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GEOLOGICAL SURVEY OF CANADA
OPEN FILE 8273

Geoscientific studies of Champlain Sea sediments, Bilberry Creek, Ottawa, Ontario: on-site preservation of cores

S. Alpay, H. Crow, M.J. Hinton, and A. Grenier

2020

Canada

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2020

Permanent link: https://doi.org/10.4095/326109

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

Recommended citation
Alpay, S., Crow, H., Hinton, M.J., and Grenier, A., 2020. Geoscientific studies of Champlain Sea sediments, Bilberry Creek, Ottawa, Ontario: on-site preservation of cores; Geological Survey of Canada, Open File 8723, 23 p. https://doi.org/10.4095/326109

Publications in this series have not been edited; they are released as submitted by the author.

SUMMARY

This Geological Survey of Canada (GSC) Open File report documents a practical field method for core preservation in Shelby tubes, using a wax mixture, immediately after retrieving sediment samples at the drill site. Experience with core tube sealing techniques in the field, during a research study of Champlain Sea deposits, generated the opportunity to describe the protocols in stepwise detail for others to use or modify for their purposes.

RÉSUMÉ

Ce dossier publique de la Commission géologique du Canada (CGC) documente une méthode de terrain pratique et efficace pour la préservation des carottes de sédiments dans des tubes Shelby, en utilisant un mélange de cire, immédiatement après avoir récupéré les échantillons sur le site de forage. L'application de cette technique de scellage sur le terrain, pendant la réalisation d'un projet de recherche sur les dépôts de la mer de Champlain, a donné l'opportunité de décrire la procédure étape par étape afin que d'autres l'utilisent ou la modifient selon leur besoin.

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INTRODUCTION

It is essential to maintain the integrity of sediment cores and their in-situ characteristics as much as possible if there is to be a time delay between coring in the field and sample extrusion and testing in the laboratory. Preservation of sediment samples is particularly important for analyses which require maintenance of the original moisture content. The Geological Survey of Canada (GSC) has adopted techniques for core preservation and handling, guided by standard practices (American Society for Testing and Materials (ASTM) International, 2014), recommendations (e.g., Lessard and Mitchell, 1985), and empirical testing of the methods at different project sites (e.g., Crow et al., 2017).

The GSC began field work for a study of Champlain Sea deposits in autumn 2019 at Bilberry Creek, Ottawa (Orleans), with research objectives to investigate:

1. the hydrogeological and geochemical factors contributing to changes in the geotechnical properties of Champlain Sea muds;
2. the hydrogeology of Champlain Sea deposits with implications for geochemistry and groundwater flow; and
3. a comprehensive suite of geological, geotechnical, geophysical, hydrogeological, and geochemical characterizations at two new sites in Champlain Sea sediments.

To fulfill these objectives, the GSC required a field protocol to preserve Champlain Sea sediments immediately after recovery of core tubes at the drill site. In particular, preservation of geotechnical properties, moisture content, and sedimentary textures were of high priority.

References to core preservation methods typically do not provide explicit procedures. This GSC Open File documents techniques to seal and preserve sediment samples in stepwise detail with photographs. The report is available for practical transfer to others interested in using these or similar techniques, adjusted for their applications. It is also available to those interested in evaluating the effectiveness of core preservation methods and in developing standard protocols.

This GSC Open File is one of a series for this research activity to document pertinent methods and results from Champlain Sea deposits in Ottawa and its vicinity. This is the first report of the series.

CORE PRESERVATION, TRANSPORT, AND STORAGE

For convenience, a list of recommended materials needed for core preservation is included in Appendix A. The GSC arranged these items by task in the portable field lab, housed in a cube truck, during the autumn of 2019.

HANDLING CORES AFTER SAMPLING

After raising the drill string from the ground, a flexible vinyl end cap secured the Shelby tube at its base. Once detached from the drill string, it was necessary to tilt the Shelby tube to pour out the drilling fluid above the sediment core. Otherwise, the core remained upright while walked over to the field lab for preservation. Mounting holes for the Shelby tubes are located at the top of the core, making their upright position evident (Fig. 1). Cleaning the outside of the tubes to remove smeared soil or mud residue allows the wax sealant to adhere to the tube more effectively. A cloth dipped in a pail of water is sufficient for wiping the exterior of the tube clean. The inside walls at the top of the Shelby tube can also be wiped with paper towels to clear away sediment residue and residual drilling fluid.

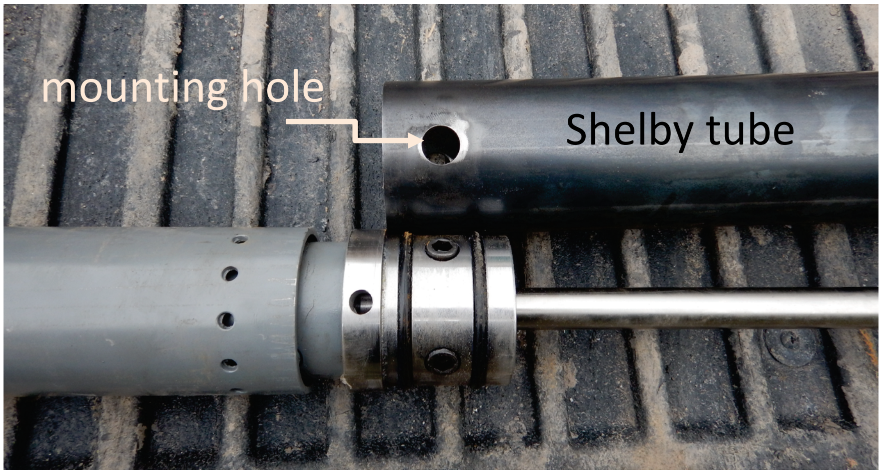

Figure 1. Mounting holes at the top end of the 3-inch diameter Shelby tube; Photo: GSC, Oct 2019

It is important to handle the Shelby tubes with care to minimize bumping or sudden movement that can disturb the sediments and compromise the integrity of some analyses. For highly sensitive sediments, avoiding physical shock (e.g., tapping) and excessive vibration is particularly important because disturbance can alter shear strength significantly (e.g., Hvorslev, 1949; Lefebvre and Poulin, 1979; Amundsen et al., 2015). Maintaining the Shelby tubes upright, as much as possible, during the preservation process helps to preserve sample integrity (Hvorslev, 1949). Likewise, exposures to extremes of temperature, particularly freezing (Hvorslev, 1949), will cause significant irreversible changes to geotechnical properties (e.g., La Rochelle et al., 1986). In this study, an insulated wooden core storage box, fashioned after the shipping box recommended by ASTM International D4220 (2014), provided support for the cores in an upright position for most of the sample handling (Fig. 2). While the wax mixture is cooling, foam or bubble wrap around the outside of the tube inside the storage box helps to secure the cores from shifting.

Figure 2. Sediment samples in Shelby tubes supported by the insulated shipping box and cushioning materials; Photos: GSC, Oct 2019

PREPARATION OF WAX SEALANT

A wax compound to seal the core is critical to avoid moisture loss, oxidation, and physical shifting of the core inside the Shelby tube. ASTM International (2014) cautions that paraffin wax is reliable for no more than three days as a sealant. Paraffin wax, used alone, is too brittle when solidified, creating cracks and fissures that allow oxidation of sediment samples (e.g., La Rochelle et al., 1986). Abdellaziz et al. (2019) used a 50:50 ratio of paraffin wax to petroleum jelly as recommended by La Rochelle et al. (1986) and Lade (2016). The wax mixture is more pliable than paraffin wax alone and increases adherence of the wax seal to the inside wall of the Shelby tube, thereby delaying potential oxidation and moisture loss during storage. There is evidence that the 50:50 compound also conserves many geotechnical and physical properties of glaciomarine clays for extended periods of time under specific storage conditions (i.e., up to 27 years; Abdellaziz et al., 2019). ASTM International (2014) recommends using microcrystalline wax, with a higher melting point than paraffin wax, or a combination of microcrystalline wax with up to 15% beeswax or resin for sealing cores. Considered to shrink less than paraffin wax, microcrystalline wax serves a similar purpose for sealing sediments in Shelby tubes (Poullain, 2012) and is available from numerous suppliers (e.g., Forney, 2020; Hole Sampling and Monitoring, 2020, Humboldt Mfg., 2020, Myers Construction Materials and Testing, 2020). Of note, microcrystalline wax is a component of petroleum jelly or petrolatum.

There remain numerous recommendations for the composition of the wax compound. Among practitioners and within the literature, there is anecdotal evidence for effective use of paraffin wax (Hvorslev, 1949; Engineering Forum, 1999-2003; NRC, 2013), beeswax (USDA-NRCS, 2012), a combination of paraffin wax and petroleum jelly (Engineering Forum, 1999-2003), microcrystalline wax (ASTM, 2014; Hunt, 2019), a combination of beeswax and paraffin wax (Hvorslev, 1949; USDA-NRCS, 2012; NRC, 2013), and a combination of beeswax and petroleum jelly (Engineering Forum, 1999-2003).

Previous GSC studies of Champlain Sea sediments (2010-14) successfully used a combination of 25% unscented petroleum jelly (by weight) with beeswax (Crow et al., 2017). The GSC adopted this wax compound of petroleum jelly and beeswax, a natural substance, to maintain the desired wax sealant properties of reducing brittleness, less shrinkage, and cracking in colder temperatures.

The GSC has used both pure (filtered; unbleached) and industrial grade natural beeswax for sealing the ends of cores in Shelby tubes with similar results. Provincial beekeepers associations are active and frequently provide a listing of local suppliers. If using industrial grade beeswax, impurities may be evident upon melting (e.g., honeycomb or bee parts; Fig. 3), but they do not interfere with the effectiveness of the wax sealant if applied in multiple layers.

Figure 3. Impurities in the industrial grade brown beeswax (shown on a metal ladle) do not impede the effectiveness of the wax mixture as a sealant when applied in multiple layers. Photo: GSC, Oct 2019

To avoid burning the beeswax and to help keep the wax from cooling too rapidly, the GSC used a double boiler on a camp stove or hot plate to melt it gently with the petroleum jelly (Fig. 4). It is easier to melt the solid beeswax after reducing it in size from the large 1-kg or 1-lb blocks to ≤ 6 cm shards (e.g., using a small hatchet). Alternately, beeswax comes in pellet form, although typically at a higher cost.

Figure 4. The field lab, including a camp stove and double boiler to melt the wax mixture, was located in close proximity to the drill site (in the background). Photo: GSC, Oct 2019

If the wax mixture is too hot, it may not adhere well to the core ends and the layers will be thin and not uniform. A qualitative measure of the ideal temperature for the wax mixture is to observe when it begins to melt solid wax coated on a stirring utensil. For the wax mixture in this study, the optimal temperature range for sealing the ends of the Shelby tubes and maintaining a molten wax compound was 68-80 °C. When the liquid wax mixture begins to form a thin crust on the surface, typically at a temperature of 64-66 °C, it is time to re-heat gently. The mixture requires periodic reheating and replenishment throughout the process. During the study, it took 552 g of solid industrial grade beeswax (with 184 g of petroleum jelly; half a standard large size tub) to seal 4-5 Shelby tubes.

SEALING THE TOP END OF THE SHELBY TUBE

The sediments at the top are not flush with the end of the Shelby tube. Therefore, it is necessary to pour or ladle the molten wax mixture on top of the sediments to seal the surface of the sample (≥ 0.5 cm thick; Fig. 5). The wax mixture must cool, turning pale once it has solidified (Fig. 6), before advancing to the next step. If the top of the sediment is deep within the tube, a flashlight can help for visual confirmation that the wax has solidified.

Figure 5. Ladling the molten wax mixture onto the sediment surface to preserve the sample at the top end of the Shelby tube; Photos: GSC, Oct 2019

Figure 6. Gradual cooling and solidification of wax mixture on the surface of the sediments. Photos: GSC, Oct 2019

Disks cut from rigid polystyrene insulation fill the void in the top end of the tube to secure the sediment sample from sliding. The disks fit snugly in the void area, and ideally, are flush with the end of the Shelby tube. In this study, the GSC cut disks from polystyrene sheets of different thicknesses (1″, 1½″, and 2″ thick). In practice, the top-most polystyrene disk stuck out ≤ 1 cm beyond the end of the tube (Fig. 7). In retrospect, allowing the disk to protrude past the top end of the Shelby tube can invite involuntary de-structuring of the sediments if the top end is subject to inadvertent force. In this study, there was little opportunity during handling and transport to put undue pressure on the tops of the Shelby tubes. For future implementation, however, the GSC will adopt the practice of cutting the top-most polystyrene disk to be flush or ≤ 0.5 cm below the top of the tube.

Figure 7. Rigid polystyrene insulation disks fill the void space above the surface of the sediments in the Shelby tube. In future coring, the top-most polystyrene disk will be cut to be almost flush with the top end of the Shelby tube. Photo: GSC, Oct 2019

The GSC used strips of aluminum foil tape (used to seal heating ducts) to cover over the entire top of the tube and along the sides, snugly sealing in the insulation disks. The tape should completely cover the mounting holes on the sides of the Shelby tube (Fig. 8). If the foil tape extends more than 5 cm down the sides of the Shelby tube, it makes easy work of removing the outer wax seal when it comes time to open the top end of the core tube. Edges or wrinkles in the tape can be finger-smoothed. For the GSC study, three strips of tape (4.8 cm width) covered a 3-inch diameter Shelby tube with two mounting holes (Fig. 8). The aluminum foil tape secures the insulation disks and creates an additional barrier to help prevent moisture loss and oxidation of the sample.

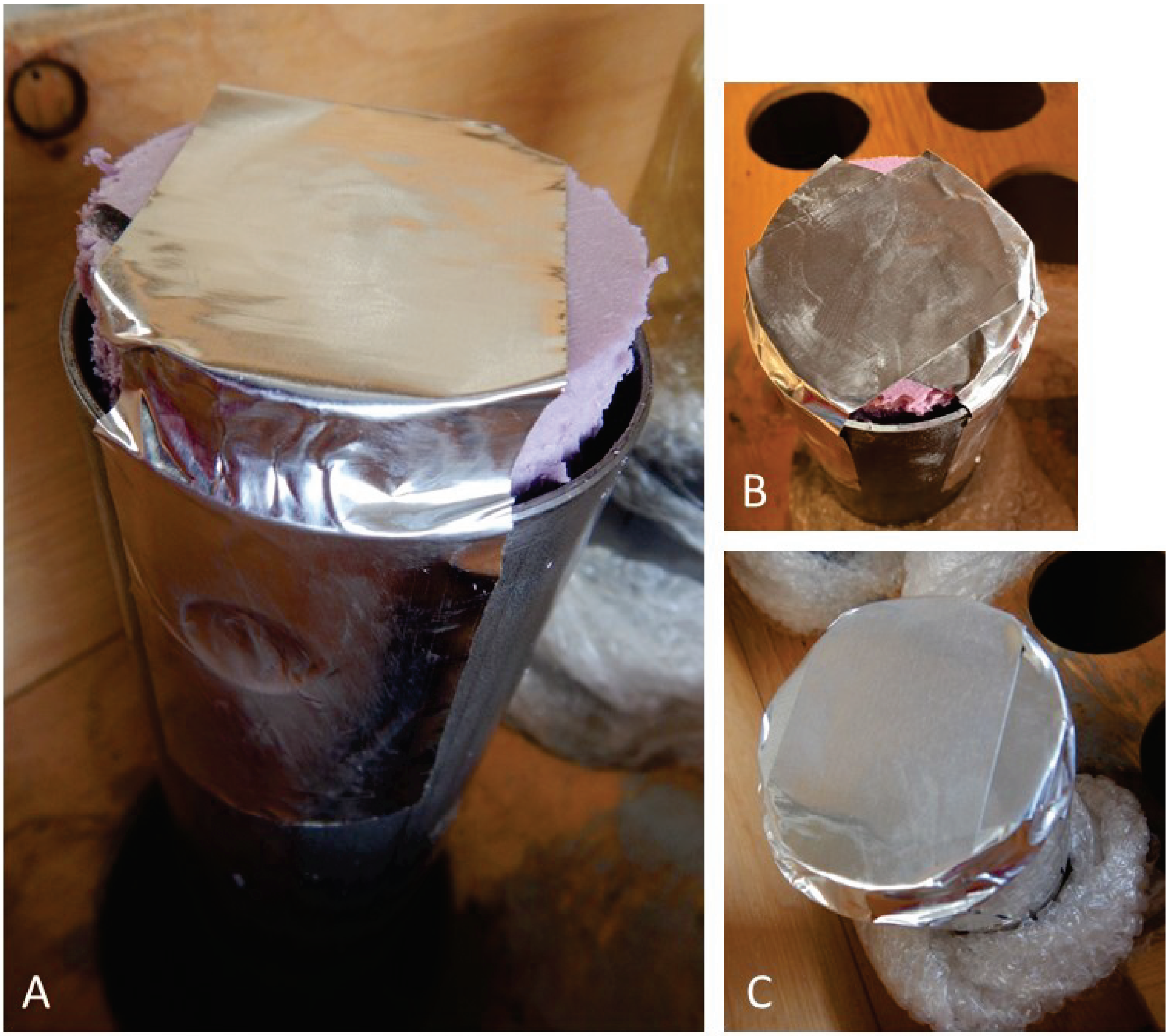

Figure 8. Strips of aluminum foil tape applied to the end of the Shelby tube. **(A)** The first strip clearly covers and seals the mounting holes. **(B), (C)** The next two strips completely seal the top end. Photos: GSC, Oct-Nov 2019

The Shelby tube can be inverted gently to dip the top end into the warm wax mixture (≤ 5 cm up the tube) and lifted out to cool and solidify (Fig. 9). The first layer of wax on the aluminum foil tape does not consistently adhere well, particularly if the wax compound is too hot. Repeated layers resolve this issue. While the hot wax begins to cool, rotating the Shelby tube at an angle above the pot helps to spread the wax mixture out evenly and allows any excess to drip back into the pot. If the air temperature is warm, it is helpful to dip the top end of the tube into a container of cool water to speed the cooling of the wax. The same process of dipping and cooling should be repeated 3-4 times to create solid thin layers of wax.

Figure 9. Adding the first wax layers on top of the aluminum foil tape **(A)** Gently inverting the top of the Shelby tube to dip it into the warm wax mixture. **(B)** The first layer of wax does not usually apply a full homogeneous coating. **(C)** and **(D)** Subsequent coatings allow for better coverage of the core end. **(E)** Once sealed, the thickness of the wax mixture coating is more even. Photos: GSC, Oct-Nov 2019

At this stage, 1-2 layers of cheesecloth added on the top, overlapping the sides of the tube, provide additional strength to the wax layers (Fig. 10). If the pieces of cheesecloth are too large, trimming them back keeps them out of the way before dipping the end of the tube into the molten wax mixture again, smoothing it with a putty knife or gloved finger. Dipping and cooling 3-4 more times typically results in a wax thickness of 1 cm, the minimum recommended (ASTM International, 2014). A thicker wax coating, if required, can be achieved by repeating the cheesecloth and wax layering procedure. Several thin layers of wax are more effective than one thick layer (ASTM International, 2014).

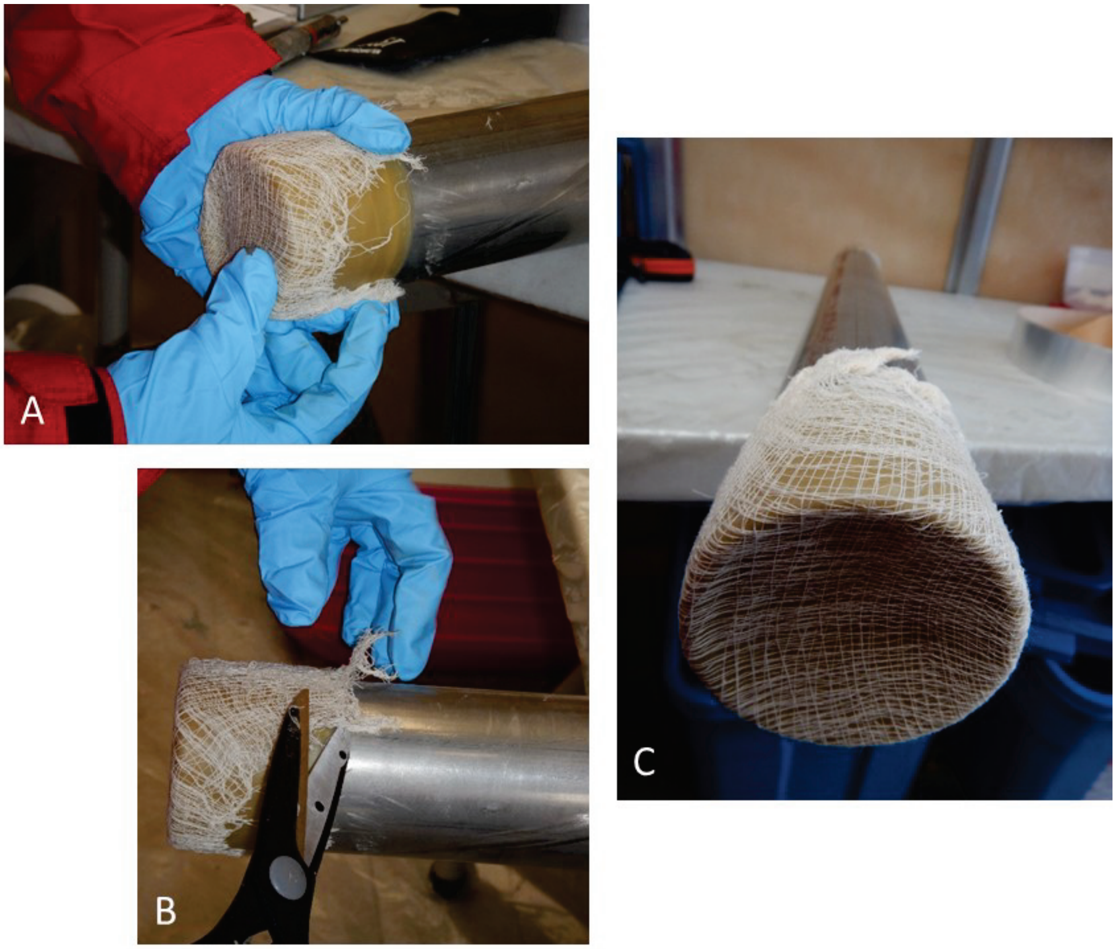

Figure 10. Adding one or two layers of cheesecloth. **(A)** Pressing cheesecloth into the wax layer is easy because the wax remains tacky. **(B)** Trimming long ends of the cheesecloth keeps them out of the way. **(C)** Adding the cheesecloth layer strengthens the next wax layers to come. Photos: GSC, Nov 2019

The wax layer is tacky even when solid. A final layer of plastic wrap prevents the wax sealant from sticking to other objects (Fig. 11). When the top end is completely sealed, the base of the Shelby tube is ready for treatment.

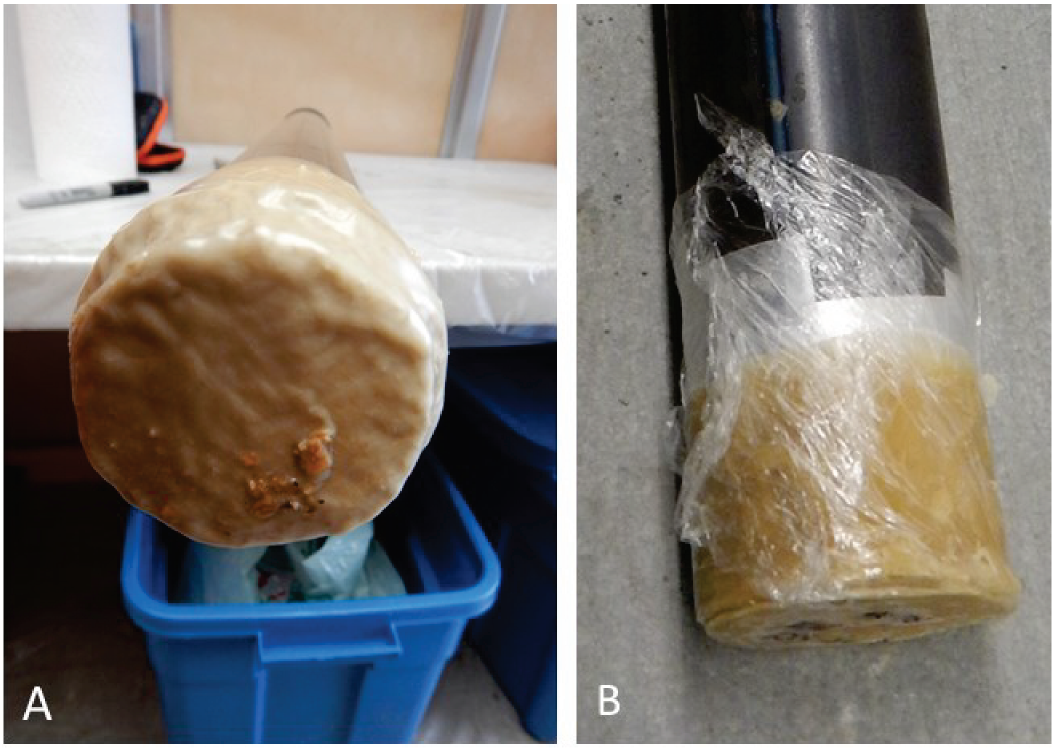

Figure 11. (A) Example of a thick wax seal at the top end of the core tube. Small fragments of impurities from the industrial grade beeswax generally do not compromise the seal if there are enough layers. **(B)** A layer of plastic wrap keeps the end from sticking to other objects. Photos: GSC, Oct and Dec 2019

SEALING THE BOTTOM END OF THE SHELBY TUBE

If the sample is flush with the bottom end of the Shelby tube

In this study, the majority of sediment samples were nearly flush with the base of the Shelby tube, simplifying the sealing method. The procedure is similar to the one used for the top end of the Shelby tube without the insulation disks. After removing the cap, the bottom end of the Shelby tube is dipped ≤ 5 cm into the molten wax compound 3-4 times, allowing the wax to solidify between each coating (Figure 12). If the bottom sediment surface is irregular, it may be necessary to dip the end into the molten wax mixture at an angle and rotate it there to get a consistent coating. As the wax starts to cool, rotating the Shelby tube above the pot will control drips.

For some test samples in core tubes, the GSC tried applying 1-2 layers of cheesecloth directly on the sediment surface first, followed by alternating layers of wax mixture and cheesecloth. The layer of cheesecloth in direct contact with the sediments reduced the loss of sediment adhering to the wax seal when unwrapping the bottom end of the core tube. Therefore, the GSC will adopt this option for future coring in similar deposits.

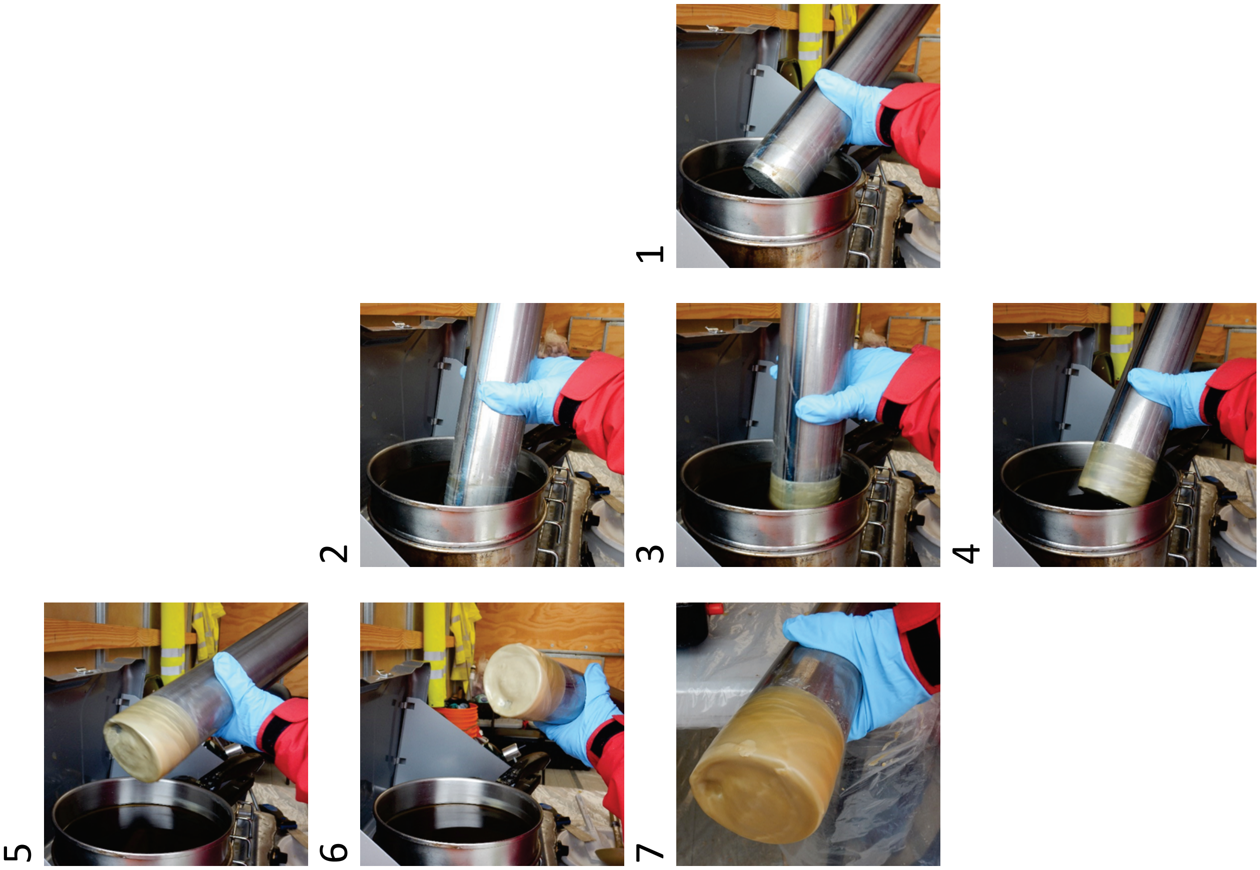

Figure 12. Sequence of dipping the bottom end of the Shelby tube into the molten wax mixture and rotating it to achieve a uniform coating, 3-4 layers thick, before adding cheesecloth. Photos: GSC, Nov 2019.

When the wax mixture has solidified, one or two layers of cheesecloth can be pressed onto the wax at the end of the tube, overlapping the sides, to give the seal more strength. Dipping the bottom end of the Shelby tube into the wax mixture 3-4 times creates a seal of approximately 1 cm, sufficient for most purposes. Fewer wax layers will not provide the desired sealing properties (ASTM International, 2014; Fig. 13). As the wax cools, a putty knife or gloved finger can smooth the surfaces. A second layer of cheesecloth and 3-4 more layers of wax yield a thicker seal, if required.

Figure 13. One layer of wax after applying the cheesecloth does not provide a uniform coating. In this example, 2-3 more dips into the wax mixture were required. The concave base of the sediment core is typical of most sediment cores in this study. Photo: GSC, Nov 2019

Once the wax is cool and solidified, the end of the tube can be covered in plastic wrap to prevent sticking (Fig. 11B).

If the sample is not flush with the bottom end of the Shelby tube

Although not encountered frequently in this study, if the gap between the sample and the base of the Shelby tube was larger than the thickness of the smallest polystyrene disk (1″), the GSC used the same procedure as for the top of the Shelby tube. Disks of rigid insulation, cut to slightly less than the diameter of the tube, will fill the void between the wax-coated sample and the bottom end of the tube. The bottom disk should not extend beyond the base of the tube because placing additional pressure on the sediment core, which rests on its base during transport and storage, could compromise the internal structure of the sample. Cutting the bottom-most polystyrene disk to be ≤ 0.5 cm shorter than the base of the Shelby tube will help to avoid direct force on the core. In this study, the majority of the sediment samples were flush with the base of the Shelby tube or slightly concave (e.g., Figs. 10, 13).

Aluminum foil tape secures the insulation disks and prevents downward sliding of the sample inside the Shelby tube. Final sealing of the bottom end follows the same sequence as the top of the core tube with alternating applications of the wax mixture and cheesecloth, followed by plastic wrap.

If the sample is protruding beyond the base of the Shelby tube, there are at least three options for core preservation. To leave the protruding end in place, it is necessary to paint a few coats of wax over it or to dip it in the wax mixture 3-4 times, allowing it to solidify in between layers. The first applications of the wax compound create a substrate for adding 1-2 layers of cheesecloth. The GSC repeated this procedure at least two more times to get a stronger seal at the base of the tube. After applying the plastic wrap, a thorough wrapping of the bottom end of the tube in duct tape provided additional support (Fig. 14). This is not an ideal method, as the upright core rests on its base.

In some instances, it may be better to leave the lower core cap in place and duct tape it there, followed by alternating layers of the wax compound and cheesecloth. In this study, it was not apparent that the sediment core extended beyond the end of the tube until removal of the cap.

If the sample protrudes well beyond the end of the tube, or is dry and brittle, it may be necessary to cut off the protruding end cleanly and wax it separately from the Shelby tube, by painting the wax mixture on it in several layers, covering it in plastic wrap, and labeling the top and bottom ends.

Figure 14. Additional sealing for a sediment sample that protruded beyond the bottom of the Shelby tube; Photo: GSC, Dec 2019

TRANSPORT AND STORAGE OF THE SHELBY TUBES

Sealed tubes remained upright (bottom end down) in the wooden insulated storage containers (ASTM International, 2014), well packed in foam or bubble wrap to transport to cold storage (Fig. 2). In cold weather, it was necessary to warm the insulated boxes to keep the cores from freezing. Two or three water bottles filled with hot water served this purpose with a lid, blanket, or sleeping bag covering the top of the box (Fig. 15A). In warmer weather, cold packs can keep the cores cool in the insulated shipping boxes (blanket or lid on top) until the samples can be stored in cold storage. A thermometer inside the insulated box monitors air temperature to allow for effective adjustments for warming or cooling of samples. In this study, daily transport of cores to GSC facilities ensured their placement upright in 3-5 ^{0}C cold storage until extrusion and subsampling (Fig. 15B).

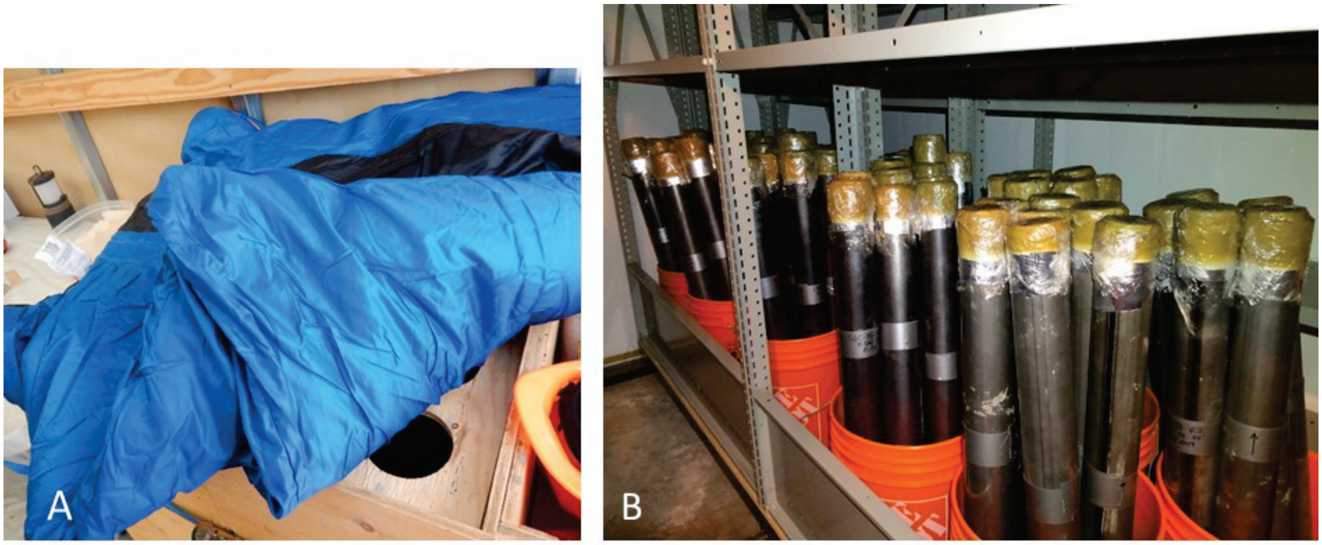

Figure 15. Transport and storage of samples. **(A)** The insulated shipping box became a core incubator with hot water bottles placed inside with the cores to avoid freezing when ambient field temperatures dropped. **(B)** For storage (several months), cores remained upright in a dedicated cold room. Photos: GSC, Nov 2019

For convenience, a one-page summary of the core preservation procedures to seal sediment samples in Shelby tubes is available in Appendix B as a quick reference tool for practitioners. These methods serve as a guide, which users can modify to suit their requirements.

CONCLUSIONS AND IMPLICATIONS

Sealing sediment cores in Shelby tubes with a wax compound preserves in-situ sample properties, by limiting moisture loss, oxidation, and physical disturbance, until core extrusion, subsampling, and laboratory testing and analyses. On the basis of experience in field conditions, published recommendations (Lessard & Mitchell, 1985), and ASTM International (2014) guidelines, the Geological Survey of Canada is providing this stepwise approach for core preservation to share with others interested in using or adapting the technique for their applications.

The stepwise description of the preservation protocol allows for reproducibility of the method. It also permits more scrutiny of the technique's effectiveness. Furthermore, availability of the detailed core sealing method opens the opportunity to test and develop improvements, particularly by those who study the effects of different core preservation techniques. Further investigations into the methods for core preservation can influence standardized procedures and provide detailed guidelines for practitioners.

ACKNOWLEDGMENTS

The City of Ottawa, Rideau Valley Conservation Authority, and both the Public Safety Geoscience and Groundwater Geoscience Programs at the Geological Survey of Canada (Natural Resources Canada, Lands and Minerals Sector) provided funding to support research and characterizations of Champlain Sea deposits at project drill sites. The authors gratefully acknowledge D. Perret for his review of this GSC Open File report. Core preservation methods in this study adopted recommendations generously shared by T. Skinner and C. Mangione (retired) during a previous GSC drilling project in Champlain Sea deposits. M. Celejewski provided valuable field support for these methods.

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APPENDIX A - EQUIPMENT AND SUPPLIES

A cube truck housed the portable field lab for core preservation procedures. The GSC set up the lab close to the drill site for easy transfer of the samples to the lab.

Figure A1. Rental cube truck, as a portable field lab, containing the essentials for core preservation; Photos: GSC, Oct 2019

Equipment and Supply list

- Pail with clean water
- Cloths, paper towels
- Insulated shipping box to hold Shelby tubes securely (ASTM International, 2014) during waxing and transport
- Ratchet straps to secure equipment inside the field lab
- Tables (2), one for camp stove, the other for treating the ends of the Shelby tube
- Plastic sheeting to cover surfaces (i.e., tables, floor)
- Camp stove and fuel
- Double boiler – glass lid can be useful to monitor the condition of the wax mixture
- Ladle for mixing and pouring wax
- Putty knife to smooth wax surface, if needed
- Spatula to cut out sections of petroleum jelly to add to the wax mixture
- Natural beeswax – pure or industrial grade
- Petroleum jelly
- Sheets of extruded polystyrene rigid insulation. GSC used 1″, 1½″, and 2″ thick sheets. Disks were cut with a holesaw bit on a drill to a diameter slightly smaller than the inside diameter of Shelby tubes
- Aluminum foil tape (used for sealing duct work; 4.8 cm width)
- Duct tape to secure samples that protrude from the bottom of the Shelby tube

- Cheesecloth, cut into squares large enough to cover the end of the Shelby tube and fold over the outside walls
- Scissors for cutting plastic sheeting, aluminum foil tape, and cheesecloth
- Plastic food wrap
- Shelby tube end caps for sealing the lower end of the core tube temporarily while waxing the upper end.
- Thermometer to monitor air temperature in the insulated shipping box if temperature extremes are expected
- Blanket, sleeping bag or lid for the insulated shipping box
- Freezer packs or water bottles to control air temperatures inside the insulated shipping box as required
- Personal protective equipment – nitrile or PVC gloves; protective eyewear, hard-toe footwear

During the waxing procedure, work surfaces (e.g., floor, tables, or counters), covered with clean plastic sheeting, catch stray drips and spills. It is also advisable to wear personal protective equipment, such as polyvinylchloride (PVC) or nitrile gloves to handle tubes and hot wax during the process, protective eyewear, in addition to closed, hard-toe footwear for handling heavy Shelby tubes and working near a drill site. Additionally, it is important to ensure proper ventilation for the camp stove, which was near the open end of the cube truck and not in an enclosed space.

Figure A2. Supplies for wax sealant of samples in Shelby tubes, clockwise from top left: Double boiler on camp stove; Plastic food wrap, industrial-grade brown beeswax, petroleum jelly; Disks cut from rigid insulation sheets; Aluminum foil tape for duct work; Cheesecloth layers cut into squares; Implements for handling the wax mixture – metal ladle, putty knife, spatula. Photos: GSC, Oct-Nov 2019

APPENDIX B – QUICK REFERENCE