Practical guide to the use of EasySensor DGT products in sediments

1. Principle of DGT

Please refer to "Introduction to EasySensor DGT products".

2. Assembly of the flat-type DGT probe

A new flat-type DGT holder configuration has been developed for deployment in sediments (Ding *et al.,* 2016). The flat-type probe has an exposure window (150 mm length and 20 mm width) with an effective area of 30 cm^2. The surface of the device exposed to the sediment is in a flat plane, which minimizes the disturbance and mobilization of dissolved or particulate substances caused by insertion of the probe into the sediment. Please refer to "Introduction to EasySensor DGT products" for detailed information on the composition and assembly of DGT probe devices.

References:

Ding SM, Wang Y, Zhang LP, et al. New holder configurations for use in the diffusive gradients in thin films (DGT) technique. RSC Advances, 2016, 6(91): 88143-88156.

3. Storage of DGT devices

The loaded DGT device should be stored at ambient temperatures of >5 °C to < 40 °C, with AgI and ZrO-AgI DGT devices requiring storage under dark conditions. Unpacked DGT probes are generally sealed in a clean plastic bag containing a small volume (~0.2 mL) of 0.01 M NaCl solution, to maintain moisture and ionic-strength conditions, with regular checking to ensure that the moisture level is maintained throughout the storage period.

To avoid the contamination of the DGT probe, please do NOT open the package to expose the DGT probe to air prior to use.

Prior to use, the DGT probe should filled with nitrogen gas for more than 16 hours for deoxidization.

4. DGT Deployment in sediments or wetland environments

4.1 *Ex situ* deployment (laboratory use)

The deoxidized device should be vertically and slowly inserted into the sediment, leaving 2 to 4 cm length remaining in the overlying water (with an effective length of 15cm). The probe is generally deployed for 24 h. The water temperature must be recorded.

4.2 On-site deployment (field-study use)

Method of marking the sediment-water interface:

When performing *in situ* deployment in the field, the sediment particles adhering on the filter membrane of the DGT probe can be easily removed when withdrawing the probe from the sediment. As a consequence, it can be hard to identify the sediment-water interface (SWI) using the existing DGT probe (Ding *et al.,* 2015).

A new method has been developed to overcome this shortage. Attaching a sheet of sponge on the back of the probe, provides a clear SWI mark, as when the probe is inserted into sediments, particles become embedded in the numerous small holes on the surface of the sponge. In addition, a sheet of plastic membrane can be fixed on the top of the probe prior to deployment, to be suspended in the overlying water after the probe is inserted into the sediment. Once the probe is withdrawn from the sediment, the membrane immediately covers both the front and back sides of the probe, preserving the SWI mark from water flow impact during withdrawal of the probe (Ding *et al.,* 2015).

References:

Ding SM, Han C, Wang YP, et al. In situ, high-resolution imaging of labile phosphorus in sediments of a large eutrophic lake. Water Research, 2015, 74(0): 100-109.

Deployment procedure

For deployment in shallow lakes or wetlands, the DGT probe can be inserted in sediments by hand. In areas where the water is deep, a releasing device or a diver is required for probe placement. Here are the recommended procedures for probe deployment using a releasing device (to be purchased separately).

1) Fasten a fishing line through the holes in the hand shank of the DGT probe. Attach the probe to the bottom of the releasing device with the fishing line.

2) Fasten a rope to the top of the releasing device. Raise the loaded-probe handle by dragging both the fishing line and the releasing device together and place the device with the DGT probe into the water column vertically. The releasing device will reach the surface of the sediment when the rope is no longer stressed, at which point, the DGT probe will have been smoothly pushed into the sediment using the gravity of the releasing device.

3) Loosen the fishing line, leaving the DGT probe in the water. Retrieve the releasing device from the water column by drawing the rope. Tie a buoy to the end of the fishing line and leave it floating on the water surface.

4) Record the position of the deployment site using GPS, as well as the deployment time, water temperature and water depth, along with any other required parameters.

The DGT probe is generally deployed for 24 h. The temperature should be recorded at regular intervals throughout the deployment period.

5. DGT Retrieval

5.1 *Ex situ* deployment (Laboratory experiment)

Retrieve the DGT probe by drawing it out from the sediment by hand. Mark the SWI on the surface of the window frame using an indelible marker pen. Rinse the surface of the DGT probe with deionized water. Retrieve the binding gel and carefully place it in a clean sealable bag. A small amount of deionized water should be added in the bag to maintain moisture levels. Seal the bag and store it at room temperature until analysis. Record the water temperature at the point of withdrawal.

5.2 On-site deployment

Locate the DGT probe according to the GPS and buoy position and retrieve the DGT probe by drawing the fishing line up through the water column. Mark the SWI position on the surface of the window frame using an indelible marker pen, according to the mud print on the sheet of sponge attached to the back of the probe. Retrieve the binding gel and carefully place it in a clean sealable bag. A small amount of deionized water should be added in the bag to maintain moisture levels. Seal the bag and store it at room temperature until analysis. Record the water temperature at the point of withdrawal.

6. Sample analysis and date treatment

6.1 Measurement of phosphate

Measurement of phosphate (P) is performed using Zr-oxide DGT. The accumulated mass of P in the Zr-oxide gel after DGT retrieval, can be determined using two methods. Firstly, P can be eluted using 1.0 M NaOH and detected using the molybdenum blue method (Ding *et al.,* 2010). Secondly, the degree of surface coloration of the Zr-oxide binding gel can be assessed using computer imaging densitometry (CID) (Ding *et al.,* 2013).

6.1.1 Slice-extraction-detection

1) Retrieve the Zr-oxide gel strip from the DGT device and rinse the gel surface with a small amount of ultrapure water.

2) Cut the Zr-oxide gel strip according to the procedure outlined below:

   A) Put the ceramic cutter (to be purchased separately) on a board, with the cutting edge facing up. The spatial resolution of the cuts can be set from 1.0 mm to 5.0 mm by adjusting the interval between the adjacent ceramic blades.

   B) Place the gel strip (150 × 20 mm as standard) on the cutter surface, with the Zr-oxide settled side facing down. Cover the surface of the gel strip with a thin and clean plastic membrane.

   C) Press the gel vertically by hand until the whole gel areas is within the grooves of the cutter.

3) Remove each gel slice from the cutter grooves individually, using sterile plastic tweezers. Put each slice into a separate tube (typically a 1.5 ml centrifuge tube is used for a slice with a 1-2 mm width).

4) Add 1.0 M NaOH to the tube to elute P from the gel slice. A volume of 0.4 mL is recommended for a 1.0 mm gel slice.

5) Leave the tube to stand for 24 h at room temperature, then remove gel residues and collect the elution.

6) Micro-colorimetric determination of P concentrations in the eluent can be performed using a 96-microwell plate spectrophotometer, according to the method of Xu *et al.,* (2012):

A) Withdraw a volume of elution solution and add to a micro-well of a 96-microwell plate, then add 2.0 M H_2SO_4 (add a v:v ratio of 1:4 for H_2SO_4:elution solution) to neutralize the eluent solution. Add deionized water to reach a total volume of 200 μL and then add 20 μL mixed reagent (preparation of the mixed reagent is described below).

B) Place the microwell plate on an oscillator and agitate at 35 °C for 45 min.

C) Read the absorbance at 700 nm using a microtiter plate spectrophotometer.

Preparation of the mixed reagent:

20 g ammonium molybdate and 0.5 g potassium antimonyl tartrate should be dissolved in a volume of 805 ml deionized water. The solution should be slowly mixed with 194.6 ml concentrated H_2SO_4 and stored in a glass bottle under room temperature conditions. The mixed reagent used for micro-colorimetric determination of P, should be freshly prepared by dissolving 1.5 g ascorbic acid into 100 ml of the mixed reagent solution. All reagents used to prepare solutions should be of analytical grade.

7) Calculate the measured DGT concentration according to the procedure below:

A. Calculate the mass of P in the Zr-oxide gel (M) using Eq. (1) (Zhang *et al.,* 2014).

$$M = \frac{C_e(V_e + V_g)}{f_e} \qquad (1)$$

Where C_e is the P concentration in the 1.0 M NaOH eluent solution (μg mL^{-1}); V_e is the volume of 1.0 M NaOH added to the Zr-oxide gel; V_g is the volume of the Zr-oxide gel; f_e is the elution efficiency for P (0.95) (Ding *et al.,* 2010).

B. Calculate the DGT measured concentration (C_{DGT}) using Eq. (2) (Zhang *et al.,*

2014).

$$C_{DGT} = \frac{M\nabla g}{DAt} \quad (2)$$

Where Δg is the thickness of the diffusive layer (typically comprising the diffusive gel and the filter membrane) (cm); *D* is the diffusion coefficient of P in the diffusive layer (cm^2 sec^{-1}); *t* is the deployment time (sec); and A is the exposure area (cm^2).

For the diffusion coefficients (*D*) for P refer to "Parameters of EasySensor DGT Products".

References:

Ding SM, Xu D, Sun Q, et al. Measurement of dissolved reactive phosphorus using the diffusive gradients in thin films technique with a high-capacity binding phase. Environmental Science & Technology, 2010, 44(21): 8169-8174.

Zhang CS, Ding SM, Xu D, et al. Bioavailability assessment of phosphorus and metals in soils and sediments: a review of diffusive gradients in thin films (DGT). Environmental Monitoring and Assessment, 2014, 186 (11), 7367-7378.

Xu D, Wu W, Ding SM, et al. A high-resolution dialysis technique for rapid determination of dissolved reactive phosphate and ferrous iron in pore water of sediments. Science of the Total Environment, 2012, 421-422(0), 245-252.

6.1.2 Gel coloration procedure for obtaining high spatial resolution information

Principle:

This technique allows two-dimensional and high-resolution determination of P by combining Zr-oxide gel surface coloration with computerized image density (CID) measurement. A blue

complex is formed on the surface of Zr-oxide gel, based on the principles of the phospho-molybdenum blue method. The calibration curve should be established by integrating the gray values obtained by scanning, with the accumulated amounts of P in the gel, allowing the distribution of DGT-measured P to be obtained at a sub-millimeter scale (Ding *et al.,* 2013).

Main instruments:

Scanner (Canon-5600F), thermostat water bath.

Main reagents:

KH_2PO_4 stock solution (100 mg L^{-1}), ammonium molybdate [$(NH_4)\ 6Mo_7O_{24}.4H_2O$], antimony potassium tartrate [$K(SbO)C_4H_4O_6 \cdot 1/2H_2O$], ascorbic acid.

Preparation of the mixed reagent used for gel coloration:

The mixed reagent used for coloration should be prepared by separately dissolving 20 g ammonium molybdate tetrahydrate and 0.5 g potassium antimonyl tartrate into 100 mL of deionized water each. The two solutions should then be slowly mixed and combined with 194.6 mL concentrated sulfuric acid. After cooling to room temperature, the mixed solution should be diluted to a volume of 1000 mL using deionized water, forming a stock solution that can be stored in a brown bottle at ambient temperatures for 3 months. Prior to colorimetric analysis, 1.5 g ascorbic acid is added to 100 mL of the mixed solution, which is then mixed with 1000 mL deionized water pre-heated to 35 °C (the temperature required for color development). The final mixed solution should contain 0.113 M MoO_4^{2-} and 8.6 mM Vc^-, with a pH of 0.48. The final mixed reagent solution used for colorimetric determination should be prepared fresh prior to analysis and used within 2 hours.

Determination Procedure

1) Retrieve the Zr-oxide gel from the DGT probe.

2) Put the gel strip into a plastic container with the Zr-oxide settled side facing upwards. Add mixed reagent at a volume of 200-fold that of the gel, ensuring that the mixed reagent is stabilized at 35±1°C before addition. 300mL mixed reagent is the recommended volume.

3) Leave the container to stand in a water bath at 35°C for 45 min.

4) Retrieve the gel strip and rinse it using cool deionized water pre-stored in refrigerator at 4 ± 1°C. Then immerse the gel in cool deionized water for at least 5 min to stop the color development.

5) Remove the water adhered to the surface of the gel and scan (using a device such as a Canon Canoscan 5600F Scanner) the surface of the Zr-oxide settled side of the gel, at a defined resolution (typically 150 to 600 dpi, corresponding to a pixel size of 169 × 169 μm^2 to 42 × 42 μm^2).

6) Obtain the grayscale intensity of the scanned images using imaging software (such as ImageJ downloaded from http://rsbweb.nih.gov/ij).

7) Calculate the mass of P in the gel using the established calibration equation shown in Eq. (3) below:

$$y = -177e^{-x/4.46} + 223 \qquad (3)$$

Where y is the grayscale intensity; and x is the mass of P (μg cm^{-2})

8) Calculate the DGT measured concentration using Eq. (2).

Attention:

The coloration procedure has been simplified from the method originally reported (Ding et al., 2013). In the original procedure, pre-treatment of the gel in hot water (85 °C) for 5 days was required prior to coloration. This pre-treatment step can be discarded in the revised procedure

described above, as the preparation of the Zr-oxide binding gel has been revised to enable smaller size Zr-oxide particles and a more even distribution.

Temperature is a vital factor influencing gel coloration. It must be controlled strictly throughout the process. Ensure that the Zr-oxide settled side faces upwards before addition of the mixed agent.

References:

Ding SM, Wang Y, Xu D, et al. Gel-Based Coloration Technique for the Submillimeter-Scale Imaging of Labile Phosphorus in Sediments and Soils with Diffusive Gradients in Thin Films. Environmental Science & Technology, 2013, 47(14): 7821-7829.

6.2 Measurement of inorganic arsenic

Measurement of total inorganic As is performed using Zr-oxide DGT. The accumulated inorganic As (As(III) and As(V)) can be eluted using NaOH, with analysis of As in the eluent performed using a HG-AFS or an ICP-MS (Sun *et al.,* 2014).

1) Retrieve the Zr-oxide gel strip from the DGT probe and rinse the gel surface with a small amount of deionized water.

2) Cut the Zr-oxide gel strip according to 6.1.1.

3) Retrieve each gel slice individually from the cutter grooves using plastic tweezer and place each slice into a tube (typically a 1.5 ml centrifuge tube is used for a 1-2 mm width slice).

4) Add 1.0 M NaOH (for freshwater matrix) or a mixed solution containing 1.0 M NaOH and 1.0 M H_2O_2 (1.0 M NaOH-1.0 M H_2O_2) (for seawater matrix) into the tube. A volume of 0.4 mL is recommended for a 1.0 mm gel slice.

5) Leave the tubes to stand for 24 h at room temperature, then remove gel residues.

6) Quantification of As concentrations in the eluent solution can be performed using a HG-AFS or an ICP-MS, after appropriate dilution.

7) Calculation of the DGT measured concentration (C_{DGT}) can be done using Equ. (1) and (2). For the efficiency and diffusion coefficient for As refer to "Parameters of EasySensor DGT Products".

References:

Sun Q, Chen J, Zhang H, et al. Improved diffusive gradients in thin films (DGT) measurement of total dissolved inorganic arsenic in waters and soils using a hydrous zirconium oxide binding layer. Analytical Chemistry, 2014, 86(6): 3060-3067.

6.3 Simultaneous measurement of eight oxyanions

Simultaneous measurement of eight anions **(**P(V), As(V), Cr(VI), Mo(VI), Sb(V), Se(VI), V(V) and W(VI)) can be performed using Zr-oxide DGT. The accumulated mass of oxyanions in the Zr-oxide gel can be simultaneously eluted using a mixed solution containing 0.2 M NaOH and 0.5 M H_2O_2 (Ding *et al.,* 2016).

1) Retrieve the Zr-oxide gel from the DGT probe and rinse the gel surface with a small amount of deionized water.

2) Cut the Zr-oxide gel strip according to 6.1.1.

3) Remove out each gel slice individually from the cutter grooves, using plastic tweezers. Put each slice into a tube (typically a 1.5 ml centrifuge tube is used for a 1-2 mm width slice).

4) Add the mixed solution of 0.2 M NaOH and 0.5 M H_2O_2 into reaction tubes, with a volume of 0.4 mL recommended for a 1.0 mm gel slice. The mixed solution should have been stabilized in a refrigerator at 4 °C prior to use, with continued storage of

the gel-loaded tubes at 4 °C. The elution time should be controlled to within 3-5 hours.

5) The concentration of P in the eluent solution can be quantified using the micro-colorimetric method (See 6.1.1). The concentrations of other oxyanions can be established by ICP-MS after appropriate dilution.

6) Calculate the DGT concentration (C_{DGT}) using Eq. (1) and (2). For the elution efficiency and diffusion coefficients for the eight oxyanions, refer to "Parameters of EasySensor DGT Products".

Attention:

The mixed solution of 0.2 M NaOH and 0.5 M H_2O_2 should be prepared fresh daily for analysis. The main stock solutions (NaOH and H_2O_2) and deionized water must be stabilized at 4 °C prior to mixing. The eluent solution should be diluted using deionized water or HNO_3 (1-3%) immediately after elution, to limit precipitate formation in the elution solution. The degree of dilution should be determined by micro-colorimetric and ICP-MS methods.

References:

Ding SM, Xu D, Wang YP, et al. Simultaneous measurements of eight oxyanions using high-capacity diffusive gradients in thin films (Zr-oxide DGT) with a high-efficiency elution procedure. Environmental Science & Technology, 2016, 50(14): 7572-7580.

6.4 Simultaneous measurement of eight metals

Measurement of eight metals (Fe(II), Cd(II), Co(II), Cu(II), Mn(II), Ni(II), Pb(II) and Zn(II)) can be performed using Chelex DGT. The accumulated mass of metals in the Chelex gel can be eluted using 1.0 M HNO_3, followed by determinations and quantification of the metals using colorimetric or ICP-MS methods (Wang *et al.,* 2016).

1) Retrieve the Chelex gel from the DGT probe and rinse the gel surface with a small amount of deionized water.

2) Cut the Chelex gel strip according to 6.1.1.

3) Retrieve each gel slice from the cutter grooves individually, using plastic tweezers. Place each slice into a tube (typically a 1.5 ml centrifuge tube is used for a 1-2 mm width slice).

4) Add 1.0 M HNO_3 to each tube, with a volume of 0.4 mL recommended for a 1.0 mm gel slice.

5) Leave the tubes to incubate for 16 hours at room temperature. Remove the gel residues and collect the elution.

6) Fe concentrations in the eluent solution can be detected using the phenanthroline colorimetric method after appropriate dilution as follows (Xu *et al.,* 2013):

   A. Transfer the appropriate volume of eluent solution into a micro-well of a 96-microwell plate, dilute with deoxygenated water to reach a total volume of 100 μL. Then add 100 μL mixed reagent and 10 μL reducing agent.

   B. Place the microwell plate on an oscillator and agitate at 35°C for 30 min.

   C. Read the absorbance at 520 nm using a microtiter plate (such as an Epoch Microplate Spectrophotometer, BioTek, USA)

7) Concentrations of the other seven elements can be established by ICP-MS after appropriate dilution.

8) Calculate the DGT concentration (C_{DGT}) using Eq. (1) and (2). For the elution efficiency and diffusion coefficients for the eight metals, refer to “Parameters of EasySensor DGT Products”.

References:

Wang Y, Ding SM, Gong MD, et al. Diffusion characteristics of agarose hydrogel used in diffusive gradients in thin films for measurements of cations and anions. Analytica Chimica Acta, 2016, 945: 47-56.

Xu D, Chen YF, Ding SM, et al. Diffusive gradients in thin films technique equipped with a mixed binding gel for simultaneous measurements of dissolved reactive phosphorus and dissolved iron. Environmental Science & Technology, 2013, 47(18): 10477-10484.

6.5 Measurement of S(-II) and simultaneous measurement of S(-II) and P

Measurement of S(-II) and the simultaneous measurement of S(-II) and P, can be performed using AgI DGT and ZrO-AgI DGT, respectively. The accumulated concentration of S(-II) in the gel can be determined directly using computer-imaging densitometry (CID). The accumulated concentration of P in the gel can be eluted using NaOH according to 6.1.1.

1) Retrieve the binding gel from the DGT device and rinse the gel surface with a small amount of deionized water.

2) Remove the water adhered on the surface of the gel and scan (using a scanner such as a Canon 5600F) the surface of the AgI settled side of the gel, at a defined resolution (typically 150 to 600 dpi, corresponding to a pixel size of 169 × 169 μm^2 to 42 × 42 μm^2).

3) Obtain the grayscale intensity of the scanned images using imaging software (such as ImageJ downloaded from http://rsbweb.nih.gov/ij).

4) Calculate the mass of S in the gel using the established calibration equation shown below in Equ. (4):

$$y = -171e^{-x/7.23} + 220 \quad (4)$$

Where y is the grayscale intensity; and x is the mass of S(-II) (μg cm^{-2}).

5) Calculation of the DGT measured concentration (C_{DGT}) can be performed using Equ. (1) and (2). For the efficiency and diffusion coefficient for S(-II) refer to "Parameters of EasySensor DGT Products".

6) Determination of P should be performed according to 6.1.1.

References:

Ding SM, Sun Q, Xu D, et al. High-resolution simultaneous measurements of dissolved reactive phosphorus and dissolved sulfide: the first observation of their simultaneous release in sediments. Environmental Science & Technology, 2012, 46(15): 8297-8304.

6.6 Simultaneous measurements of 16 elements (metals and oxyanions)

Simultaneous measurement of eight metals (Fe(II), Cd(II), Co(II), Cu(II), Mn(II), Ni(II), Pb(II) and Zn(II)) and eight oxyanions (P(V), As(V), Cr(VI), Mo(VI), Sb(V), Se(VI), V(V) and W(VI)) can be performed using ZrO-Chelex DGT. Cations and anions in the ZrO-Chelex gel can be eluted using the two-step procedure reported by Wang *et al.,* (2017). 1.0 M HNO_3 is first used to extract the cations bound to the gel in a 16-hour extraction, followed by rinsing with deionized water and then the addition of the 0.2 M NaOH and 0.5 M H_2O_2 solution for 3 to 5 hours to elute oxyanions (Wang *et al.,* 2017).

1) Retrieve the gel from the DGT probe and rinse the gel surface with a small amount of deionized water.

2) Cut the gel strip according to 6.1.1.

3) Retrieve each gel slice from the cutter grooves individually, using plastic tweezers. Put each slice into a tube (typically a 1.5 ml centrifuge tube is used for a 1-2 mm

width slice).

4) Add 1.0 M HNO_3 to each tube to elute cations, with a volume of 0.4 mL recommended for a 1.0 mm gel slice.

5) Leave the tubes to stand for 24 hours at room temperature. Collect the eluent and add deionized water into each tube to remove the remaining HNO_3 from the gel.

6) After 2 hours, remove the deionized water and add 0.4mL of the 0.2 M NaOH and 0.5 M H_2O_2 solution to each tube, to elute oxyanions using the same procedure as 6.3.

7) Concentrations of P and Fe are quantified using a Microplate Spectrophotometer as outlined above. Quantification of the other 14 elements can be performed using an ICP-MS.

8) Calculate the DGT concentration (C_{DGT}) using Eq. (1) and (2). For the elution efficiency and diffusion coefficients for the eight metals, refer to "Parameters of EasySensor DGT Products".

References:

Wang Y, Ding SM, Shi L, et al.Simultaneous measurements of cations and anions using diffusive gradients in thin films with a ZrO-Chelex mixed binding layer. Analytica Chimica Acta, 2017, 972: 1-11.

6.7 Simultaneous measurements of 17 elements (metals, oxyanions and S(-II))

Simultaneous measurement of **S(-II) ,** eight metals (Fe(II), Cd(II), Co(II), Cu(II), Mn(II), Ni(II), Pb(II) and Zn(II)) and eight oxyanions **(**P(V), As(V), Cr(VI), Mo(VI), Sb(V), Se(VI), V(V) and W(VI)) can be performed using ZrO-Chelex-AgI DGT. The accumulated concentration of S(-II) in the gel can be determined directly using

computer-imaging densitometry (CID). Cations and anions in the ZrO-Chelex-AgI gel can be eluted using the two-step procedure reported by Wang *et al.,* (2017). After scanning the gel, 1.0 M HNO_3 is first used to extract the cations bound to the gel in a 16-hour extraction, followed by rinsing with deionized water and then the addition of the 0.2 M NaOH and 0.5 M H_2O_2 solution for 3 to 5 hours to elute oxyanions (Wang *et al.,* 2017).

1) Retrieve the gel from the DGT probe and rinse the gel surface with a small amount of deionized water.

2) Remove the water adhered on the surface of the gel and scan (using a scanner such as a Canon 5600F) the surface of the AgI settled side of the gel, at a defined resolution (typically 150 to 600 dpi, corresponding to a pixel size of 169 × 169 μm^2 to 42 × 42 μm^2).

3) Obtain the grayscale intensity of the scanned images using imaging software (such as ImageJ downloaded from http://rsbweb.nih.gov/ij).

4) Cut the gel strip according to 6.1.1.

5) Retrieve each gel slice from the cutter grooves individually, using plastic tweezers. Put each slice into a tube (typically a 1.5 ml centrifuge tube is used for a 1-2 mm width slice).

6) Add 1.0 M HNO_3 to each tube to elute cations, with a volume of 0.4 mL recommended for a 1.0 mm gel slice.

7) Leave the tubes to stand for 24 hours at room temperature. Collect the eluent and add deionized water into each tube to remove the remaining HNO_3 from the gel.

8) After 2 hours, remove the deionized water and add 0.4mL of the 0.2 M NaOH and 0.5 M H_2O_2 solution to each tube, to elute oxyanions using the same procedure as

6.3.

9) Calculate the mass of S according to 6.5.

10) Calculate the concentration of cations and oxyanions according to 6.6.

References:

Wang Y, Ding SM, Ren MY, et al. Enhanced DGT capability for measurements of multiple types of analytes using synergistic effects among different binding agents. Science of the total environment, under review.

6.8 Simultaneous measurements of 15 rare earth elements (REE)

Simultaneous measurement of 15 REEs (La (III), Ce (III), Pr (III), Nd (III), Sm (III), Eu (III), Gd (III), Tb (III), Dy (III), Ho (III), Er (III), Tm (III), Yb (III), Lu (III), and Y (III)) can be performed using Chelex DGT.

1) Retrieve the gel from the DGT probe and rinse the gel surface with a small amount of deionized water.

2) Cut the Chelex gel strip according to 6.1.1.

3) Retrieve each gel slice from the cutter grooves individually, using plastic tweezers. Place each slice into a tube (typically a 1.5 ml centrifuge tube is used for a 1-2 mm width slice).

4) Add 2.0 M HNO_3 to each tube, with a volume of 0.4 mL recommended for a 1.0 mm gel slice.

5) Leave the tubes to incubate for 24 hours at room temperature. Remove the gel residues and collect the elution.

6) Detection: Concentrations of REEs can be established by ICP-MS after appropriate

dilution.

7) Calculate the DGT concentration (C_{DGT}) using Eq. (1) and (2). For the elution efficiency and diffusion coefficients for the eight metals, refer to "Parameters of EasySensor DGT Products".

References:

Yuan YM, Ding SM, Wang Y, et al. Simultaneous measurement of fifteen rare earth elements using diffusive gradients in thin films. Analytica Chimica Acta, 2018, 1031, 98-107.

6.9 Simultaneous measurements of Hg^{2+} and CH_3Hg^+

Simultaneous measurement of Hg^{2+} and CH_3Hg^+ can be performed using Tulsion® CH-95 DGT.

1) Retrieve the gel from the DGT probe and rinse the gel surface with a small amount of deionized water.

2) Cut the binding gel strip according to 6.1.1.

3) Retrieve each gel slice from the cutter grooves individually, using plastic tweezers. Place each slice into a tube (typically a 1.5 ml centrifuge tube is used for a 1-2 mm width slice).

4) Stable elution efficiencies were obtained for CH_3Hg^+ and Hg^{2+} using a mild reagent containing 0.1 M HCL and 2% thiourea. Add the mixed solution to each tube, with a volume of 0.4 mL recommended for a 1.0 mm gel slice.

5) Leave the tubes to incubate for 24 hours at room temperature. Remove the gel residues and collect the elution.

6) Detection: CH_3Hg^+ and Hg^{2+} were measured using cold vapor atomic fluorescence spectrometry (CV-AFS). Two standard methods, EPA 1630 and EPA 1631, were

applied for the analysis of the CH_3Hg^+ and total Hg, respectively. The difference between the CH_3Hg^+ and total Hg was used to yield Hg^{2+}.

7) Calculate the DGT concentration (C_{DGT}) using Eq. (1) and (2). For the elution efficiency and diffusion coefficients for the eight metals, refer to "Parameters of EasySensor DGT Products".

References:

Ren MY, Wang Y, Ding SM, et al. Development of a new diffusive gradient in the thin film (DGT) method for the simultaneous measurement of CH_3Hg^+ and Hg^{2+}. New Journal of Chemistry, 2018, 42(10), 7976-7983.