### **TEST METHODS MANUAL**

Laboratory Services Division Bureau of Soils and Water Management

## TM-LSD-04-06

Department of Agriculture		
SECTION : SOIL CHEMISTRY	Issue No.: 3	Effective date: April 15, 2024
SUBJECT : AVAILABLE PHOSPHORUS (BRAY NO. 1 METHOD)	Revision No.: 1	Page 1 of 7

### SCOPE

The Bray Method is best suited to acid soils ( $pH \le 5.5$ ) with a moderate CEC and base saturation, and organic soils. The Bray No. 1 Method removes "adsorbed" phosphorus (Al-P, Fe-P, and Mn-P). In soils with pH above 7.2, the Bray No. 1 may significantly underestimate the amount of available P.

Slightly acid and neutral soils (pH 6.0 to 7.0) may contain both Ca- and Al- phosphates. The NaHCO<sub>3</sub> extractant (in Olsen Method) can remove Ca- phosphates and phosphate adsorbed on soil surfaces of calcium and magnesium carbonates along with Al-phosphates and is considered the most suitable P test for these soils.

The Bray extractants should not be used on alkaline soils because (1) the acid tends to be neutralized and/or (2) excessive calcium phosphates may be extracted, giving a false high test for available P. Olsen Method provides a method of determining soil P in those alkaline soils where the Bray No. 1 Method is unsatisfactory.

### PRINCIPLE

Phosphorus is known as the master key to agriculture because lack of available P in the soils limits the growth of both cultivated and uncultivated plants. It has also been established that phosphorus is relatively unavailable for plant uptake in highly weathered soils. It is firmly fixed in soil and its proportion is relatively stable and dependent on soil reaction values. Adequate phosphorus results in higher grain production, improved crop quality, greater stalk strength, increased root growth, and earlier crop maturity.

Acidic ammonium fluoride extractant in acid or neutral soils is said to remove both adsorbed and acid-soluble phosphorus from the "insoluble" iron and aluminum phosphates and also dissolves acid-soluble calcium phosphate. Using the same solution in calcareous soils will reduce or destroy the acidity of the extractant depending on the amount of carbonate present and the ratio of the soil solution. Phosphate in the extract is determined colorimetrically with molybdenum blue method using ascorbic acid as reducing agent.

$3 \text{ NH}_{4}\text{F} + 3 \text{ HF} + \text{AIPO}_{4} \rightarrow \text{H}_{3}\text{PO}_{4} + (\text{NH}_{4})_{3}\text{AIF}_{6}$	Aluminum phosphate reaction
$3 \text{ NH}_{4}\text{F} + 3 \text{ HF} + \text{FePO}_{4} \rightarrow \text{H}_{3}\text{PO}_{4} + (\text{NH}_{4})_{3}\text{FeF}_{6}$	Iron phosphate reaction

 $\begin{array}{c} H_{3}PO_{4}+12 \ H_{2}MoO_{4} \rightarrow H_{3}P(Mo_{3}O_{10})_{4}+12 \ H_{2}O \\ \text{yellow} \\ \end{array} \text{(acidic medium)}$ 

### **TEST PRECAUTIONS**

Fluoride, arsenic or ferric ions may interfere with color formation. The addition of boric acid (*Bray and Kurtz, 1945*) to form non-interfering fluoroborate, and reduces as  $As^{+5}$  to  $As^{+3}$  and  $Fe^{+3}$  to  $Fe^{+2}$  by adding sodium bisulfite (*Jackson, 1958*), may be required to prevent such interference.

Slightly colored extracts (similar to weak tea) can be used without difficulties. However, dark colored extracts need to be decolorized by shaking 20 mL extract with 0.02 g activated carbon for 30 minutes. Then filter through Whatman no. 2 filter paper. Since such a treatment may introduce an uncertainty about the result (possible adsorption or desorption of phosphate by the activated carbon), it should be reported with the results.

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Use micropipette when delivering volumes as small as a microliter. This is to provide more accurate means for measuring volumes. Always check the appropriate conditions. The amount of P extracted will vary with temperature and shaking speed.

### EOUIPMENT

- a) UV-VIS spectrophotometer capable of measuring absorbance at 882 nm.
- b) Analytical Balance, precision of at least 0.001g
- **Reciprocating shaker** c)
- d) Vortex Mixer

### LABORATORY WARE

- a) Erlenmeyer flasks, 125 mL
- b) Funnel
- c) Beakers; 5L, 1L
- d) Polyethylene bottles with lid, wide-mouth type, 50 mL capacity
- e) Volumetric flasks; 1L, 250 mL, 100 mL
- f) Graduated cylinder; 100 mL, 50 mL
- g) Test tubes, 20 mL capacity
- h) Micropipette; 1-10mL, 10-1000μL
- Volumetric pipette; 50 mL, 20 mL, 10 mL i)
- CHEMICALS AND REAGENTS
- a) Deionized water
- b) Ammonium fluoride solution, 1 M. Dissolve 3.7 g NH<sub>4</sub>F in deionized water and make to 100 mL (store in polythene bottle).
- c) Hydrochloric acid, 0.5 M. Dilute 8.3 mL 6 M HCl (or 4.3 mL concentrated HCl, 37%) to 100 mL with deionized water.
- d) Extracting solution Bray 1 (0.03 M NH<sub>4</sub>F and 0.025 M HCl). Add 15 mL 1 M NH<sub>4</sub>F and 25 mL 0.5 M HCl to approx. 400 mL deionized water and fill up to 500 mL with deionized water.
- e) Sulphuric Acid, 2.5 M. Slowly add 35 mL conc. H<sub>2</sub>SO<sub>4</sub> (96%) to 150 mL deionized water under constant stirring. After cooling make to 250 mL with deionized water.
- Ammonium molybdate solution, 4%. Dissolve 4 g of (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>.4H<sub>2</sub>O in deionized water and make to 100 mL. f) Store in polythene or pyrex bottle.
- g) Potassium antimony tartrate solution, 0.275% (1000 mg/L Sb). Dissolve 0.275 g KSbOC<sub>4</sub>H<sub>4</sub>O<sub>6</sub> in deionized water and make to 100 mL.
- h) Ascorbic acid solution, 1.75%. Dissolve 1.75g ascorbic acid in deionized water and make to 100 mL. Prepare fresh daily.
- Mixed Reagent. Successively add with a graduated cylinder to a 500 mL beaker and homogenize after each i) addition:

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- 200 mL deionized water
- 50 mL of 2.5 M H<sub>2</sub>SO<sub>4</sub>
- 15 mL of NH<sub>4</sub>-molybdate solution
- 30 mL of ascorbic acid solution
- 5 mL of KSb-tartrate solution

Prepare fresh daily.

- j) Boric acid solution, 1%.Dissolve 1 g H<sub>3</sub>BO<sub>3</sub> in 100 mL deionized water.
- k) Standard Phosphate Solution, 100 mg/L P. Pipette 100 mL of NIST Traceable 1000 mg/L Phosphorus Standard Solution into a 1 L volumetric flask an Limited Preview<sup>acting solution.</sup>

Alternatively, dissolve 0.4390g KH $_2$ PO $_4$  (dried for 2 h at 110 °C) in extracting solution in a 1L  $\sim$ 

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- I) Standard phosphate solution, 12 mg/L P. Pipetter Print of the 100 mg/L P standard solution into a 250 mL volumetric flask and make to volum Google Form link:
- m) Standard series. https://forms.gle/RbCgCdA54prTS66N7<sup>P</sup> standard solution. Make to volume with extracting solution. The standard series is then 0-1.2-2.4-3.6-4.8-6.0 mg/L P.

Remarks:

- 1. The range of working phosphate standards can be adjusted according to the equipment specifications and the concentration of P in the analyzed soil samples.
- 2. Excess fluorides produce negative interference in the molybdenic-blue reaction. Boric acid is added before the color development to remove this interference in the horizontal reacts with the fluoride, and a fluoroborate is formed by the following reaction (Kurtz, 1942).  $4F + H_3BO_3 + 3H^2 \rightarrow (BF_4)^2 + 3H_2O$

### HEALTH AND SAFETY

Wear proper personal protective equipment. Use laboratory coat, close shoes, gas mask or dust mask and appropriate gloves when performing chemical analysis to mitigate the harmful effects of exposure on chemicals.

Observe careful and proper handling of chemicals when using strong alkali bases, strong acids and oxidizing agents. Potential direct body contact (e.g. ingestion, inhalation) may cause severe irritation and inflammation to the skin, eyes, respiratory and digestive tracts. Severe exposure to these corrosive substances may cause serious health damage or death.

Avoid mixing incompatible chemicals to reduce risks of fire and explosion inside the laboratory. Sulfuric acid may react violently when mixed with water because of its high affinity for water. Always pour the acid into water to avoid splattering.

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