Colorimetric analysis of P using the molybdenum blue method.

Many laboratories routinely measure phosphate using the molybdenum blue method, often using automated systems. We describe here a simple batch version of the method. Automated methods can be adapted if they account for the composition of the DGT eluent.

The successful use of the method depends on ensuring that the composition of the DGT elution solution is compatible with the reagents to be used in the first steps of the analysis. The first reagent used in this method contains 0.25 M H_2SO_4.

When ferrihydrite is used in the binding layer, 5 mL of 0.25 M H_2SO_4 can be used as an eluent. This solution is then ideal for colorimetric analysis, as there is no need to have H_2SO_4 in the first reagent added, designated below as mixed reagent 2 (MR2).

Alternative eluents are used when other elements are measured simultaneously with P. Commonly nitric or hydrochloric acid are used with binding layers containing ferrihydrite and sodium hydroxide is used with binding layers containing Metsorb (TiO_2). With appropriate precautions for polyatomic interferences, P can be measured along with the other elements by ICP-MS. However, P can alternatively be measured colorimetrically in a portion of the eluent if it is first neutralised using high purity reagents to avoid contamination. This neutral solution can then be analysed conventionally by the molybdenum blue method.

Analytical procedures

Reagents

A) H_2SO_4 (2.5 M)
B) Ammonium molybdate (20 g into 500 mL water)
C) Potassium antimonyltartrate (0.28 g into 100 mL water)
D) Ascorbic acid (1.76 g into 100 mL water)

Procedure 1

Mixed reagent 1 (MR1) for neutralised eluents.
10 mL A + 3 mL B + 1 mL C + 6 mL D.
Add 1 mL the mixed reagent MR1 into a 5 mL sample of eluent (diluted if necessary), wait for 15~20 minutes for colour to develop and then measure.

The concentration in the eluent or its derived diluted solution can be measured by preparing a calibration curve using standard phosphate solutions. Ensure that the standard solutions contains the same concentration of salt as in the eluent, resulting from the neutralisation. The same procedure as above is followed using 5 mL of these standard solutions.

Procedure 2

Mixed reagent 2 (MR2) for elution solutions containing 0.25 M H_2SO_4.
10 mL water + 3 ml B + 1 ml C.
Add 0.7 ml (MR2) into 5 ml of elution solution followed by 0.3 ml reagent D (ascorbic acid), wait for about 15 ~ 20 minutes for colour to develop and then measure.

The concentration in the eluent can be measured by preparing a calibration curve using standard phosphate solutions prepared in 0.25 M H_2SO_4. The same procedure as above is followed using 5 mL of these standard solutions.

DGT Research Ltd
Skelmorlie, Bay Horse Rd, Quernmore, Lancaster, Lancashire, LA2 0QJ
Phone : +44 (1524) 593899, 2nd Phone : 01524 751451
Email : info@dgtresearch.com

Calculating the DGT Measured Concentrations:

The measured concentration of analyte, c_e, in the eluent prior to any dilution, of volume V_e (usually 5 mL when using H_2SO_4, but other volumes for other eluents), can be used to calculate the mass, M, of analyte on the binding layer, of volume, V^{bl} (usually 0.20 mL for a DGT solution or soil device). Remember to take into account any subsequent dilution.

$$M = \frac{c_e(V^{bl} + V_e)}{f_e}$$

Where c_e is in nmol mL^{-1} or ng mL^{-1}, M is in nmol or ng. The appropriate elution factor, f_e, for the chosen eluent and binding layer should be used. When eluting a ferrihydrite binding gel with 0.25 M H_2SO_4, f_e = 1.

In most situations where DGT is deployed in water that is flowing or subject to convection currents the standard DGT equation is appropriate. For sediment or soil deployments this equation gives the mean concentration during the deployment period of analyte at the DGT surface.

$$c_{DGT} = \frac{M\Delta_g}{D^{mdl}A_p t}$$

c_{DGT} (nmol mL^{-1} or ng mL^{-1}) is the time averaged concentration of analyte in the deployment medium measured by DGT.

M (nmol or ng) is the mass of analyte accumulated in the binding layer. It is obtained from the analysis (see above)

Δ_g (also known as δ_g) (0.094 cm for a standard solution device) is the total thickness of the materials in the diffusion layer (diffusive gel and filter membrane).

D_{mdl} (cm^2 s^{-1}) is the diffusion coefficient of analyte in the material diffusion layer for the deployment temperature (see diffusion coefficients).

A_p (3.14 cm^2 for a standard solution device) is the physical area of the exposed filter membrane.

t (s) is the deployment time.

Recommended units to facilitate easy calculation are shown. This calculation procedure should work well for most situations. For more accurate methods of calculation that incorporate estimates of the flow regime see FAQs on the web site.

DGT Research Ltd
Skelmorlie, Bay Horse Rd, Quernmore, Lancaster, Lancashire, LA2 0QJ

Phone : +44 (1524) 593899, 2nd Phone : 01524 751451
Email : info@dgtresearch.com