

INDIANA DEPARTMENT OF TRANSPORTATION DIVISION OF MATERIALS AND TESTS

DETERMINING PHOSPHORUS CONTENT IN SOILS BY SPECTROPHOTOMETRIC METHOD ITM No. 519-24

1.0 SCOPE.

- 1.1 This test method measures the soluble phosphorus concentration in topsoil by using Spectrophotometry.
- 1.2 This ITM may involve chemical handling during operations and may not address all the safety problems associated with the use of the test method. The user of the ITM is responsible for establishing appropriate safety and health practices and determining the applicability of regulatory limitations prior to use.
- 1.3 Latex gloves are required to be used when handling the phosphorus reagent. The reagent is toxic, and care is required to be taken to avoid ingestion or contact with the skin or eyes.

2.0 REFERENCES.

2.1 AASHTO Standards.

R 58 Dry Preparation of Disturbed Soil and Soil-Aggregate Samples for Test

M 231 Weighing Devices Used in the Testing of Materials

2.2 HACH Test Methods

Phosphorus, Reactive (Orthophosphate)-PhosVer 3 (Ascorbic Acid) Method 8048

3.0 TERMINOLOGY.

- **3.1 Terms and Abbreviations.** Definitions for terms and abbreviations will be in accordance with the Department's Standard Specifications, Section 101 and the following:
- **3.2 Filtrate.** Soil/water slurry that has passed through a filter
- 3.3 Parts Per Million (ppm). A measure of concentration
- **3.4 Spectrophotometry.** Spectrophotometry is an experimental technique that is used to measure the concentration of solutes in a specific solution by calculating the amount of light absorbed by those solutes.
- **4.0 SIGNIFICANCE AND USE.** This ITM shall be used to determine the water-soluble phosphorus content in soil. The results are used to determine if chemical stabilization

of the soil is appropriate.

5.0	Δ	PP	\mathbf{A}	R A	T	HS

- **5.1** Balance, Class G 2, in accordance with AASHTO M 231
- **5.2** Beakers, 100 ml
- **5.3** Buchner funnel (polyethylene funnel if vacuum is not available)
- **5.4** Spectrophotometer and accessories (HACH DR 1900 or similar)
- **5.5** Dry, lint-free tissue
- **5.6** Filter paper, fine porosity, 8 microns
- **5.7** Graduated Cylinder, 25 ml
- **5.8** Latex gloves
- 5.9 Oven, capable of maintaining a temperature of $140 \pm 9^{\circ}F$
- **5.10** Small scoop or spoon
- **5.11** Pipettes, 2 ml disposable
- **5.12** Mehlich 2 Soil Extractant (Diluted in 1:10 with Deionized water)
- **5.13** Sample cell (vial), glass, 10 ml
- **5.14** Side arm flask
- 5.15 Sieves, No.10 conforming to the requirements of AASHTO M 92
- **5.16** HACH PhosVer 3 Phosphate Reagent
- **5.17** Vacuum (Not required if using polyethylene funnel)
- **5.18** Wash bottle, 500 ml, for distilled or de-ionized water
- **5.19** Wide-mouth rectangular or round high-density polyethylene (HDPE) or glass bottles, 100 ml

6.0 MATERIAL.

6.1 Distilled or de-ionized water

7.0 SAMPLE PREPARATION.

7.1 The sample may be air dried, or oven dried at a temperature not exceeding 140°F.

- 7.2 After the sample is dried, the sample is crushed, ground, and split to obtain an approximately 100g sample that is passing the No.10 sieve.
- 7.3 The sample is split to obtain three approximately 2g samples and each sample is weighed to the nearest 0.1g.

8.0 PROCEDURE.

8.1 Soil Slurry.

- **8.1.1** Take 20 ml of Mehlich 2 solution (Diluted 1:10) into the HDPE or glass bottle
- **8.1.2** Put one sample of 2 g soil into it.
- **8.1.3** Cap the bottle and shake the soil slurry for 5 minutes

8.2 Filtration.

- **8.2.1** Place a filter paper into the Buchner funnel and slightly moisten the filterpaper with distilled or de-ionized water
- **8.2.2** Place the stem of the funnel through the rubber stopper and affix the stopper/funnel assembly onto the top opening of the side arm flask
- **8.2.3** Shake the soil slurry sample for few seconds before the filtration
- **8.2.4** Start the vacuum, pour the soil slurry sample into the funnel, and collect the filtrate in the side arm flask. Do not rinse material into the Buchner funnel or add any water. A filtration of 10-15 ml would be sufficient to proceed further. No vacuum is required if using polyethylene funnel, filter it by gravity.
- **8.2.5** Repeat the filtration procedure for the remaining two soil slurry samples, collecting the filtrate in separate flasks.

8.3 Measurement.

- **8.3.1** Using a clean, uncontaminated disposable pipette, measure 1ml of filtrate and transfer the filtrate into the graduated cylinder 25 ml.
- **8.3.2** Fill the cylinder up to 25 ml mark with deionized water.
- **8.3.3** Place the cap, invert it several times and set it for rest up to 3 minutes.

8.3.4 After 3-minute pour 10 ml liquid in both vial(10ml). Place the cap on first one and keep it aside for blank.

- **8.3.5** Add the contents of PhosVer 3 Phosphate Reagent Powder Pillow to the second vial according to the requirement for 10 ml sample.
- **8.3.6** Immediately close the sample, shake it for 20-30 seconds, and set it aside for rest up to 2 minutes. (Neither less than 2 nor more than 5 minutes)
- **8.3.7** Press the power button in spectrophotometer and select the program 490 P react.
- 8.3.8 Clean the outside of the blank vial with lint guard wipes and place it in spectrophotometer to set zero. Close the lid and press zero. The display shows $0.00 \, \text{mg/L}$.
- **8.3.9** When 2-minute timer ends, clean the outside of the second vial with lint guard wipes, remove the blank from spectrophotometer and place the prepared sample into it.
- **8.3.10** Press the READ/ENTER key. The display will show "---" followed by the results in mg/L phosphorus.
- **8.3.11** Take a minimum of three readings for each sample and average the results

9.0 CALCULATIONS.

9.1 Calculate the phosphorus content in ppm by multiplying the average of the three Spectrophotometer tests by the Dilution Ratio from Table 1.

SPECTROPHOTOMETER METHOD						
Mehlich 2/De-ionized Water, ml	Extractant / De- Ionized Water	Dilution Ratio	Phosphorus Content Range ppm			
1:1 (Undiluted)	1:25	25	0.5-62.5			
1:10 (Standard)	1:25	250	5-625			
1:15	1:25	375	7.5-937.5			
1:20	1:25	500	10-1250			

Table 1. DILUTION RATIO FOR PHOSPHORUS TESTING

Example:

Average of three spectrophotometer results = 2 mg/L

Dilution Ratio = 250 (From Dilution Ratio Table)

Phosphorus Content (ppm) = $2 \times 250 = 500 \text{ ppm}$

- 9.2 The phosphorus test only displays the result in spectrophotometer (HACH DR 1900 or Similar) in between the range 0.02 mg/L to 2.5 mg/L. Any results apart from this range will be displayed as under range or over range.
 - **9.2.1** If the display shows over range or (+++), increase the dilution of Mehlich 2 and Deionized water and change the dilution factor according to table 1.
 - **9.2.2** If the display shows under range or (---), decrease the dilution of Mehlich 2 and Deionized water or use the undiluted form of Mehlich 2 and change the dilution factor according to table 1.
 - **9.2.3** If the display shows a negative value, it could be because of mistakenly putting the treated sample in place of blank and blank in place of treated sample, so try once doing vice-versa. If it does not work, repeat 9.2.2.
 - **9.2.4** In prepared sample, the more the color is blue then the higher the content of phosphorus. Similarly, less the color is blue, then the lower the content of phosphorus.
- **10.0 REPORT.** Report the concentration of phosphorus in parts per million (ppm)

APPENDIX

Dilution method for Mehlich 2 Solution with Di-Ionized Water				
1:1 (Undiluted)	This means it contains only one part of concentrated Mehlich 2 Solution. No need to add any Deionized water			
1:05	This means it contains total 5 parts (1 Part Mehlich 2 solution and 4 parts Deionized water). Example- If total solution is 50 ml, then 10 ml Mehlich solution and 40 ml deionized water			
1:10 (Standard)	This means it contains total 10 parts (1 Part Mehlich 2 solution and 9 parts Deionized water). Example- If total solution is 100 ml, then 10 ml Mehlich solution and 90 ml deionized water			
1:15	This means it contains total 15 parts (1 Part Mehlich 2 solution and 14 parts Deionized water). Example- If total solution is 150 ml, then 10 ml Mehlich solution and 140 ml deionized water			
1:20	This means it contains total 20 parts (1 Part Mehlich 2 solution and 19 parts Deionized water). Example- If total solution is 200 ml, then 10 ml Mehlich solution and 190 ml deionized water			