Natural Resources Canada | Ressources naturelles Canada

Epoxy impregnation of unconsolidated marine sediment core subsamples for the preparation of thin sections at the Geological Survey of Canada (Atlantic)

A. Normandeau, O. Brown, K.A. Jarrett, P. Francus, and A. De Coninck

Geological Survey of Canada
Technical Note 10

2019

Canada

Geological Survey of Canada
Technical Note 10

Epoxy impregnation of unconsolidated marine sediment core subsamples for the preparation of thin sections at the Geological Survey of Canada (Atlantic)

A. Normandeau, O. Brown, K.A. Jarrett, P. Francus, and A. De Coninck

2019

ISSN 1914-525X
ISBN 978-0-660-28494-1
Catalogue No. M41-10/10-2019E-PDF
https://doi.org/10.4095/313055

A copy of this publication is also available for reference in depository libraries across Canada through access to the Depository Services Program's Web site at http://dsp-psd.pwgsc.gc.ca.

This publication is available for free download through GEOSCAN (http://geoscan.nrcan.gc.ca).

Recommended citation

Normandeau, A., Brown, O., Jarrett, K., Francus, P., and De Coninck, A., 2019. Epoxy impregnation of unconsolidated marine sediment core subsamples for the preparation of thin sections at the Geological Survey of Canada (Atlantic); Geological Survey of Canada, Technical Note 10, 10 p. https://doi.org/10.4095/313055

Critical review

K. Jenner

Authors

A. Normandeau (alexandre.normandeau@canada.ca)
O. Brown (owen.brown@canada.ca)
K.A. Jarrett (kate.jarrett@canada.ca)
Geological Survey of Canada
1 Challenger Drive
Dartmouth, Nova Scotia
B2Y 4A2

P. Francus (Pierre.Francus@ete.inrs.ca)
A. De Coninck (Arnaud.De_Coninck@ete.inrs.ca)
Institut national de la recherche scientifique
490, rue de la Couronne
Québec, Quebec
G1K 9A9

Correction date:

Epoxy impregnation of unconsolidated marine sediment core subsamples for the preparation of thin sections at the Geological Survey of Canada (Atlantic)

Normandeau, A., Brown, O., Jarrett, K., Francus, P., and De Coninck, A., 2019. Epoxy impregnation of unconsolidated marine sediment core subsamples for the preparation of thin sections at the Geological Survey of Canada (Atlantic); Geological Survey of Canada, Technical Note 10, 10 p. https://doi.org/10.4095/313055

Abstract: Micromorphology is the study of arrangements of particles and matrix and is primarily carried out using thin sections. It is a useful tool to characterize sedimentary facies because material is not disaggregated and the particles are examined in their original position relative to other particles, thus increasing the reliability of sedimentological interpretations. This technical note presents a method for the impregnation of marine sediment core subsamples for the preparation of thin sections. To achieve this, the pore water has to be replaced by an epoxy resin. The method consists of five main steps, described in detail: subsampling preparation; subsampling; sediment dehydration; impregnation; and drying and cutting.

Résumé : La micromorphologie est l'étude de l'arrangement des particules et est principalement effectuée à l'aide de lames minces. Les lames minces sont efficaces pour la caractérisation des faciès sédimentaires puisque les particules sont examinées dans leur position originale par rapport aux autres particules, ce qui augmente la fiabilité des interprétations sédimentologiques. Cette note technique décrit une méthode d'imprégnation d'échantillons de carottes de sédiments marins pour la préparation de lames minces. Pour ce faire, le contenu en eau des sédiments doit être remplacé par une résine époxy. La méthode comporte cinq étapes qui sont décrites en détail : la préparation pour l'échantillonnage; l'échantillonnage; la déshydratation du sédiment; l'imprégnation; et le séchage et le découpage.

INTRODUCTION

Past depositional environments are interpreted from the description of sedimentary facies. Increasingly, high-resolution images of sediment cores are needed to characterize the sedimentary structure, fabric, and grain size of facies. Typical methods for this characterization include X-rays, Computed Tomography (CT) scans, and grain-size analyses. Although X-rays and CT scans allow high-resolution images of sediment cores to be collected, they cannot image at the grain level (Fortin et al., 2013). On the other hand, grain-size analysis is a destructive method that precludes the preservation of structures. Therefore, combining both information into one method is key for a high-resolution characterization of facies. Micromorphology is the study of arrangements of particles and matrix and is primarily carried out using thin sections (van der Meer and Menzies, 2011). It is a useful tool to characterize sedimentary facies because material is not disaggregated, as it is for grain-size analysis, and the particles are examined in their original position relative to other particles, thus increasing the reliability of sedimentological interpretations.

Thin sections are commonly used by sedimentologists studying bedrock outcrops, but are seldom used by sedimentologists studying unconsolidated sediments. One reason for this is that there are few observations linking the microscopic scale to processes (van der Meer and Menzies, 2011). Another reason is that thin sectioning of bedrock samples is straightforward and is done using standard thin-sectioning procedures. Unconsolidated sediments, however, need to be dehydrated and impregnated and the method requires a rigorous time-consuming procedure.

In the past decade, thin sections have been proven particularly useful to limnogeologists for the characterization of laminated sediments, particularly varves (Lamoureux, 1994; Ojala et al., 2012; Francus et al., 2013). In this case, thin sections are important for an accurate representation of grain sizes and for varve counting (Lapointe et al., 2012). In marine settings, thin sections are not as widely used, even though they are very useful for the characterization of turbidite deposits (Fig. 1) (Köng et al., 2016; Normandeau et al., 2017) and other finely laminated sediment (Schimmelmann et al., 2016).

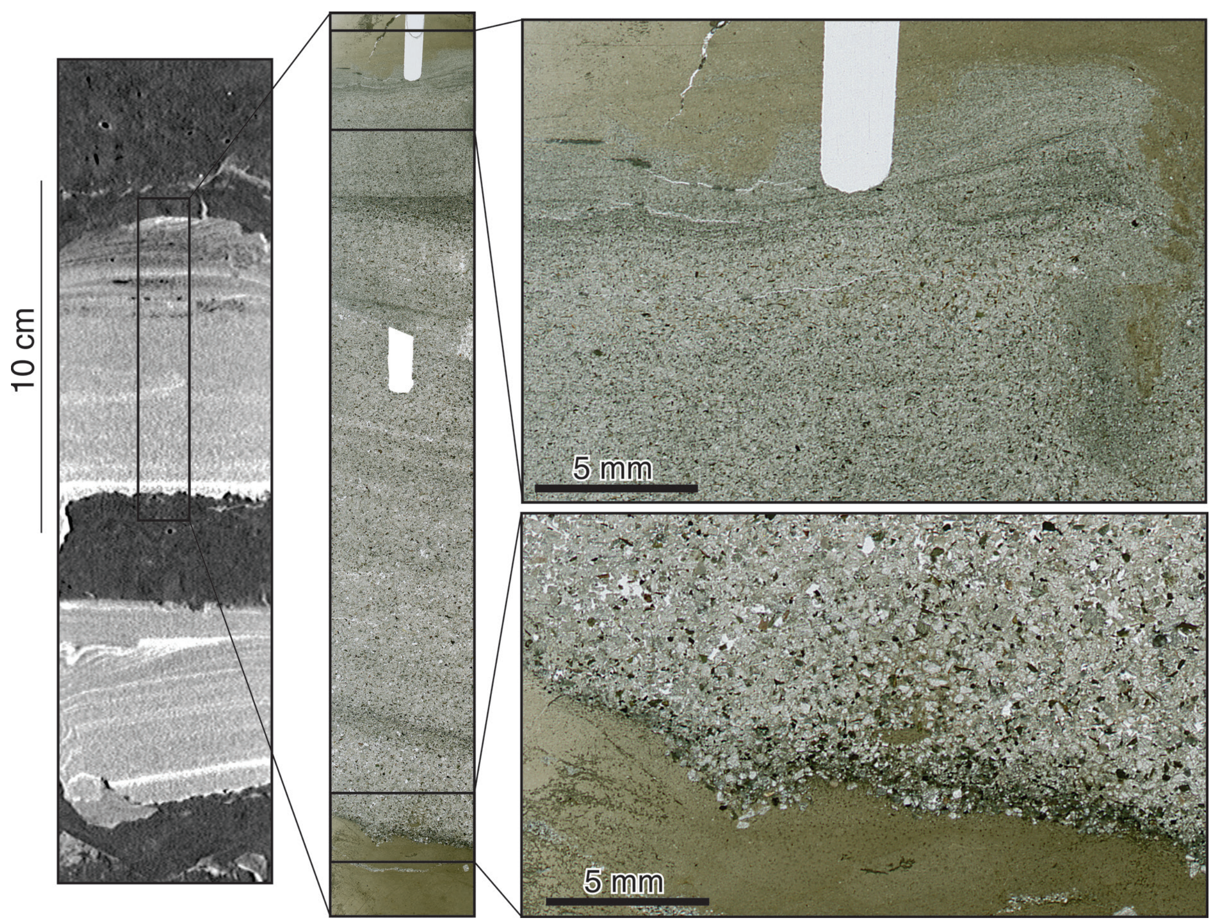

Figure 1. Example of a thin section collected on a river-generated turbidite in the St. Lawrence River estuary (Normandeau et al., 2017). Left image is a medical CT scan image.

This report presents a method for the impregnation of marine sediment core for the preparation of thin sections. This method relies heavily on methods developed at the Institut National de la Recherche Scientifique (INRS) (Quebec), in von Merkt (1971) and Lotter and Lemcke (1999) that were adapted at the Geological Survey of Canada (Atlantic). The preparation of marine sediment core thin sections requires the replacement of the sediment pore water by an epoxy resin. To achieve this, the method consist of five main steps, described in detail below: 1) subsampling preparation; 2) subsampling; 3) sediment dehydration; 4) impregnation; and 5) drying and cutting.

IMPREGNATION METHOD

Subsampling preparation

Sediment cores are subsampled using aluminum boxes (Fig. 2). The type of aluminum is the same as the one used for offset printing (Francus and Asikainen, 2001) and can be purchased at local printing stores. The aluminum is then cut into rectangles of 19.5 cm x 3.5 cm. A manual press, developed by the late E. Karabonov at the University of South Carolina, used at Institut national de la recherche scientifique (INRS) and reproduced at GSC Atlantic, is used to fold the aluminum into boxes with six holes at the bottom that allow epoxy impregnation. Once the aluminum is folded, the ends are cut 0.5 cm to close the boxes (Fig. 2).

The holes made for epoxy impregnation in the boxes can potentially lead to loss of sand-size sediment. To prevent this, a consumer-purchased two-part 5 minute epoxy-based glue (e.g. the authors used Gorilla Brand™) can be used to glue a synthetic mesh (e.g. A.S.T.M. specification nylon monofilament cloth) of fine sand-size to the bottom of the box. The mesh allows the epoxy to penetrate the sediment from the bottom and prevents coarse sediment from falling through the holes. Each box contains six holes and each box can make three separate thin sections.

The next step is to hand-write three separate identifying numbers on the outer part of the box, one for each section. First, an engraving pencil is used to permanently etch the outer surface (Fig. 2g). This etched surface is then overwritten using a permanent marker for easy identification. The permanent marker often wears off during the process, but the

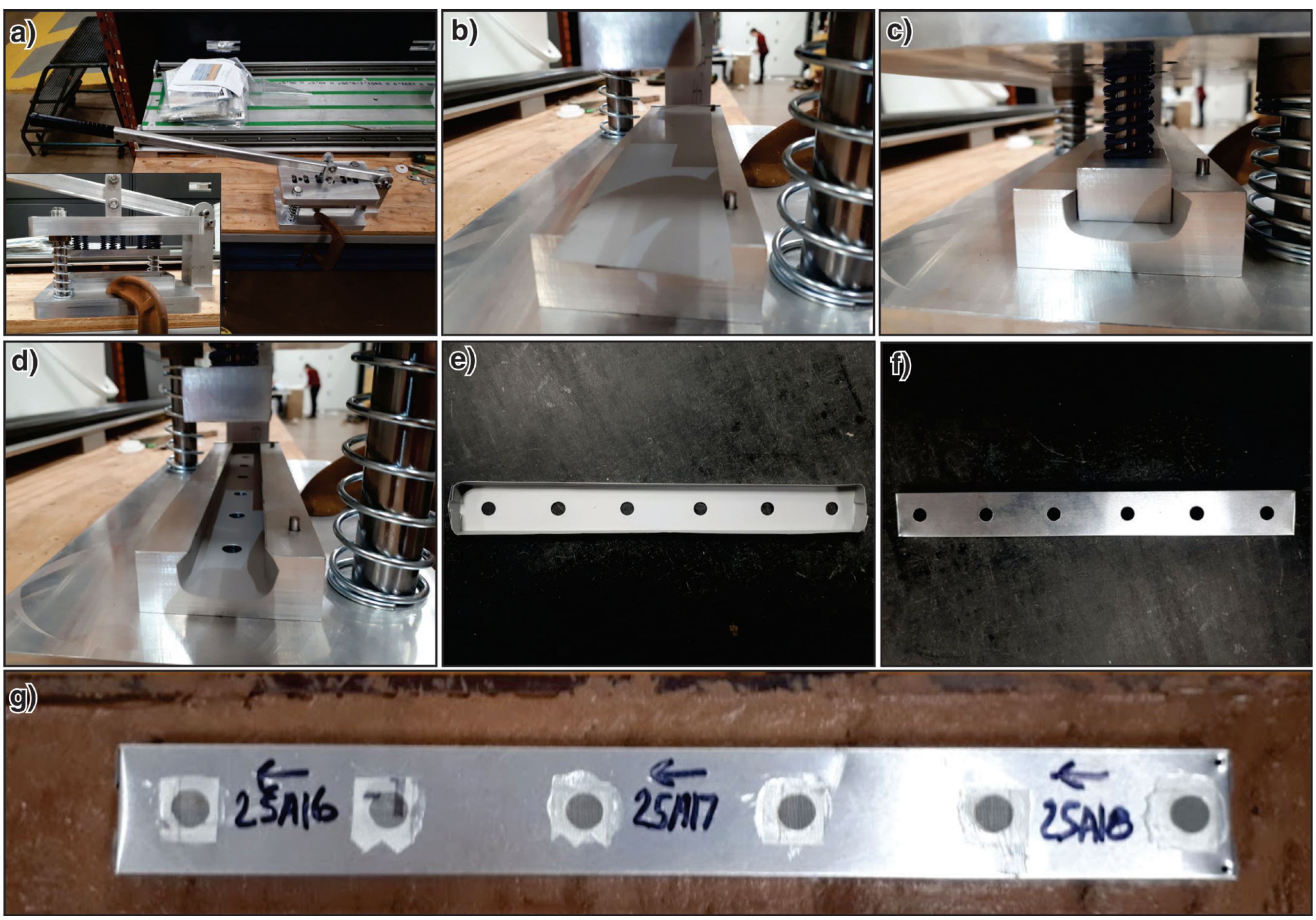

Figure 2. Steps for the preparation of aluminum boxes for subsampling: **a)** manual press, 2018-292, inset 2018-304; **b)**, **c)**, **d)** folding the aluminum box, b: 2018-284, c: 2018-289, d: 2018-301; **e)**, **f)** interior and exterior of aluminum box, e: 2018-294, f: 2018-286; **g)** final box ready for sampling, 2018-306. All photographs by A. Normandeau.

etched identification is permanent. Due to the limited space on the base of the box the identifying labels must be concise. It is recommended that the core number, the first letter of the core section, and a sequential number be used. For example, for core 2016011PHASE1–000**2**PC-**D**E, the label could be: 2D1, 2D2, 2D3, etc. An arrow indicating the top of the core should be drawn and two pin holes at the bottom can help to quickly identify the top of the box (Fig. 2g).

Subsampling

Before placing the aluminum boxes on the split sediment core, the surface of the core needs to be prepared by cleaning and levelling it; companies making the thin sections often take them close to the bottom of the box, which corresponds to the prepared surface of the core. Not preparing the core surface can lead to artifacts within the thin sections. Once prepared, the boxes are placed on the split sediment-core surface at locations of interest for the thin sections. If the continuous characterization of sedimentary facies is important, the boxes should have an overlap of either 1 cm or of a recognizable laminae (Fig. 3b).

The boxes are then gently inserted into the sediment, ensuring that the sides of the boxes enter the sediment without any deformation. Once inserted, a photograph is taken to document the position of the subsamples within the core. The downcore depth of each box is recorded to ensure that the thin section is associated with a specific core and depth.

A cheese-cutter–type tool, designed by Francus and Asikainen (2001), is inserted at the top of each aluminum box and gently pulled downcore (Fig. 3a). Once the tool is passed under the aluminum box containing the sediment, the box is gently moved from side to side (Fig. 4) to prevent resealing of the cut made by the wire of the cheese cutter. The boxes must be constantly checked during removal from the core to ensure that the sediment remains flush with the bottom of the box. Often, this process is difficult because fine sediment within the box sticks to the underlying core despite using the wire cheese cutter to break the sediment seal. If this happens, a folded joint knife can be used to separate the sediment from the core (Fig. 3a, 4).

Once the sediment is removed, the surface of the aluminum boxes are cleaned to remove excess sediment (Fig. 4f), which will also reduce the quantity of epoxy used for the impregnation step. To protect the sediment prior to the next step, the subsamples should be wrapped with plastic wrap.

Sediment dehydration

Fine-grained sediments cannot be air dried because of the risk of sediment shrinking and distorting during the procedure (Tanner and Leong, 1995). Therefore, there are three methods for sediment dehydration: 1) acetone in liquid phase; 2) acetone in vapour phase; and 3) freeze-drying. Both dehydration methods using acetone require weeks to complete and are time-consuming (Lotter and Lemcke, 1999; Zaragosi et al., 2006). Therefore, the authors opted for the freeze-drying technique (lyophilization), a method used by LacCore and INRS. The freeze-drying technique removes the pore water by sublimation; however, the subsamples must first be frozen. Freezing the sediments in a freezer can lead to large hexagonal ice-crystal formation (Francus, 1998). In order to avoid deformation, the sediment subsamples are shock-frozen in liquid nitrogen, which allows the formation of small cubic ice (Boës and Fagel, 2005). If the shock-freezing is too rapid, it can lead to cracks within the sediment structure.

Using personal protective equipment, liquid nitrogen is poured in an enclosed container (Fig. 5). The subsamples are gently lowered into the container until only the base of the subsamples touches the liquid nitrogen and they remain in this position until frost reaches the subsample surface. The subsamples are then completely submerged into the liquid nitrogen. This controlled freeze-drying prevents sediment cracking. The subsamples then remain submerged for a minimum of 2 minutes (or until bubbling slows) and until they are put into the freeze-dryer. At GSC Atlantic, a custom-built drying rack was designed to accommodate up to 15 subsamples at a time (Fig. 6a). Once the subsamples are frozen, they are freeze-dried for a minimum of 48 hours (Fig. 6). Freeze-drying removes the pore water from the pores without changing the sediment microstructure (Lotter and Lemcke, 1999).

Figure 3. a) Tools used to subsample; 2018-290; **b)** boxes placed on a core ready for subsampling. Note the overlapping boxes to have a continuous thin section; 2018-293. **c)** Subsampling using the cheese cutter from top to bottom of core; 2018-307. All photographs by A. Normandeau.

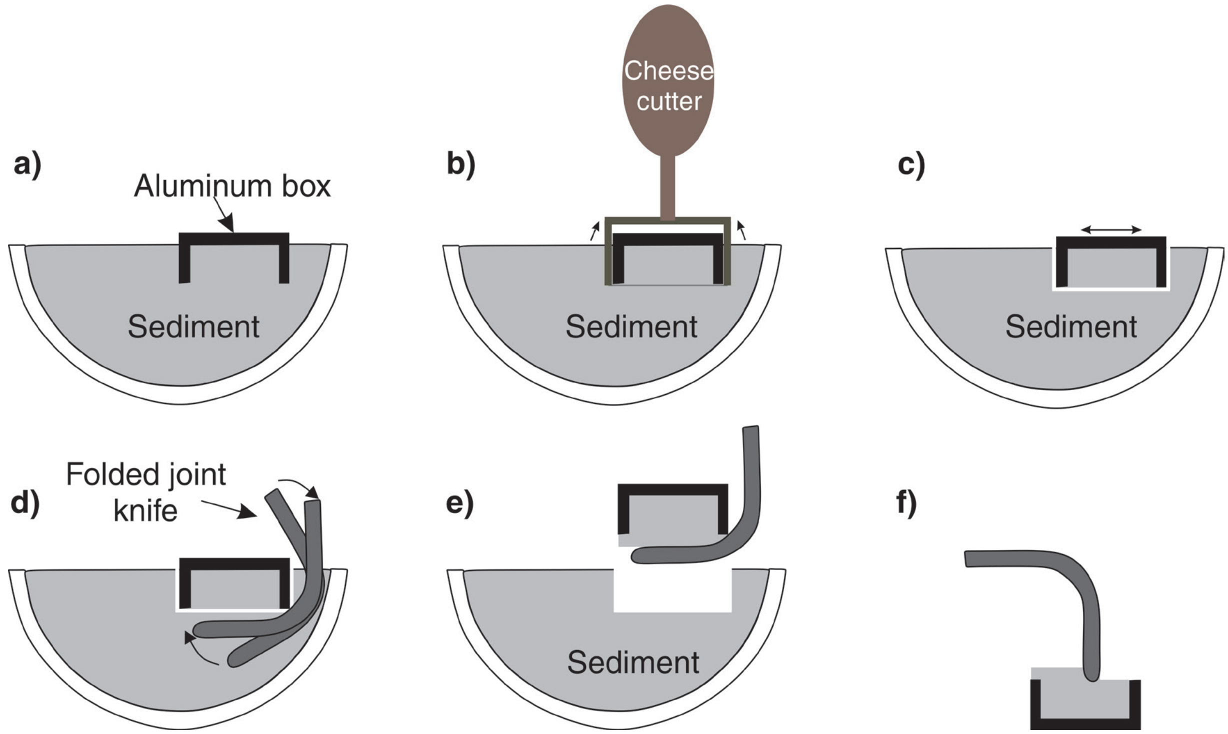

Figure 4. Subsampling of aluminum boxes) using the **b)** cheese-cutter and **d)**, **e)**, folded joint knife sampler (d–e). *Modified from* Lamoureux (1994).

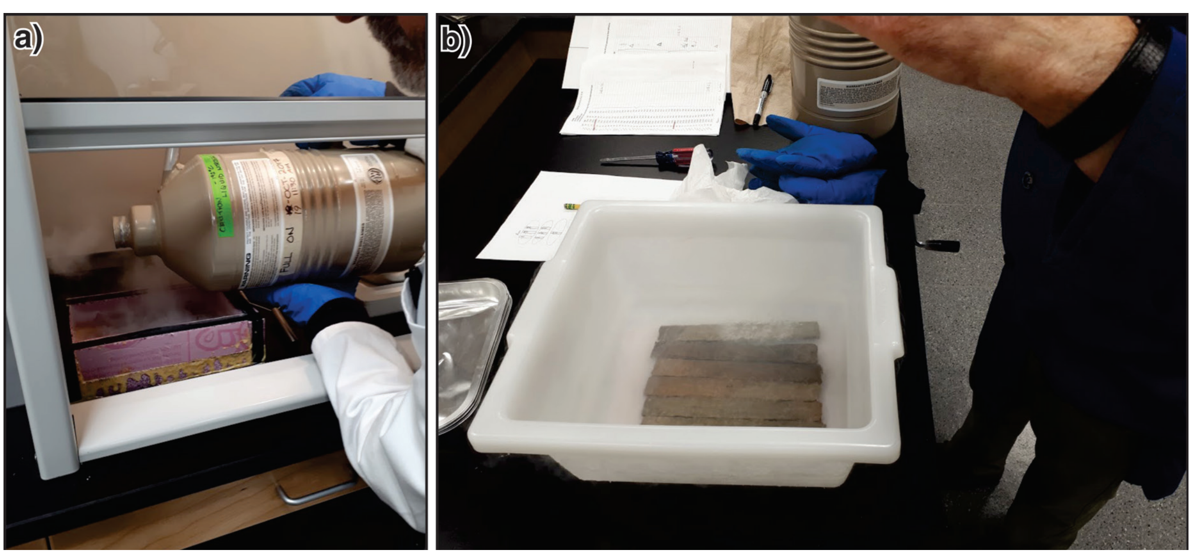

Figure 5. a), **b)** Freezing of subsamples using liquid nitrogen, a: 2018-309, b: 2018-283; photographs by A. Normandeau.

Figure 6. a) Rack built to accommodate up to 15 boxes (45 thin sections) at a time; 2018-285; **b)**, **c)** freeze-dryer used at GSC Atlantic; b: 2018-282, c: 2018-305. All photographs by A. Normandeau.

Impregnation

The impregnation is done using a special low-viscosity and optically clear epoxy resin system (comprised of a resin, hardener, plasticizer, and catalyst available in kit form from SPI Supplies, a Division of EMicron Research, Ltd., London, Ontario). Once the subsamples are completely freeze-dried, they are removed from the freeze-dryer and gently set in aluminum trays. It is critical that the subsamples are completely dehydrated or the impregnation step will not work properly and affect the end result. It is recommended to minimize the time between the removal of the subsamples from the freeze-dryer and the sediment impregnation and to perform the procedure in an environmentally controlled environment. Two types of trays are used at GSC Atlantic: 1) disposable trays (Fig. 7e) and 2) reusable aluminum trays (Fig. 7f). The advantage of using the reusable trays is that once the epoxy is dry, the impregnated aluminum subsample trays are square within the tray and easier to separate from each other once removed from the tray.

The first step after freeze-drying is to put the subsamples in an aluminum tray ensuring that they are elevated from the base, and then putting the tray into a desiccator. The subsamples are elevated, using toothpicks, to allow epoxy impregnation through the holes in the base of the subsample trays. In addition, small spacers are put between the trays to facilitate the cutting and separation process. The epoxy resin components are mixed in the order and using the weight values presented in Table 1. Handling these chemicals requires the use of gloves and a fume hood since one of them (Dimethylaminoethanol, DMAE) is highly toxic. Once the first three ingredients are in the container (Fig. 7a, b), they are mixed for 1 minute until the mixture is homogenized. The last chemical (DMAE) is added using a pipette. Once all of the chemicals are mixed, the container is placed in a desiccator, under vacuum, to remove trapped air that forms as bubbles. Using the vacuum in the freeze-dryer (with the refrigeration turned off) has proven to be particularly effective at initially removing bubbles from the resin before the actual impregnation step begins. The presence of bubbles will inhibit impregnation and create pockets of resin-starved sediment.

Before pouring the resin into the trays, they are identified, either by engraving on the back of the trays (disposable trays) or identifying the location of each subsample (reusable trays). The impregnation is preferably done using a large pipette. It could also simply be poured, but the large pipette allows better control on the pouring of the resin. It is critical not to rush this step. The low viscosity and long working time at room temperature easily permits one to add resin from one side only, at least until the bottom of the trays get covered. As it slowly flows across the tray it should displace any trapped air. After this point, resin can be added around the edges of the tray. The resin is poured on the side of the trays and until it reaches about one half the height of the subsamples, without covering the subsamples. Covering the subsamples would lead to additional air being trapped in the sediment, again preventing effective impregnation. The impregnation needs to be done from the bottom, by capillarity and is facilitated by holes made at the base of the aluminum boxes (Fig. 2). Once the resin fills the trays half way, the trays are put into the desiccator under a vacuum for at least 15 minutes and up to 1 hour. The vacuum enables the release and removal of bubbles. After 1 hour, the resin should have moved up into the sediment, thereby lowering its level in the trays. The procedure is repeated as many times as required until the sediment surface is wet, but without pouring the resin on top of the subsamples. Once the sediment is thoroughly wet, resin is then poured over the subsamples. The trays are then put into the desiccator for 12 hours or until no more bubbles are observed at the surface.

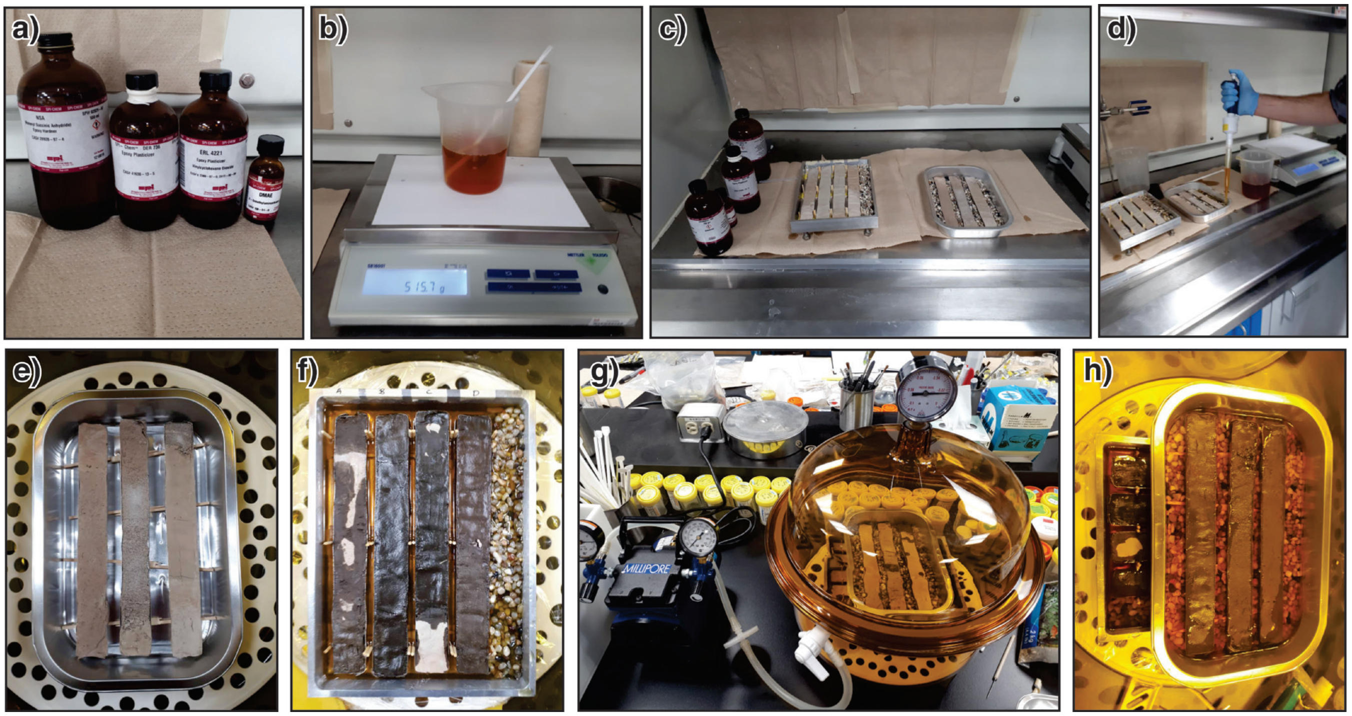

Figure 7. Impregnation procedure: **a)** chemicals used for the resin; 2018-308; **b)** mixing of the resin on a two-decimal balance; 2018-287; **c)** subsamples in the reusable (left) and disposable (right) aluminum trays; note the presence of gravel between the subsamples, which minimizes the amount of resin required; 2018-302; **d)** pouring of resin using a large pipette; 2018-291; **e)** disposable aluminum tray; 2018-300; **f)** reusable aluminum tray; 2018-281; **g)** desiccator; 2018-298; **h)** subsamples in the desiccator showing the impregnation process from top to bottom (note that the resin does not cover the subsamples, but that the surface of the subsamples are impregnated by capillarity action); 2018-297. All photographs by A. Normandeau.

Table 1. Amounts (in grams) for epoxy resin mixing

	1 tray	Cumulated	2 trays	Cumulated	3 trays	Cumulated
ERL 4221	60.31	60.3	120.6	120.6	180.9	180.9
DER 736	21.04	81.3	42.1	162.7	63.1	244.0
Nonenyl succinic anhydre (NSA)	86.79	168.1	173.6	336.3	260.4	504.4
Dimethylaminoethanol (DMAE)	1.47	169.6	2.9	339.2	4.4	508.8

Drying and cutting

Once the subsamples are well impregnated, the resin needs to be hardened by heat curing. The aluminum trays are put into an oven at 80°C for 48 hours. This process hardens the resin so that it is no longer sticky. After cooling, the subsamples are relabelled with the correct name and an up arrow, using a correction fluid (e.g. liquid paper™) (Fig. 8). The aluminum trays can then be removed. For the disposable trays, the aluminum peels off quite easily. The reusable trays require more work to remove the impregnated subsamples and screws are used to help to push the epoxy out of the tray (Fig. 8a).

As a final step, a rock saw is used (Fig. 9a, b) to cut the resin-infused subsample blocks lengthwise into individual slabs. Each slab is then cut twice crosswise to create the three separate labelled sections of 6–7 cm (Fig. 8d). These two final cuts are done at an angle, which will allow any lamina from one thin section to also be observed on the other section (Fig. 8d, 9c). A short incision at the top of each section is then made to clearly identify the top of the thin sections. If the impregnation process was successful, the sediment should look well impregnated and homogenous (Fig. 9d).

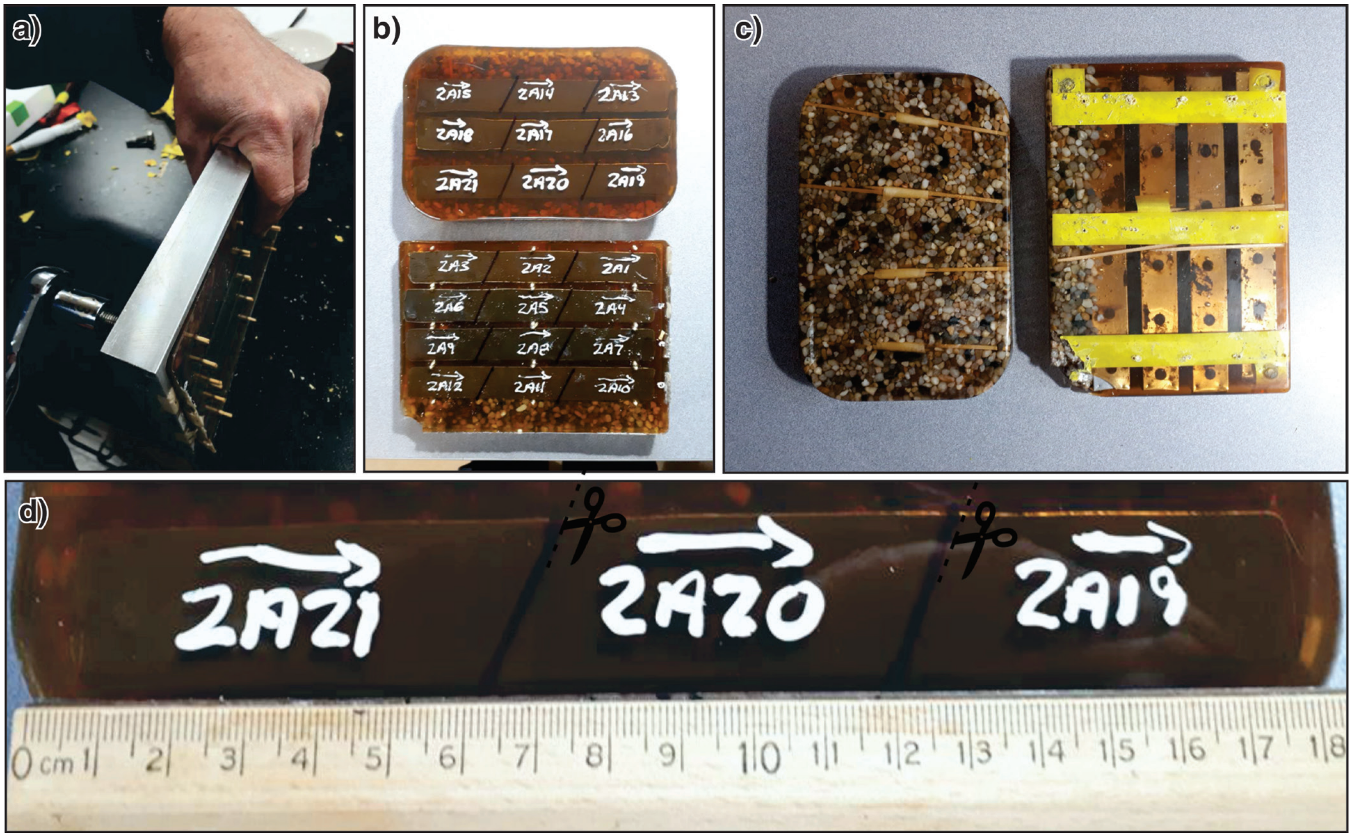

Figure 8. **a)** The reusable trays with screws used to remove the subsamples; 2018-280; **b)** impregnated subsamples with naming convention; 2018-303; **c)** bottom of the subsamples showing the near-absence of bubbles and therefore, a good impregnation. The gravel on the bottom was placed to limit the use of resin; 2018-288; **d)** lines drawn on the subsamples at an angle to show where the cutting will take place; 2018-299. All photographs by A. Normandeau.

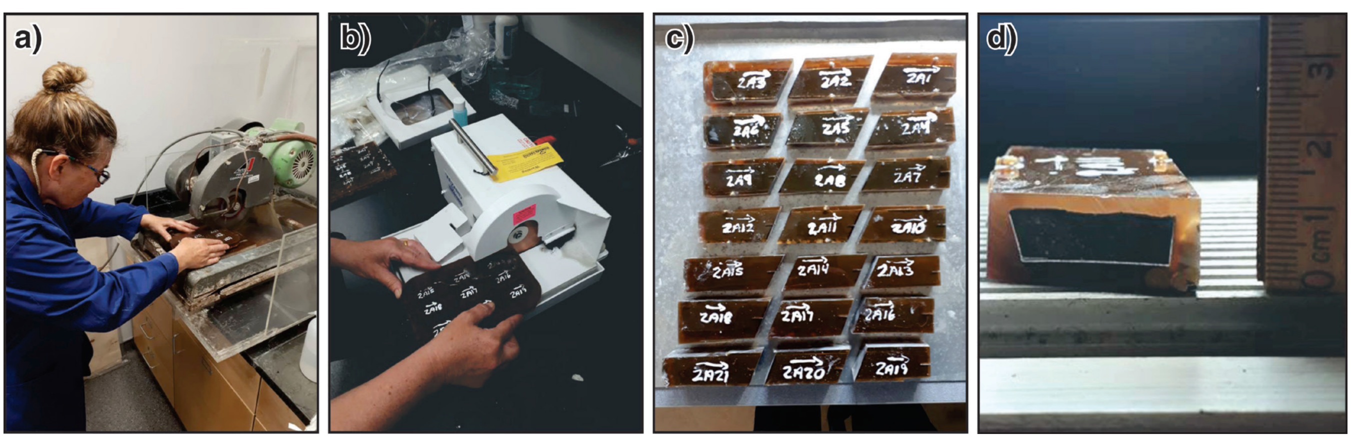

Figure 9. **a)** Large rock saw used for separating the boxes; 2018-278; **b)** small rock saw used for separating the boxes and making the top incisions on each subsample; 2018-295; **c)** final impregnated subsamples ready to ship to a petrographic laboratory; 2018-279; **d)** example of a well impregnated subsample ready for thin sectioning. 2018-296. All photographs by A. Normandau.

CONCLUSIONS

The impregnation of unconsolidated sediment requires special care and is a week-long process. It can be subdivided into five steps, from subsampling preparation, subsampling, sediment dehydration, impregnation, and cutting and drying. Once all of these steps are completed, the subsamples can be sent to a petrographic laboratory where the thin sections are made to a thickness of approximately 30 µm. The thin sections can then be interpreted at the microscopic scale and provide insights into past depositional processes (Fig. 10).

REFERENCES

Boës, X. and Fagel, N., 2005. Impregnation method for detecting annual laminations in sediment cores: an overview; Sedimentary Geology, v. 179, p. 185–194. https://doi.org/10.1016/j.sedgeo.2005.05.001

Fortin, D., Francus, P., Gebhardt, A.C., Hahn, A., Kliem, P., Lisé-Pronovost, A., Roychowdhury, R., Labrie, J., and St-Onge, G., 2013. Destructive and non-destructive density determination: method comparison and evaluation from the Laguna Potrok Aike sedimentary record; Quaternary Science Reviews, v. 71, p. 147–153. https://doi.org/10.1016/j.quascirev.2012.08.024

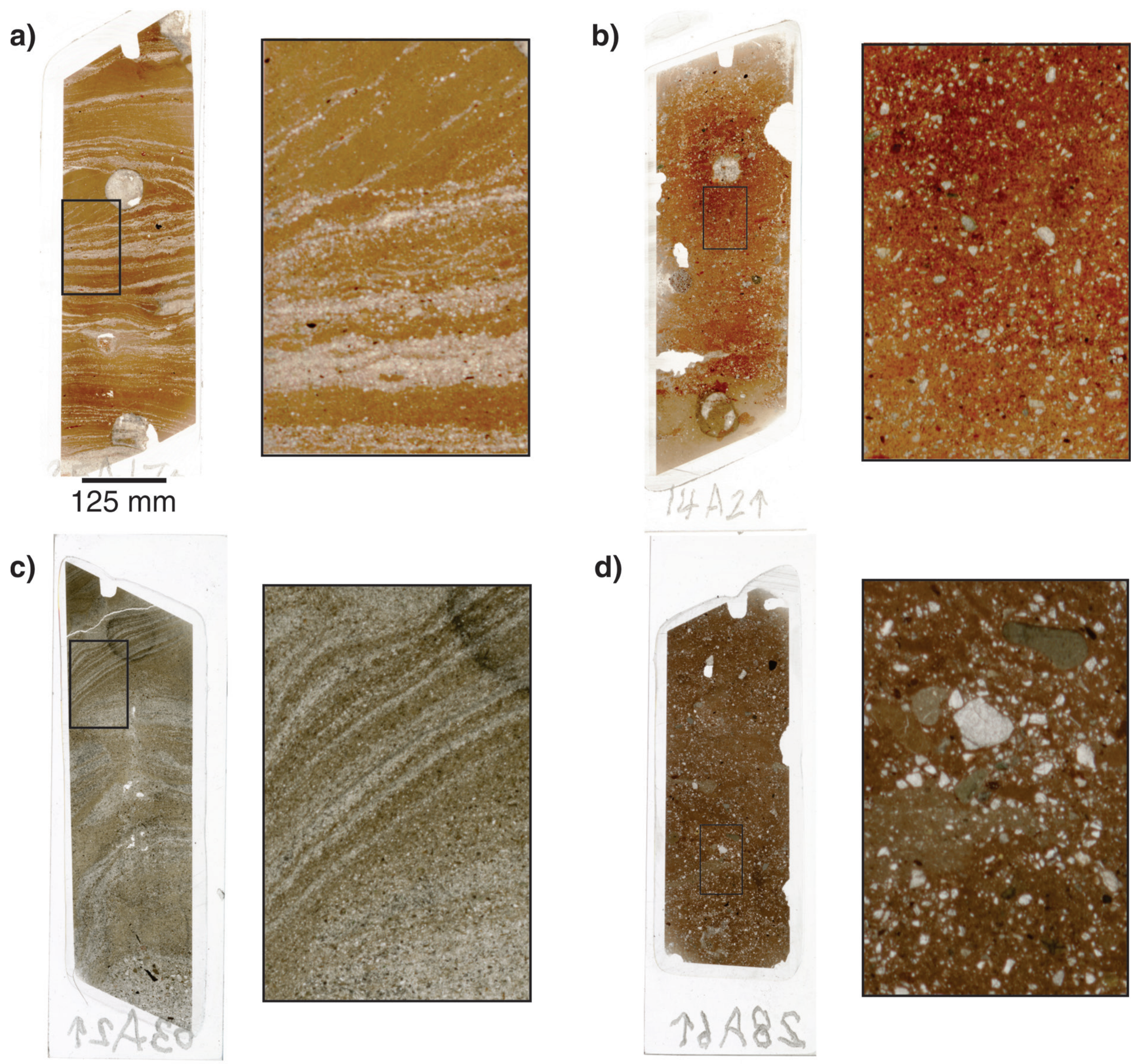

Figure 10. Preliminary results obtained at GSC Atlantic: **a)** thin-bedded turbidite sample in the Gulf of St. Lawrence; **b)** brick-red mud in the Gulf of St. Lawrence that represents a major ice-calving event in the Laurentian Channel; **c)** turbidite offshore of Pond Inlet; **d)** diamict on the continental slope of Nova Scotia.

Francus, P., 1998. An image-analysis technique to measure grain-size variation in thin sections of soft clastic sediments; Sedimentary Geology, v. 121, p. 289–298. https://doi.org/10.1016/S0037-0738(98)00078-5

Francus, P. and Asikainen, C.A., 2001. Sub-sampling unconsolidated sediments: a solution for the preparation of undisturbed thin-sections from clay-rich sediments; Journal of Paleolimnology, v. 26, p. 323–326. https://doi/org/10.1023/A:1017572602692

Francus, P., von Suchodoletz, H., Dietze, M., Donner, R.V., Bouchard, F., Roy, A.-J., Fagot, M., Verschuren, D., and Kröpelin, S., 2013. Varved sediments of Lake Yoa (Ounianga Kebir, Chad) reveal progressive drying of the Sahara during the last 6100 years; Sedimentology, v. 60, p. 911–934. https://doi.org/10.1111/j.1365-3091.2012.01370.x

Köng, E., Zaragosi, S., Schneider, J.L., Garlan, T., Bachèlery, P., San Pedro, L., Seibert, C., and Racine, C., 2016. Untangling the complex origin of turbidite activity on the Calabrian Arc (Ionian Sea) over the last 60 ka; Marine Geology, v. 373, p. 11–25. https://doi.org/10.1016/j.margeo.2015.12.010

Lamoureux, S.E., 1994. Embedding unfrozen lake sediments for thin section preparation; Journal of Paleolimnology, v. 10, p. 141–146. https://doi.org/10.1007/BF00682510

Lapointe, F., Francus, P., Lamoureux, S.F., Saïd, M., and Cuven, S., 2012. 1750 years of large rainfall events inferred from particle size at East Lake, Cape Bounty, Melville Island, Canada; Journal of Paleolimnology, v. 48, p. 159–173. https://doi.org/10.1007/s10933-012-9611-8

Lotter, A.F. and Lemcke, G., 1999. Methods for preparing and counting biochemical varves; Boreas, v. 28, p. 243–252. https://doi.org/10.1111/j.1502-3885.1999.tb00218.x

Normandeau, A., Dietrich, P., Lajeunesse, P., St-Onge, G., Ghienne, J., Duchesne, M.J., and Francus, P., 2017. Timing and controls on the delivery of coarse sediment to deltas and submarine fans on a formerly glaciated coast and shelf; Geological Society of America Bulletin, v. 129, p. 1424–1441. https://doi.org/10.1130/B31678.1

Ojala, A.E.K., Francus, P., Zolitschka, B., Besonen, M., and Lamoureux, S.F., 2012. Characteristics of sedimentary varve chronologies – a review; Quaternary Science Reviews, v. 43, p. 45–60. https://doi.org/10.1016/j.quascirev.2012.04.006

Schimmelmann, A., Lange, C.B., Schieber, J., Francus, P., Ojala, A.E.K., and Zolitschka, B., 2016. Varves in marine sediments: a review; Earth-Science Reviews, v. 159, p. 215–246. https://doi.org/10.1016/j.earscirev.2016.04.009

Tanner, P.A. and Leong, L.S., 1995. The effects of different drying methods for marine sediment upon moisture content and metal determination; Marine Pollution Bulletin, v. 31, p. 325–329. https://doi.org/10.1016/0025-326X(95)00172-J

van der Meer, J.J.M. and Menzies, J., 2011. The micromorphology of unconsolidated sediments; Sedimentary Geology, v. 238, p. 213–232. https://doi.org/10.1016/j.sedgeo.2011.04.013

von Merkt, J., 1971. Zuverlässige Auszählungen von Jahresschichten in Seesedimenten mit Hilfe von Gross-Dünnschliffen; Archiv fuer Hydrobiologie, v. 69, p. 145–154.

Zaragosi, S., Bourillet, J.-F., Eynaud, F., Toucanne, S., Denhard, B., Van Toer, A., and Lanfumey, V., 2006. The impact of the last European deglaciation on the deep-sea turbidite systems of the Celtic-Armorican margin (Bay of Biscay); Geo-Marine Letters, v. 26, p. 317–329. https://doi.org/10.1007/s00367-006-0048-9

Geological Survey of Canada Project 330401NU62