1.0 SCOPE

1.1 This method is utilized for determining extractable P in soils with pH > 7.2.

2.0 DEFINITIONS

2.1 Laboratory Control Sample: The laboratory control sample is an intralaboratory developed sample whose true Olsen P value is approximated by the average of repeated measures.

2.2 Duplicate Samples: A duplicate test involves splitting a sample to sub-samples and processing each through the same sample preparation procedure in order to determine the precision of the method.

2.3 Preparation Blank: The Preparation Blank is a sample that contains only the reagents used in the extraction procedure. The preparation blank undergoes the same procedure as other samples and therefore gives an indication of any contamination picked up during the sample preparation process.

2.4 ICP-OES: Inductively Coupled Plasma-Optical Emission Spectrometry.

3.0 EQUIPMENT AND SUPPLIES

3.1 Bottletop dispenser, 20 mL capability

3.2 pH Meter

3.3 Laboratory Balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.01 grams)

3.4 50 mL centrifuge tubes

3.5 Stir plate and stir bar.

3.6 ≥18 MΩ deionized water (DI).

3.7 Horizontal reciprocating shaker.

3.8 15ml Falcon tubes.

3.9 Centrifuge with rotor that can spin fourteen 50 mL tubes at 10,000 g.

3.10 NaHCO₃ (f.w.: 84.01 g)
3.11 5M NaOH
3.12 2L volumetric flask

4.0 PROCEDURE

4.1 Calibrate pH meter using pH 7 & 10 buffer solutions. Record calibration % slope in appendix.

4.2 Calibrate bottletop dispenser at 20.0 ± 0.1 mL volume using DI H2O. Record dispenser accuracy in appendix.

4.3 Prepare extraction solution (0.5 M NaHCO3) by dissolving 84.01 g NaHCO3 in approximately 1.5 L DI H2O within 2 L volumetric flask. Once NaHCO3 is dissolved, add DI H2O until solution level reaches neck of flask. While stirring on stir plate, adjust pH of solution to 8.5 using 5 M NaOH. Bring solution to volume with DI H2O; invert to mix. Check solution pH once more to confirm that it is 8.5. Prepare fresh extraction solution daily. Transfer extraction solution to glass bottle fitted with bottletop dispenser.

4.4 Weigh 1.00 g of air-dry, < 2 mm soil into 50 mL centrifuge tubes. Work with batches of fourteen 50 mL centrifuge tubes. Each batch will consist of 11 samples, 1 duplicate, 1 blank, and 1 check soil.

4.5 Add 20 mL of extraction solution to sample.

4.6 Shake on horizontal reciprocating shaker for 30 min.

4.7 Centrifuge at 10,000 g for 15 min.

4.8 Using autodiluter, dilute x 5 with DI H2O into 15 mL falcon tubes. Add 1 drop concentrated HCl for sample preservation and store samples in refrigerator.

4.9 Determine P concentration in sample using ICP-OES.

5.0 QUALITY CONTROL

5.1 Laboratory Control Sample (LCS): The laboratory control sample must fall within ± 20% of the known value.

5.2 Sample Duplicates: The relative percent difference (RPD) must be no more than ±20%.

\[
RPD = 100 \times \left( \frac{S - D}{S} \right)
\]
5.3 Preparation Blank: If any analyte concentration is above the detection limit in the preparation blank, the lowest concentration of the analyte reported in associated samples must be ≥ 10 times the preparation blank concentration.

6.0 REFERENCES

7.0 APPENDIX

Extraction Solution pH day of extraction

Batches completed

Initials/Date

Bottletop Dispenser Calibration

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pH Calibration

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%Slope