

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

HJ 712-2014

**Solid waste-Determination of total phosphorus. Ammonium
metamolybdate spectrophotometric method**

固体废物 总磷的测定 偏钼酸铵分光光度法

(English Translation)

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Foreword

To implement the "People's Republic of China Environmental Protection Law" and "People's Republic of China Solid Waste Pollution Prevention Law" Protecting the environment, safeguarding human health, specifications can be crushed in solid, semi-solid determination of total phosphorus in the solid waste of the enactment of this standard.

This standard specifies the determination of total phosphorus in the solid waste Partial molybdate spectrophotometric method.

This standard is the first release.

This standard is developed by the Ministry of Environmental Protection Science, Technology organization.

Drafting of this standard. Nantong Municipal Environmental Monitoring Center.

The standard verification. Jiangsu Provincial Environmental Monitoring Center, Suzhou Municipal Environmental Monitoring Center, Yangzhou Municipal Environmental Monitoring Center Station, Zhenjiang Municipal Environmental Monitoring Center, Nantong City Agricultural Products Quality Testing Center and Nantong, Tongzhou District Environmental Monitoring Station.

This standard MEP November 27, 2014 for approval.

This standard since January 1, 2015 implementation.

The standard explanation by the Ministry of Environmental Protection.

Determination of Solid Waste 1 TP Partial molybdate spectrophotometry

1 Scope

This standard specifies the determination of total phosphorus in the solid waste Partial molybdate spectrophotometric method.

This standard applies to be crushed solid or semi-solid determination of total phosphorus in the solid waste.

When the sample volume is 0.5 g, volume by volume of 50 ml, using a 30 mm cuvette when the standard method detection limit of 3 mg/kg, detection limit of 12 mg/kg.

2 Normative references

This standard refers to the contents of the following documents or the terms. For undated references, the effective version suitable for this standard.

HJ/T 20 industrial solid waste sampling sample preparation Technical Specifications

3 PRINCIPLE OF THE METHOD

Solid waste system by nitric acid digestion, in which the phosphorus soluble salts and organic matter is completely converted to soluble orthophosphate Salt, under acidic conditions to form a yellow heteropoly metavanadate and ammonium molybdate react with acids to measure absorbance at a wavelength of 420 nm Luminosity. Within a certain range of concentrations, the phosphate content of the absorbance values conform to the Lambert - Beer law.

4 and elimination of interference Microwave digestion - Partial molybdate spectrophotometric determination of total phosphorus in the solid waste, the color liquid Fe³⁺ concentration of less than 100 mg/L, Cr⁶⁺ concentrations below 18 mg/L when measured without interference.

5 Reagents and materials

Unless otherwise indicated, the use of chemical reagents analytical line with national standards of analysis, to test water for the preparation of the new Ionized or distilled water.

5.1

concentrated nitric acid. $\rho(\text{HNO}_3) = 1.40 \text{ g/ml}$, pure class distinctions.

Concentrated sulfuric acid 5.2. $\rho(\text{H}_2\text{SO}_4) = 1.84 \text{ g/ml}$.

5.3 Sulfuric acid solution. $c(\text{H}_2\text{SO}_4) = 0.5 \text{ mol/L}$

Measure 5.5 ml of concentrated sulfuric acid (5.2) was slowly poured into a small amount of water and dilute to 200 ml.

5.4 Sodium carbonate solution. $w(\text{Na}_2\text{CO}_3) = 10\%$

Weigh 10 g of sodium carbonate (Na_2CO_3) was dissolved in 100 ml of water.

5.5 potassium dihydrogen phosphate (KH_2PO_4). pure class distinctions.

Proper amount of potassium dihydrogen phosphate (of KH_2PO_4) in a weighing bottle is placed 105 °C drying 2 h, cooling the oven and set aside.

5.6 Ammonium molybdate solution. $\rho[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}] = 62.5 \text{ g/L}$

Weigh 25 g of ammonium paramolybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ was dissolved in 400 ml of water.

5.7 Metavanadate solution. $\rho[\text{NH}_4\text{VO}_3] = 2.27 \text{ mg/L}$

Weigh 1.25 g of ammonium metavanadate (NH_4VO_3) was dissolved in 300 ml boiling water, after cooling, was added 250 ml of concentrated nitric acid (5.1), Cooled to room temperature.

25.8 Molybdate - a mixed solution of ammonium metavanadate

The ammonium molybdate solution (5.6) was slowly added a solution of ammonium metavanadate (5.7), diluted with water to 1000 ml. Placed in the refrigerator 2 ~ 5 °C preservation, stable for at least a year, if turbidity occurs, the discarded reconfiguration.

5.9 P standard stock solution. $\rho(\text{P}) = 1000 \text{ mg/L}$ Weigh 4.3940 g of potassium dihydrogen phosphate (5.5) dissolved in about 200 ml of water, add 5 ml of concentrated sulfuric acid (5.2) to move to 1000 ml volumetric flask, add water to the mark, and mix. The solution was stored in a brown reagent bottle, valid for one year. Or direct Purchase a commercially available certified reference solution.

5.10 Phosphorus standard solution. $\rho(\text{P}) = 20 \text{ mg/L}$ Pipette 10.00 ml phosphate standard stock solution (5.9) to 500 ml flask, water volume, and the solution with the existing Pro.

5.11 Indicator. 2,6-dinitrophenol ($\text{C}_6\text{H}_4\text{N}_2\text{O}_5$) or 2,4-dinitrophenol ($\text{C}_6\text{H}_4\text{N}_2\text{O}_5$) Weigh 0.2 g 2,6- or 2,4-dinitrophenol dinitrophenol is dissolved in 100 ml of water.

6 instruments and equipment

6.1 Visible Spectrophotometer. with 30 mm glass cuvette.

6.2 Microwave Digestion System. Maximum power 1600 W.

6.3 Electric digester. special microwave digestion tank (50 ~ 200 °C, the output power of 1600 W).

6.4 Electric board. 50 ~ 200 °C, the output power of 3000 W.

6.5 Analytical balance. accuracy of 0.0001 g.

6.6 plug colorimetric tube. 50 ml.

6.7 General Commonly used laboratory instruments and equipment.

7 Sample

7.1 collection, preservation and preparation Collection and preservation of samples were prepared in accordance with HJ/T relevant provisions 20.

7.2 Preparation of the sample it weighs about 0.2 ~ 0.5 g sample (accurate to 0.0001 g), placed in a microwave digestion vessel, with the amount of water wet sample, Add 10 ml of concentrated nitric acid (5.1), stamped with a cold digestion overnight (at least 16 hours), and then placed in a microwave digestion (L Temperature program refer to Table 1), after digestion is complete cooling. The microwave digestion tank into the digester to about 160 °C electric rush acid sample was adhered to Viscous. If after a hot plate to catch the acid digestion solution can be completely transferred to a glass beaker on a hot plate heated to about 160 °C sample it was sticky. As the test solution does not show then the digestion is not completely white, etc., cool to room temperature, plus the amount of concentrated nitric acid (5.1), following the Continued hurry microwave digestion and acid until the sample was gray. Remove the microwave digestion vessel was cooled to room temperature, the samples were all transferred to 50 ml colorimetric tube, add water to 50 ml, shake, standing, and the supernatant tested.

Table 1 microwave digestion instrument reference temperature program

Heating step	heating time (min)	digestion temperature (°C)	retention time (min)
the first step	5.00	1202	Step 4.00
1605	the third step	4.00	19025

Note 1: The rush acid sample digestion was complete, after digestion solution was allowed to stand colorless, clear shape. If reddish-brown nitrogen oxides do not catch, can continue to catch the acid straight to reddish brown disappear.

38 Analysis steps

8.1 Calibration curve Pipette 0.00,2.00,4.00,6.00,8.00,10.00,12.00,14.00 ml phosphorus standard solution (5.10) to 50.0 ml colorimetric tube, add water to 25 ml mark.

Subsequently, 2 drops of indicator (5.11), with a sulfuric acid solution (5.3) or carbonate Sodium solution (5.4) solution was adjusted to pale yellow, then add 10 ml ammonium molybdate - ammonium metavanadate mixed solution (5.8), water volume to 50.0 ml, allowed to stand at room temperature for 30 min. Phosphorus concentrations were 0.00