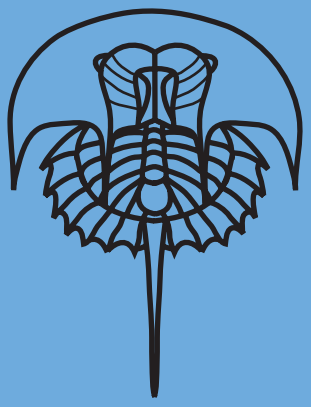

TREATISE ONLINE

Number 65

Part V, Revised, Chapter 11: Graptolite Preparation and Illustration Techniques

Denis E. B. Bates, Jörg Maletz,
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2015

KU PALEONTOLOGICAL INSTITUTE
The University of Kansas

Lawrence, Kansas, USA
ISSN 2153-4012 (online)
paleo.ku.edu/treatiseonline

PART V, REVISED, CHAPTER 11: GRAPTOLITE PREPARATION AND ILLUSTRATION TECHNIQUES

Denis E. B. Bates,[1] Jörg Maletz,[2] and Jan Zalasiewicz[3]

[1]Institute of Geography and Earth Sciences, Aberystwyth University, Aberystwyth, Ceredigion SY23 3DB, United Kingdom, denisbates@uwclub.net;
[2]Freie Universität Berlin, Institut für Geologische Wissenschaften, Malteserstrasse 74-100, D-12249 Berlin, Germany, Yorge@zedat.fu-berlin.de;
[3]Department of Geology, Leicester University, University Road, Leicester, LE1 7RH, United Kingdom, Jaz1@leicester.ac.uk]

INTRODUCTION

The collection, preparation, and illustration techniques for graptolites—as for all fossils—have changed considerably over the last 150 years to enable increased precision in collecting and documentation of scientific material. Nonetheless, certain basics are still followed and have remained unchanged. New discoveries are often made in museum collections, while the most important source of material remains the tireless fieldwork of geologists and paleontologists. The value of collections is enhanced if their exact origins are carefully noted prior to subsequent deposition in public repositories of National Museums and Geological Surveys.

With the change in collection methods, documentation of the material in scientific publications has also changed dramatically. In earlier publications, woodcuts and line drawings were the common method of illustration. The photographic illustration of fossil specimens in scientific books and other publications was rare at the time of Joachim Barrande, James Hall, Hanns Bruno Geinitz, Charles Lapworth, Sven Leonhard Törnquist, Gerhard Holm, and others, as graptolites were just too small to photograph with existing technology. The eventual use of the camera lucida greatly improved the accuracy of drawings. The quality of the scientific illustrations, therefore, is quite variable in earlier publications and often unreliable, hampered by lack of understanding of the fossils investigated and their construction and fossil preservation (see, for example, *Nautilus veles* Richter, 1871, now *Cochlograptus veles*: Maletz, 2001). This situation sometimes results in uncertain identification of type specimens and subsequent taxonomic attribution. Techniques such as SEM and TEM investigation and computer techniques (e.g., computer tomography, X-ray imaging) have provided new insights into graptolite research and will likely provide some unique perspectives on these enigmatic fossils.

COLLECTING GRAPTOLITES

Collecting graptolites does not require any special techniques that are not common to most paleontological collecting. Care should be taken to collect with a precise knowledge of the individual horizons and to document the faunal assemblages. Faunas can change considerably from horizon to horizon, not just in terms of biostratigraphical changes, but also in terms of ecological influences; all available data (e.g., enclosing lithology) concerning these factors should be collected.

Detecting graptolites in rock may require astute attention. Although the classic graptolitic facies is marine black shale, the fossils can occur in various rock types, such as greywackes, sandstones, or carbonate in which the tubaria may not be orientated parallel

Denis E. B. Bates, Jörg Maletz, & Jan Zalasiewicz. 2015. Part V, Revised, Volume 1, Chapter 11: Graptolite Preparation and Illustration Techniques. Treatise Online 65:1–20, 17 fig.

to bedding planes. Graptolites may be more apparent on weathered surfaces than in fresh rock where they may appear whitish from clay coatings or reddish from iron oxide formation. Graptolites that can be easily seen in newly broken fresh rock may become less visible as the inherent moisture dissipates from the surface. Wetting surfaces with alcohol (isopropyl is recommended) usually enhances graptolite definition on both fresh and weathered rock and sometimes reveals otherwise undetected specimens.

Where possible, counterpart pieces should be collected. These can include fragments of the tubarium material on both parts, as well as molds. Material in friable shales should be particularly protected from abrasion of the surface. Wrapping is best done using pliable paper (such as newspaper or paper towels), rather than kitchen paper (waxed paper). Packages of specimens can be held together using tape and put into bags that are labeled with indelible pens. The orientation of material may be recorded; for example, where there appears to be an alignment of specimens. Bulk material, collected for subsequent splitting or acid digestion, is usually more robust. If appropriate, the geologic sections from which specimens are collected should be carefully logged. Both local and national rules on collecting should be obeyed, and appropriate permissions obtained to access private property and other sites.

Splitting shale samples with a bladed hammer or with the help of a chisel is usually easily achieved. In tectonized areas, however, it can be difficult to persuade the rock to split along bedding rather than cleavage planes. After extracting a piece of rock, one needs to identify a bedding plane going through it, which may have graptolites on it. The rock is held in one hand, with the bedding plane dipping towards the hand (Fig. 1), and the other end of the rock is hit by the chisel end of a hammer, with the chisel held parallel to the bedding (Palmer & Rickards, 1991, p. 61).

The immediate marking or labeling of the rock slabs collected is very important. In the past, much care was exercised and scientists labeled each individual slab with the name of the locality and a mark to recognize the important fossil specimens (Fig. 2). Proper labeling is often neglected, and samples are sometimes just placed in boxes with paper notes. Misplacement of these samples in the wrong boxes can easily go unnoticed and may lead to misinterpretations. In Germany, many samples were rendered useless during World War II, as samples and labels were separated and partly lost, leaving the curated material without the essential locality data.

PHYSICAL PREPARATION OF GRAPTOLITES

Developing is the term given to the physical excavation of graptolites by removing rock matrix that overlies and obscures the fossil. Such work is not always necessary. When graptolite-bearing rocks split cleanly along the bedding, then the graptolites (particularly when flattened in fine-grained and well-laminated shales) can emerge more or less entirely visible, and no further work is needed.

However, when graptolites occur in more massive lithologies or, particularly, in tectonically cleaved rocks that do not split easily along the bedding, then all that may initially be seen is the fractured end of a graptolite that seems to disappear into the rock (Fig. 3.1,5). In such cases, a good deal of work must follow to reveal the rest of the fossil.

The typical implement used to develop these fossils is a strong needle mounted in a handle such as a pin vice (available from some hardware shops) or an engineer's scribe. A typical sewing needle is usually too flexible to be effective, while an old-fashioned steel gramophone needle (admittedly hard to find) or something similar is nearer the ideal. The needle-point is best kept very sharp (using an emery stick), though some workers also shape them into tiny, very sharp chisel ends. One may also use a small mechanical drill with a grinding wheel, or an electric engraver (vibro-tool)—particularly

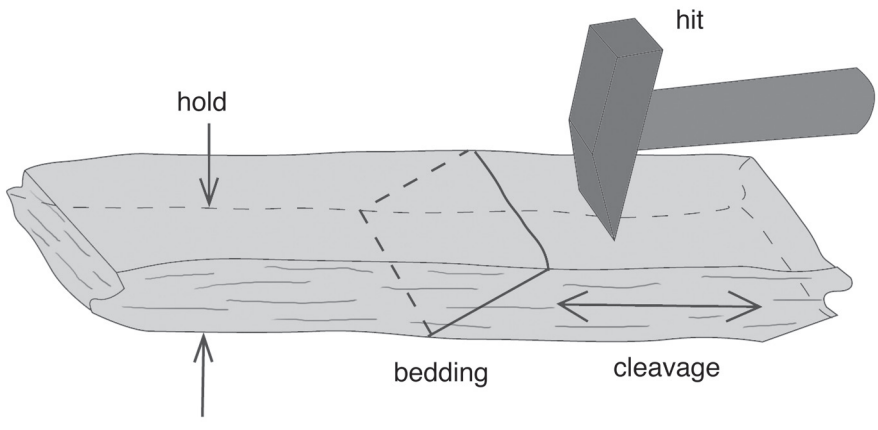

FIG. 1. Splitting tectonized rock slabs to gain fossil specimens (new).

for graptolites encased in very hard rock such as chert (though the approach here is a little different to that described below).

Some people prefer to use a scalpel or a razor blade for preparation. A scalpel is best, as it is easier to handle than a razor blade that has to be mounted for this purpose. These tools work well for softer rocks, such as shales that are not overly tectonized or thermally altered. Very precise excavation work can be done with a scalpel, and even larger specimens are easily and cleanly prepared.

Preparation should invariably be carefully done while using a binocular microscope under good light to follow the work exactly. The specimen should be comfortably and firmly held in place, either simply by hand or with the help of wooden blocks or small sandbags.

Many graptolites are a good deal more delicate than the enclosing rock, though this varies, depending on the nature of the rock and the type of preservation of the graptolite (e.g., pyrite-filled, weathered,

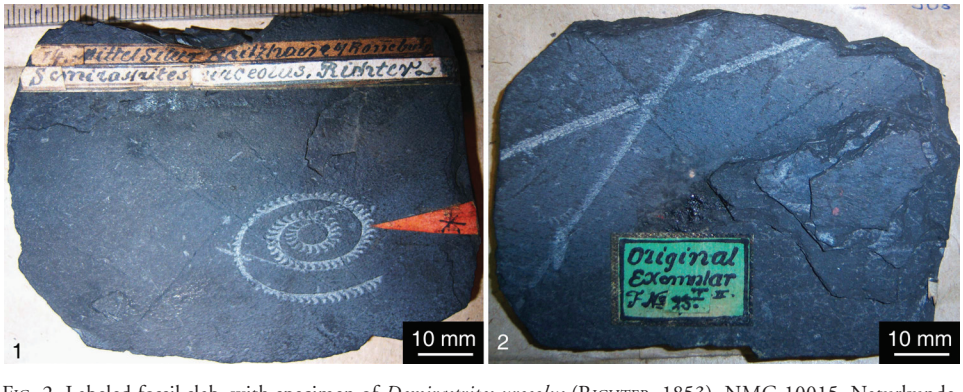

FIG. 2. Labeled fossil slab, with specimen of *Demirastrites urceolus* (RICHTER, 1853), NMG 10015, Naturkundemuseum Gera, Germany; labels indicate the locality (14. Mittelsilur Raitzhain/Ronneburg) and identification (*Demirastrites urceolus* RICHTER) of specimen (marked with *red arrow*) on upper side *(1)* of slab, as well as its scientific value (Original Exemplar) on back side *(2)* of slab, indicating that the specimen was illustrated in a scientific publication (Eisel, 1912, pl. 2, fig. 25).

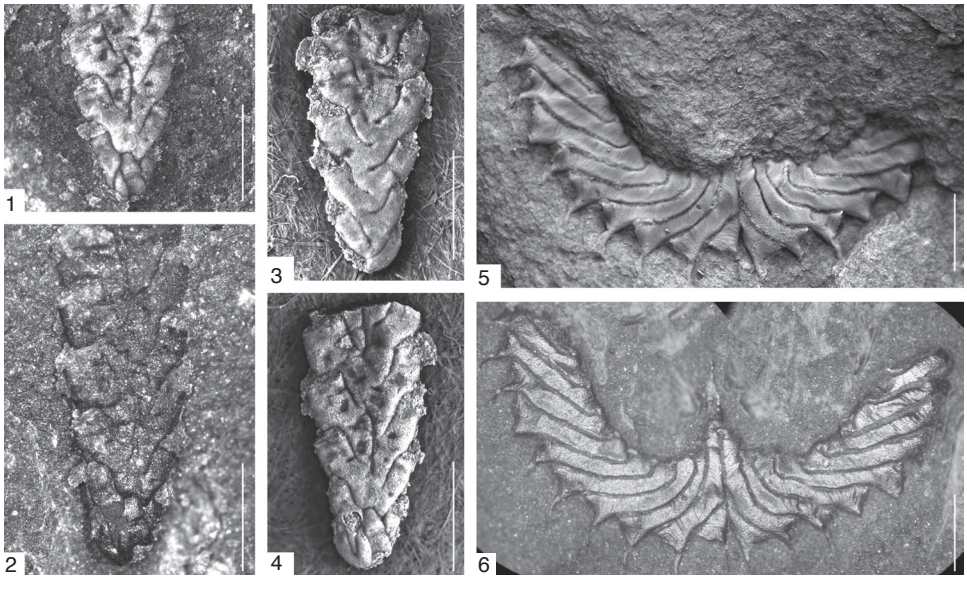

FIG. 3. Preparation of graptolites; all scale bars, 1 mm; specimens from the Lerhamn and Krapperup drill cores of Scania, Sweden. *1–4, Skanegraptus janus* MALETZ, 2011, LO 11196T, holotype (Maletz, 2011); *1*, unprepared specimen, coated for photography; *2*, prepared specimen becoming loosened on slab (see shadow on lower left side); *3–4*, specimen separated from slab and coated for photography to show reverse *(3)* and obverse *(4)* views of the same individual; *5–6*, *Arienigraptus geniculatus* (SKEVINGTON, 1965), LO 10601t, *5,* unprepared, coated specimen (new); *6,* prepared, uncoated specimen (Maletz & Ahlberg, 2011).

flattened film of organic material). The technique, however, remains essentially the same in all cases. Where the rock matrix cover is thin, the needle is simply pressed down—firmly but carefully—*vertically* into the rock above, or just adjacent to, the graptolite; scraping laterally is *not* recommended. This should free a small flake of matrix from the graptolite, exploiting the plane of weakness that almost always exists between graptolite and matrix. One then works progressively along the graptolite, theca by theca (Fig. 4). If the rock matrix above the graptolite is thick, then one can more quickly excavate a small "quarry" above the graptolite, before proceeding more carefully to break through the "quarry floor" to the graptolite beneath. Careful work is *slow*, with many breaks to blow or wash away (using alcohol) the dust and rock chips produced off the graptolite; excavating a single graptolite may take a couple of hours or more.

Accidents do happen and many museum specimens, including type specimens, have all too obviously been damaged (butchered) by hasty and careless excavation: it is all too easy to push the needle through the rock into the graptolite itself. Even in these instances where the fossil is accidentally damaged, the organic material of the graptolite almost invariably breaks away, leaving an impression, or a pyrite internal cast (that is still taxonomically useful) beneath. Graptolites are often coated with pale phyllosilicates, commonly regarded as pressure shadow minerals formed during the tectonic deformation of the surrounding rocks (UNDERWOOD, 1992). One may aim to retain this coating (for example, in the case of flattened, white graptolites on black shale, this essentially represents the graptolite itself) or remove it (as in the case of the so-called chlorite sheath around pyritic Welsh graptolites, though the surface of this coating typically mimics the external outline of the graptolite).

Practice is essential to graptolite preparation—and best done on spare graptolite fragments. Repairs are sometimes possible: a fragment of an accidentally broken graptolite may be put back in place with a drop of glue, carefully applied from the tip of a fine brush or needle (for this, it is best to use water-soluble glue, strongly diluted with water to make it very runny). Often, however, it may be better to keep specimens flaked off a slab in a separate container (glass bottle, etc.) in glycerin or dry on a slide, so they can be viewed from both sides (Fig. 3.3–3.4).

One can almost never entirely free the graptolite from its matrix—and one should not try. The idea is to expose as much of the graptolite as possible, and as needed for identification of relevant characters, while leaving it firmly bound into the rock slab. The needle technique works for most graptolites and is mostly done dry, though occasionally it may be best done under alcohol, which necessitates frequent washing of the specimen with the same fluid.

Mechanical drills are best used on very hard rocks, too tough to yield to pressure from a hand-held mounted needle. These drills require care, as it is more difficult to have the same fine control as in the hand-held excavation. It is important to use ear protection, as most such drills are painfully loud. Typically, the most effective drill bit is the sharpest and finest one, which is used to slowly excavate through enclosing matrix, with special care at the interface with the graptolite (at which point, it may be advisable to revert to using the hand-held mounted needle).

Although friable material has often been fixed by varnishing the surface, varnish is difficult to remove, may darken, and invariably cracks with age, so this method is not recommended. An alternative is Gum Tragacanth. Another material, which can be removed using acetone, is an ethyl methacrylate copolymer, Paraloid/Acraloid B72, diluted to a 20% w/v concentration. Removing specimens that are in the state of

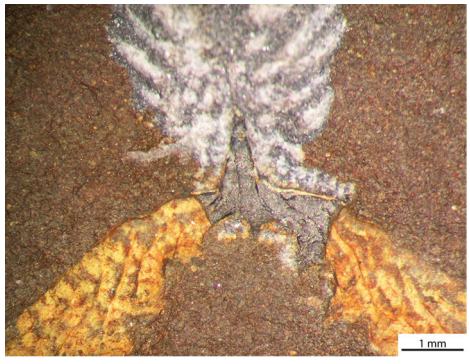

Fig. 4. *Baltograptus jacksoni* Rushton, 2011, holotype, Ht 1260a; proximal end excavated from rock matrix with the help of a preparation needle; proximal end with preserved fusellum (dark grey), distal stipes preserved as imprint outlined with orange-colored pressure shadow minerals.

separating from the shale surface (Fig. 3.2) is recommended, and they can be kept in separate bottles or on slides. In these instances, latex casts can be taken from the imprint left in the shale, if the shale is hard enough. A small glass bottle can be used to keep the separated specimens in glycerin, as it prevents them from being broken on transport. Loose, shifting specimens on slides may easily be damaged, as evidenced by the isolated specimens in the Holm collection, Naturhistoriska Riksmuseet (Natural History Museum) in Stockholm, Sweden. Many weathered slabs with graptolites from Victoria, Australia, have been completely destroyed by covering the specimens with varnish, which later cracked and started to flake off, as the shale is very soft and deeply weathered.

Serial sectioning has long been used to reconstruct the internal structure of isolated three-dimensional material. Pioneered by in the 19th century by Holm (1890, 1895) and Wiman (1895, 1897a, 1897b, 1901), it was used to great effect in the 20th century by Bulman (1944–1947) and Kozłowski (1949). Holm used paraffin wax, and Bulman used collodion and paraffin wax. However, a low-viscosity epoxy resin, such as that used to embed material for TEM (Transmission Electron Microscopy) sectioning, is

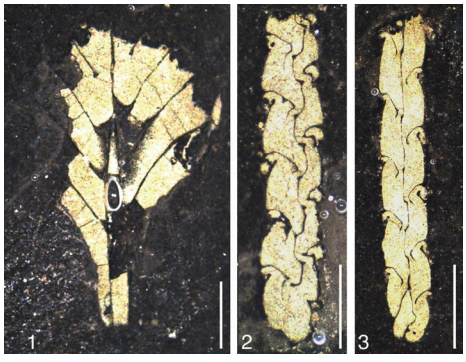

Fig. 5. Specimens from Tommarp, Scania, Sweden, preserved as polished sections on dark shale slabs; sections prepared and illustrated as drawings by Törnquist (1893); scale bar, 1 mm. *1, Petalolithus palmeus* (BARRANDE, 1850), LO 1119t, obverse view (Törnquist, 1893, fig. 35); *2, Metaclimacograptus internexus* (TÖRNQUIST, 1890), LO 1111t, reverse view (Törnquist, 1893, fig. 27); *3, Metaclimacograptus internexus* (TÖRNQUIST, 1890), LO 1110t reverse view (Törnquist, 1893, fig. 26).

a better material. Although sectioning has, to some extent, been superseded by SEM (Scanning Electron Microscopy) examination of isolated specimens, it is still a useful, if time-consuming, technique (see DUMICAN & RICKARDS, 1985). Modern techniques include the use of computer programs to reconstruct the sections (SUTTON & others, 2001). A disadvantage of the method is, however, the destruction of the original specimen, which can be avoided using CT (computer tomography) scans.

TÖRNQUIST (1893, p. 2) described a simpler, but highly effective, method for the preparation of sections for graptolite specimens filled with pyrite in hard shales. He ground the slabs down on a slab of sandstone to the desired level and subsequently polished it. The results are beautiful sections of pyritic graptolites showing the internal features (Fig. 5). LOYDELL and MALETZ (2009) used a similar method to investigate the internal features of *Normalograptus scalaris:* chemically isolated specimens were embedded in epoxy resin, ground down on one side, mounted on a glass slide, and ground down from the other side to get sections of the specimens.

Natural molds of graptolites (Fig. 6.1,3), often the result of pyrite oxidation or weathering, can be studied by making latex replicas to improve the visibility of important features (Fig. 6.2). These latex replicas are sometimes good enough to be examined by the SEM. As latex dries clear, a few drops of Indian Ink can be added to the latex before applying. This will produce a matt black peel that, after whitening, photographs well. The application of liquid latex must be preceded by wetting the slab: this lowers the surface tension of the latex and allows it to seep into every small surface feature to form a perfect cast without producing air bubbles. Latex can also be used to clean weathered surface areas of shales and make graptolites more easily visible. A first latex cast can be used for cleaning of the shale surface, while a second one provides the useful casts of specimens.

In some instances, the latex (dyed black) slightly dyes the specimens, but not the surrounding shales, and enhances the contrast of the graptolite specimens. Conversely, it may dye the shale and not the smooth surface of the graptolite imprints. Thus, applying latex often makes the specimens more easily visible on the shale surfaces.

Loss of paleontological specimens by pyrite oxidation is common in geological collections. It may be a serious problem for fossil collections that include pyritic specimens and considerable effort is necessary to preserve these (NEWMAN, 1998). Pyritic internal molds of graptolites often suffer from mineral decomposition and can easily be lost completely (BIRKER & KAYLOR, 1986). Pyritic specimens in shale should be kept in dry conditions: in the presence of oxygen they break down to ferrous sulfate ($FeSO_4$) and sulfur dioxide (SO_2). If water is present, sulfuric acid (H_2SO_4) is also produced, which can cause damage to labeling and storage containers. The most effective method of preventing rapid decay from pyrite oxidation is to store specimens within

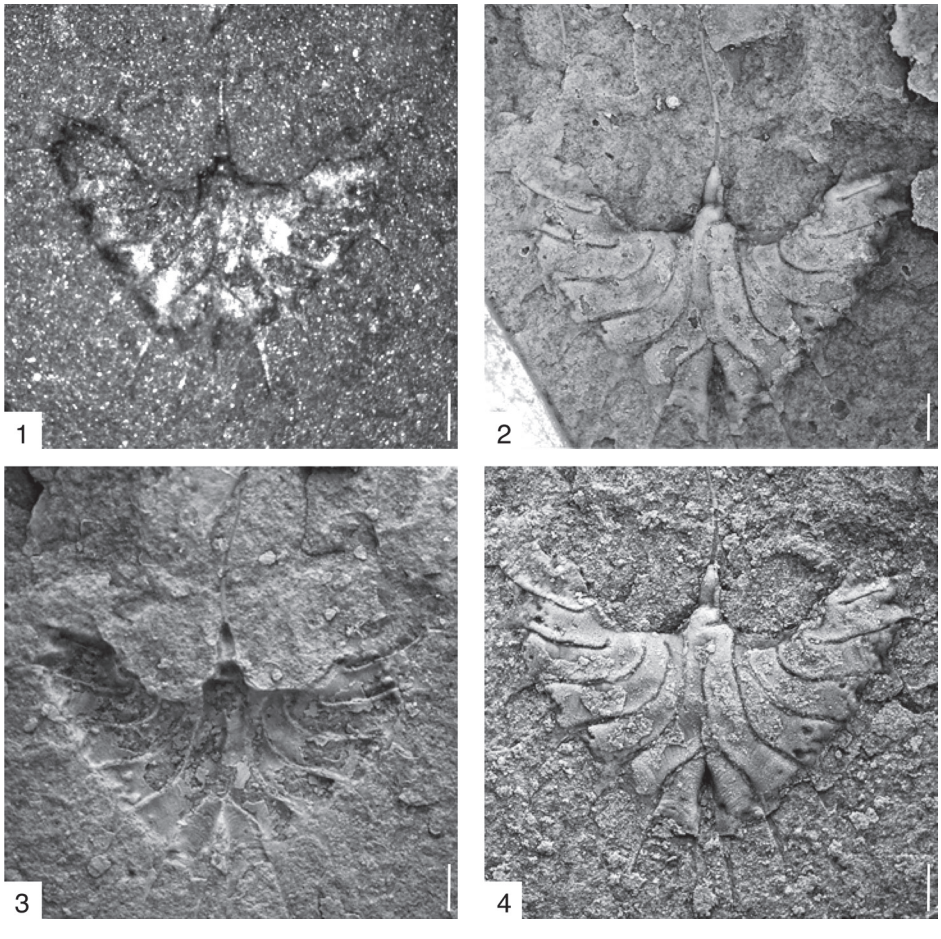

FIG. 6. *Arienigraptus zhejiangensis* YU & FANG, 1981, Krapperup drillcore, 59.30–59.35 m; scale bars, 1 mm. *1*, mold of specimen; *2*, latex cast of mold; *3*, coated mold; *4*, counterpart, relief specimen for comparison. Specimens in *2–4* lightly coated with ammonium chloride to enhance visibility of features (new).

a moisture and oxygen barrier containing an oxygen scavenger. Further oxidation can be reduced or eliminated by storing specimens in an environment with a humidity level below 30%. Ammonium gas and ethanolamine thioglycollate treatments neutralize sulfuric acid and remove ferrous sulfate, and they are reportedly effective in partly or completely removing oxidation reaction products (SHINYA & BERGWALL, 2007). HUTT and RICKARDS (1967) suggested a transfer method to preserve pyritic graptolite specimens in polyester resin.

CHEMICAL ISOLATION OF GRAPTOLITES

Detailed information about the structure and development of graptolites is obtained almost entirely from specimens that have been isolated (dissolved) from their matrix (Fig. 7) and, in some cases, rendered more or less transparent by the use of various oxidizing agents (Fig. 8). The actual processes and reagents employed depend, of course, on the degree of carbonization of the fossil. Not only is it possible

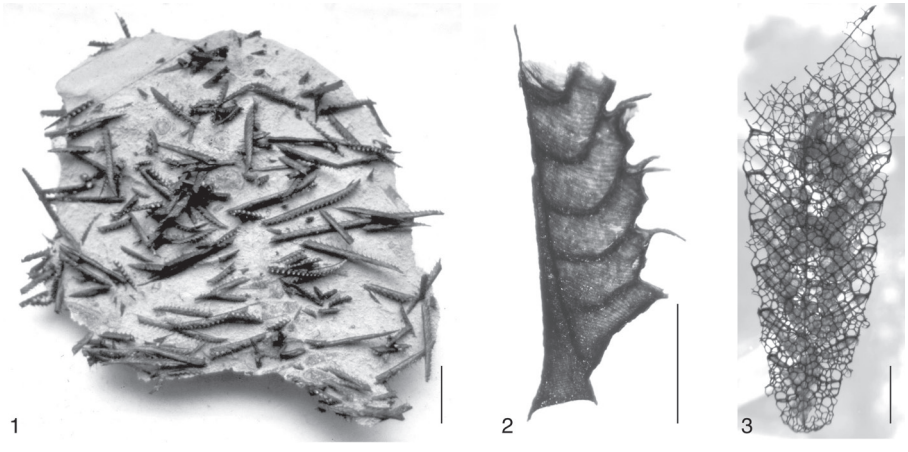

Fig. 7. Chemical isolation of graptolites. *1–2*, *Saetograptus leintwardinensis* (Hopkinson in Lapworth, 1880); *1*, numerous specimens partially isolated from limestone, glacial boulder, northern Germany, FGWG 125, University Greifswald, Geology, scale bar, 10 mm (new); *2*, SMF 68 294, individual specimen from the sample, scale bar, 1 mm (Maletz, 1997, fig. 4.12); *3*, *Retiolites geinitzianus* (Barrande, 1850), isolated specimen, SMF XXIV450, scale bar, 1 mm (Maletz, 2008, fig. 1a).

to isolate three-dimensionally preserved specimens, but also flattened ones, as, for example, from shales using hydrofluoric acid (HF; see, for example, Albani & others, 2001). Chemical isolation produces the best and most complete assemblages, allowing for recovery of small specimens and delicate forms not easily recognizable on rock surfaces. Gümbel (1878) first attempted the chemical isolation of graptolites, but Holm (1890, 1895) deserves much credit for successfully isolating numerous graptolites and describing them in great detail. Pure limestone matrix containing graptolites can be readily dissolved with hydrochloric acid (HCl) or acetic acid (Fig. 7.1). For fragile material, acetic acid is preferable, on account of its more gentle action. Dolomite can only be dissolved using HCl, and the concentration should be adjusted so that effervescence is very gentle and is maintained by the repeated addition of drops of concentrated acid. If the concentration of acid becomes too dilute, there could be access to the container and a danger of fungal growth occurring and entangling with the specimens. This can be counteracted by adding Thymol.

The physical preservation of the graptolite's organic material is an important factor. Some limestone material, otherwise seemingly suitable, should not be subjected to chemical isolation if the graptolite remains are too highly carbonized. Moreover, some graptolites are so brittle that they crumble to a powder when freed from matrix. It is often impossible to know this in advance, and a trial is invariably useful. Before examination by SEM, it is advisable to treat already isolated specimens with hydrofluoric acid, which can remove any fine clay or siliceous material adhering to them. Some graptolite material, however, is surrounded by diagenetic silica and dissolution of this mineral growth will destroy the specimens, as the silica helps to keep the broken graptolite fragments in place (see Maletz, 2009); in these instances, it is advisable to check whether the specimens show broken tubarium walls or are well preserved.

Impure limestone generally requires a double treatment, involving the solution of the calcareous matter first and then, after washing out all trace of HCl, the solution or disintegration of the arenaceous or argillaceous remainder with HF. Non-calcareous

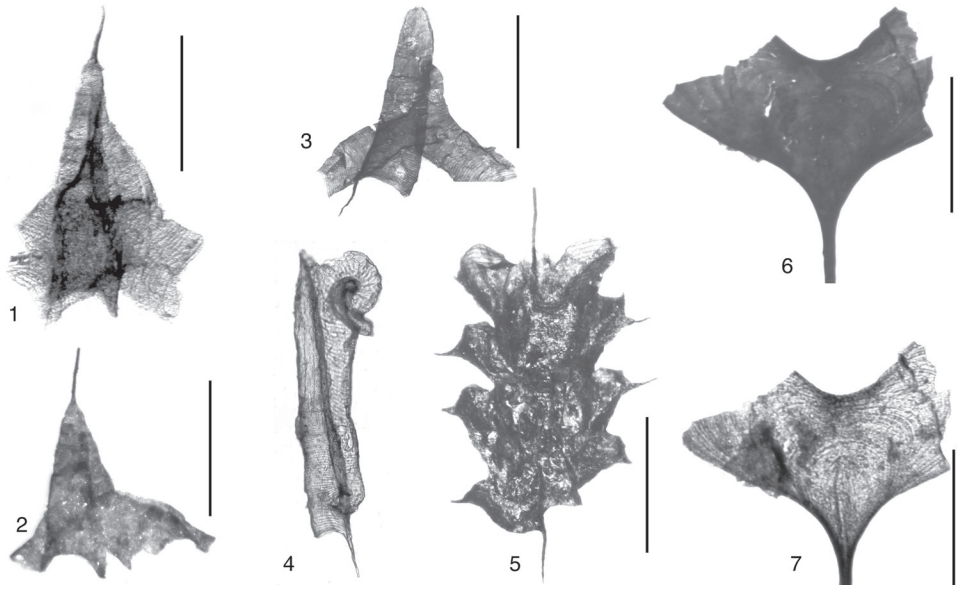

FIG. 8. Photography of isolated specimens; scale bars, 1 mm (new). *1*, *Tetragraptus* sp., chemically bleached flattened specimen, showing some fusellar construction; *2*, *Tetragraptus* sp., unbleached, flattened, not showing fuselli; *3*, *Xiphograptus primus* MALETZ, 2010, bleached, flattened specimen, showing well-preserved fuselli; *4*, *Streptograptus* sp., 3D specimen, IR-photo; *5*, *Oepikograptus bekkeri* (ÖPIK, 1927), isolated 3D specimen, fuselli visible on distal thecae, unbleached; *6–7*, *Archiclimacograptus decoratus* (HARRIS & THOMAS, 1935), nematularium, transmitted light photo *(6)* and IR-photo *(7)* of same specimen.

material, such as shale or chert, can be treated directly with HF. Repeated washing and decanting is necessary to remove all HF before the graptolite remains can be picked out with a pipette or a small brush under a low-power binocular microscope. Much of the fine mud can be removed by elutriation. Some workers wash the insoluble residue through one or a series of sieves, although this risks greater breakage of specimens.

JAROCHOWSKA and others (2013) described a new, acid-free method of extraction of graptolites and other fossils from clay-rich sediments. The surfactant Rewoquat can be used to disintegrate samples and to isolate fossils from sediments with little damage. It is a very gentle method that can produce excellent results and even preserve features like the delicate membranes in retiolitids.

A bleaching agent can be used to render material more or less transparent. Graptolites that have been dissolved out of calcareous rocks may contain bubbles of CO_2, which should be removed in a vacuum dessicator before further treatment. Clearing is most usually done in a watch-glass with potassium chlorate and concentrated nitric acid, though bleaching agents (e.g., *eau de Javelle*) have been used. The treatment time varies with the thickness of the fusellum and the degree of carbonization and can only be judged individually by constant observation through a low-power binocular microscope. In general, the treatment cannot be prolonged much beyond 20–30 minutes without the specimens becoming too brittle to handle; however, some workers use a much lower concentration of bleaching agent and a correspondingly greater period of time. After bleaching, clove oil can be used to neutralize the acids. Quite a high proportion of material successfully dissolved from its matrix proves unsuitable for further treatment of this kind. The process of bleaching

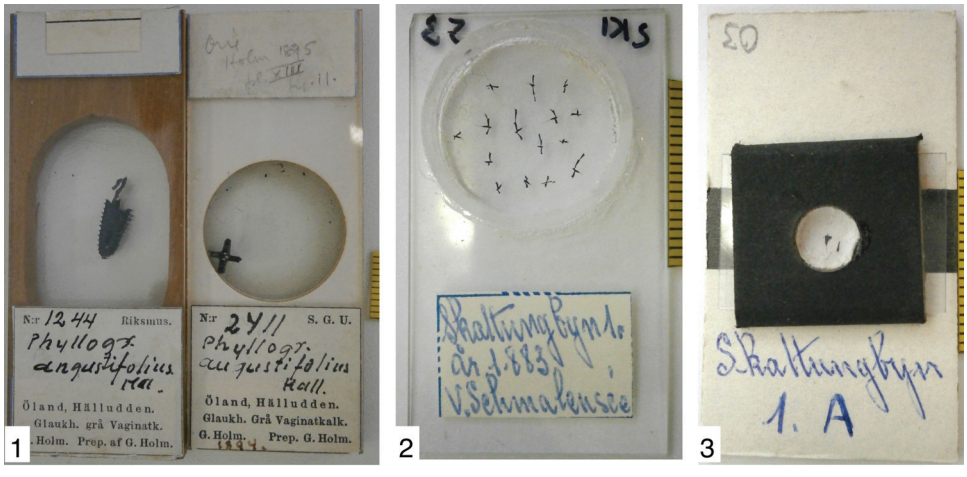

FIG. 9. Isolated and dry-mounted graptolites. *1, Phyllograptus angustifolius* (J. HALL, 1865), RM Holm Nr. 1244 and SGU Holm 2411, both specimens mounted by Gerhard Holm (new, possibly originals for HOLM, 1895, pl. 13–14); *2, Acrograptus* sp. specimens, SGU-SK 1.23, originally mounted by Roland Skoglund in liquid (alcohol or glycerin), which has now evaporated; *3, Pseudophyllograptus* sp., juvenile, SGU-SK 1A.03, mounted dry by Roland Skoglund (Skoglund material is unpublished).

has now been largely superseded by Infrared (IR) photography. If IR photographs do not show details such as growth lines, bleaching will not help.

All chemical treatments should be carried out in controlled conditions, with due regard for safety. These activities should be done in appropriately ventilated spaces, equipped with fume cupboards (hoods); protective clothing and eye goggles should be worn; and all written safety instructions consulted and followed.

Long-term preservation of chemically isolated specimens is problematic, as the specimens are generally fairly fragile and easily break through handling when kept dry. Various methods have been used in the past to ensure their preservation. Robust specimens can be mounted dry, since surface features are more easily seen than when mounted in a relatively high-refractive index medium. Gerhard Holm mounted specimens between glass slides, so they can be observed from both sides. He left enough space between the two slides so the specimens are not damaged (Fig. 9.1). Specimens can be affixed between two glass slides with a minute drop of gum Arabic. These specimens are generally in good shape even after more than 100 years of museum storage. Roland Skoglund used a similar method for flattened graptolite specimens from Dalarna. He mounted individual dry specimens on cardboard, covered with a thin glass slide (Fig. 9.3). More problematic is the preservation of his numerous specimens in small containers glued on glass slides (Fig. 9.2). As the liquid in the containers dried out over time, the specimens started to break apart and often show considerable damage (see also MALETZ & SLOVACEK, 2013, fig. 1).

Transparent specimens may be mounted in Canada balsam. Euparal is an alternative to Canada balsam, which has the advantage of not requiring perfect desiccation in absolute alcohol, thus eliminating processes in which damage to the specimen may occur.

Most specialists agree that graptolites are best kept in glycerin (glycerol) for long-term storage, which allows the specimens to be moved and viewed from all sides. They can also be removed from the glycerin and washed with warm water prior to SEM photography of the dry specimens; thorough removal of the glycerin is necessary, as surface features may be covered by residual

fluid. However, ALLINGTON (2006) provided a warning and suggests the use of silicone oil instead. Graptolites in the Natural History Museum (formerly the British Museum [Natural History]) that were stored in glycerin to prevent pyrite decay apparently allowed pyrite oxidation and deterioration of the material. The containers with graptolite specimens should invariably be tightly closed to prevent absorption of water from the atmosphere by the hygroscopic medium.

Other liquids can be used for long-term storage such as alcohol, formalin and Thymol. However, long-term preservation in alcohol requires special care, as the alcohol needs to be refilled from time to time due to evaporation, even in the most tightly sealed containers.

Special care is also necessary to preserve the delicate and fragile graptolite specimens on SEM stubs. These have to be kept in a dry place and need to be protected from dust: plastic boxes in which the SEM stubs are mounted securely is one option.

ILLUSTRATIONS

In the 19th century, graptolites, like other groups of fossils, were originally illustrated with drawings or paintings. Even with the advent of light photography, there was no major change to illustration methods—the nature of preservation of most material did not lend itself to photography. As a result, freehand drawing or painting has continued to be a standard method of illustration to the present day.

The illustrations of the Scandinavian authors Holm and Wiman and the British authors Elles, Wood, and Bulman mark the high point in freehand graptolite illustration. In Holm's publications, the illustrations were by Georg Gideon Liljevall (1848–1928) (Fig. 10.1). However, such standards are impossible to achieve for most people, and most papers are illustrated with outline drawings, often with use of the camera lucida.

Illustrations should be at as large a scale as possible to show all necessary information, but precise guidelines cannot be given. Overall illustrations of most graptolites should be at a smaller scale, while details should be shown at greater magnifications. Early scientific works often have small illustrations at original size that are inadequate in detail for modern use, considering the amount of detail available in many specimens and necessary for modern identifications (e.g., LAPWORTH, 1876; ELLES & WOOD, 1901–1918). However, even some earlier authors (e.g., WIMAN, 1893) provide incredible insight from drawings of highly magnified specimens (Fig. 10.4–10.5). The drawings by Ethel Wood (in ELLES & WOOD, 1901–1918), finely reproduced on the plates (at original scale) remain useful today to understand colony shapes, though they often lack the detail necessary for a proper taxonomic identification.

DRAWINGS

Most non-isolated and matrix-bound graptolites can be drawn using a binocular microscope with camera lucida attachment, essentially a drawing mirror. The image of the specimen under the microscope is superimposed by a split screen onto a sheet of paper on the side under a mirror, and the fossil outline can be traced directly. The results obtained can give a clearer and cleaner image of the graptolite than can be obtained even by careful close-up photography. This is because the photograph (if not heavily retouched) will show every visible feature, petrological as well as paleontological, regardless of its taxonomic significance. In drawing a graptolite, the illustrator should focus on the morphology of the graptolite, although including such features as fractures, compactional or tectonic crumples that are deemed significant. The drawing is thus an interpretation, albeit one that is tightly constrained by the physical evidence and dimensionally accurate.

It is best to draw at as high a magnification as is practically possible—considerably higher than is intended for the final published figure. To start, one needs to balance the lighting on both the graptolite

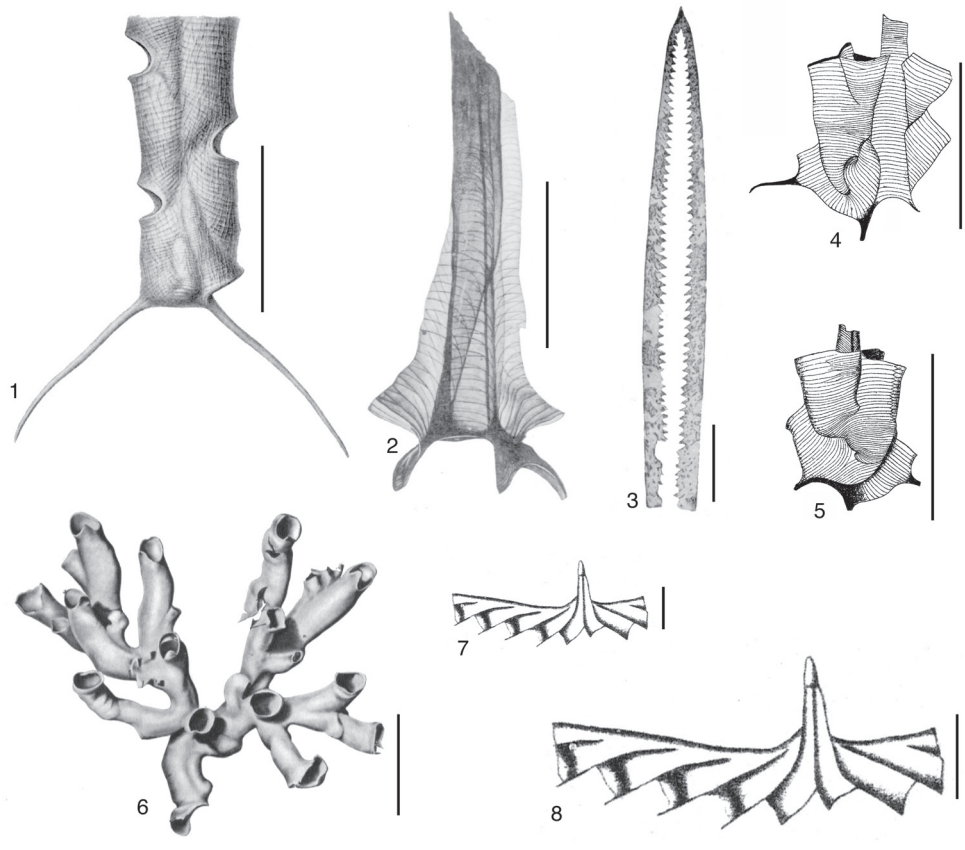

Fig. 10. Illustrations of hand-drawn graptolites. *1, Diplacanthograptus spiniferus* (Ruedemann, 1912), drawn by G. Liljevall, note even the presence of cortical bandages on the surface of the thecae, scale bar, 1 mm (Bulman, 1932, pl. 3, fig. 7); *2, Corynoides* cf. *calicularis* Nicholson, 1867, drawn by O. M. B. Bulman, scale bar, 1 mm (Bulman, 1945, pl. ii, fig. 12); *3, Didymograptus murchisoni* Beck, drawn by E. M. R. Wood, scale bar, 10 mm (Elles & Wood, 1901, pl. iii, fig. 1f); *4–5, Rectograptus gracilis* (Roemer, 1861), line drawings of specimen showing growth lines and proximal development in all available detail in obverse *(4)* and reverse *(5)* views (identified as *Diplograptus* sp. in Wiman, 1893, fig. 7), corrected from Wiman's (1893, p. 104) originals, as he stated that the plate was accidentally prepared as a mirror image of the original drawings, scale bar, 1 mm (adapted from Wiman, 1893, fig. 7); *6, Rhiphidodendrum samsonowicz*i Kozłowski, 1949, scale bar, 1 mm (Kozłowski, 1949, pl. 10, fig. 1); *7–8, Expansograptus latus constrictus* (J. Hall, 1865), originally magnified ×4.5, showing even the differentiation of the prosicula in a relief specimen in black shale, scale bar, 1 mm (Törnquist, 1901, pl. 2, fig. 15).

and on the sheet of paper (the latter provided by a separate lamp), so that both graptolite and paper (and the moving pencil-point) can be clearly seen. For some makes of camera lucida, there is some distortion near the edge of the field of view; therefore, it is best to work in the middle three-fourths or so of the field of view. It is usually easiest to start at the proximal end and work towards the distal end, theca by theca. Once the segment of graptolite in the working area of view has been drawn, then it should be carefully moved until the next segment comes into view; the drawing also is moved (while looking through the microscope) until the completed line-work and the specimen are exactly aligned. Some workers, such as Ethel Wood, scored a straight line adjacent to the graptolite to gage its position.

A scale must be added to each drawing. This must be done at the time of drawing by placing a scale (e.g., a ruler with millimeter

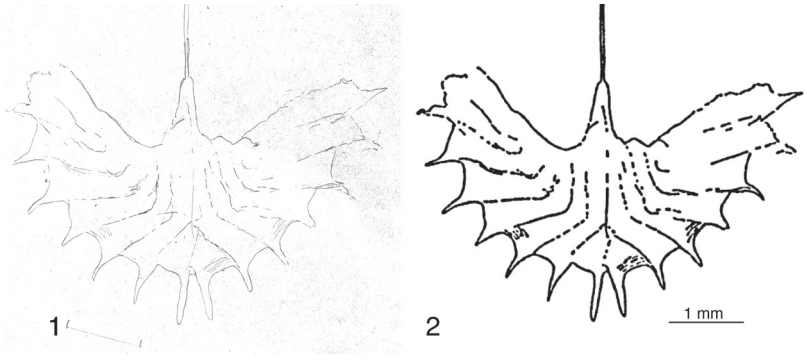

FIG. 11. Example of camera lucida pencil drawing *(1)* and ink drawing *(2)* of *Arienigraptus angulatus* (GEH & YIN in MU & others, 1962), reverse view, from *Levisograptus dentatus* Biozone, Côte Fréchette, Québec, Canada, GSC 102702a (new).

divisions) against the graptolite and then drawing a few of the millimeter divisions using the camera lucida.

For final publication, the drawing must be reproduced as a line or half-tone drawing in black ink (Fig. 11). This is best done on a photocopy or scanned reproduction of the pencil drawing (with a dark enough setting to show all line-work and shading). There are also computer programs that can assist a rendering of the original pencil drawing by digital means. Some workers use a range of different nib sizes to achieve different line weights for their illustrations.

The shading can be detected as different densities of dots and, for the darkest shades, simply completely inked-in areas. Small mistakes can be corrected on the film: erroneous lines can be delicately scratched out with a razor blade and re-inked. Bulman's early line drawings have also been produced from photos (STRACHAN & others, 1991, p. 66), a method still used by some people. The lines were inked on a highly magnified photo and the photo was subsequently bleached.

VISIBLE LIGHT, ULTRA-VIOLET, AND INFRA-RED PHOTOGRAPHY

Flattened specimens can be very difficult to photograph, unless there is a good contrast between specimen and matrix. Flooding by alcohol, commonly isopropyl alcohol or industrial methylated spirits, can increase contrast, and also reduce unwanted reflections, although it evaporates quickly. An alternative is a mixture of 50% alcohol and 50% glycerin (Loven's reagent). This can be washed off using alcohol. Wetting the surface can improve the contrast between matrix and graptolite, but it may cause the clay minerals to swell and the organic material of the graptolite to flake off.

For specimens in relief, including latex peels and even isolated pyritic specimens, the material can be whitened using either MgO ribbon burning or ammonium chloride, applied as a sublimate. This method improves the visibility of structural details in relief specimens (Fig. 12). Great care must be used for the process.

The documentation of internal details of the graptolite colonies has always been difficult, and a number of methods have been used, including various measures of preparation and photographic experimentations (Fig. 13). Good results are achieved by using infrared (IR) photography of chemically isolated graptolites. Infrared photography was explored by EISENACK (1935) to investigate graptolites, though it presented too many difficulties to be a successful method at that time; it was reintroduced by MELCHIN and ANDERSON (1988) using a modern IR-Video camera (Fig. 13.1). Infrared video microscopy (IVM) uses a relatively inexpensive video camera that is sensitive

through the visible and near IR range, up to wavelengths of 1300 nm. This camera can be connected to a normal biological or petrographic microscope. The best results are obtained when visible light is filtered with a 1000 nm, long-pass filter, although the crossed polarizers on a petrographic microscope can serve a similar function. The images obtained can be viewed on a video monitor, printed by direct connection to a video printer or electronically stored using a computer video image capture system. The stored images can then be analyzed using image analysis software.

Provided the specimens are not of high thermal alteration or very thickly covered in cortical tissues, and are not covered or infilled with IR-opaque mineral material, the resulting images reveal surface morphology, fusellar banding (and occasionally cortical bandages), as well as all internal walls and structures (Fig. 13.1). Optimum image quality requires that the specimens be immersed in a very high refractive index liquid, preferably 1.76, the refractive index of the graptolite fusellum. The best visualization of internal structures can be achieved using stereo-pairs of prints of the video images. These can be obtained by using a universal stage and tilting the specimens approximately 8° between prints. Stereo-pairs are useful for relief material, but flattened specimens can also be examined with IR light and provide useful data (Maletz, 2010).

With the advent of digital photography and the use of computers, it has become much simpler to improve the quality of images. Contrast can even be improved selectively, for example in the darker areas of the image. Unwanted backgrounds can be removed electronically, instead of physically. This is particularly effective for SEM photographs. Tectonic distortion can be removed easily using an appropriate computer program.

SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy (SEM) has become, since the early 1970s, a widely used method for illustrating complete tubaria and revealing ultrastructure in three dimensions. It is applicable to isolated material and to fossils on rock surfaces where there is relief present or where the graptolite wall material is still preserved. SEM can even be used on artificial molds or casts. The SEM shows only surface features (Fig. 14), not internal details, unless the specimens are broken open. SEM photography is especially important to show the delicate and complex meshwork of the retiolitids (Fig. 14.3).

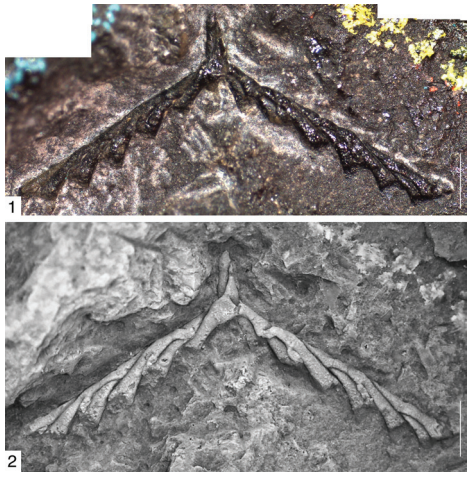

Fig. 12. *Kiaerograptus* (?) *supremus* Lindholm, 1991, LO 5970T, holotype; scale bars, 1 mm. *1,* original specimen showing traces of preparation on rock (light color); *2,* same specimen coated with ammonium chloride for photography, showing the tubarium construction more clearly.

In preparation for SEM photography, isolated material, which may be stored in glycerin, water and formalin, or dilute acid, should first be transferred to a small container and then washed in several changes of distilled water. This is best done using a pipette to draw off the liquid and then add distilled water. After washing, the specimens can either be directly placed on the stub or coated in liquids, either pure alcohol (IMS: industrial methylated spirits is satisfactory) or acetone before placing on the stub. Because these liquids have a lower surface tension than water, they may cause less damage to the specimens when being

transferred to the stub. If the specimens are small, they can be lifted and transferred in a pipette: it may be possible to manipulate a specimen on the stub surface, before the liquid evaporates. A fine artist's brush may also be used to lift a specimen. The specimen will adhere to the brush while the liquid evaporates (holding it above an electric bulb will speed up evaporation), and it can then be gently lowered onto the stub. For very large specimens, it is possible to submerge the stub in the container of liquid and specimen, manipulate the specimen onto the stub with a brush, and then pipette off the liquid until the stub emerges from it.

Specimens may also be completely dried before mounting on SEM stubs. Specimens originally preserved in glycerin can be cleaned using warm water and then dried. A small brush with very few hairs can be used to transfer the specimens to an SEM stub. Rubbing the brush slightly on one's skin provides enough moisture for the specimen to stick to the brush.

A variety of adhesive surfaces can be used to attach the specimens to the stub. Double-sided tape is clean and efficient, and can be cut from a roll. However, it can release vapor under vacuum, which may affect the working of the instrument. Alternatively, circular patches of tape are specially made to apply to the stubs. Other liquid glues can be used, such as fingernail polish, spread on the stub and allowed to become tacky before

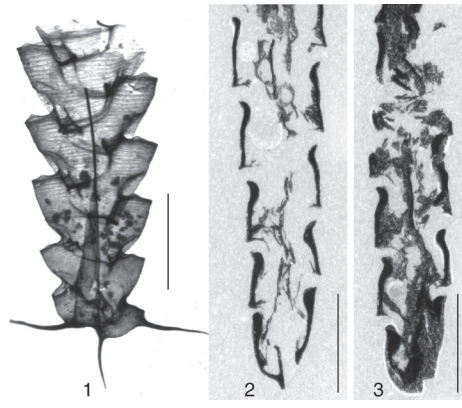

Fig. 13. Documenting internal details of graptolite colonies; scale bars, 1 mm. *1, Rectograptus intermedius* (Elles & Wood, 1907), USNM 542831, infrared photo of relief specimen with internal structures (Štorch & others, 2011, fig. 17C); *2–3, Normalograptus scalaris* (Hisinger, 1837), showing internal thickenings close to the thecal apertures in sectioned specimens (Loydell & Maletz, 2009, fig. 2).

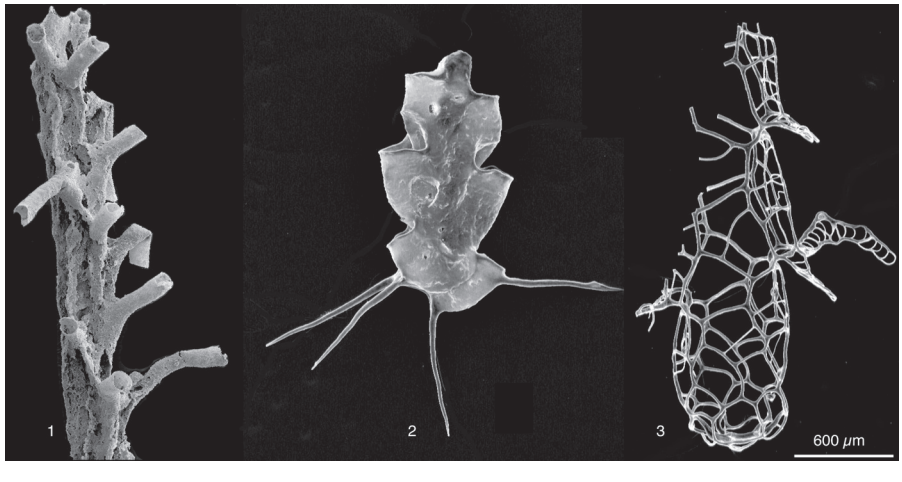

Fig. 14. SEM photos of specimens; scale bar, 600 µm. *1, Acanthograptus* sp. in obverse view. Öland, Sweden, JM 39.05 (new); *2, Rectograptus* sp., Viola Limestone, JM26/21 (new); *3, Neogothograptus ornatus* Maletz, 2008, glacial boulder, northern Germany, coll. Jaeger (Maletz, 2008, fig. 10M).

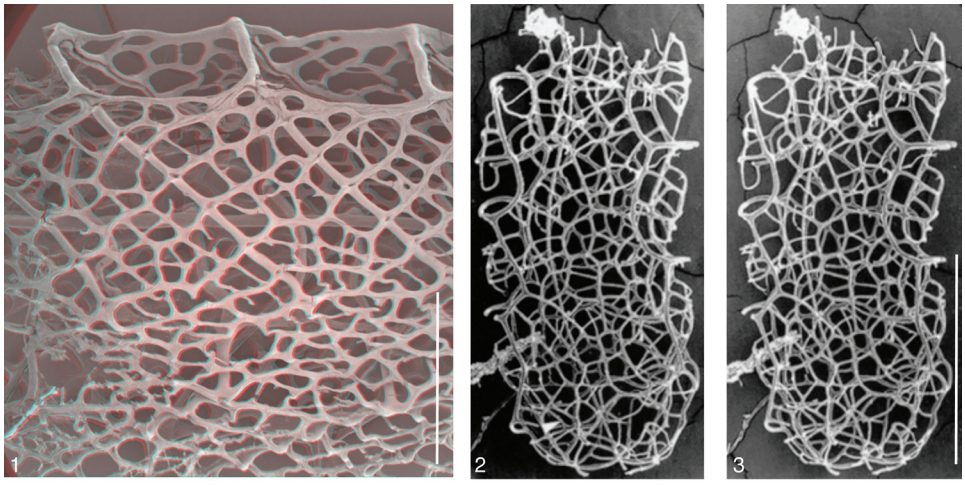

FIG. 15. Stereo photography, scale bars, 1 mm. *1*, Anaglyph image of retiolitid fragment (new); *2–3*, Stereopair photographs of retiolitid *Sagenograptoides arctos* (LENZ & KOZŁOWSKA-DAWIDZIUK, 2001) (Lenz & Kozłowska-Dawidziuk, 2001, pl. 8).

mounting the specimens. In this case, check that the glue used will not react with any liquid remaining on, or in, the specimens.

The standard stub for most instruments has a diameter of about 12 mm, and this will suffice for much material. Indeed, a great number of siculae can be attached to a single stub. If the specimen is larger, bigger stubs can be bought or made using a lathe. An alternative method is to glue a piece of thin sheet metal, such as brass or nickel silver, to a standard stub, using an epoxy glue. Conductive paint should be applied to the underside of the metal to ensure good electrical contact with the stub.

To view a specimen from as wide a range of directions as possible, it may be possible to mount it on a wire, which is glued to the stub. It is not possible to use double-sided tape, but the wire can be smeared with a suitable glue, and the specimen stuck to it.

Material on rock can easily be glued to a stub using epoxy resin, after trimming to size. BATES (1996) used a pair of carpenter's nail pincers to break up pieces of the Viola Limestone from Oklahoma, USA, fracturing it along bedding planes to give counterpart specimens, and also trimming the pieces to size.

Material that was fossilized in anaerobic conditions is often infilled with and/or coated in pyrite. In these conditions, the actual wall material of the tubarium may still be present, showing up as a thin brown-to-black coating on the surface of the pyrite. Where the tubarium wall is not present, the pyrite surface may bear the extremely fine mold of the internal surface, including that of the cortical fibrils.

For most work, specimens will be coated, usually with a gold-palladium target in the coater. Occasionally (e.g., where it desired to remove the specimen from a stub, and return it for storage in glycerin), it may be better to view the specimen uncoated, with a low accelerating voltage. Some field emission microscopes can also be used with uncoated material.

The SEM can be used to take stereopair photographs, particularly useful in illustrating retiolite graptoloids (e.g., BATES, 1996; LENZ & KOZŁOWSKA, 2006). The specimen can be rotated by about 5° between photographs. The resulting stereopair is normally illustrated using two small prints, with a separation of 60 mm between the images (Fig. 15.2–15.3). It can be viewed through a pocket stereoscope. An alternative method of reproduction uses anaglyph

images (Fig. 15.1), typically red and green for the left and right images, the printed image being viewed with green and red spectacles.

Although the SEM normally shows surface detail, the electron beam does penetrate beneath the surface: the amount of penetration depends on the nature of the specimen, the angle of tilt in the microscope, and the accelerating voltage. In particular, if the fusellum is particularly thin, it may be possible to see such features as fuselli, bandages, and even stolons (e.g., BATES & KIRK, 1997, fig. 99a,d).

To obtain SEM images at a higher power than normal, specimens can be examined using a transmission electron microscope in scanning mode. Material, however, must be very small (about 4 mm wide by 3 mm thick by 13 mm long), and it can be tilted to about 60°.

If the back-scattered electron signal is used, an image with mean atomic number contrast is produced. Michael Steiner (described in MALETZ, STEINER, & FATKA, 2005) first used the method for graptolites. MALETZ, STEINER, and FATKA (2005, p. 78) stated that the method worked best with wall thicknesses of less than 5 µm. Thicker wall material did not produce sufficient information and fuselli were not visible. ZHANG and ERDTMANN (2004) have also applied the Back-Scattered Electron Microscopy (BSEM) technique to study graptolites. BSEM images illustrate both the chemical compositional differences on the surface layer and the surface morphology of the sample. On the BSEM pictures of *Psigraptus jacksoni* RICKARDS & STRAIT, 1984 (ZHANG & ERDTMANN, 2004, fig. 11), the carbonized fuselli are marked as dark lines, whereas the rock matrix is brighter because of the dominance of silicon and heavy elements. The surface morphology of those areas, where the fusellum has broken off and only films have remained, has also been illustrated in the BSEM images. The method also shows the composition of the surrounding sediment and of the pyrite growth (Fig. 16).

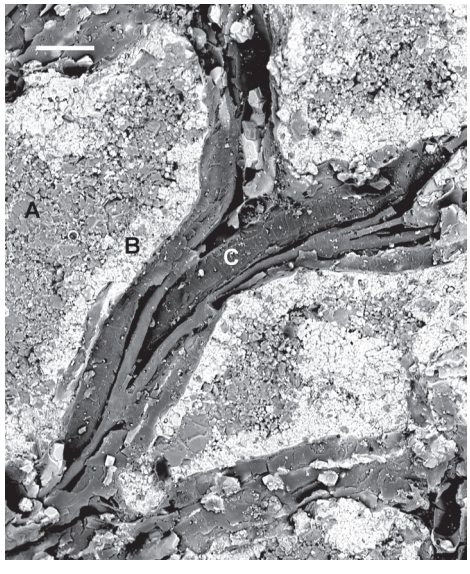

FIG. 16. SEM back-scatter image of *Desmograptus micronematodes* (SPENCER, 1884), PE60371; *A,* limestone matrix, *B,* pyrite coating of the tubarium, *C,* graptolite; scale bar, 100 µm (Saunders & others, 2009, text-fig. 3).

TRANSMISSION ELECTRON MICROSCOPY

The use of the transmission electron microscope (TEM) started with the work of WETZEL (1958) and KRAATZ (1964, 1968), though the seminal work in this field comprises the two monographs by URBANEK and TOWE (1974, 1975). Standard TEM techniques have been described in a number of papers and books in the biological field (e.g., HUNTER, 1993) and can be readily applied to graptolites (Fig. 17). However, graptolite wall material is harder than most biological tissue, so that a diamond knife is essential for good results. In general, a high degree of skill is needed in cutting the sections.

Isolated material is needed for production of sections, using the techniques described above. Unbleached specimens are then embedded in a resin such as Durcupan.

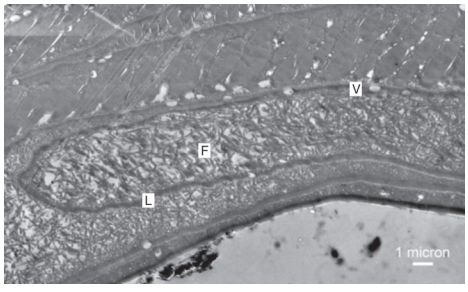

Fig. 17. ?*Dendrograptus* sp., TEM longitudinal section of the closure of a fusellus; parallel fibrils *(F)* of the cortical fabric of the lamella bounding the fusellus are seen as elliptical cross sections that are thicker than the fusellar fibrils, but thinner than those of the cortex itself; *L,* basal granular fabric; *V,* vesicles; scale bar, 1 µm (adapted from Bates, 1997, pl. 5).

Blocks are then sectioned using an ultramicrotome provided with a diamond knife. Sections of about 900 Å are suitable. They are then placed on copper grids, which have been coated with a film of Parlodion and carbon.

The TEM work provides ultrastructural details of the tubarium material, showing the development of fibrils in the fusellum and cortex. It offers information on very fine details and helps to compare fossil tubarium material with the tubaria of extant pterobranch specimens.

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