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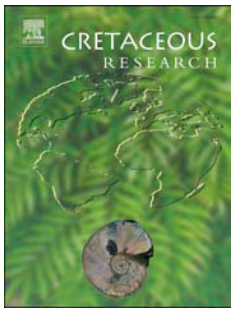

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The resin transfer technique: an application to insect fossils in laminated limestones of the Crato Formation (Lower Cretaceous) of north-east Brazil

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Abstract

Documented here is a variation of the widely used resin transfer technique, allowing for the preparation of fragile articulated fossil arthropod material preserved in laminated limestone. The extensive use of the resin transfer technique in palaeontology has traditionally been restricted to palaeobotany and vertebrate palaeontology. The parameters discussed here allow for its application to three-dimensional arthropod fossils preserved in acid resistant minerals (e.g. iron oxides such as goethite, the sulphides pyrite and marcasite, and silica) in laminated limestone. In the experiments described here we have utilised fossils from the Lower Cretaceous (Aptian) Crato Lagerstätte of north-east Brazil. The equipment and methodology are outlined, along with cautions and concerns when using this technique.

Key Words: Resin transfer, fossil preparation, acid digestion, limestone, Arthropoda, Brazil, Early Cretaceous.

1. Introduction

When a fossil is first exposed from within a laminated limestone, it typically splits into part and counterpart specimens, not necessarily in equal halves. The fossil can remain entirely on one surface, producing a perfect external mould on one slab and the entire body fossil on the other, but such an occurrence is unusual. In many (probably most) cases, the plane of splitting extends through the fossil, leaving its remains on both surfaces and causing considerable damage. The freshly exposed surfaces are also vulnerable to further damage during excavation, transport, preparation, curation, and analysis, and are especially vulnerable to reunification of the part and counterpart, which should be avoided. Palaeontological preparators have long sought to remedy this issue with the application of translucent resins to set exposed surfaces, stabilising and protecting the fossil.

Several resin-transfer methodologies have been developed to prepare or preserve fossils, typically for palaeobotanical or vertebrate material (Kühne, 1961; Mayr et al*.,* 2006; Escapa et al*.*, 2010; Graham and Allington-Jones, 2015, among others). In all methodologies, an epoxy or polyester resin is used to embed the specimen such that, in multi-component fossils, removal of the matrix can proceed without losing contextual information of the fossil's individual elements, or to preserve the integrity of a delicate fossil when the matrix is highly unstable (e.g. oils shales such as the Eocene Messel shales of Germany) (Micklich and Klappert, 2001; Smith and Wuttke, 2012). Usually, the fossil is removed completely from the matrix and is left partially embedded within the epoxy resin, although variations in its application will determine different outcomes. For example, in some techniques, the specimen is simply protected from further damage by a transparent resin coating (Holm, 1890; Lepage and Basinger, 1993). In others, the fossil is embedded in resin, then the opposite pristine surface exposed via mechanical or chemical preparation (Cridland and Williams, 1966). Alternatively, a fossil may be completely entombed in resin, and re-exposed for examination via serial sectioning and polishing (Escapa et al., 2010). Resin entombment of fossils was first described in the 1800s (Young, 1877; Holm, 1890), but resin setting and the subsequent removal of

surrounding sediment using acids was not practiced until the following century (Bather, 1908; Walton, 1923). The technique has evolved through several iterations, allowing the development of acetate peel techniques (Abbott, 1950; Abbott and Abbott, 1952). Over the last century, numerous museum specimens (particularly vertebrate fossils) have been embedded in resin, either as a simple coating or a complete transfer (Bather, 1908; Toombs and Rixon, 1950; Kühne, 1961; Cridland and Williams, 1966; Bonde and Christiansen, 2003; Graham and Allington-Jones, 2015). Some of these fossils were originally coated in resin and their surrounding sediment was not chemically removed until decades later (Barthel et al., 1990; Bonde and Christiansen, 2003; Bonde and Leal, 2015). In many of these 20th century preparations, the resins used were phenolic (Bakelite) or polyvinyl alcohol, with Bakelite sometimes bulked with ground-up sediment to be used as an adhesive (Graham and Allington-Jones, 2015). Time has shown that the clear resins can discolour and deteriorate with age, or may shrink while curing (Lindsay, 1986, 1995; Rutzky et al. 1994). Discolouration is particularly problematic when in the presence of ultraviolet light, which readily causes the resin to discolour yellow (Down, 1986).

While the use of the resin transfer technique has become widespread in palaeobotany and vertebrate palaeontology, it has seen limited application to other fossil groups. The refined technique presented here discusses how resin transfer (setting the fossil) can be applied to arthropod fossils preserved in laminated limestones, as well as addressing some of the key concerns regarding the long-term survival of the prepared fossil. For this technique to be successful, the specimen must possess several key characteristics. The rock surrounding the fossil must be soluble in the selected acid, whereas the fossil material and resin must be insoluble in the same acid. The matrix surrounding the fossil must be sufficiently porous for the resin to penetrate around the fossil, setting it securely, but not so porous that the resin penetrates past the fossil, entombing it entirely; a factor that can be controlled by varying the resin viscosity. Finally, there must be no diagenetic alteration or mineral precipitation around the fossil that acts as an insoluble coating, such as silica 'haloing' or insoluble concretion formation (Mizutani, 1970; McCoy, 2013). The suitability of a

specimen for resin transfer usually can be assessed by dropping 1 – 2 drops of the chosen acid on the matrix and observing its effectiveness in dissolving it. For a more precise assessment, the specimen can be subject to energy dispersive X-ray analyses to determine the chemical composition of the matrix.

The methodology presented here sets the fossil with a single resin coating (on the fossil baring surface only), allowing the matrix-entombed part of the fossil to be subsequently exposed via matrix dissolution. This allows for pristine fossil material to be subject to analytical procedures where direct observation is essential (e.g. scanning electron microscopy).

2. Equipment

2.1. Specimens

The example specimens used here are a collection of fossil insects from the Lower Cretaceous (Aptian) Nova Olinda Member of the Crato Formation, north-eastern Brazil (Barling et al., 2015). The Crato Formation is a world famous Konservat-Lagerstätte that yields a diverse flora and fauna, and has been studied extensively (Martill et al., 2007 and the references therein). Importantly, these fossils are preserved primarily as goethite (FeO(OH)), apatite ($Ca_5(PO_4)_3(OH,F,Cl)$), and pyrite (FeS_2) replacements, whereas the surrounding matrix is 99% re-crystallised calcium carbonate ($CaCO_3$) of varying porosity (Barling et al., 2015; Osés et al., 2016). Rarely, galena, sphalerite and barite also occur as part of the diagenetic mineral assemblage (Martill et al., 2007).

The specimens were donated by an anonymous German collector for a Ph.D. project at the University of Portsmouth. Somewhat ironically, they were donated as ‘poor-quality’ fossils considered of little scientific or commercial value. When Crato Formation fossil insects are found, quarry workers usually rub them ‘clean’ to remove loose sediment. This ‘cleaning’ seriously damages the exposed fossil, often obliterating any microscopic surface detail. As such, these fossils are a

prime example of how seemingly low-quality specimens to be prepared via resin transfer to reveal otherwise obscured high-fidelity preservation.

2.2. Resins

Polymer resins can be used as embedders, consolidants, adherers, and barriers in the preparation and curation of fossil specimens. In this project, resins are used to consolidate the exposed surface of the specimen and set the fossil material. Many resins are commercially available for this technique (Horie, 1987; Davidson and Alderson, 2009; Graham and Allington-Jones, 2015). Amongst the most highly recommended resins for the preparation of fossils is Synolite 0328-A-1, a pre-accelerated, thixotropic, low styrene emission, orthophthalic based unsaturated polyester resin (Graham and Allington-Jones, 2015), although it is largely untested on arthropod fossils. Unfortunately, Synolite 0328-A-1was not available for this project and instead two epoxy resins were used to examine different viscosities and prices. Buehler EpoThin Epoxy Resin is used as a relatively expensive 'high-quality' low-viscosity resin and Bisphenol A (and its associated hardener m-xylylenediamine) is used as an inexpensive 'poorer-quality' high-viscosity resin. While Buehler EpoThin Epoxy Resin is available through specialised scientific suppliers, Bisphenol A is available from several high-street retailers. Of these two resins, the cheaper Bisphenol A proved most effective. It, and similar high-viscosity resins are recommended here. Lower-viscosity resins frequently penetrate past the fossil, masking it in an insoluble opaque resin-sediment mix. Bisphenol A cures at a high temperature and, while this had no apparent effect on the specimens herein, must be considered before preparing fragile heat-sensitive specimens.

2.3. Acids

Acetic acid (CH_3COOH) at 10% concentration was used to remove the limestone matrix around the fossils. On rare occasions, this did not completely dissolve the sediment (possibly due to the presence of calcium phosphate cements), in which case 10% hydrochloric acid (HCl) was a suitable alternative. Further alternative acids (such as formic (CH_2O_2) and sulphamic (H_3NSO_3)) were not

investigated in this project but may be suitable. However, many Crato Formation insect fossils preserve labile internal tissues (e.g. flight muscles) in apatite (Barling et al., 2015; Osés et al., 2016), which readily dissolves in hydrochloric acid. Consequently, the use of HCl may damage the fossils and it must be used with caution. The duration of acid digestion varies between specimens. This is dependant largely on specimen size and rock porosity, which, in the Crato Formation limestones, is greatly controlled by the extent of weathering.

2.4. Buffering agents and other solutions

The role of a buffer in acid digestions is to prevent a sudden change in pH when a new acid or base is added to the solution (Jeppsson et al., 1985). In this project, a buffer was used to protect normally acetic acid-resistant fossil tissues (preserved in apatite) from dissolution when the digestion solutions were 'topped up' (explained below in section 3.1). Without a buffer, 'topping up' the solution would have resulted in a sudden decrease in pH and the dissolution of these fossil tissues. Here, the guidelines for using 'acetate soup', as outlined by Jeppsson et al. (1985), are followed albeit with the 'topping up' solution composed of 20% 'spent' acetic acid (containing calcium acetate) and 80% 'fresh' acetic acid. On the rare use of hydrochloric acid, a similar 'soup' of spent acid and fresh acid can be used as a buffer, albeit using spent hydrochloric acid (or calcium orthophosphate) (Kim and Park, 2008; Bonde and Leal, 2015). A buffer was not required for the initial immersion of a specimen in acetic acid, as these vulnerable fossil tissues were not yet exposed.

At each stage of removal from a liquid medium, one-to-two drops of 2-5% Decon 90™ (an emulsion base of anionic and non-ionic surface active agents, stabilising agents, non-phosphate detergent builders, alkalis, and sequestering agents) (Decon Laboratories Ltd., 2018) was added as a 'flow aid'. This reduces water surface tension and helps prevent damage to fragile fossil structures as they break the surface tension. An ample supply of pure water and acetone are also required.

2.5. Initial sample preparation

Acid preparation is a destructive technique, and for security a photographic record of the specimens should be made prior to matrix dissolution, ideally including a detailed written report of their condition. Specimen numbers and additional information must be recorded separately, with labels and adhesives removed. Any information written onto the specimens must also be removed, as permanent marker ink and pencil graphite will dislodge during matrix dissolution, contaminating the specimens. The specimens should be lightly washed with acetone to remove grease and pure water to remove dust.

Broken specimens that have split into several pieces can be reconnected and should be physically held in place by clamps rather than adhesives (resins, glues, or cements) before being embedded in the resin. Using adhesives to repair a broken specimen will cause its final exposed surface to be partially obscured. Part and counterparts should not be reunited. If a specimen has already been glued or cemented, the glues and cements should be removed where possible without compromising the integrity of the fossil.

Excess rock should not be removed as vibration from rock cutting equipment may damage the fossil. Instead, excess rock can be removed later, after the fossil has been set in resin.

2.6. Extra kit

All chemical and resin preparation should be carried out under a fume hood with appropriate PPE (personal protective equipment) when handling all chemical components (including gloves, protective goggles, and a laboratory coat). Industrial rock cutting equipment may be required, depending on the amount of excess rock surrounding the specimen and full health and safety protocols must be followed when using such industrial equipment.

Disposable containers and stirring implements are required for mixing and pouring epoxy resins, including disposable cardboard containers. Plasticine® (a non-drying putty-like modelling clay) is used here to create a reservoir for the resin around the fossil, although many other alternative

products could be used to create a reservoir. In this project, resin and hardener were mixed in beakers. Alternatively, measuring cylinders can be used for increased accuracy when measuring resin-to-hardener ratios.

3. Methodology

The technique outlined below leaves fossils particularly fragile. Extreme care should be taken when handling resin transferred specimens and it is suggested that they are stored in protective containers. In this project, resin transferred fossils were subsequently mounted on steel stubs and stored in the same manner as scanning electron microscope specimens. If specimens do not require direct observation, a second translucent resin coating can be applied to the exposed surface to set it as well.

Here, an instructional step-by-step guide for the resin transfer technique is presented, accompanied by a simplified diagram (Fig. 1) and example photographs (Fig. 2). Figure 1 illustrates how the fossils are initially damaged during extraction, prior to the instructional guide itself.

3.1. Instructional guide

- A > 2 cm high Plasticine® reservoir should be constructed around the specimen on the bedding surface, encircling it completely. Care must be taken to ensure there is an adequate seal around the specimen to prevent resin escaping the intended area. If the fossil is centred on a slab less than 4 cm in diameter, the resin should encircle the margins of the slab rather than be placed solely on the bedding plane surface.
- The specimen with its Plasticine® reservoir should be placed in a disposable low-sided cardboard container on a level surface. This will prevent resin leakages from damaging any other equipment or surfaces.

- Prepare the resin. For Bisphenol A and its associated hardener m-xylylenediamine, thoroughly mix at a ratio of 1:1 for 2 minutes. The ratio of hardener can be altered to affect the rigidity when cured. Experimentation may be required to ascertain which ratio provides the best result for the specimens being transferred.
- The resin must be carefully and slowly poured over the specimen, until the desired thickness is achieved. Bisphenol A is a relatively flexible resin, and it is recommended here that it is set at least 1 cm thick to protect the fossil. Any thickness above this will depend on the size of the fossil. For fossils larger than 4 cm in length, the resin should be cast thicker. Avoid introducing bubbles to the resin where possible. Small bubbles will usually rise to the surface of the resin and can be teased to the margins or popped using a needle. However, this is generally not necessary as the uppermost surface of the resin during curing (where bubbles accumulate) forms the back side of the final specimen and does not interfere with the set fossil.
- The resin should be left to cure fully. This may take several days, depending on its thickness and ratio of hardener.
- Once the resin has cured, the Plasticine® reservoir can be gently peeled away. Some small traces of Plasticine® may need to be scraped or buffed away. Care should be taken to remove all of the Plasticine®, as it can contaminate the specimen during matrix dissolution and analyses.
- Excess rock can now be removed using a rock saw or pincers if the slab is sufficiently thin. The resin sets the fossil, stabilising it and reduces the risk of vibration damage during cutting. To further stabilise the specimen, the set resin could be protected and sealed with a temporary liquid proof cover or film, however this was not undertaken for the specimens prepared herein.
- The matrix is now subject to dissolution in an appropriate acid. Gently pour the acid into an appropriate container under a fume hood and place the specimen in it, resin-side-down. We

recommend acetic acid as there is a chance that some internal soft tissues are preserved in apatite and will be damaged if hydrochloric acid is used.

- Matrix dissolution will likely take several days, depending on the amount of and solubility of the matrix. It may be preferable to monitor acid pH during digestion to maintain consistent dissolution. During this time, the spent acid will periodically require decanting and 'topping up' with 'acetate soup' (Jeppsson et al., 1985). Any decanted acid must be appropriately neutralised. In this project, acids were neutralised with sodium bicarbonate ($NaHCO_3$). Care must be taken while doing this to not damage the specimen, especially in the last few hours of matrix dissolution. The acid must never be allowed to evaporate below the specimen as this will result in contaminant mineral growth (calcium acetate) and surface tension effects that may damage the exposed fossil. Evaporation rates can be controlled by keeping a lid on the container.
- Depending on the content of the matrix, a gritty and foamy surface may form on the acid surface that should be decanted every 6–12 hours. This can be gently scooped off with a spoon. A soupy mixture of insoluble material may also form at the bottom of the container. This can generally be ignored, unless it accumulates to a level where it may contact the fossil. If there is too much insoluble material, carefully transfer the specimen into a mixture of 80% 'fresh' acid and 20% 'spent' acid to prevent partial dissolution of phosphatic minerals (Jeppsson et al., 1985; Kim and Park, 2008; Bonde and Leal, 2015; Graham and Allington-Jones, 2015).
- Post-matrix dissolution, the specimen will be extremely fragile. Consequently, at all stages of moving it in or out of a liquid, 1–2 drops of 2–5% Decon 90 (or another flow-aid) should be added to prevent damage to the specimen while breaking surface tension.
- Once matrix dissolution is complete, carefully remove the specimen from the acid, gently clean it in two or three baths of deionised water for 12–24 hours to thoroughly dissipate any

remaining acid. Neutralising agents should not be added, as these contaminate the specimen with salts (Fig. 3).

- Once cleaned, the specimen should be left to dry in a sealed desiccator for 1–2 days to prevent dust contamination.
- Finally, the specimen should be stored in a small portable desiccator or sealed container with blue indicating silica granules to control relative humidity. In this project, specimens were also stub mounted and gold/palladium coated for scanning electron microscopy and are stored securely in stub holders.

4. Discussion

4.1. Comparison to vertebrate transfers

Currently, the most comparable and widely used resin transfers are performed on vertebrate fossils (Lindsay, 1986; Mayr et al., 2006; Graham and Allington-Jones, 2015). However, the methods differ in several key ways from that of the insect resin transfers described here. First is the scale, which requires different aspects of the transfer to be prioritised. For example, Synocryl 9122x (poly (butyl methacrylate) thermoplastic acrylic polymer in xylene) has been used as an acid-resistant protective coating as fossil material is revealed during dissolution , masking exposed material because it can be removed easily with acetone or by mechanical preparation later (Schiele, 2008). If this method were applied to arthropod fossils, the fossil may disintegrate during resin dissolution or be damaged during mechanical preparation. Additionally, vertebrate fossils are often preserved almost entirely in calcium phosphate and so ‘spent’ acetic acid (containing calcium acetate) or calcium orthophosphate must be added to prevent damage to the fossil during matrix dissolution (Kim and Park, 2008; Bonde and Leal, 2015). Although the insect fossils used here appeared to be preserved almost entirely in goethite, many later revealed internal soft-tissue preservation in apatite. As such, the addition of ‘spent’ acetic acid as a buffer was vital to protect those soft tissues.

The fragility of fossil arthropods is a serious point of concern during resin transfer. Typically, the appendages and articulations of arthropods are much smaller than those of vertebrates. When fossilised, these can be exceptionally fragile, disarticulating or breaking under even the slightest pressure. Consequently, the entire transfer must be designed around protecting these fragile structures. Slow, weak dissolutions are preferable, and cleaning of specimens should also be as careful as possible. The preparatory method presented here was developed while preparing specimens for a PhD project, and so dissolutions were undertaken with 10% acetic acid due to time constraints. Future dissolutions could be undertaken with much lower acid concentrations (possibly as low as 0.5%) but may take several weeks.

Due to the diminutive size of many fossil arthropods (particularly insects), further analyses often require an electron microscope. If transferred specimens are to be examined under a scanning electron microscope, micron-scale contamination must also be taken into consideration. All precautions should be taken to prevent contamination, including the non-use of neutralising agents. It is always recommended that pure water be used when preparing specimens that will be viewed under a scanning electron microscope (Fischer et al., 2012).

4.2. Examples of successful transfers

The quality of preservation revealed by a successful transfer is invariably controlled by the original fidelity of preservation of the fossils being prepared. What is so extraordinary about the resin transfer technique is that this fidelity can be revealed to the naked eye in a manner unmatched by other forms of preparation. If executed properly, the technique will completely remove all obscuring material, leaving only pristine fossil material. In fossils with high-fidelity preservation, this will reveal sub-micron scale replications of arthropod cuticle and internal soft tissues (Fig. 4; Barling et al., 2015). Fossilised internal tissues can be exposed carefully, without causing excessive damage to the surrounding cuticle (Fig. 5; Barling et al., 2015). A down-side of this technique is that it can fail if

certain criteria are not met, leaving the fossil exceptionally fragile, and presenting a series of additional challenges.

4.3. Challenges

The resin transfer technique is not always successful and there are several variables that can cause it to fail. Consequently, it is not recommended to attempt a resin transfer on type specimens, rare specimens, or otherwise irreplaceable specimens. The primary controls on the success of a transfer are the viscosity of the resin, the properties of the host sediment, the three-dimensionality of the fossil, and the degree of mineralisation. Most importantly, if the resin viscosity vs matrix porosity is mismatched, the fossil may be destroyed. The resin may penetrate past the fossil, entombing it in an indigestible mix of matrix and resin, or the fossil will not be secured and will disintegrate during matrix dissolution (Fig. 6). Careful attention should be paid to the host sediment and disposable specimens should be experimented upon first. For the Crato Formation, the sediment porosity is largely controlled by the extent of weathering. The plasticity of the resin when cured is also important to consider. Soft flexible resins will not adequately protect the fossil. Ideally, each fossil should be uniquely considered (in terms of specimen size and matrix porosity) and an appropriate resin chosen.

Plasticine® was used to create a reservoir for these specimens, however this can be difficult to remove after the resin has cured. Unremoved Plasticine® will contaminate the specimen during matrix dissolution. While this had little impact on specimens viewed under a light microscope, it was particularly problematic for specimens examined with scanning electron microscopy. Additionally, permanent marker ink and pencil graphite will enter suspension during matrix dissolution and, much like the Plasticine®, may contaminate specimens. It may be easier to use a solid reservoir (i.e. the end of a plastic tube could be cut off to make a solid ring reservoir) and simply cut away cured leaked resin, or to use a separator such as Vaseline®. Dental wax can be used as an easy-to-remove alternative, but this is considerably more expensive.

The preservation of arthropods in laminated limestones is a complex and greatly varied process (Martínez-Delclòs et al., 2004). Each individual fossil has a unique micro-diagenetic history and may behave differently during a resin transfer. Specimens that have minor precipitation of siliceous minerals (or another insoluble mineral) may still be masked by that mineral after the transfer. Although in some cases these superfluous insoluble mineral phases can be removed by mechanical preparation, their presence will usually result in a failed transfer. Some fossil arthropods possess deeply penetrating calcite cements. As these dissolve, they can destabilise the fossil, resulting in disarticulation or even partial disintegration, especially where effervescence forces bubbles between cuticular layers (Fig. 7).

Ultimately, some transfers will simply fail regardless of all efforts to select an appropriate fossil, resin, or acid.

5. Conclusions

Many previous iterations of the resin transfer technique have varying levels of success (Graham and Allington-Jones, 2015), and the technique outlined here is no exception. Nevertheless, when executed correctly, pristine fossils with high preservational fidelity can be exposed. For Crato Formation insect fossils, heavily abraded specimens that appear to have little scientific value can reveal remarkably high fidelity. Many specimens currently confined to university and museum storage could yield a wealth of new data if prepared in this manner.

The application of this technique to other fossil sites is yet to be explored, but may yield similar results. Although many Solnhofen Formation fossil insects are described as preserved in calcite and pyrolousite (Ponomarenko, 1985; Grimaldi and Engel, 2005), they are also preserved in calcium phosphate (Martínez-Delclós et al., 2004). These calcium phosphate specimens could be prepared via resin transfer, with the explicit exclusion of hydrochloric acid. However, the detail revealed will

ultimately be controlled by the original preservational fidelity of the formation, which is reportedly low (Ponomarenko, 1985; Martínez-Delclós et al*.,* 2004; Grimaldi and Engel, 2005). Alternatively, the Yixian Formation fossil insects may be more suitable, as they can be preserved with high-fidelity in goethite or pyrite (Wang et al*.,* 2012). However, the silicaceous content of the Yixian sediments may make selecting an appropriate acid difficult (Fürsich et al., 2007; Zhang and Sha, 2012).

In addition to these two localities, there are several other fossil arthropod sites for which the resin transfer technique may be applicable (e.g. the Calizas de la Huérgina Formation (Las Hoyas) (Buscalioni and Fregenal-Martínez, 2010) and the Tiaojishan Formation (Daohugou) (Wang et al., 2009)). Further experimentation is recommended to assess the use of this methodology for these sites.

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475

476 **FIGURE CAPTIONS**

477

478 **Fig. 1.** Simplified step-by-step process for resin transfer. A, Fossil insect (odonate nymph) enclosed in
479 laminated limestone; B, Black line shows plane of splitting across fossil; C, Typical separation of part

and counter-part specimens. Thick brown line represents fragments of fossil separated on counter-part and arrow highlights damaged exposed fossil surface; D, Reservoir created around specimen (dark blue); E, Resin poured into reservoir, covering fossil; F, Resin allowed to cure, then reservoir removed; G, Specimen immersed in acid to dissolve remaining matrix; H, Matrix dissolved (although sometimes a thin surface film remains), exposing pristine surface of insect with high relief.

Fig. 2. Photographic guide to the resin transfer technique using unnumbered specimen (Blattodea) from the University of Portsmouth collection. A, Overview of entire specimen prior to resin transfer. Red rectangle highlights area magnified in image B. B, Higher magnification image of fossil shown in image A. Pale creamy-brown area of thorax has been damaged by abrasion. C, Approximately 1 cm high Plasticine® reservoir created around the fossil. D, Plasticine® reservoir filled with resin. E-F, Plasticine® reservoir removed, leaving a block of cured resin on the specimen, consolidating the topmost laminae around the fossil and setting the fossil itself. G, Specimen post acid emersion and matrix dissolution, revealing a well-preserved pristine side of the fossil. H, Lower angle photograph, revealing three-dimensionality of the fossil. Scale bars = 1 cm.

Fig. 3. Scanning electron micrograph of contaminating sodium acetate crystals formed from the addition of neutralising agents to a specimen undergoing resin transfer (specimen JW522). Scale bar = 5 µm.

Fig. 4. Scanning electron micrographs demonstrating the exceptional preservation of Crato Formation fossil insects revealed by the resin transfer technique. A, Overview of Hemiptera (specimen FLO15). Scale bar = 1 mm; B-C, Blattodea (specimen JW291) cerci with partially disarticulated setae. B, Arrow highlights area viewed in C. B, Scale bar = 100 µm. C, Scale bar = 10 µm; D, High magnification image of diamond-shaped cuticular scales of Hemiptera (specimen FLO38). Scale bar = 10 µm; E, Broad thin cuticular scales covering a large area of cuticle of Blattodea (specimen NBRL036). Scale bar = 10 µm; F, Cuticular scales of Orthoptera (specimen NBRL059) with sub-micron spines extending along their margins, demonstrating that sub-micron structures can be

retained during resin transfer. Scale bar = 10 µm; G, Remarkably preserved intact and articulated ommatidia of orthopteran, probably an elcanid (specimen NBRL044). Scale bar = 10 µm; H, Sub-micron cuticular meshwork on gena of same specimen. Scale bar = 5 µm.

Fig 5. Scanning electron micrographs demonstrating the potential for further mechanical preparation of resin transferred specimens. Specimens can be prepared carefully with a needle (or stiff single-haired brush) under a light microscope. A, Posterior ventral cuticle of abdomen removed from fossil dipteran (specimen FLO19) revealing preserved ovary, highlighted by arrow. Scale bar = 100 µm; B, Mechanically prepared dipteran (specimen FLO19) limb, revealing 'Orsten-type' preservation of muscle fibres, highlighted by arrow. Scale bar = 10 µm; C, Ventral thorax of same dipteran (specimen FLO19) mechanically prepared to reveal long tracheal tubing. Scale bar = 10 µm; D, High magnification image of thoracic muscle fibres of unidentified insect, possibly Hymenoptera (specimen FLO43). Arrow highlights interlocking repeating unit. Scale bar = 1 µm; E, Mechanically prepared caudal filament of Ephemeroptera nymph (specimen FLO37), revealing complex spiral internal gill structure within, highlighted by arrow. Note that there is still some calcite remaining in this specimen. Scale bar = 10 µm.

Fig. 6. Examples of failed resin transfers on unnumbered test specimens. A, Bisphenol A failing to adhere to Orthoptera specimen, resulting in the loss of the vast majority of cuticle and the creation of an external mould, rather than a resin transfer; B, Buehler EpoThin Epoxy Resin penetrating past the fossil, forming an insoluble mix of resin and sediment masking most of the specimen. Mechanical preparation allows for some of the specimen to be exposed, but is time consuming and risks damaging the specimen further. Scale bars = 5 µm.

Fig 7. Example of resin transfer damage. Scanning electron micrograph of Crato Formation fossil orthopteran (specimen NBRL026) hindlimb damage caused by the dissolution of internal calcite cements. Arrows highlight layers of cuticle that have fragmented and disarticulated. Scale bar = 1 mm.