SHIMADZU APPLICATION NEWS

LAAN-A-UV-E002

● SPECTROPHOTOMETRIC ANALYSIS

No.A354

Measurement of Total Phosphorus in Water by Absorption Spectophotometry

Phosphorus, widely found in various products such as fertilizer, agricultural pesticides, dyes, processed food and alkaline detergent, has greatly enriched our lives. However, it also contaminates the environmental waters by flowing into rivers and lakes along with residential and industrial waste water, or agricultural runoff. Phosphorous and nitrogen, which enhance the growth of plankton in lakes, are used as indices of eutrophication. Consequently, measurement of phosphorus concentration is important for water quality management.

The spectrophotometric absorption method has long been used to measure the total phosphorus concentration in water quality testing. Reported here is an investigation to determine the concentration range within which the quantitation of phosphorus can be performed using Shimadzu's UV-VIS spectrophotometer UV-2550.

■ Measurement Flow

The outline of the procedure of phosphorus measurement is presented below.

First, phosphorus standard solutions are prepared to generate a calibration curve. To these, ammonium molybdate + ascorbic acid mixeacid solution is added then the solution will become blue, and the absorbances are measured at 880nm. These values are used to generate a calibration curve. Here, phosphate standard solution (P: 1000mg/L, KH_2PO_4) was obtained from a reagent production company.

In pretreatment of the unknown samples, in addition to the above, a high-pressure steam sterilizer is used to decompose substances in the solution that contain phosphorus (P) under high temperature and high pressure, so that all the phosphorus is in the form of phosphate ions. (This process is unnecessary for the phosphorus standard solutions used to generate the calibration curve.)

"Total phosphorus" expresses the total amount of phosphorus (P) contained in the compounds in an aqueous solution, and is measured and expressed in the form of phosphate ions.

The above procedure is merely an outline, as various other operations are also required. For details, refer to JIS K 0102 "Testing Mmethod for industrial wastewater." Fig. 1 shows the measurement flow based on this JIS method.

Note : To measure phosphorus, in addition to the measurement of total phosphorus, measurement of just the phosphate ions (PO_4^{3-}) is also available. In that case, the process of decomposition under high temperature and high pressure is omitted.

Standard Sample Preparation Flow Chart
(In case of 10mm cell used)

(1) Transfer phosphorus standard solution (P: 5μg/mL) of 0mL, 1mL, 2.5mL, 5mL, 10mL and 20mL to 100mL flasks, and bring to 100mL using water.
↓
(2) As a result, the following concentrations of solution are prepared.
0μg/mL, 0.05μg/mL, 0.125μg/mL, 0.25μg/mL, 0.5μg/mL, 1μg/mL
↓
(3) Add 2mL of an ammonium molybdate - ascorbic acid mixed solution to 25mL of each of (2), shake and set aside for 15minutes.
↓
(4) Transfer a portion of solutions to absorption cells, take zero reading using the 0μg/mL solution, and measure absorbance of standard solutions near 880nm.
↓
(5) Generate a calibration curve using those absorbance values.

Fig.1 Standard Solution Preparation Flow Chart

Unknown Sample Measurement Flow Chart
(in case of 10 mm cell used)

(1) Prepare the following solutions.
- potassium peroxodisulfate (40g/L)
- ammonium molybdate – ascorbic acid mixed solution
↓
(2) Transfer 50mL of unknown sample to a decomposition flask.
↓
(3) Add 10mL of potassium peroxodisulfate (40g/L), stopper and mix.
↓
(4) Place in a high-pressure steam sterilizer, heat to about 120˚C and continue decomposition heating for 30minutes.
↓
(5) Remove the decomposition flask, let it cool, transfer 25mL of supernatant to a stopper-equipped test tube.
↓
(6) Add 2mL of ammonium molybdate – ascorbic acid mixed solution, shake and set aside for about 15 minutes at 20 - 40˚C.
↓
(7) Transfer a portion of solution to an absorption cell, and measure absorbance near 880nm.
↓
(8) As a blank, use 25mL of water, perform steps (6) and (7), measure absorbance, and use this value to correct the absorbance values of the samples (subtract the blank value).
↓
(9) The concentration value is determined by applying 6/5 (correction from (3)) to the concentration obtained from the calibration curve.

Note : There are additional details and many notes given in JIS K 0102. When performing actual analysis, refer to JIS K 0102.

Fig.2 Unknown Sample Measurement Flow Chart

■ Determining Quantitation Range

The possible concentration range within which quantitation could be performed using the UV-2550 was determined based on the calibration curve generated from measurement of the phosphorus standard solutions (Refer to Fig. 1). The slit width was set to 5nm, and a 10mm square cell was used. In this case, unknown samples were not measured.
The standard sample spectra are shown in Fig. 3. Here, the calibration curve was generated using absorbance values measured at 880nm (Fig. 4). A good calibration curve whose correlation coefficient r2 was 0.99959 was obtained. The relationship between the standard sample concentrations and absorbance values are as shown in Table 1.
The possible concentration range within which quantitation could be performed was estimated from the calibration curve result at about 0.05mg/mL-3mg/mL. Here, a 10mm square cell was used, however, using a 50mm square cell would provide five times the sensitivity, or a quantitation range of 0.01mg/mL-0.6mg/mL.

Note: The obtained quantitation range was based on ideal standard solutions, so it is likely that this range would be a bit narrower using unknown solutions containing other constituents.

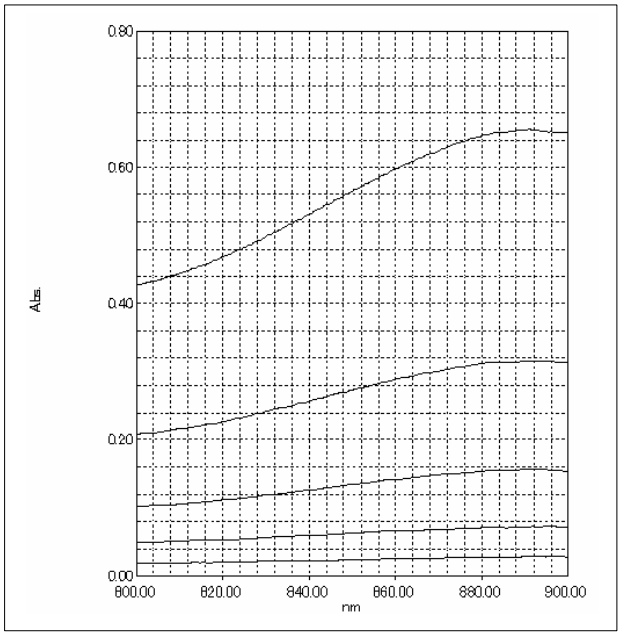

Fig.3 Spectra Curves

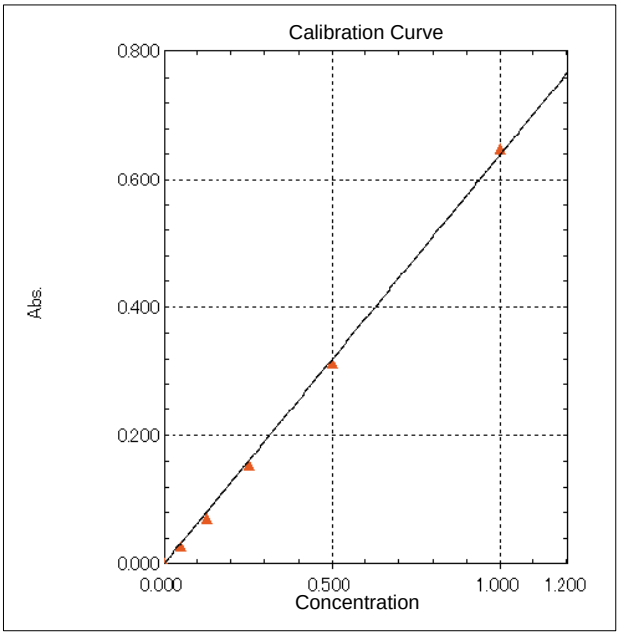

Fig.4 Calibration Curve

Table 1 Concentration and Absorbance in standard samples.

STD sample	Conc.(µg/mL)	Abs.(880.0nm)
1	0.000	0.000
2	0.050	0.027
3	0.125	0.070
4	0.250	0.152
5	0.500	0.312
6	1.000	0.646

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