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A New Method for the Restoration of Palaeontological Specimens Mounted in Canada balsam

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Abstract
Many museums contain slides mounted with Canada balsam. If this resin is poorly prepared, it can become crazed. Examples can be found within the British Type Graptolite Collection at the Natural History Museum, London. These are delicate dendroids prepared using the transfer technique. A search of the available literature and communication with museum workers highlighted suggestions for methods to rescue the cracked slides. These methods were tested, and the most suitable method proved to be a double transfer technique utilising carbowax. This technique may be used to rescue any specimen which is mounted in Canada balsam and which possesses an exposed surface. It is particularly important for the conservation of fragile specimens.

Introduction
The British Type Graptolite Collection at the NHM contains dendroids mounted in Canada balsam (a turpentine semi-fluid resin from the *Abies balsamea* fir) (Mills and White, 1987). These slides have been prepared from the collection of Mrs Robert Gray, which was acquired in 1920 and consists of dendroid graptolites from Girvan, Scotland. The preparations were carried out by O. M. Bulman and date from the 1920s. Professor Bulman was a distinguished palaeontologist and for many years the leading world authority on graptolites. He worked at Imperial College London, Cambridge University, Sidney Sussex College and Sedgwick Museum, Cambridge. He was editor of the Geological Magazine from 1934 and the president of the Geological Society from 1962 to 1964. His publications included 'Monograph of British Dendroid Graptolites', 'The Graptolites Prepared by Holme' and a section for Raymond C. Moore's 'Treatise on Invertebrate Palaeontology' (The Times, February 20, 1974). These specimens, therefore, represent an important period of publication.

Currently no suitable technique has been documented which is suitable for removing delicate specimens from Canada balsam and re-mounting them. This article explores the methods recommended within the published literature and compares the results with a newly proposed technique.

Production Technique
The slides were prepared using the 'transfer' technique, developed by palaeobotanists desiring to see both sides of fossil leaves (Elvers ,1988). The rock surface was coated with Canada balsam and a glass microscope slide pressed on top. The slide was then coated with a protective layer of wax. The rock was dissolved away using hydrofluoric acid, leaving the resistant fossil, in reverse, on the Canada balsam.

Canada balsam is of importance in optics because its refractive index (1.53 for the sodium D lines) is close to that of glass (Liu, 1971). It has been used as a mounting medium since the 1830s. Within the entomology collection at the NHM, 150 year old slides have not crystallised or absorbed moisture (Brown, 1997). Canada balsam is considered by Mound and Pitkin (1973) to be the only mountant that can be kept in a variety of climates without deteriorating. It does yellow with age but most researchers do not see this as a major problem (Brown, 1997).

> "Resin-based mountants such as Canada balsam and Euparal, which have stood the test of time, are probably the best option for permanent mounts." (Carter and Walker, 1999)

This is echoed by other workers: An informal internet discussion by Halliday (web 1) found that most workers consider Canada balsam to be the most suitable mounting medium. Galtier and Phillips (1999) state that "natural balsam remains unaltered by oxidation over long periods of time". After an extensive survey of the microscope slide mountants within the entomology collection at the NHM Brown (1997) also concludes that Euparal and Canada balsam are unsurpassed by modern materials. Euparal (a mixture of eucalyptol, camsal, paraldehyde and sandarac) is the best alternative to Canada balsam. This is because Euparal does not need to be dissolved in carcinogenic xylene (isomer of dimethyl benzene) and does not yellow with age (Brown, 1997). Euparal, however, can damage fine structures due to early development of a meniscus and can also craze if poorly prepared (Hood, 1940).

Current Condition of Specimens

Currently the Canada balsam is cracked, pieces are flaking off and being lost (Fig. 1). This is because the resin was heated too much during preparation and volatiles were lost too rapidly (Shute, C., Palaeontology Dept, NHM and Brown, P., Entomology Dept, NHM, *pers comms*, 2004). This has severely reduced the longevity of the mountant. In some cases the resin has separated from the glass rendering microscopical investigation impossible.

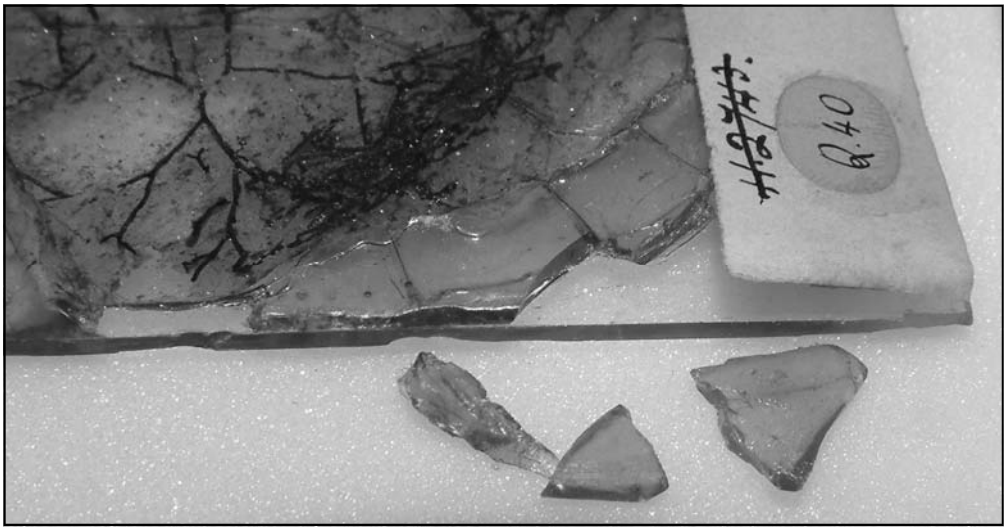

Fig. 1. Crazed Canada balsam slide containing dendroid graptolite.

Discussion of Possible Techniques

The advice of colleagues and expertise from the literature is invaluable when choosing what methods and products to use. Inspiration for new methods can also be gained from extensive reading. A literature survey was undertaken to enable decisions on how to conserve the slides and from that experimental methods were adopted. Personal communication with colleagues at the NHM and other institutions was utilised to enhance ideas developed from the literature and to identify the most appropriate methods and equipment to enable investigation.

According to Jones and Rowe (1999) successfully removing specimens from resin and reusing them is a "luxury [that] only applies to some fossil plants and spores." Carter and Walker (1999) state that it is impossible to recover specimens from cracked resin. A method for removing the graptolites and re-embedding them or repairing their existing mounting medium must be established. The curator would prefer that the graptolite should remain the same way up and that the surface of the specimen remains exposed. It is important to preserve the integrity of an object, especially if it is figured, as well as maintaining an exposed surface is important for light microscopy.

The suitability of Canada balsam as a storage medium has been established. It can therefore be used in methods to restore the specimens. Brunner and Blueford (1986) recommend the use of microwaves to re-heat Canada balsam slides. They state that conventional re-melting methods, using water baths, sand baths, bunsen burners and electrical hotplates, pose a risk of damaging or losing specimens. They believe that microwaves are superior because they heat the balsam from inside outward, the process does not heat the slide, heat intensity and duration is easy to control, and small ovens can be placed within fume cabinets. A microwave oven uses radio waves which are converted directly into atomic motion. At standard frequencies they are not absorbed by most plastics, glass or ceramics, and hence will not heat up the slide. In microwave cooking, the radio waves penetrate the food and excite water and fat molecules evenly throughout. The whole heating process is different than in an oven because it does not rely on the conduction of heat. Radio waves can, however, penetrate unevenly if the subject varies in thickness, and there are also "hot spots" caused by wave interference (web 2). The microwave method was used by Brunner and Blueford (1986) to reposition robust specimens. For their slides with cracked Canada balsam they added a drop of xylene and allowed them to stand over night under a dust cover.

One contributor in the survey of Halliday (web 1) found that specimens in balsam could be recovered by soaking in xylene and remounted. The process involves soaking the slides until the fossil floats freely, then slowly replacing the xylene with glycerol until it traps the specimens against the floor of the container. Approximately 5mm deep extra layer of glycerine would then be added and the alcohol removed with a pipette (Rickards, B., Dept of Earth Sciences, University of Cambridge, *pers comms* 2004). The material in question was, however, more robust than a graptolite. The dendroids would be too fragile for this technique. Another possible method would be to inject the slide with xylene and gradually add Paraloid B72 (poly (ethyl methacrylate-*co*-methylacrylate)) to the solution, thereby replacing the Canada balsam.

Transferring the slide by inversion into fresh resin would not fulfil the curator's request, but may be the only way to preserve the specimens. The fossil could be covered in Canada balsam, a slide pressed on top and the old slide removed with a hot knife or xylene. Canada balsam is a highly resistant medium and it becomes less soluble with age as volatiles are progressively released. The new layer of resin may dissolve before the old layer can be removed. A much simpler method would utilise an epoxy resin, such as Fynebond® (web 3), which is insoluble in xylene: the new resin would be poured on top of the fossil and the old resin dissolved with xylene, leaving the overturned fossil in the epoxy. This would pose less risk of damage to the specimen, but the result would be irreversible and the original surface would lie underneath. An alternative could be a double transfer: once into wax and then, when the old balsam has been removed, back into fresh balsam. The most suitable option would be carbowax™ (Polyethylene Glycol 1500), which is often used in palaeontology to make water-soluble supports during preparation. Carbowax is insoluble in xylene and should not dissolve whilst the old Canada balsam is being removed.

Crizzled glass can be impregnated with polymeric materials to consolidate the surface but this has been found to be ineffective (Brill, 1975). This process has been used to create fake amber fossils: Dr Ross at the NHM discovered a Canada balsam and amber hoax, where a fly "fossil" had been created within a central hollow. Fresh Canada balsam could therefore be added to the graptolite slides to fill up the cracks. This, however, obscures the surface desired by the curator.

Investigation and Discussion of Possible Methods

Slides were selected from a range kindly donated by various members of the palaeontology department. Only two slides were graptolites which had been prepared by O.M.B. Bulman. The remaining slides were chosen for their similarities: crazed Canada balsam holding delicate fossils. Several experiments were set up according to methods in the published literature, recommendations by colleagues or based on personal inspiration.

Microwave: The slides were heated at various temperatures and durations in an Amana® Commercial 1100 Watt microwave. At low temperatures and duration of up to 10 minutes no effect was made on the Canada balsam. At the highest temperature setting and 5 minutes bombardment duration, one sample achieved plasticity and two of the cracks sealed up but were still visible. One of the cracks opened up and a new crack appeared along side the embedded fossil, separating part of it from the main area. Many small vesicles appeared within the Canada balsam and it became darker in colour. Another sample was even more adversely affected. Many of the shards of resin flew off the slide completely. Those that were left melted. At high temperature and 6 minutes of bombardment the Canada balsam liquefied and darkened further, the vesicles were still present and the fossil became distorted. In Brunner and Blueford (1986) this method was used to free-up fossil radiolarians and reposition them, it was not used for repairing cracks or on large fragile fossils like the graptolites.

Xylene drops: Droplets of xylene were applied to the crazed slides using a pipette but evapourated before any effect was perceptible. Xylene is harmful or fatal if swallowed. Its vapour is also harmful and can affect the central nervous system. Xylene can also cause severe eye irritation, causes irritation to the skin and the respiratory tract. Chronic exposure can cause adverse liver, kidney and blood effects. The liquid and vapour are both flammable (MSDS number X2600). The slides were then placed in a polypropylene box on a layer of sympatex. Xylene was poured in until it covered the slides by 0.5mm depth. The lid of the box was then sealed and progress monitored every 5 minutes. After the first five minutes the Canada balsam appeared to be much clearer and cracks were beginning to heal, but crystals were forming on the surface. Over time the upper level cracks continued to improve but the crystals worsened. There were no changes between 20 and 45 minutes. The slides were then left to soak in the xylene for 3 days. When dried the crystals had disappeared and many of the cracks had healed but other cracks had opened up wider than before. One of the fossils suffered severe damage.

Exchange with Paraloid: The initial theory proposed injection of xylene, gradually changing the solution into Paraloid B72. The slide was soaked for an hour in xylene and the excess surrounding resin removed with a metal spatula. Then the xylene and paraloid solution was injected. Unfortunately Paraloid also dissolves in xylene and the resins became mixed. A drop of Paraloid was added on top of the slide to stabilise it. Around 30% of the fossil was lost in this process.

Transfer: Drops of Canada balsam in xylene were added to the slide, a new glass slide was pressed on top and the resin was allowed to set. A hot wire was then passed in between the two slides to melt the old Canada balsam and release the fossil. This method did not work: the fresh Canada balsam, richer in volatiles, melted preferentially and the fossil was disturbed by the wire's progress.

Double Transfer: Carbowax was heated in a microwave on full power for 1 minute and then cooled for 30 mins until crystals began to form. This prevented melting of the Canada balsam. The label was removed from the slide using a methyl cellulose poultice and reinforced with repair paper to prevent damage during treatment. Carbowax was poured over the fossil and allowed to cool. The slide was soaked in xylene until the resin had dissolved (this can take a few days or may take weeks, depending on the age and thickness of the resin). The fossil remained embedded in the carbowax. A new layer of Canada balsam in xylene was applied to the exposed fossil and a new glass slide pressed on top of it. After 2 weeks the carbowax was removed by soaking in water over night. The label was then reapplied to the slide using Paraloid B72 in acetone.

Conclusion

The microwave method produced very unsatisfactory results; the resin darkening and developing vesicles. The addition of fresh Canada balsam worked well to stabilise the slides, but does not fulfil the curator's requirement for exposure of the original surface. Adding drops of xylene was ineffectual, whilst soaking the whole slide in xylene led to damage of the fossil. The attempt to exchange Canada balsam with Paraloid B72 was completely unsuccessful; the two resins blended. During the inversion method the fossil was disturbed and the requirements of the curator were not met. The double transfer method, utilising carbowax, worked extremely well, although one of the graptolites became slightly disturbed during an early trial which had not allowed sufficient time for the fresh Canada balsam to set (Fig. 2). This was because the fresh resin was put into the water bath before it had set properly.

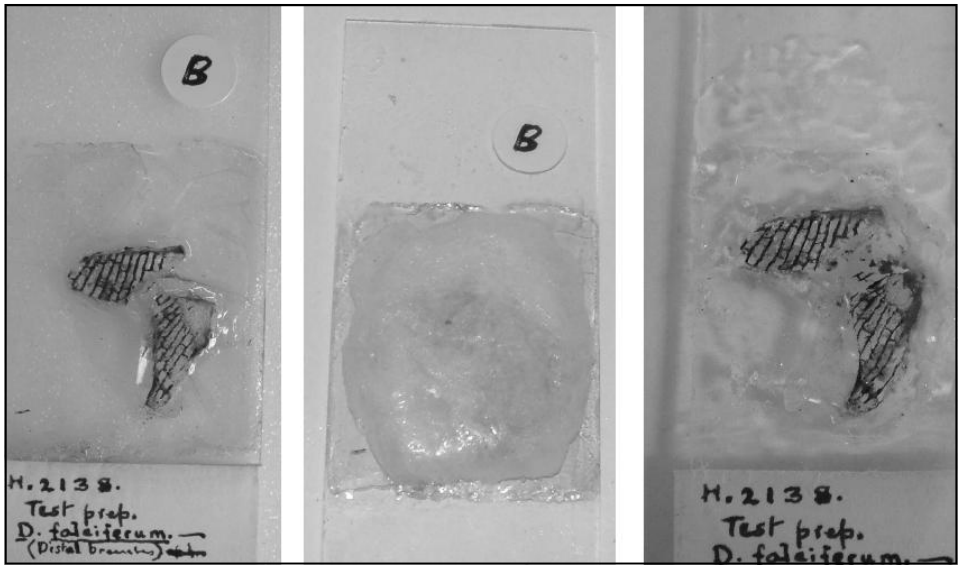

Fig. 2. An example of one of the slides treated with the double transfer technique. The photograph on the left is of the slide before treatment, the photograph in the centre shows carbowax during treatment and the photograph on the right is the slide after treatment. This particular example suffered disturbance because not enough time was allowed for the fresh resin to set.

Experimentation indicated that the best method for restoring the cracked Canada balsam slides is the double transfer technique. This method was subsequently applied to all of the slides mounted in Canada balsam within the graptolite collections in the Palaeontology Department. The process can take many weeks but

each step in itself is not time consuming. The total time actually taken is less than an hour per slide and relative time can be reduced if several slides are treated together. Care must be taken to ensure that the specimens remain undamaged. The greatest risk is from indelicate handling, the application of carbowax and fresh Canada balsam does not affect the fossils adversely. During the treatment the fresh resin is sandwiched between the wax and the slide, allowing a very small surface area for off-gassing and making it difficult to tell when the central areas are fully set. This causes a potential risk of premature transferral into the water bath. The fresh Canada balsam must therefore be allowed at least 4 weeks to set to prevent movement of the specimen, before the slide is immersed in water (for removal of the carbowax). If the fresh Canada balsam is not completely cured before the slide is immersed in water, it can also take on a slightly clouded appearance. This can be very simply remedied by applying a drop of xylene to the upper surface and allowing it to dry in a fume cabinet. As with all conservation treatments, the double transfer technique should only be performed if neglected specimens would be damaged or lost. It should not be carried out on stable slides. If appropriate care is taken, however, this treatment poses no risk to the specimens.

The use of this method on fossilised material prone to pyrite oxidation should be carefully considered, since the process involves water. If this method is used on such specimens, then the slides must remain immersed for the minimal amount of time (check after an hour) and dried as quickly as possible. Pyritic fossils should subsequently be stored in low humidity or oxygen-free microenvironments.

The resulting mount is reversible using the same method and the original surface is exposed, as requested by the curator. This is a valuable new method for use in restoring any delicate Canada balsam mounted slides.

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References

Web 1 - Halliday, R. B., 'Microscope slide mounting media: results of informal survey', http://www.nhm.ac.uk/hosted_sites/acarology/archive/summary.html (accessed 27 November 2006).

Web 2 - Marshall, B., 'How microwave cooking works', http://home.howstuffworks.com/microwave2.htm (accessed 27 September 2005).

Web 3 - 16 Vidler, K., 'Conservation of a tortoiseshell book cover', http://www.vam.ac.uk/res_cons/conservation/journal/number_50/tortoiseshell_cover/ (accessed 12 October 2005).

Brill, R. H., 'Crizzling: A Problem in Glass Conservation', *Conservation in Archaeology and the Applied Arts*. Reprints of the Contributions to the Stockholm Congress, 2-6 June 1975, The International Institute for Conservation of Historic and Artistic Works, London (1975) 121- 131.

Brown, P. A., 'A Review of Techniques used in Preparation, Curation and Conservation of Microscope Slides at the Natural History Museum, London', *The Biology Curator* **10** supplement, (1997) 1- 33.

Brunner, C. A., and Blueford, J. R., 'Restoration of Radiolarian Strewn Slides made with Canada Balsam', *Micropaleontology* **32** (1) (1986) 43- 45.

Carter, D., and Walker, A., 1999. *Care and Conservation of Natural History Collections.* The Natural History Museum, Butterworth Heinemann, Oxford (1999).

Elvers, W. B.,. Preparation of fossil specimens for acid development by the transfer method. In Forty-eighth annual meeting, Society of Vertebrate Paleontology; abstracts of papers. *Journal of Vertebrate Paleontology* **8**(3) Supplement:14A, (1988).

Galtier, J., and Phillips, T. L., 'The acetate peel technique', in *Fossil Plants and Spores,* ed. T. P. Jones and N. P. Rowe, Geological Society, London (1999) 67- 70.

Hood, J. D., *Microscopical Whole Mounts of Insects* Cornell University (1940).

Jones, T. P., and Rowe, N. P., 'Embedding techniques: adhesives and resins', in *Fossil Plants and Spores,* ed. T. P. Jones and N. P. Rowe, Geological Society, London (1999) 71- 75.

Liu, T. S., *A Monograph of the Genus Abies,* Department of Forestry, College of Agriculture, National Taiwan University, (1971).

Mills, J. S., and White, R., *The Organic Chemistry of Museum Objects*, Butterworth and Co Ltd, London (1987).