

## Phosphate using DGT with a ferrihydrite binding layer

These instructions are written for LSNP-NP (phosphate in solution). The procedure for analysis is the same for LSLP-NP (phosphate in soils) and LSPP-NP (phosphate in sediment), but different deployment strategies are used as detailed in [soil deployments](#) and [sediment deployments](#).

|                             |  |
|-----------------------------|--|
| <b>Type:</b>                | LSNP-NP  |
| <b>Measurable analytes:</b> | Phosphate P(V)   |
| <b>Holder:</b>              | Standard DGT holder  |
| <b>Filter :</b>             | Polyethersulphone membrane (thickness: 0.14 mm)  |
| <b>Diffusive gel:</b>       | 0.8 mm APA diffusive gel   |
| <b>Binding layer:</b>       | Fe <sub>2</sub> O <sub>3</sub> .xH <sub>2</sub> O (Ferrihydrite) freshly prepared before gel casting |

Using ferrihydrite as the binding layer provides the simplest elution and analysis strategy for phosphate and it has been used successfully tens of thousands of times. Other analytes can be measured with the same device, but a different elution strategy is required (see [detailed guide for oxyanions](#)).

### Water types and Limiting Conditions

**Appropriate waters:** Suitable for freshwater through to seawater. **pH:** recommended range 5 to 8.5. **Ionic strength:** recommended range 1 to 700 mM. Can be used at lower ionic strengths, but as there is a possibility of gel charge affecting performance, laboratory control tests at the same ionic strength as the field solution, are then advised.

**Deployment time:** Optimum deployment time depends on the concentration of phosphate in the sample water. Deployment times up to 4 days have been validated, but shorter times of a day can also be used ([see FAQs](#)).

**Measured species:** The dominant forms are the various protonation states of phosphate, which is considered to be the analyte measured in most cases.

### Storage

Store the DGT devices in a refrigerator (4°C). Do not freeze. The DGT devices are provided in sealed clean plastic bags containing a few drops of 0.01M NaCl solution. Do not open them until immediately prior to deployment. Check the devices about once a week to make sure they are under moist conditions. Add a few more drops of trace-metal-clean 0.01M NaCl solution if it is necessary.

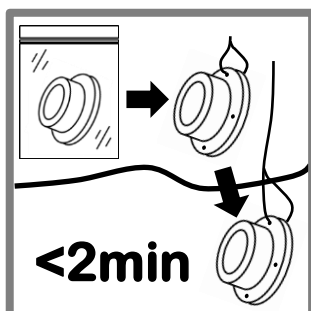
### Handling

The main consideration when using DGT devices is to prevent contamination of the sample. Clean handling procedures should be adopted during deployment and recovery of the DGT devices and all subsequent handling during the sample treatment step. Do not touch or contact the white filter membrane at the face of the device. In general, the highest quality reagents should be used and all equipment and laboratory apparatus cleaned appropriately.

## General deployment considerations

**Biofouling:** The extent of any biofouling is very dependent on local conditions, particularly light, temperature and productivity, but it is not usually a problem for deployments less than one or two weeks.

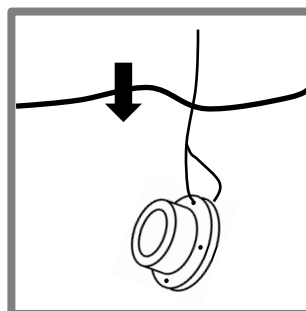
## Deploying DGT devices



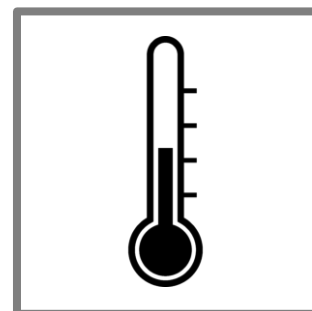
**1.** Having placed the DGT unit in its deployment holder or simply attached it to any deployment device by tying it with a fishing line threaded through the hole on the rim of the unit, deploy the unit immediately (minutes).



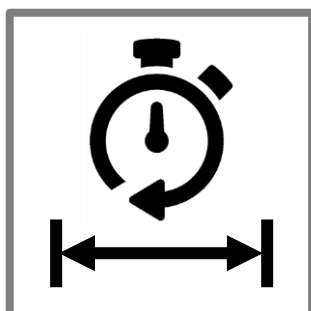
**2** Ensure the DGT device is deployed in flowing (or moving) water, but avoid excessive turbulence, particularly bubbles. Large open waters including lakes usually have sufficient natural flow through wave action.



**3.** Ensure that the white face of the DGT unit is fully immersed during the deployment period.



**4.** Accurately record the temperature of the water during the deployment period. If the variation is within  $\pm 2^{\circ}\text{C}$ , a mean (or start and end) temperature will suffice. If the variation is greater, ideally the mean temperature should be obtained from an integrated record of temperature (data logger).



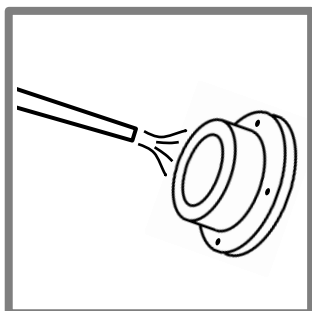
**5.** Provide an accurate record to the nearest minute of the deployment time

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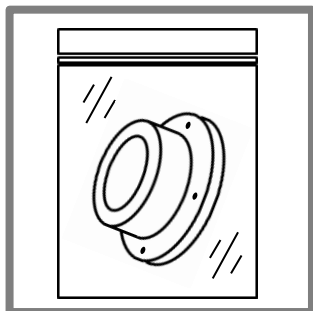
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## Procedure for analyzing DGT samples

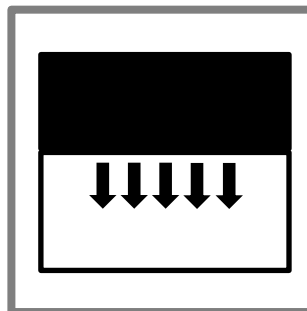
### DGT Recovery and Sample Treatment



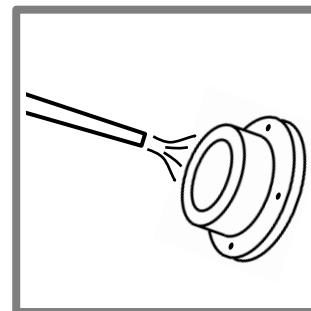
**1.** After retrieving the DGT device from the deployment environment thoroughly rinse the DGT device with ultrapure water.



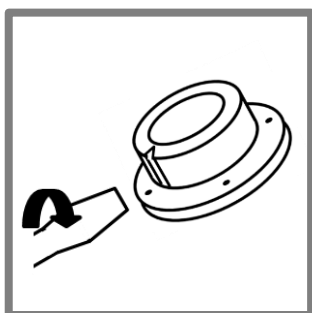
**2.** Place in a clean plastic bag for storage and transportation back to the lab for sample treatment.



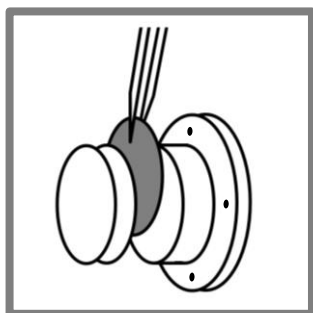
**3.** For ultra trace concentrations, consider undertaking sample treatment in a laminar flow hood to avoid contaminating the sample.



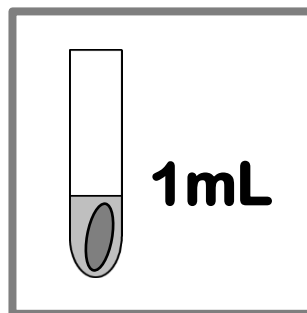
**4.** Remove from the plastic bag and thoroughly rinse the device with ultrapure water.



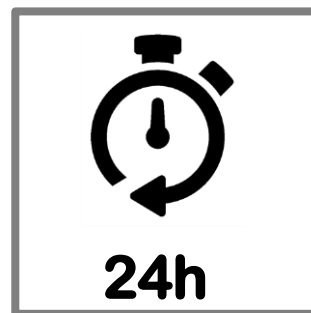
**5.** To retrieve the resin gel after deployment, insert a suitable screwdriver into the groove in the cap and twist it. The cap will be broken at the weak point.



**6.** Remove the broken cap and then peel off the filter and diffusive gel layer to reveal the bottom binding-gel layer.

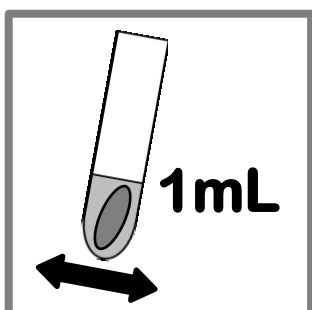


**7.** Place the binding layer (or sliced portion of it for sediment probes) in 5 ml of high purity water for 1 hour. Then place it in a clean sample tube and add 5 ml of 0.25 M H<sub>2</sub>SO<sub>4</sub>.

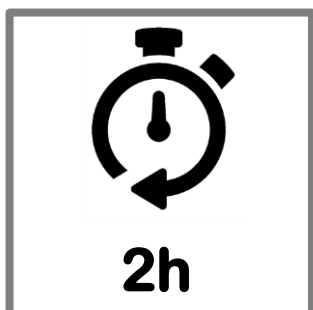


**8.** Make sure the binding gel is fully immersed in the H<sub>2</sub>SO<sub>4</sub> solution. Leave to stand for at least 24 hours before analysis. Analyse using the [colorimetric phosphate method](#) based on molybdenum blue.

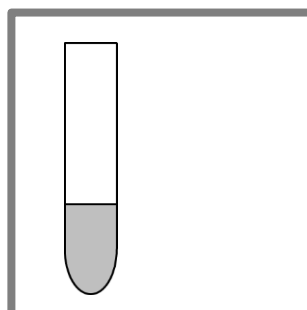
### For urgent analysis



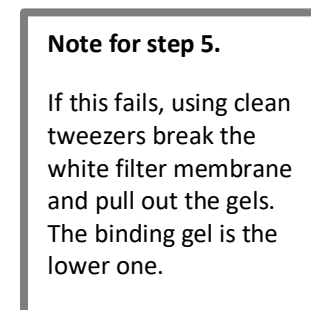
**7a.** Agitate the tube containing the binding gel and 5 ml of 0.25 M H<sub>2</sub>SO<sub>4</sub> on a shaker.



**8a.** Shake for at least 2 hours before analysis.



**9.** Analyse using the [colorimetric phosphate method](#) based on molybdenum blue.



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## Analysis

Phosphate is readily analysed colorimetrically using the molybdenum blue method. The above validated elution strategy has been designed to be compatible with this analysis, as detailed in [analysis of phosphate colorimetrically](#).

## Method and field blanks

To ensure accurate results it is recommended to determine DGT laboratory<sup>1</sup> and field blank<sup>2</sup> concentrations.

<sup>1</sup>The laboratory blank is an unexposed DGT device carried through all steps of the measurement process (from extraction through analysis). A laboratory blank is typically analyzed with each sample batch.

<sup>2</sup>The field blank is designed to identify levels of contamination from DGT devices exposed in the field as the field. In summary, field blanks consist of additional DGT devices, which are transported to the monitoring site, exposed briefly at the site when the samples are exposed (but no deployment is carried out), and transported back to the laboratory for analysis, similar to a field sample. It is advisable to have at least one field blank for each test series.

## Elution efficiency, $f_e$

For the procedure described here, the elution efficiency is 1.

## Calculation

In most situations where DGT is deployed in water that is flowing or subject to convection currents the standard DGT equation is appropriate.

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

$C_{\text{DGT}}$  (nmol mL<sup>-1</sup> or ng mL<sup>-1</sup>) 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 below)

$\Delta_g$  (also known as  $\delta_g$ ) (usually 0.094 cm) is the total thickness of the materials in the diffusion layer (diffusive gel and filter membrane).

$D^{\text{mdl}}$  (cm<sup>2</sup> s<sup>-1</sup>) is the diffusion coefficient of analyte in the material diffusion layer for the deployment temperature (see [diffusion coefficients](#)).

$A_p$  (3.14 cm<sup>2</sup> for the standard DGT solution holder, 2.54 cm<sup>2</sup> for soil devices) 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](#).

The mass,  $M$ , of analyte on the binding layer, of volume,  $V^{\text{bl}}$  (usually 0.20 mL for standard solution devices), is calculated from the measured concentration of analyte,  $c_e$ , in the acid eluent, of volume  $V_e$  (usually 5 mL), remembering to take into account any subsequent dilution.

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

For the usual set up for solution devices, if  $f_e = 1$ ,  $M = 5.2c_e$  (where  $c_e$  is in nmol mL<sup>-1</sup> or ng mL<sup>-1</sup>,  $M$  is in nmol or ng).

Using the same DGT equation for soils and sediment gives the mean concentration of analyte at the probe surface during the deployment time, where, for sediments,  $A_p$  is the area of the gel slice used. The mass calculation is the same for soil probes. For sediment probes the appropriate volume of the binding gel slice must be used.

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## References

Diffusive Gradients in Thin-Films for Environmental Measurements, Ed. W. Davison, Cambridge University Press, 2016, Cambridge. Chapters in this book, which are particularly relevant are:

1. Introduction to DGT (covers the basic principles)  
William Davison and Hao Zhang
2. Principles of measurements in simple solutions (explains procedures for calculations)  
William Davison and Hao Zhang
3. Diffusion layer properties (provides and critiques diffusion coefficients)  
William Davison and Hao Zhang
4. Binding layer properties (provides properties including elution efficiencies for a range of binding agents and analytes)  
William W. Bennett, Maja Arsic, Jared G. Panther, David T. Welsh, and Peter R. Teasdale
10. Practicalities of working with DGT (practical issues such as deployment, quality control and sensitivity)  
Dianne F. Jolley, Sean Mason, Yue Gao and Hao Zhang

### Prime publications for this analyte:

- S. Mason, R. Hamon, A. Nolan et al., Performance of a mixed binding layer for measuring anions and cations in a single assay using the diffusive gradients in thin films technique, *Anal. Chem.*, **77**: (2005), 6339-6346.
- H. Zhang, W. Davison, R. Gadi, and T. Kobayashi, In situ measurement of dissolved phosphorus in natural waters using DGT, *Anal. Chim. Acta.* **370**: (1998), 29-38.
- H. L. Price, P. R. Teasdale and D. F. Jolley, An evaluation of ferrihydrite- and Metsorb™-DGT techniques for measuring oxyanion species (As, Se, V, P): Effective capacity, competition and diffusion coefficients, *Anal Chim Acta*, **803**: (2013), 56-65.

### Relevant reviews:

- W. Davison and H. Zhang, Progress in understanding the use of diffusive gradients in thin-films – back to basics, *Environ. Chem.* **9**: (2012), 1-13.
- H. Zhang and W. Davison, Use of DGT for studies of chemical speciation and bioavailability, *Environ. Chem.* **12**: (2015), 85-101.

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