DGT® Research
For measurements in waters, soils & sediments

LSNB - AP Loaded DGT device for mercury and monomethylmercury in solution

Type:	LSNB-AP
Measurable analytes:	As(III), Hg^{2+} and MeHg
Holder:	Standard DGT holder
Filter :	Polyethersulphone membrane (thickness: 0.14 mm)
Diffusive gel:	0.8 mm agarose diffusive gel
Binding layer:	3-mercaptopropyl functionalized silica gel

Water types and Limiting Conditions

Freshwater through to seawater. **pH:** recommended range from 3 – 8.5. (Gao et al., 2014)

Ionic strength: recommended range 0.001 M- 0.7 M. Can be used at lower ionic strengths, but as there is a possibility of gel charge affecting performance, control tests at the same ionic strength as the field solution, are then advised.

Binding limitations: resin gel can effectively accumulated MeHg in a pH range of 3 – 9. At pH outside of this range, competition between H^+ and MeHg and formation of stable MeHg hydroxide complexes prevents these species from being effectively absorbed by the resin. (Clarisse and Hintelmann, 2006)

Deployment time: Optimum deployment time depends on the quantification limits of the analytical technique used to determine analyte concentrations. Deployment times between 3 and 30 days are generally optimal, but shorter times of a day can be used (see FAQs). If the concentrations of the metals are low (less than a few micrograms per litre or less than a few nanogram per litre for Hg), as in an offshore marine environment, and there is no indication of biofilm growth on the surface of the devices, longer deployment times may be appropriate.

Storage

Store the units in a refrigerator (4°C). The DGT units provided are kept in the heat-sealed clean plastic bags containing a few drops of 0.03 M NaCl solution. Do not open them until immediately prior to deployment. Check the units about once a week to make sure they are under moist conditions. Add a few more drops of trace metal clean 0.03 M NaCl (or $NaNO_3$ for sea water) solution if it is necessary.

Handling

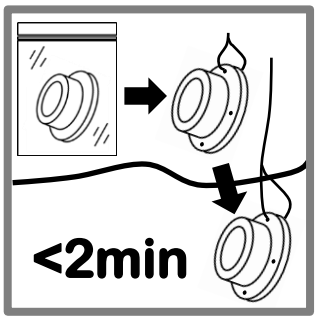
The main consideration when using DGT devices is to prevent contamination of the sample. Clean handling procedures should be adopted during deployment and recovery of the DGT devices and all subsequent handling during the sample treatment step. In general, the highest quality reagents should be used 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. Biofouling is not usually a problem for deployments less than a week. In pristine or deep waters, very long-term deployments (months extending to a year) have been unaffected by biofilms.

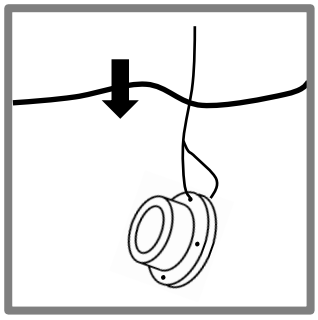
DGT Research Ltd
Skelmorlie, Bay Horse Rd, Quernmore, Lancaster, Lancashire, LA2 0QJ
Phone : +44 (1524) 593899, 2nd Phone : 01524 751451
Email : info@dgtresearch.com

Deploying DGT devices

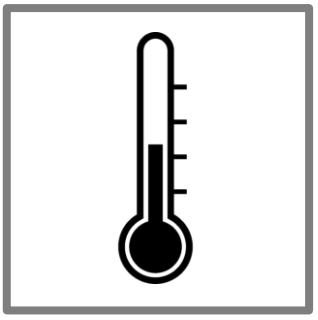


1. Having placed the DGT unit in its deployment holder or simply attached it to any deployment device by tying it with a pre-cleaned fishing line threaded through the hole on the rim of the unit, deploy the unit immediately (minutes).

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.



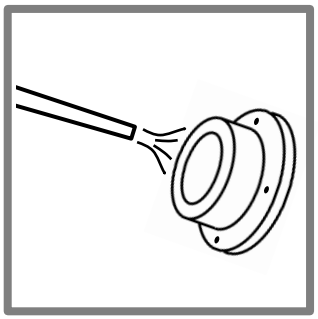
3. Ensure that the white face of the DGT unit is fully immersed during the deployment period. Provide an accurate record to the nearest minute of the deployment time



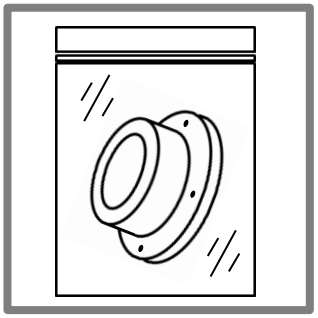
4. Accurately record the temperature of the water during the deployment period. If the variation is within ± 2°C, a mean (or start and end) temperature will suffice. If the variation is greater, ideally the mean temperature should be obtained from an integrated record of temperature (data logger).

Procedure for analyzing DGT samples

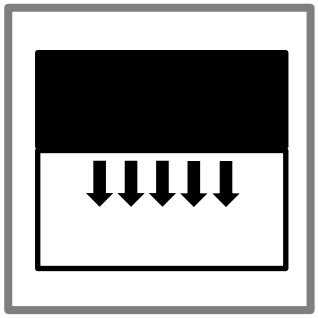
DGT Recovery and Sample Treatment for Total Hg



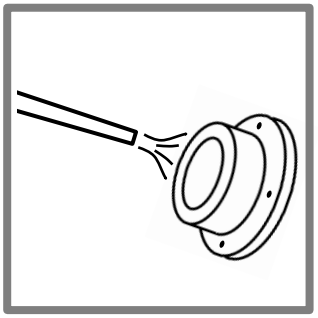
1. After retrieving the DGT device from the deployment environment thoroughly rinse the DGT device with ultrapure water.



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



3. For analytes at trace concentrations, all sample treatment should be carried out in a laminar flow hood to avoid contaminating the sample.



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

DGT Research Ltd
Skelmorlie, Bay Horse Rd, Quernmore, Lancaster, Lancashire, LA2 0QJ
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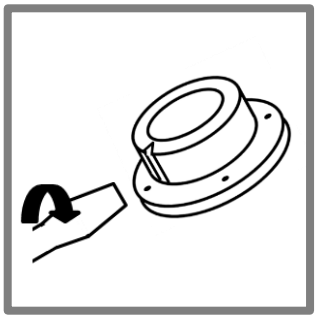
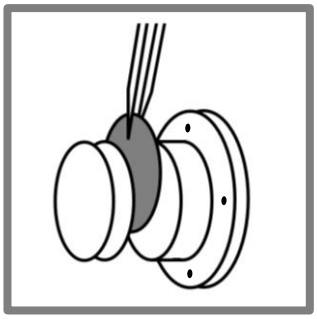
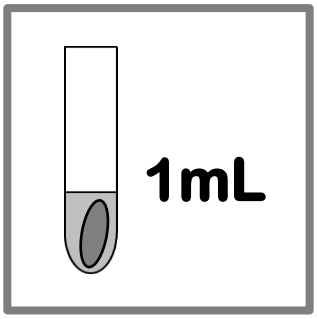
Email : info@dgtresearch.com



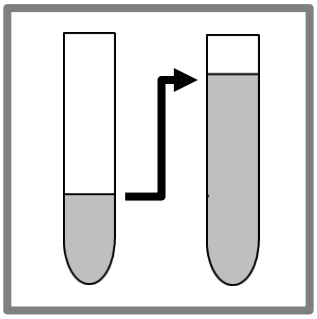
5. To retrieve the resin gel after deployment, insert a suitable screwdriver into the groove in the cap and twist it. The cap will be broken at the weak point.



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

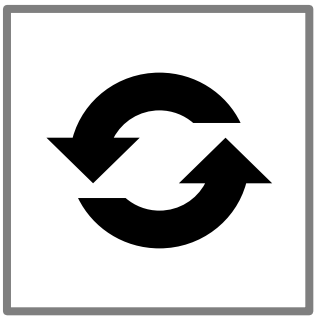


7. For Total Hg, place the binding layer in a glass vial/ or Teflon vial/ or High-Density Polyethylene vial containing 1 ml of Hg-free aqua regia and shake overnight. Or analyse directly with AMA-254 (Advance Mercury Analyser-254)

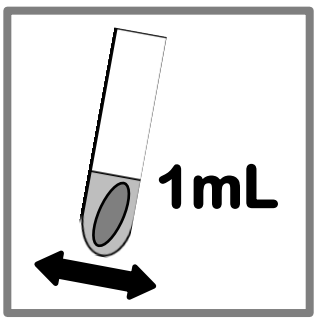


8. Remove the binding layer from the tube, squeezing out any residual liquid. Then dilute the eluate by adding 9 mL of MilliQ water (depending on the concentration levels of Hg) prior to analysis using Cold-Vapor AFS or ICPMS.

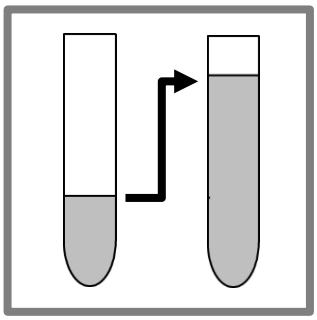
DGT Recovery and Sample Treatment for MeHg



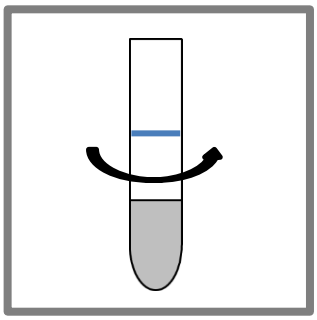
1a. Repeat the above steps 1 – 6.



2. Extracte the resin gel with 1 mL 6 M HNO_3 in a glass vial for 6 hours at 70 °C.



9. After cooling down to room temperature, add 9 mL of MilliQ water.



10. Centrifuge the eluate at 3000 rpm for 10 min, then keep the supernatant for further analysis using for example GC-AFS (in this case, ethylation of the supernatant is necessary).

Analysis for total Hg

Typically, analysis of the total Hg for this type of DGT can be performed by Advanced Mercury Analyzer-254 (AMA-254) with direct combustion of resin gels (no elution needed) or by inductively coupled plasma mass spectrometer (ICP-MS) after acid extraction as outlined above (aqua regia is recommended, however other strong acid extractions can work too). To control the quality of the analysis, NIST 2704 reference should be used for total Hg.

Analysis for MeHg

Analysis of Methylmercury (MeHg) is highly depended on availability of instruments. If the lab is equipped with HPLC-ICP-MS, different Hg species can be extracted (acid extraction) from the resin gels and Hg speciation can be carried out with HPLC-ICP-MS. Or if the lab is equipped with a specific MeHg analyzer, then the DGT user is recommended to use their own extraction protocols for MeHg (for example, extraction of MeHg from sediment sample, extraction of MeHg from biological sample), which will normally work for the extraction of MeHg from our DGT resin gels. Then the analysis can be carried out with their own MeHg analyzer.

Method and field blanks

To ensure accurate results it is recommended to determine DGT laboratory[1] and field blank[2] concentrations.

[1]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.

[2]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 and a 10x dilution of the eluent prior to analysis, there is some agreement that appropriate elution efficiencies are 1.0 for total Hg and MeHg for the elution method with analysis by ICP-MS.

Calculation

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

$$c_{\mathrm{DGT}} = \frac{M\Delta_{\mathrm{g}}}{D^{\mathrm{mdl}}A_{\mathrm{p}}t}$$

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 below)
Δ_g (also known as δ_g) (0.094 cm) 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 (taken from Diffusion Coefficients under the What DGT Does heading).
A_p (3.14 cm^2) 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.

The mass, M, of analyte on the binding layer, of volume, V^{bl}(usually 0.2 mL), is calculated from the measured concentration of analyte, c_e, in the eluent, of volume V_e, remembering to take into account any subsequent dilution. f_e is the elution factor.

$$M = \frac{c_{\mathrm{e}}\left(V^{\mathrm{bl}} + V_{\mathrm{e}}\right)}{f_{\mathrm{e}}}$$

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 these analytes:

Y. Gao, S. D. Craemer and W. Baeyens, A novel method for the determiniation of dissovled methylmercury concentrations using diffusive gradients in thin films technique, *Talanta*, **120**: (2014), 470-474.

O. Clarisse and H. Hintelmann, Measurement of dissolved methylmercury in natural waters using diffusive gradients in thin film (DGT), *J. Environ. Monit.*, **8**: (2006), 1242-1247.

A. Bratkic, K. Klun, Y. Gao, Mercury speciation in various aquatic systems using passive sampling technique of diffusive gradients in thin-film, *Science of the Total Environment,* **663**: (2019), 297-306.

M. Reichstadter, P. Divis, E. Abdulhur-Alfakhoury, Y. Gao, Simultaneous determination of mercury, cadmium and lead in fish sauce using Diffusive Gradients in Thin-Films technique, , *Talanta*, **217**: (2020), 121059.

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.