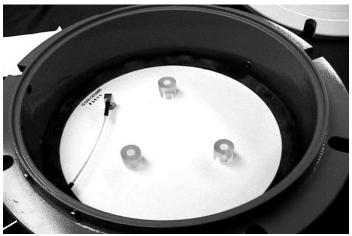


Figure 7. Plastic spacers on the ceramic plate.

Page 8 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

4.4 Placing the soil samples in the extractor

4.4.1 Place the rubber soil sample retainer rings (called rubber soil rings in this SOP) on the ceramic plate. The rubber soil rings can hold around a 25 g soil sample and are 1 cm high by 5.5 cm in diameter. The bottom side of the rubber soil ring is thicker than the top of the ring (Figure 8). The first rubber soil ring should be placed next to the outflow tube. The other rubber soil rings are placed close to each other, lined up in a ring with a distance of about half a centimeter from the neoprene diaphragm. Each plate can accommodate a maximum of 13 rubber soil rings.



Figure 8. The rubber soil ring.

4.4.2 Measure out enough soil to reach the 25 cc marked line of a scooper; pour into the rubber soil ring. The amount of soil should weigh approximately 22 g (Figure 9).



Figure 9. Soil (22 g) is poured into the soil ring.

Page 9 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

- 4.4.3 Using the bottom of the scooper, level the soil surface in the rubber soil ring (Figure 10).
- 4.4.4 Cut out a 1- by 2-cm white paper strip. Label the sample number on the top of the paper strip with a pencil.
- 4.4.5 Embed the paper strip labeled with corresponding sample number into the leveled soil sample in the rubber ring (Figure 10).

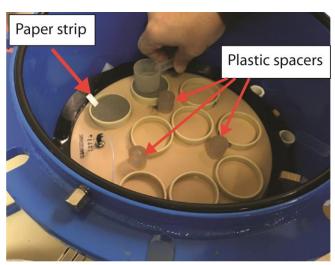


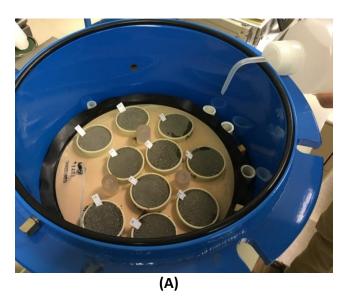
Figure 10. Leveling the soil surface with the scooper.

- 4.4.6 Clean soil from the scooper with a Kimwipe.
- 4.4.7 Repeat steps 4.4.1 to 4.4.6 for the remainder of the soil samples.
- 4.4.8 Slowly apply DI water onto the ceramic plate between the ring samples to saturate the samples from the bottom and to ensure hydraulic continuity between the ceramic plate and the rubber ring samples (Figure 11A). Add DI water until the standing water level on the ceramic plate is 1-2 mm in height. Avoid directly spraying water onto the soil samples. Darkened soil indicates complete soil saturation (Figure 11B).

Page 10 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor



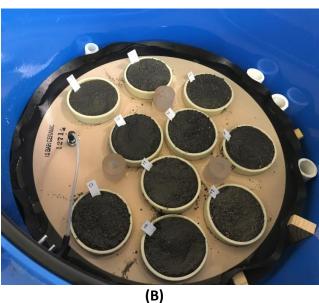


Figure 11. (A) Additional application of DI water until **(B)** the soil is completely saturated.

4.4.9 Place the second ceramic plate onto the spacers (Figure 12). Repeat the step 4.3.4 for holding the water on the ceramic plate surface.

Page 11 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

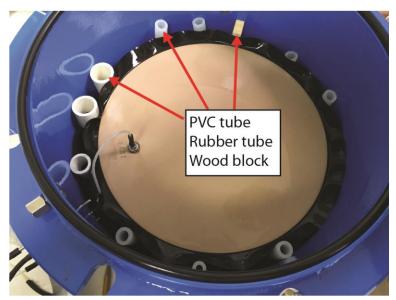


Figure 12. Last ceramic plate at the top with rubber and PVC tubes and wood block

4.4.10 Repeat steps 4.3.6-4.4.9 for the second and third ceramic plates if required. The last ceramic plate at the top does not need the plastic spacers, so step 4.3.6 can be skipped (Figure 12).

4.5 Closing the extractor

- 4.5.1 Close the extractor lid.
- 4.5.2 Attach one wingnut at a time.
- 4.5.3 Ensure the washer of the wingnut is on top of the extractor lid (Figure 13A).
- 4.5.4 The rectangular head of the wing nut should properly fit into the constraining groove on the bottom side of the lower clamping ring (Figure 13B).
- 4.5.5 Push upward on the bottom of the wingnut to set it in place when tightening.
- 4.5.6 After the wingnuts are in place, simultaneously tighten two opposing wingnuts in sequence until all six wingnuts are secure. Double check that all the wingnuts are securely tightened.

Page 12 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

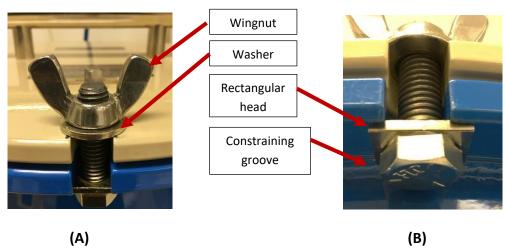


Figure 13. Alignment of wingnut to secure the lid to the pressure vessel.

4.6 Setting up the nitrogen gas as a pressure source

4.6.1 Obtain two full compressed nitrogen tanks (300 ft³ gas volume or 49 L in water capacity). One is connected to the pressure extractor and the other will be a spare (Figure 14). The nitrogen tank can be used until the pressure gauge drops to 300 psi, at which point the nitrogen tank should be replaced.

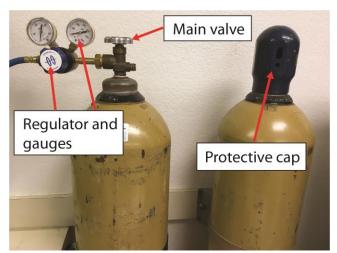


Figure 14. Nitrogen tank in use (left) and spare nitrogen tank (right).

4.6.2 For the release valve, a vertical (0°) lever position indicates open and a horizontal (90) position indicates closed (Figure 15). For all other valves and regulators, turn

Page 13 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

counterclockwise to open and/or release pressure and turn clockwise to close and/or load pressure. The pressure gauge should show "0" pressure. If the pressure gauge shows some pressure, release the pressure with Regulator I by turning it counterclockwise (Figure 15). Ensure that the exhaust valve is closed.

- 4.6.3 Where extreme accuracy is needed, use "double regulation" to eliminate variation in applied source pressure. This means putting and using Regulators I and II in series. Regulator I is set to a higher pressure than Regulator II in order to supply reasonable constant pressure to Regulator II. Pressure from Regulator II will be constant with source pressure variations reduced at least 1/100.
- 4.6.4 For routine determinations of the 15 bar equivalent moisture content, a set up using only Regulator I will be adequate. Ensure that while the lower valve is closed, the upper valve is open (Figure 15).

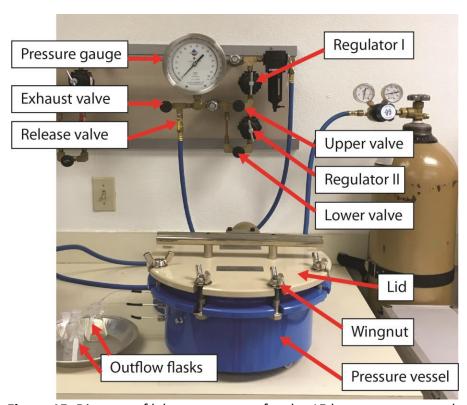


Figure 15. Diagram of laboratory setup for the 15 bar pressure vessel using a regulated air system.

Page 14 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

- 4.6.5 Check that Regulator I is initially fully open (Figure 15). Close the release valve to avoid pressure load to the pressure vessel.
- 4.6.6 Open the main valve on the nitrogen gas tank connected to the pressure extractor (Figure 14).
- 4.6.7 Set the pressure to 15 bar with Regulator I by observing the pressure gauge (Figure 15).
- 4.6.8 Place each outflow tube into a 500 mL beaker to capture the excess water (Figure 16).



Figure 16. 500 mL beaker to capture excess water.

- 4.6.9 Slowly open the release valve to pressurize the pressure plate extractor. There will be initial airflow from the outflow tubes as pressure builds inside the extractor. This is normal since the diaphragm and screen collapsing under the pressure in the extractor causes reduction in the gap between the ceramic plate and the neoprene diaphragm. The airflow will last for several minutes before a steady flow of water begins.
- 4.6.10 Once the excess water flow decreases, transfer the tubes into the 50 mL flasks, which are taped to record the water levels. Cover the openings of the flasks with Parafilm (Figure 17).

SOP Number: METH017.00 Previous SOP: None

Page 15 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

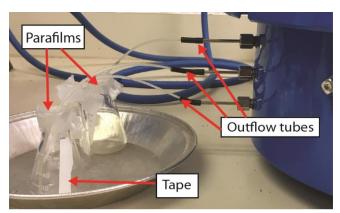


Figure 17. The 50 mL beakers to capture and measure water.

4.6.11 Mark the water heights twice per day, once at the beginning and then at the end of the day (Figure 17). Once the water flow into the tubes ceases for approximately 24 hours, the soil samples inside the pressure vessel have reached the equilibrium water content at the applied pressure. This process should take about 10 days.

4.7 Changing the nitrogen tank during the experiment

As mentioned in section 4.6.1, the nitrogen tank can be used until the pressure gauge drops to a 300 psi pressure level. During the measurements, if the pressure level drops below 300 psi, the tank should be replaced with a spare full tank. To replace the tank, take the following steps:

4.7.1 To remove the empty tank:

- 4.7.1.1 First, close the release valve by turning it to a horizontal (90) position (Figure 15). This maintains the 15 bar pressure level in the pressure vessel.
- 4.7.1.2 Close the main valve on the nitrogen gas tank (Figure 14). Please do not touch Regulator I at the system (Figure 15).
- 4.7.1.3 Remove the regulator connected to the empty nitrogen gas tank using an adjustable wrench (26 mm).
- 4.7.1.4 Place the cap to the top of tank to protect its valve from damage (Figure 14).

4.7.2 To install the full tank:

Page 16 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

- 4.7.2.1 Remove the cap of the spare nitrogen gas tank and connect the regulator to the tank using the adjustable wrench (26 mm).
- 4.7.2.2 After ensuring all connections are secured, open the main valve on the tank and watch for the pressure gauge to maintain a 15 bar pressure. If the pressure level changes, adjust the pressure level with Regulator I accordingly. After ensuring a 15 bar pressure on the gauge, open the release valve on the system (Figure 15). A full tank generally starts at between 2300 and 2400 psi.

4.8 Releasing the pressure from the pressure vessel to remove the soil samples

When the soil samples inside the pressure vessel have reached the equilibrium water content at the applied pressure as mentioned in section 4.6.11, it is time to remove the soil samples from the extractor:

- 4.8.1 Turn Regulator I counterclockwise to exhaust the air pressure in the extractor.
- 4.8.2 Wait until the sound of escaping gas has stopped and the pressure gauge has reached "0".
- 4.8.3 Loosen and remove all the wingnuts from the extractor lid. Open the lid.
- 4.8.4 Unplug the outflow tubes from the outflow ports.

4.9 Weighing the soil samples after pressure extraction

4.9.1 Record the soil sample numbers on the outer wall of the aluminum can at two places with a dark colored permanent marker (Figure 18).



Figure 18. Aluminum can to contain the soil sample.

Page 17 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

4.9.2 With a metal spatula, carefully transfer each soil sample into its corresponding can (Figure 19 A and B). Place the soil ring to the side.

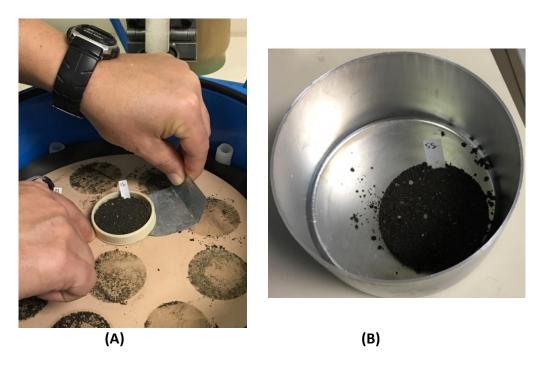


Figure 19. (A) Removing soil sample. **(B)** Completed transfer of soil sample without the soil ring into the aluminum can.

- 4.9.3 Record the final weight of the samples (aluminum can and soil sample) in Table 1.
- 4.9.4 With both hands, lift up the top ceramic plate. Place the ceramic plate onto the table.
- 4.9.5 Repeat steps 4.9.1 to 4.9.4 for all the soil samples.
- 4.9.6 Transfer the soil samples in the cans to a drying oven (Figure 20A).

SOP Number: METH017.00 Previous SOP: None

Page 18 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor





Figure 20. (A) Samples in the drying oven. (B) Drying oven turned on.

- 4.9.7 Place the soil samples into a drying oven in the laboratory (Figure 20A). To turn on the oven, press and hold the power button. The temperature of the oven should be set to 105 °C as indicated in the right hand corner of the display (Figure 20B).
- 4.9.8 Allow the soil samples in the aluminum cans to dry in the oven for 24 hours.
- 4.9.9 After 24 hours, turn off the oven by pressing and holding the power button.
- 4.9.10 Using oven mitts, transfer the ring samples in the aluminum cans from the oven into a desiccator with an active desiccant for cooling. The desiccator can hold 11 samples at a time (Figure 21). To open the desiccator, slide the lid off. The samples can be stacked into three levels. The first level has four samples, the second level has four samples, and the third level has three samples. Slide the lid(s) on to close the desiccator(s).
- 4.9.11 The rest of the soil samples waiting to be placed in the desiccator should be kept in the hot oven until their turn. This prevents the soil samples from absorbing additional moisture from the air.

Page 19 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor



Figure 21. Samples stacked in the desiccator.

- 4.9.12 Allow the hot soil samples in the desiccator to cool for 40 min.
- 4.9.13 When the samples have cooled, slide the desiccator's lid off and record the weight of the cooled soil samples in the aluminum cans in Table 1.
- 4.9.14 Transfer the soil in the aluminum cans into the corresponding nylon bags.
- 4.9.15 Wash the aluminum cans with soap and water.
- 4.9.16 Completely dry the aluminum cans with paper towels or by placing them in the oven.
- 4.9.17 Record the weight of the dry aluminum cans in Table 1.

5.0 Calculating soil moisture content

5.1.1 Calculate the gravimetric water content (GWC, g/g)

$$GWC = \frac{(Weight\ of\ wet\ soil\ (g) - Weight\ of\ oven\ dry\ soil\ (g))}{Weight\ of\ oven\ dry\ soil\ (g)}$$

Page 20 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

- 5.1.2 The oven-dry bulk density, $\rho_{\rm b}$ (g cm⁻³), in the equation below should have been determined using the SOP method of Fong and Tuli (2018).
- 5.1.3 Calculate soil moisture content of the samples on a volume basis (volumetric water content, VWC) (cm³ cm⁻³) at 15 bar applied pressure using the formula shown below:

$$VWC = \frac{GWC (g/g)}{\rho_{w} (g/cm^{3})} \times \rho_{b} (g/cm^{3})$$

where $\rho_{\rm w}$ is the density of water at laboratory temperature (~ 1 g cm⁻³)

6.0 TROUBLESHOOTING

6.1 For any problems with the pressure plate extractor, please refer to its operating instructions at

http://www.ictinternational.com/content/uploads/2016/01/Resource Instructions 0898-1500 1500-15-Bar-Pressure-Plate-Extractor.pdf.

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SOP Number: METH017.00 Previous SOP: None

Page 21 of 22

STANDARD OPERATING PROCEDURE

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SOP Number: METH017.00 Previous SOP: None

Page 22 of 22

STANDARD OPERATING PROCEDURE

Determination of Soil Water Characteristic Curve Using 15 Bar Ceramic Plate Extractor

Table 1: Datasheet for soil weights

Ring	Wet Soil +	OD Soil +	Can	Wet soil	GWC	BD	VWC
Sample #	Can (g)	Can (g)	(g)	(g)	(g/g)	(g/cm ³)	(cm ³ /cm ³

OD= \underline{O} ven \underline{D} ried; Can = aluminum \underline{Can} ; GWC= \underline{G} ravimetric \underline{W} ater \underline{C} ontent; VWC= \underline{V} olumetric \underline{W} ater \underline{C} ontent

Volume = $L*A= 5.10 \text{ cm} * 19.63 \text{ cm}^2 = 100.08 \text{ cm}^3$

BD= \underline{B} ulk \underline{D} ensity is generally in the range of 0.90 - 1.70 g/cm³