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A REVIEW OF THE LABORATORY PREPARATION OF PALYNOMORPHS WITH A DESCRIPTION OF AN EFFECTIVE NON-ACID TECHNIQUE

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ABSTRACT – The preparation of palynomorphs for microscopy has traditionally used hydrochloric acid (HCl), hydrofluoric acid (HF) and nitric acid (HNO_3). The use of these acids is both expensive and hazardous. An effective technique of preparation using sodium hexametaphosphate [$(NaPO_3)_6$] has been developed. The cleaned, crushed and softened sample is treated with $(NaPO_3)_6$. The deflocculated clay is then sieved away and the residue centrifuged. This method has been successfully tested on seven Jurassic to Quaternary sample sets from the United Kingdom (UK) and Antarctica. In five of these sets, the rock/sediment was prepared using the mineral acid technique and the $(NaPO_3)_6$ procedure. Four of these five sample suites were prepared by both methods quantitatively, so that the concentrations of palynomorphs can be compared. The $(NaPO_3)_6$ method largely proved to be equally as effective as the mineral acid procedure. The Lower Toarcian Whitby Mudstone Formation of Leicestershire and the Middle and Upper Albian Gault Formation of Kent both produced similar palynomorph/kerogen associations. Some differences between the two procedures were, however, noted. The $(NaPO_3)_6$ method produced significantly better results than acid preparations for the uppermost Cretaceous (Maastrichtian) of Antarctica and the Pleistocene Till of northern England. By contrast, the majority of the samples from the *in situ* late Campanian-early Maastrichtian White Chalk Subgroup of north Norfolk prepared using HCl, were significantly richer in palynomorphs than those treated with $(NaPO_3)_6$.

Key words: palynology, preparation techniques, Mesozoic, Cenozoic.

RESUMO – A preparação de palinomorfos para microscopia tem usado tradicionalmente os ácidos clorídrico (HCl), fluorídrico (HF) e nítrico (HNO_3). O uso destes ácidos é, ao mesmo tempo, dispendioso e arriscado. Como alternativa, foi desenvolvida uma técnica eficiente de preparação que utiliza hexametafosfato de sódio [$(NaPO_3)_6$]. A amostra limpa, fragmentada e finamente britada é tratada com $(NaPO_3)_6$. A argila desfloculada é então tamisada e eliminada e os resíduos centrifugados. Este método foi testado com sucesso em sete amostras do Jurássico ao Quaternário do Reino Unido (UK) e Antártida. Em cinco destas amostras, a rocha/sedimento foi preparada usando a técnica de ácido e a de hexametafosfato de sódio $(NaPO_3)_6$. Quatro destas cinco amostras foram submetidas a tratamentos quantitativos, tanto que se pode comparar as concentrações de palinomorfos. O método $(NaPO_3)_6$ provou ser tão efetivo quanto o processo de ácido. A Formação Whitby Mudstone, Toarciano inferior de Leicestershire, e a Formação Gault, Albiano médio e superior de Kent, ambas produziram associações similares de palinomorfo/querogênio. Contudo, foram notadas algumas diferenças entre os dois procedimentos. Para o Cretáceo superior (Maastrichtiano) da Antártida e as argilas glaciais do Pleistoceno do nordeste da Inglaterra, o método $(NaPO_3)_6$ produziu resultados significativamente melhores do que as preparações ácidas. Em contraste, a maioria das amostras do Campaniano-Maastrichtiano inferior, provenientes do Subgrupo White Chalk do norte de Norfolk, preparadas com HCl apresentaram-se mais ricas em palinomorfos do que aquelas tratadas com $(NaPO_3)_6$.

Palavras-chave: palinologia, técnica de preparação, Mesozóico, Cenozóico.

INTRODUCTION

Palynology is the study of microscopic fossils made of resistant organic material. It was originally known as 'pollen analysis' and encompassed the study of Quaternary pollen grains and plant spores (von Post, 1916). Hyde & Williams (1944) coined the term palynology to refer to the analysis of all microfossils that are resistant to aggressive chemicals such as hydrochloric acid (HCl) and hydrofluoric acid (HF). Organic-walled microfossils are termed palynomorphs (Tschudy, 1961), and may have plant or animal affinity and may be derived from either the marine or terrestrial realms. They include acritarchs, dinoflagellate cysts, chitinozoa, fungal spores, green/blue algae, plant spores, pollen grains and scolecodonts (Jansonius & McGregor, 1996a). Palynomorphs, however, do not include organic non-

microfossil elements such as wood fragments, plant cuticle and amorphous organic material. The generic term for palynomorphs and these other kerogen fragments is phytoclasts (Bostick, 1971).

Palynology is, despite its relative youth as a subdiscipline of geology and palaeontology, a mature subject with an extensive literature. It is used in many integrated geological studies from the Proterozoic to the Holocene for providing detailed biostratigraphical and palaeoecological information. By virtue of their small size, high levels of preservability and hence virtual ubiquity in sedimentary rocks, palynomorphs have been extensively used in the oil and gas exploration and production industry, principally as relative age indices. This economic application has significantly stimulated the study of Phanerozoic palynomorphs and a contemporary overview of this subject has been given by Jansonius & McGregor (1996b). Ehrenberg (1838) first recorded fossil marine organic microfossils from translucent flakes of flint and chert. The microscopical study of siliceous flakes was continued by palynologists including Wetzel (1933), Deflandre (1936; 1937) and Foucher (1975). Palynomorphs are currently normally extracted from sediments and sedimentary rocks by digestion of the sediment fabric using strong mineral acids. This procedure is both hazardous and expensive, both in terms of chemicals and operator time. The purpose of this contribution is to review palynological processing techniques, including the provision of an extensive bibliography, and to describe, using examples, a new preparation method using a sediment disaggregant that avoids the need to use hazardous chemicals.

THE CHEMICAL NATURE OF PALYNOMORPHS

Palynomorphs are composed of sporopollenin, a diverse group of highly complex macromolecular biopolymers formed by the oxidative polymerisation of carotenoids and carotenoid esters (Shaw & Yeadon, 1964; Brooks *et al*., 1971; Shaw, 1971; Brooks & Shaw, 1968a,b, 1972, 1978). A generalised chemical formula is $C_{90}H_{142}O_{36}$, but the nature of the molecular structure of sporopollenins is variable (Fawcett *et al*., 1970). There has been, however, significant progress made recently in regard to the chemical and physical structure of the pollen wall (Rowley, 1976, 1990). Sporopollenin is known to include several unbranched aliphatic chains (Traverse, 1988; Killops & Killops, 1993); it is closely related to lignin and cutin, is present in certain fungi and algae and modern material, and has a specific gravity of *c*. 1.4 (Flenley, 1971). Juvigné (1973a,b) discovered that the density of sporopollenin increases with time, and quoted values of 1.9 and 2.1 for material from the latest Quaternary (Devensian) and Palaeogene respectively. These specific gravity changes are at least partially related to the effects on sporopollenin of the geothermal gradient in fossil material. Sporopollenin is probably the most resistant organic material of direct biological origin in nature. Manskaya *et al*. (1973) attributed this robustness to the presence of condensed aromatic structures formed partially from lignin. It is resistant to aggressive mineral acids (e.g. HF) and concentrated alkalis, and can survive heating to *c*. 300° C.

The material which forms most geologically-preservable dinoflagellate cysts has been termed dinosporin because it has different fluorescence properties to terrestrially-derived sporopollenin and responds differently to natural and artificial stains such as Bismarck Brown, Fuchsin, Methyl Green or Safranine O (Fensome *et al*., 1993). Furthermore, Kokinos *et al*. (1998) found that dinosporin contains a distinctive biomacromolecular material with a largely aromatic structure. These differences are believed to be responsible for the different susceptibilities of dinosporin and sporopollenin to oxidation (see below). Nevertheless, the chemical differences between sporopollenin and dinosporin are believed to be relatively minor, the two substances being part of a closely related complex of organic macromolecules. Dinosporin is thus best regarded as a variety of sporopollenin.

A REVIEW OF THE TRADITIONAL METHODS OF LABORATORY PREPARATION OF PALYNOMORPHS

The purpose of palynological preparation is to isolate palynomorphs from the rock/sediment matrix, and then to concentrate and present them for study in pristine condition, avoiding any modifications of shape, size and preservation and contamination of the assemblage. This is achieved by using a wide variety of physical and chemical procedures and the precise procedural path is highly variable and dependent upon factors such as lithology, mineralogy, level of induration, organic-richness etc. The tenacious, inert nature of sporopollenin makes palynomorphs geologically preservable in, and extractable from, sediments and sedimentary rocks. To prepare palynomorphs for microscopical study from a sample, the material is traditionally initially treated separately with concentrated HCl and HF. Important publications describing the traditional palynological preparatory procedure include Norem (1953, 1956), Sittler (1955), Staplin *et al*. (1960), Wilson & Goodman (1963), Hughes *et al*. (1964), Lennie (1968), Higgins & Spinner (1969), Wilson (1971a), Barss & Williams (1973), Sarjeant (1974, Appendix A), Faegri & Iversen (1975), Doher (1980), Batten & Morrison (1983), Herngreen (1983), Evitt (1984), Phipps & Playford (1984), Johnson & Fredlund (1985), Farley (1988), Traverse (1988), Litwin & Traverse (1989), Aldridge (1990), Poulsen *et al*. (1990), Moore *et al*. (1991) and Batten (1999). Two of the most recent and comprehensive accounts of this topic were given by Wood *et al*. (1996) and Green (2001, chapters 25 and 26). Wood *et al*. (1996) is a review of modern palynological preparatory techniques and part of a comprehensive, multiauthored textbook on palynology (Jansonius & McGregor, 1996a). The accounts by Green (2001) are two chapters from a modern and comprehensive textbook on palaeontological techniques and give step-by-step

instructions on all established palynological preparation methods. There is an extensive literature on palynological techniques; in particular, many contributions on this topic were published in the journal *Micropaleontology* during the 1960s. The references in this paper are deemed to be the principal contributions on this subject, but it should not be considered to be a fully comprehensive bibliography.

Normally around 20-30 g of unweathered sample material are required for a successful palynological preparation. Larger amounts of organic-lean sediment per sample are needed due to the low density of sedimentary organic material in, for example, some pure limestones (Wilson, 1971a; Nørgaard *et al*., 1991) and sand-rich sedimentary rocks. Clean sampling equipment is imperative in order to protect against the cross contamination of palynomorphs between samples. Funkhouser (1967) gave a brief description of sample types and possible sources of contamination during sampling and allochthonous palynomorphs. Contamination may be from diverse sources, such as drilling mud additives (Traverse *et al*., 1961), laboratory supplies (Fisher, 1962) and even blackboard chalk (Echols & Levin, 1964). A comprehensive and up-to-date description of field collecting procedures, laboratory documentation, curation and related issues was given by Green (2001). Wood & Segroves (1963) briefly described a method for the standardisation of palynological sample collection.

The use of HF in palynological preparation was introduced by Assarson & Granlund (1924). In sediments and sedimentary rocks other than peat/coal, the HCl and HF remove carbonate and silicate minerals respectively, thereby destroying the matrix of the rock and leaving a residue of organic material and resistant minerals (e.g. rutile and tourmaline). The HCl treatment is always performed first and the decalcified residue must be decanted to neutrality before adding HF. This is to remove all calcium ions, which would form calcium fluoride (CaF_2) on reaction with the HF. Calcium fluoride is relatively insoluble and hence difficult to remove from the residue. Glass should not be used for HF treatment because this acid rapidly corrodes glass (Cridland, 1966). The mineral acids have no significant corrosive effect on sporopollenins and other organic elements such as wood fragments and effectively etch the palynomorphs from the rock fabric. Sarmiento (1957) used orthophosphoric acid (H_3PO_3) to remove carbonates and claimed that it is a gentler reagent than HCl. Despite this, H_3PO_3 has not replaced HCl for carbonate mineral dissolution in most palynology laboratories. Following mineral acid treatment, the palynomorphs in the raw organic/mineral residue are separated and concentrated in various ways. These include briefly oxidising, normally with nitric acid (HNO_3), to clear the residue of any residual fine organic materials (Funkhouser & Evitt, 1959). Other methods of 'cleaning' the palynomorphs include ultrasonic treatment, the density separation of any resistant minerals and the sieving away of finely disseminated organic matter (Caratini, 1980; Ediger, 1986). Density separation techniques normally involve either centrifuging or 'swirling' in a large watch glass the residue in order to separate the dense mineral fraction from the light organic residue. Centrifuging media are heavy liquids such as bromoform ($CHBr_3$), sodium polytungstate ($3Na_2WO_4 9WO_3 H_2O$), stannic chloride ($SnCl_4$), zinc bromide ($ZnBr_2$), and zinc chloride ($ZnCl_2$) (Davis, 1961; Kummel & Raup, 1965; Munsterman & Kerstholt, 1996). Forster & Flenley (1993) developed a density gradient centrifugation technique that removes extraneous organic materials and can fractionate modern and fossil palynomorphs. Tschudy (1960) devised a device termed a 'Vibraflute' to separate palynomorphs from mineral grains and finely disseminated organic material. The 'Vibraflute' is a subhorizontal glass tube, which is connected to a vibrotool (Tschudy, 1960, fig.1). A sample residue is placed in the glass tube and the tube is agitated with the vibrotool. The palynomorphs tend to concentrate in the centre of the glass tube, with the resistant minerals and fine material at the proximal and distal ends respectively; these three fractions can then be readily separated. Another method of separating palynomorphs from extraneous organic material was described by Hansen & Gudmundsson (1979). Following standard acid preparation, oxidation, filtration and centrifugation, these authors treated acid-resistant residues with ethyl alcohol (C_2H_5OH), which is absorbed into the cavities of the palynomorphs thereby reducing their density. The denser, massive organic material (e.g. wood fragments, resistant minerals etc.) then settles out in a separation tube (Hansen & Gudmundsson, 1979, fig. 2), whereas the less dense palynomorphs are in suspension close to the alcohol/water interface and can be separated by decanting. This method is especially useful in highly carbonised material or rocks with low palynomorph contents. Palynomorphs can also be separated from aqueous residues by flotation using either solvents of high specific gravity or oil (Kurtz & Turner, 1957; Urban, 1961). Bond (1964) described a simple method for the removal of light colloidal material from palynological preparations. Colloidal material may also be removed by a combined $ZnBr_2$ and HF method according to Björck *et al*. (1978). Residues are also normally treated with a dilute alkali solution such as ammonium hydroxide (NH_4OH) or potassium hydroxide (KOH) in order to neutralise any humic acids present; these may be released during the oxidation process (see below). If the palynomorphs and the remaining kerogen macerals form clumps at this stage, these aggregations may be dispersed by brief (< 1 minute) ultrasonic vibration, at *c*. 50 kHz, of the residue. At this stage, if the palynomorphs are pale or have been bleached during the preparation process, they may be stained to accentuate their body colour to aid observation and make any photomicrography easier. Several staining media can be used, for example Bismarck Brown, Congo Red, Fuchsin, Methyl Green, Methylene Blue, Safranine O, Vert Green and various food colouring products (Artusy & Artusy, 1956; Clarke, 1963; Wilson & Goodman, 1963).

Following these various cleaning procedures, the aqueous palynomorph-rich residue is then concentrated by

sieving to the required level (Kidson & Williams, 1969; Raine & Tremain, 1992) and mounted on glass microscope slides. Vidal (1988) described the use of a membrane-filtering unit for maximising palynological yield in organic-lean material. Slide production is commenced by evaporating several droplets of residue onto a glass coverslip. The residue to be mounted is treated with a dispersing agent in order to form a thin film on the coverslip with an even distribution of grains (Jeffords & Jones, 1959). When dry, the coverslip is mounted to the microscope slide using a proprietary mounting medium. Schopf (1960) described a double coverslip technique. Various mounting media have been used and include Canada balsam, Cellosize, Clearcol, Elvacite, gelatin, glycerine, glycerine jelly, gum arabic, silicone oil, Vinylite and various optical adhesives (Andersen, 1960; Wilson, 1968; Barss & Crilley, 1976). No single mountant is unequivocally superior and these vary in optical properties, principally the refractive index, and longevity. If the mounting medium is alcohol-based, palynomorphs may be isolated for scanning electron microscope (SEM) study by using a solvent such as dimethyl sulphoxide ($C_2H_8OS/(CH_3)_2SO$, also referred to as 'DMSO') (Shane & Clark, 1981). Jacobsen & Schopf (1979) described a methodology for the reverse process, i.e. mounting on glass slides specimens that have been studied using an SEM. Palynomorphs mounted in glycerine jelly may also be extracted and remounted/reused (Wilson, 1971b). It should be noted that the body colour of palynomorphs mounted on microscope slides frequently fade with time. Riding & Helby (2001, fig. 16) demonstrated that specimens of the Jurassic dinoflagellate cyst *Tabularium senarium* have faded significantly over a *c.* 20-year period. This phenomenon is especially noticeable in *Tabularium senarium* because this species is characterized by prominent, dark, low relief intratabular ornamentation, however fading has been discerned in many other palynomorph taxa. The remainder of the aqueous residue is normally stored as a liquid and some laboratories favour the addition of several drops of dilute HCl or phenol (C_6H_6O) to prevent fungal infestation (Elsik, 1966a). Dempsey & Urban (1965) and Felix & Burbridge (1985) described methods for the dry storage of palynomorph residues. It is necessary to ensure that all laboratory equipment used in this process is scrupulously clean at the start of this procedure. This is to avoid the cross contamination of samples via residues from previous samples on acid vessels and other laboratory equipment. Some laboratories use a dishwasher with strong detergents, disposable labware or a powerful chemical cleaning agent such as chromic acid (CrO_3), which cleans palynomorphs and organic materials by oxidation.

Megaspores may require alternative preparatory techniques (Hills & Sweet, 1972). Furthermore, it should be noted that coal samples, which lack silicate minerals, are prepared by oxidation with Schulze's solution or fuming HNO_3. There is the 'dry' (or 'Raistrick') method (Raistrick & Simpson, 1933; Raistrick, 1934; Raistrick & Marshall, 1939) and the so-called 'wet' method (Smith & Butterworth, 1967). Schulze's solution is an extremely powerful oxidising agent and comprises 70% HNO_3 supersaturated with potassium chlorate ($KClO_3$) (Manum, 1956; Staplin *et al.*, 1960; Traverse, 1988). Staplin *et al*. (1960) described how pre-treatment of coals with acetone (CH_3COCH_3), benzene (C_6H_6) and methanol (CH_3OH) may shorten the oxidative process of coals. In the 'dry' method, the coal is crushed and Schulze's solution is added carefully. A variant of this technique is that nitric acid is added to a mixture of coal and $KClO_3$. Following the oxidation process, which varies considerably depending upon the coal being prepared, the Schulze's solution is washed away by repeated decantations and dilute alkali is washed through the residue. The spore-rich residues can then be concentrated and mounted on microscope slides. Smith & Butterworth (1967, p. 100-104) fully described the 'wet' method and various other techniques applicable to coal samples. More information on coal preparations can be found in Tschudy (1958), Staplin *et al*. (1960), Spielholtz *et al*. (1962), Lee (1964), Gray (1965a,b) and Wood *et al*. (1996). Likewise, peat samples are prepared by treatment in hot 10% KOH, washing and acetolysis using acetic anhydride [$(CH_3CO)_2O$] and concentrated sulphuric acid (H_2SO_4) in a 9:1 ratio (Brown, 1960; Erdtman, 1960; Bigelow, 1980; Moore *et al*., 1991; Green, 2001, p. 132, 133). Acetolysis, or acetylisation, is often mis-termed acetylation but is in fact a two-step process involving both acid hydrolysis and esterification (Erdtman, 1960). Acetolysis was discovered by Erdtman (1936) and eliminates or reduces cellulose from organic residues. The reaction is exothermic and water should not be added to the solution. Marret (1993) reported that acetolysis selectively destroys Quaternary dinoflagellate cysts. Specifically, this treatment was proved to adversely affect protoperidiniacean forms, whereas gonyaulacacean taxa are only slightly sensitive to acetolysis (see below). Occasionally, the need may arise to analyse solid bituminous materials and Faruqi & Copley (1966) described a technique for the extraction of palynomorphs from tar.

This brief summary gives the basic procedures to a highly complex, and potentially relatively slow, procedure that relies considerably on operator experience and sound laboratory technique, in addition to the availability of a well-appointed facilities (Wrenn, 1998).

ASPECTS OF THE SUSCEPTIBILITY OF PALYNOMORPHS TO OXIDATION

Despite their great physio-chemical robustness, palynomorphs are susceptible to oxidation, both of a sustained and rapid nature (Havinga, 1964, 1967; Kedves, 1985; Campbell, 1991; Campbell & Campbell, 1994). Oxidising reagents break up carbon chains in organic substances, forming simpler carbon compounds in a stepwise fashion (Holst, 1954). Exposed sedimentary rocks have been subjected to constant oxidation during the weathering process and the weathered crust should be thoroughly removed from an outcrop before sampling for palynology. The destruction of sedimentary organic material by

weathering is significantly greater in hot climates. Similarly, great care should be taken when treating organic residues with oxidising agents in the laboratory during the later stages of preparation. It is recommended that sample residues should be carefully monitored for palynomorph damage during the oxidising process. Chemicals such as HNO_3 and hydrogen peroxide (H_2O_2) are powerful oxidising agents and can destroy palynomorphs (Hopkins & McCarthy, 2002). This destruction can be either complete or partial degradation; for example Kempf (1971, pl. 23) demonstrated that H_2O_2 preferentially destroys the innermost layer of water-fern (*Salvinia*) megaspores by oxidation. More recently, Traverse (1990) demonstrated that hollyhock (*Althea rosea*) pollen is fundamentally altered by brief treatment with 'Clorox', a commercial bleaching agent that uses sodium hypochlorite (NaOCl) as the active ingredient. The outermost layer of the exine including the prominent, abundant spines is destroyed and the remaining exine shrinks and collapses. Other studies of the selective degradation of palynomorphs include Downie (1959), Elsik (1966b) and Schrank (1988). Certain palynomorphs have different tolerances to chemical oxidation. For example, Quaternary heterotrophic protoperidiniacean dinoflagellate cysts, such as *Brigantedinium*, are much more susceptible than autotrophic gonyaulacacean genera (e.g. *Impagidinium*, *Operculodinium* and *Spiniferites*) to oxygen-related decay (Dale, 1976; Wall *et al*., 1977; Marret, 1993; Head, 1996; Zonneveld *et al*., 1997, 2001). Savrda *et al*. (2001), in an elegant study, related this factor to ichnology and sedimentology. These authors studied the Quaternary of the New Jersey slope, eastern USA and found low gonyaulacacean:peridiniacean dinoflagellate cyst (G:P) ratios in rapidly deposited clays with low bioturbation. By contrast, Savrda *et al*. (2001) recorded associations rich in gonyaulacacean cysts from intensely bioturbated, sand-rich sediments. These differences were interpreted as taphonomic, rather than reflecting the original dinoflagellate cyst assemblages. Clearly the majority of the protoperidiniacean forms in the burrowed, sandy sediments had been oxidised *in situ* (McCarthy *et al*., 2002). Hopkins & McCarthy (2002) also noted the selective destruction of protoperidiniacean dinoflagellate cysts in the Quaternary of offshore New Jersey using H_2O_2. These authors also found that the adverse effects of H_2O_2 on pollen and spore floras were significantly less than on dinoflagellate cysts. Hopkins & McCarthy (2002) stressed that, rather than being a barrier to palaeoecological interpretations, a thorough knowledge of the differential preservation of palynomorphs can help the elucidation of geological problems. By contrast with the Quaternary, Dodsworth (1995) found that, in the laminated calcareous shales of the Mid Cretaceous Greenhorn Formation of Colorado, USA, oxidation with Schulze's solution and KOH solution selectively destroyed gonyaulacacean dinoflagellate cysts and did not affect peridiniacean taxa.

In certain cases, where sedimentary rocks contain significant levels of amorphous organic material, however, strong oxidising reagents may have to be used in the preparation process. This is because amorphogen tends to surround palynomorphs, thereby occluding them for microscopical study (Funkhouser & Evitt, 1959). In these cases, the risk of palynomorph damage or loss via oxidation is balanced by the need for their efficient extraction. Normally a swift (< 5 minutes) treatment of the sample residue with HNO_3 or fuming HNO_3 in a Buchner Flask is all that is required (Sarjeant, 1974, fig. 45). After oxidation, the residue should be washed with a dilute alkali such as 10% KOH or 5% NH_4OH; this clears away any humic acids which have been produced by the oxidising agents. The alkali treatment also helps to swell the palynomorphs, which may have shrunk during oxidation. Two Jurassic examples of organic-rich lithologies that require severe oxidation are the widespread organic-rich Lower Toarcian mudstones deposited during the Oceanic Anoxic Event (OAE) (Bucefalo Palliani *et al*., 2002) and the Upper Jurassic Kimmeridge Clay Formation (Ioannides *et al*., 1976, 1988; Nøhr-Hansen, 1986; Riding & Thomas, 1988). In these cases, the organic residues are typically treated for sustained periods with aggressive oxidising agents such as fuming HNO_3 or Schulze's solution in order to dissolve and fragment the sapropelic material so that it can be sieved away (Neves & Dale, 1963). Ultrasonic treatment can also help to fragment the amorphogen (Funkhouser & Evitt, 1959), but care must be taken as this technique can damage palynomorphs, especially if they are carbonised and therefore brittle (McIntyre & Norris, 1964). If an organic-rich sedimentary rock has been deeply buried, the amorphous organic material becomes much more tenacious and resistant to oxidation. An example of this is the Upper Jurassic (Oxfordian to Volgian) Brae Formation (Humber Goup) of the northern North Sea (Riley *et al*., 1989; Richards *et al*., 1993). Typically, Brae Formation material requires extended (> 24 hours) oxidation with Schulze's solution in order to release palynomorphs. This heavily oxidised material is frequently bleached of body colour and requires staining, to increase contrast, prior to microscopy. Experience of the laboratory preparation of these Jurassic organic-rich sediments indicates that the judicious use of strong oxidising agents does not significantly affect the palynomorph associations detrimentally. Part of the reason for this may be that Jurassic dinoflagellate cyst assemblages are dominated by gonyaulacacean taxa (Riding & Ioannides, 1996). Highly carbonised palynomorphs may be lightened to facilitate study by using Schulze's solution. However, this treatment may degrade them and the palynomorphs often redarken (Marshall, 1980). Safer methods of removing amorphous organic material have been described for pre-Quaternary material. For example, Eshet & Hoek (1996) worked on a Late Cretaceous (Campanian-Maastrichtian) limesone/marl succession from southern Israel, which is extremely rich in amorphous organic material. These authors bleached the residues with NaOCl for 8-12 hours and found that this treatment released abundant and well-preserved dinoflagellate cyst assemblages (see also Hoffmeister, 1960).

DISADVANTAGES OF THE ACID-DIGESTION PALYNOLOGICAL PREPARATION METHOD

The use of mineral acids in palynological preparation is highly expensive in terms of the cost of raw materials, laboratory infrastructure and staff time. Due to the aggressively corrosive nature of concentrated HCl, HF and HNO_3, these reagents must be used in modern, well-maintained fume cupboards, operatives should wear good quality protective clothing/footwear and all liquid acidic waste should be neutralised prior to disposal (Costa, 1983; Wood *et al*., 1996; Green, 2001). Hydrofluoric acid is the most hazardous chemical used in traditional palynological processing (see below). Safer methodologies such as the 'macerationtank-method' of Poulsen *et al*. (1990) have been developed for the use of HF. The advantage of this method is that both fresh and waste HF is carried along tubing and is never used in an open vessel. Another closed system technique is to adapt a kidney dialysis unit for HF macerations (Jackson *et al*., 1974; McKee, 1977). Certain legislations require the gases emanating from fume cupboards to be made safe ('scrubbed'), rather than merely venting them to the atmosphere. Specifically, the United Kingdom (UK) government passed the Control of Substances Hazardous to Health (COSHH) Regulations in 1988. These regulations have since been updated (Health and Safety Executive, 1999; or go to: http://www.hse.gov.uk/hthdir/noframes/coshh/). The COSHH regulations stipulate that, if a hazardous chemical is used in the workplace, a safer alternative must be used if possible. If this is not viable, the minimum amount of the hazardous substance must be used. Most other developed countries have similar legislation. The regulations pertaining to mineral acids by health and safety/environmental protection legislation also imposes administrative and logistical burdens on palynological laboratory facilities. For example in the USA, the maintenance costs of a pre-treatment system for acid waste disposal, plus licensing for use of these chemicals, varies from state to state and can be as much as $50,000 per year above normal building maintenance costs. These costs and logistical overheads also apply to modern closed vessel technologies such as microwave digestion systems (Ellin & McLean, 1994; Jones, 1994, 1998; Jones & Ellin, 1998; Simes & Wrenn, 1998). The difficulties associated with the use of mineral acids are exacerbated in mobile laboratories such as those used on offshore oilfield drilling rigs. In particular, Health and Safety issues regarding hazardous chemicals are significantly more acute in offshore settings where the risks of accidental spillage are much higher. Operators are, in the main, willing to invest in the relatively expensive rig laboratory infrastructure because many offshore drilling installations now require precise and rapid biostratigraphical data aquired during drilling operations (Payne *et al*., 1999). This is largely because modern directional drilling techniques can drill along relatively thin oil/gas reservoir beds. To maximise production levels, the drill bit must stay in the pay horizon. Any deviations from this path are detected by using palynological and/or micropalaeontological analyses of the cuttings/core material by microscopists at the rigsite (Holmes, 1999; Shipp, 1999).

THE HEALTH HAZARDS OF HYDROFLUORIC ACID

Hydrofluoric acid is by far the most hazardous and corrosive chemical used in the traditional palynological preparatory process. Its unique properties, which make HF so useful in surface cleaning and etching in addition to mineral digestion, also make it significantly more hazardous than either HCl or HNO_3. For example HF is 1000 times more undissociated than HCl and furthermore it is highly lipid soluble. Hydrofluoric acid causes severe burns and may be fatal if it comes into contact with *c*. 5 % of the body area. At low levels of exposure and/or concentrations, the initial contact burns from hydrogen ions play only a minor role in HF injuries; this is unlike other acids. Upon contact there is a delay, after which the HF disassociates via ionisation into hydrogen and fluoride radicals; it is the fluoride ions that cause the principal tissue and bone damage. This is the most insidious aspect of HF, such that, if it contacts the skin, pain is not generally felt immediately. Because it is highly lipid soluble, it penetrates the skin rapidly and, upon ionisation, fluoride ions rapidly cause prolonged destruction of deep soft tissues and bone. The fluoride ions typically initially destroy soft tissue via liquefaction necrosis and when bones are reached they are rapidly decalcified (Hodgkinson, 1991). Bone decalcification may also cause systemic fluoride poisoning in extreme cases; this condition is termed fluorosis and symptoms include weight loss, brittle bones, and anaemia. Because of the rapidity with which HF penetrates animal tissue, it is extremely difficult to neutralise and the destructive effects may last several days (Head, 1995a,b). In the gaseous phase, HF is extremely irritating to the eyes, skin and respiratory system. Airbourne concentrations of HF of 10-15 ppm will cause irritation and levels above 30 ppm are deemed to be extremely hazardous to life and health.

Any contact of HF with skin should be liberally irrigated with cold water and then treated with calcium gluconate [$(HOCH_2(CHOH)_4COO)_2Ca$] gel before urgently seeking immediate medical treatment. This substance comprises a mixture of calcium gluconate in a water-soluble gel medium, and can also be injected subcutaneously in extreme cases. Note that this gel should not be applied to eyes that, if affected, should be irrigated then treated with 1% calcium gluconate drops every few hours for several days. Calcium gluconate is an efficacious HF antidiote because it combines with the HF to form insoluble CaF_2, thereby preventing extraction of calcium from tissues and bones and the resulting painful, slow-healing burns. Calcium gluconate gel should ideally be stored in a refrigerator, however even when it is chilled it has a limited shelf life, normally of approximately 2 years. Because of the ability of HF to cause delayed tissue/bone damage without immediate pain, all incidents of skin, eye or tissue contact with this substance should receive

immediate first aid and medical evaluation, even if the areas of contact appear normal and no discomfort is felt.

The above description of the hazardous nature of HF should not preclude its laboratory use. With well-appointed laboratory facilities, suitable protective clothing/equipment and adequate training, it may be safely used in palynological preparation (see Green, 2001, chapters 8, 9). Given the extreme toxicity of this substance, it is a testament to the sustained skill and good practice of palynological technicians worldwide that relatively few HF-related accidents have occurred in preparation laboratories since its introduction by Assarson & Granlund (1924).

PREVIOUS LITERATURE ON PALYNOLOGICAL PREPARATION WITHOUT THE USE OF MINERAL ACIDS

It is clear that if palynomorphs could be extracted from sediments and sedimentary rocks without the use of mineral acids, this process would be safer, cheaper and probably quicker. Despite some mention of palynological preparation techniques which do not involve mineral acids in the literature, there is no widely practiced non-acid method currently in use. This section is a review of this subject in the literature.

Knox (1942) described a simple method of the separation

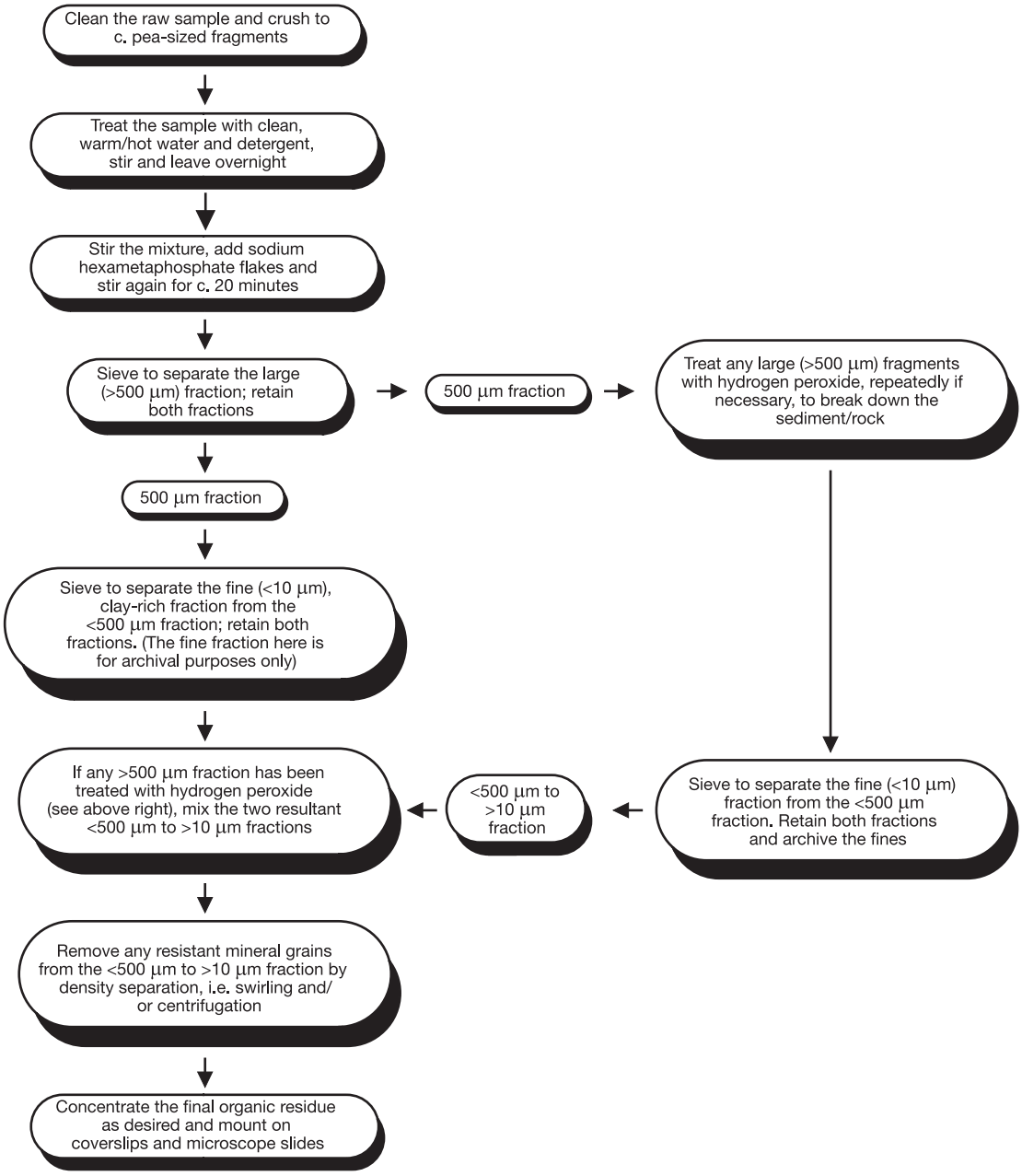

Figure 1. A flow chart illustrating the principal steps in the palynological preparation procedure using sodium hexametaphosphate.

of palynomorphs by a centrifugation technique. Firstly, the sediment is deflocculated by agitating it with CH_3COCH_3, $CHBr_3$ or water. This is especially suitable for unconsolidated or lightly lithified rocks. If the sedimentary rock is highly indurated, acids may be required to break the rock fabric down. Following deflocculation, the sediment is washed, dried and treated with a mixture of CH_3COCH_3 and $CHBr_3$ with a specific gravity of 2.3. It is then centrifuged and the sediment may require several centrifugations. The light fraction, containing the palynomorphs, floats on the surface of the heavy liquid after centrifugation and can be easily separated from the denser components.

A novel method of concentrating palynomorphs from unconsolidated sediments from semi-arid regions was described by Arms (1960). This author found that palynomorphs in material from the south-west of the USA and Mexico formed an insoluble colloid when treated with HF. To avoid this situation, the sample is placed in a 50 cm^3 centrifuge tube which is in turn placed in a 1 litre beaker (Arms, 1960, fig. 1). Distilled water is added to the sample, which is then treated with a 5% solution of water soluble depressant, 5% solution of pine oil in distilled water which has been agitated and laboratory detergent solution. Then gas is bubbled slowly through the aqueous mixture for 15 minutes. Bubbles will emerge from the centrifuge tube and these should be collected in the beaker. Mineral material, including mineralised microfossils, will remain in the tube and the bubble residue in the beaker should be rich in palynomorphs. This palynomorph-rich residue should be centrifuged if necessary and slides made up as normal. The depressant retards the removal of silicates in the bubbles, the pine oil solution adjusts the surface tension and coats the grains and the detergent produces the bubbles.

Brown (1960) described a modification for palynology of the disaggregation method used by Frizzel & Middour (1951), for the isolation of radiolaria using sodium pyrophosphate ($Na_4O_7P_2$). This chemical was found to disaggregate clays, although Brown (1960, p. 40-41) reported that it may destroy palynomorphs by oxidation, especially if heated. Shales do not consistently disaggregate when treated with $Na_4O_7P_2$. The disaggregation of some relatively tenacious mudrocks using $Na_4O_7P_2$ can be accelerated by boiling, however this has the effect of destroying the organic material, including palynomorphs, by oxidation according to Brown (1960, p. 94-95). Bates *et al*. (1978), however, stated that $Na_4O_7P_2$ does not cause the oxidation of palynomorphs. These authors described a method of preparing clay-rich Quaternary samples using $Na_4O_7P_2$. This method used the $Na_4O_7P_2$ as a clay deflocculant after the sample has been treated with either HCl or sodium hydroxide (NaOH). Following removal of the clay, samples were then still treated with HF (Bates *et al*., 1978, p. 460). Cwynar *et al*. (1979) described a sieving procedure for concentrating Quaternary pollen following both acid and $Na_4O_7P_2$ preparation procedures.

A procedure for the physical disaggregation of unconsolidated samples was described by Staplin *et al*. (1960). The fragmented and dried sample is treated with a mixture of proprietary core analysis fluid ('Soltrol C') and a miscible liquid detergent. Addition of a 1:1 CH_3COCH_3 and C_6H_6 mixture may help in some cases. This sample mixture is left to disaggregate and agitation is helpful. After settling, the clear liquor is decanted, boiling water added and the mixture stirred. It is then washed and centrifuged. This technique was envisaged as being a precursor to the acid phase of the traditional technique, but Staplin *et al*. (1960) stated that in rare cases the sample may have broken down such that further chemical treatment is superfluous. Even if the sample requires acid treatment following this disaggregation, Staplin *et al*. (1960) claimed that the pre-treatment saves both time and reagents.

Eagar & Sarjeant (1963) briefly described a technique used to extract dinoflagellate cysts from the London Clay Formation (Eocene) of southern England using a modification of the standard calcareous microfossil preparation procedure. The clay samples are soaked in water and sieved using a 64 µm sieve in order to remove the fine clay fraction. The > 64 µm fraction is then boiled with sodium carbonate (Na_2CO_3) in order to deflocculate the remaining clay and re-sieved. This procedure is repeated as necessary until the clay fraction has all been removed, leaving a clean, palynomorph-rich, residue. Eagar & Sarjeant (1963) commented that the dinoflagellate cyst assemblages obtained using this method are entirely composed of large chorate (spine-bearing) forms. It is probable that many of the smaller forms were washed away during the sieving process.

One of the most comprehensively described non-acid techniques is the mechanical disaggregation method of Felix (1963). Samples of Palaeogene/Neogene shales from the Gulf Coast, USA gave unsatisfactory results when prepared using the traditional preparation procedure. Firstly, the sample is washed for 24 hours in distilled water using a rotary washer. More indurated lithologies may require a 48 hour washing phase and, in some cases, the addition of a water-softening agent. The disaggregated sample is then centrifuged and the organic fraction dried and powdered. The powder is treated with $CHBr_3$ and toluene (C_7H_8), followed by gentle agitation in a microblender for 15 minutes. This allows the palynomorphs and kerogen to float, thereby allowing easy separation from the denser mineral fraction.

Williams & Downie (1966a, p. 20) briefly mentioned the occurrences of dinoflagellate cysts in washed preparations for foraminifera from the London Clay Formation of Isleworth, Middlesex. Murray J. Hughes, a micropalaeontologist with the British Geological Survey (BGS), had noted large clumps of abundant dinoflagellate cysts in disaggregated London Clay Formation samples and sent some picked specimens for identification to Charles Downie at the University of Sheffield in 1958. These samples had been prepared using standard, non-destructive methods for extracting calcareous microfossils (see also Eagar & Sarjeant, 1963). This action led directly to the intensive study of the marine palynology of the Palaeogene of southern England using traditional preparation techniques by Charles Downie and co-workers at Sheffield (e.g. Williams & Downie, 1966b,c; Eaton, 1976; Bujak *et al*., 1980).

Traverse (1978, p. 993) briefly described an impromptu method of palynological preparation, developed on board a drilling ship due to the late discovery that HF use was prohibited. This author disaggregated wet silty clays samples in an electric blender. This was followed by prolonged heating in 20% HCl, followed by boiling with Calgon®. This freed sufficient palynomorphs, which were isolated from the mineral-rich residue using $ZnBr_2$ gravity separation. Traverse (1978) stated that the traditional mineral acid preparation method used on the same samples onshore gave more concentrated and cleaner preparations. This non-HF method was also mentioned by Traverse (1988, p. 460). A similar method of dense-media separation for Quaternary organic-rich sediments and peats was described by Nakagawa *et al*. (1998).

Dodson (1983) described an experiment where he compared the results of a Quaternary pollen study in New South Wales, Australia using both acid digestion and ultrasonic dispersal of clays in $CHBr_3$, followed by centrifugation as preparation methods. This author found that the ultrasonic-centrifugation method produced more concentrated pollen associations and less extraneous organic matter than acid digestion. However the ultrasonic treatment damages pollen grains with thin exines such as Chenopodiaceae and Poaceae (see also McIntyre & Norris, 1964).

Herngreen (1983, p. 21) briefly mentioned the use of Darvan as a dispersant/deflocculant in palynological preparations. Darvan is a proprietary industrial dispersing agent and is known as lignosulphonic acid. It creates surface tension and causes separation of the finer, less dense, particles and settling of the coarser elements.

Heusser (1983) and Heusser & Stock (1984) described a sieve-decant method for the concentration of pollen grains in organic-lean sediments. Here, clay-sized particles are deflocculated using a solution of $Na_4P_2O_7$ and removed by sieving, leaving a palynomorph-rich residue which is then prepared in the traditional way using mineral acids. A method of palynological preparation suitable for fieldwork based on Heusser (1983) and Heusser & Stock (1984) was outlined by Van Der Kaars & Smit (1985). These authors adapted the sieve-decant method, omitting the mineral acid stages. They suspended 20 cm^3 of crushed sample in 500 ml of 10% $Na_4P_2O_7$ solution and left it to disaggregate for up to 24 hours. The sand and silt settles out relatively rapidly, leaving the bulk of the clay and palynomorphs in suspension. The suspension is then decanted and sieved at 7 μm. It was found that the addition of detergent at this stage allows the greater deflocculation of clay particles, thereby helping them to be sieved away and concentrating the palynomorphs. The palynomorph-rich residue is then washed using distilled water and alcohol, then stained and centrifuged. After the centrifuging stage, the palynomorphs are mounted on slides in the normal way (Van Der Kaars & Smit, 1985, p. 494, 495). These authors reported that the palynological preparations using $Na_4P_2O_7$, on Cretaceous and Palaeogene strata from Montana, USA are closely comparable to those prepared in the traditional way (see also Colbath, 1985).

Rueger (1986) developed a method using ethylenedinitrilotetraacetic acid (abbreviated to EDTA) for extracting palynomorphs from evaporites. This method was adapted from that of Bodine & Fernalld (1973), and involves the removal of any water-soluble minerals, the residue is then boiled for four hours in a 0.25 M solution of tetrasodium EDTA. Rueger (1986) stated that any remaining silicates could be removed using HF, however it is likely that density separation and/or clay disaggregation techniques will suffice to separate the palynomorphs from the silicate mineral fraction. Clay-Poole (1990) described a modified version of the technique described by Rueger (1986) for material from the arid south-west of the USA, which includes phases using both HCl and HF.

Clarke (1994) described an experiment involving the preparation of modern material using the traditional HF/acetolysis method and two non-acid techniques, i.e. sieving/swirling and heavy liquid separation. It was determined that all three methods have benefits and disadvantages, however the HF/acetolysis method generally proved the most effective technique.

THE EXTRACTION OF PALYNOMORPHS USING SODIUM HEXAMETAPHOSPHATE AS A DISAGGREGANT

Because of the hazardous and expensive nature of HCl, HF and HNO_3, especially in the field or at a rigsite, an effective generic method of extracting palynomorphs that does not require the use of aggressive chemicals would be highly desirable. It is known that certain non-acid preparation techniques are currently in use, but precise details of these methods are not in the public domain (Williams *et al*., 2003). Consequently, a project to test the use of certain methods of disaggregation techniques was undertaken by the BGS palynological laboratory. Because alternative palynological preparation procedures inevitably will rely on chemical/mechanical disaggregation of the mineral fabric, it was acknowledged that the best lithologies to test are relatively soft sedimentary rocks, which have not been intensively lithified, or unconsolidated sediments. Peterson *et al*. (1983) gave a brief review of the use of surfactants in disaggregating shales and other mudrocks. The technique described below is based around the deflocculating/sieving technique using Na_2CO_3 described by Eagar & Sarjeant (1963) and the HCl and Calgon® method of Traverse (1978, 1988).

The disaggregating and deflocculating agent sodium hexametaphosphate [$(NaPO_3)_6$] was used in this pilot study. This substance is the active ingredient in the dispersant Calgon® (Hodginson, 1991). Sodium hexametaphosphate has been successfully used in the BGS laboratories in the preparation of calcareous microfaunas. This is only one of several methods of disaggregating sedimentary rock samples to separate foramininfera and ostracods from the matrix (see also Kummel & Raup, 1965; Brasier, 1980). Several workers, however, have noted damage to calcareous microfossils using $(NaPO_3)_6$. These include Oda *et al*. (1975), who reported that

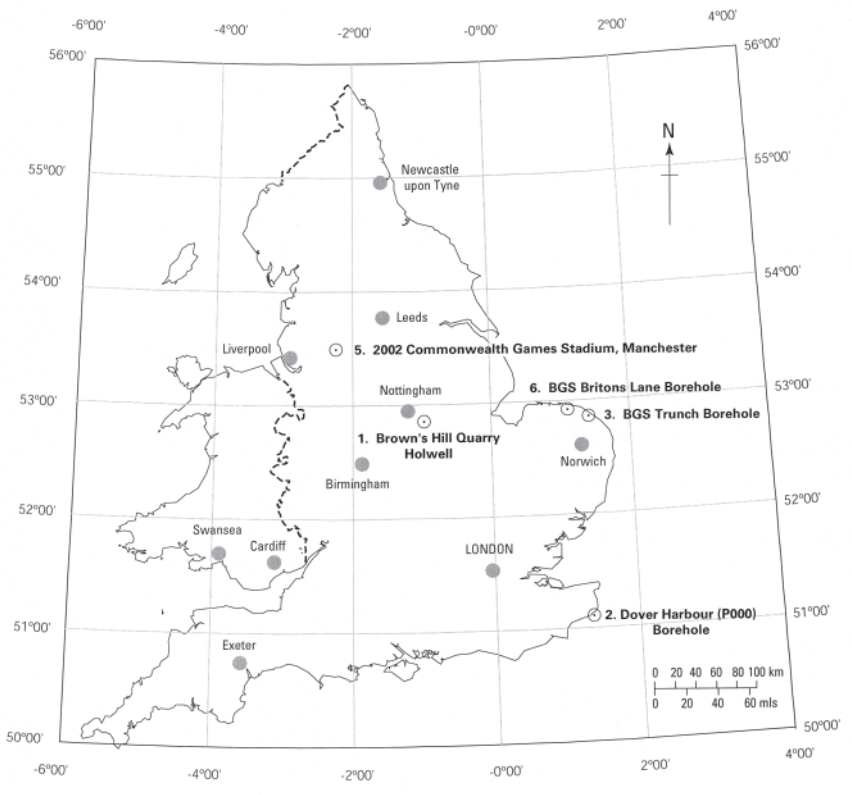

Figure 2. Location map of samples sets 1, 2, 3, 5 and 6 from England, UK.

a mixture of naptha and $(NaPO_3)_6$ damaged Holocene planktonic foraminifera, and that this dissolution effect increased with concentration and exposure time. This corrosional effect on calcareous microfossils was also noted by Van Stuijvenberg (1979) and Hodgkinson (1991). Hay (1977) stated that $(NaPO_3)_6$ at pH levels below 8 is corrosive to calcareous nannofossils. Despite this, in this study, $(NaPO_3)_6$ was not noted to have any deleterious effects whatsoever on palynomorphs.

Phosphates in solution have a high ionic charge and, even at low concentrations, affect suspensions of colloidal particles (van Wazer, 1958; Bates *et al*., 1978). The phosphates generally are known as deflocculating, dispersing and peptising reagents. They reduce the coherent nature of the clay because phosphate ions are strongly adsorbed onto the clay particles, which break up because of the high ionic charges. The surface charges prevent any reflocculation of the clay.

Sodium hexametaphosphate (EC chemical number 223-343-1) is readily available from most chemical suppliers. It is a white, odourless, crystalline, inorganic salt that is extremely soluble in water and normally supplied in flake form. The substance is a chain phosphate and has many synonyms including metaphosphoric acid and Graham's Salt. It is not a toxic chemical and there is no evidence of carcinogenic properties or mutagenic/teratogenic effects. However the manufacturers advise that, if ingested in large amounts, it may cause nausea, vomiting, gastric pain and/or diarrhoea. It also may cause irritation to the eye. These properties are typical of many inorganic salts, but it is relatively harmless if handled properly. No specific United Kingdom (UK) exposure limits have been assigned. In terms of an ecological/environmental hazard, $(NaPO_3)_6$ is not considered to be a dangerous substance, provided it is disposed of with due care and attention. Because it is a phosphate, if discharged in large amounts into a relatively small water body, it can cause eutrophication. In addition to disaggregating and deflocculating, $(NaPO_3)_6$ is used commercially as a water softener, as a dispersant in the dyeing process, as a corrosion inhibitor for metals and in industrial cleaning.

In the methodology followed here, 42 rock samples of Jurassic to Quaternary age were selected for palynological preparation using both the traditional acid digestion technique and the new $(NaPO_3)_6$ method. Selected samples were prepared using a quantitative technique (Harland, 1989), which allows palynomorphs extracted per unit mass of rock sample to be calculated. This method allows the effectiveness of both methods to be assessed objectively. The rock samples used are listed in Appendix 1. The preparation procedure using $(NaPO_3)_6$ is outlined in detail in Figure 1 and Appendix 2. In summary, the rock samples are softened overnight using a strong detergent, treated with $(NaPO_3)_6$, sieved, and mounted on microscope slides. Urgent ('hotshot') samples can be prepared omitting the overnight soak in water with detergent (step 2 of Figure 1 and step 1 of Appendix 2). The eventual palynomorph concentrate was generally found to be as rich and similarly preserved as those residues produced using the acid digestion process (see below). Another advantage with the $(NaPO_3)_6$ process is that any silicofossils in the sample are also preserved. Commonly, this treatment does not disaggregate the whole of the rock sample. The method used here of ensuring that all the rock is broken down is that any remaining material is repeatedly treated with H_2O_2 to effect full disaggregation. While this strategy ensures that all the sample material is prepared, it is possible that the fraction subjected to H_2O_2 may be oxidised (Hopkins & McCarthy, 2002). Hydrogen peroxide is an aggressive chemical and is known to corrode calcareous microfossils. Recent electronic correspondence on 'Paleonet' and other internet-based mailing lists has suggested that certain hair bleaching products, which contain peroxides at various concentrations, may also be useful in disaggregating sedimentary rocks. No recorded testing of this method, however, is available.

CASE STUDIES

In this section, seven case studies using the $(NaPO_3)_6$ palynological preparation process are described. Five of these studies have used both the acid and $(NaPO_3)_6$ methods in order to compare them and four are quantitative in order to effect objective comparisons of the two procedures. The geographical locations of all seven intervals studied and selected photomicrographs are given as Figures 2 to 7. Figure 3 is a montage of individual palynomorphs prepared using the $(NaPO_3)_6$ procedure. Relatively low magnification photomicrographs directly compare both preparation procedures in Figure 4 and illustrate examples of the $(NaPO_3)_6$ preparation technique in Figure 6. All taxa at and below species level mentioned in this contribution are listed in Appendix 3.

All the specimens illustrated in Figures 3, 4 and 6 are curated in the British Geological Survey palynological collections. The specimens in Figure 3 are curated in the figured specimen ('MPK') collection and the low magnification views in Figures 4 and 6 are of slides that are in the micropalaeontological/palynological ('MPA') collection.

1. The Lower Jurassic of Holwell Quarry, Leicestershire, UK (both preparation methods, non-quantitative)

Fourteen samples of the Lower Jurassic (Upper Pliensbachian to Lower Toarcian) Lias Group succession at Brown's Hill Quarry, Holwell, Leicestershire, central England (Figure 2; Clements, 1989), were prepared using both the traditional mineral acid method and $(NaPO_3)_6$. Holwell Quarry is a disused ironstone quarry and an important reference section for the Upper Pliensbachian and Lower Toarcian in the East Midlands; the palynology of this succession was described by Riding (2002a). This succession was chosen for study because it includes both limestones and organic-rich claystones, hence some lithological comparisons can be made. The samples are listed in Appendix 1 and the results illustrated in Table 1. These samples were not prepared quantitatively in order to test the $(NaPO_3)_6$ method under normal laboratory conditions. Routinely, most palynology samples are prepared using a non-quantitative preparatory technique.

The samples are variably palynologically productive. Samples 14 to 9 from the Dyrham and Marlstone Rock formations are sparse in palynomorphs and other plant phytoclasts. Preservation is generally poor and this is thought to be due to these sandstones and limestones being organic-lean and also possibly having being highly weathered. The palynofloras are dominated by pollen and spores but also include some marine microplankton, principally the prasinophyte *Halosphaeropsis liassica* and dinoflagellate cysts. Because the $(NaPO_3)_6$ preparation method uses this reagent as a disaggregant, it was somewhat surprising that it proved successful in preparing samples 13 to 9 from the Marlstone Rock Formation at this locality. This unit is a hard, splintery, chamosite oolite. However, the Marlstone Rock Formation is clearly primarily organic-lean at Holwell, so this may not be an entirely suitable test for limestones.

By contrast, the samples from the Whitby Mudstone Formation, numbers 8 to 1, are significantly richer and well-preserved (Table 1). The principal reasons for this major

Table 1. Numbers of palynomorphs per microscope slide from samples 14 to 1 from the Lower Jurassic of Holwell Quarry, prepared non-quantitatively using both methods. The largest within the respective pairs of figures are emboldened. The two right hand columns are a comparison between the two methods: these comprise the number of grains per slide from the acid method, minus this figure for the $(NaPO_3)_6$ procedure (Difference in number), and the percentage of this difference with respect to the overall count (Difference in %) for both methods respectively. Where the $(NaPO_3)_6$ method produced a higher number of palynomorphs per slide, these figures are emboldened. **Abbreviations**: D, dinoflagellate cysts; M, miospores; P, palynomorphs; V, various microplankton.

Sample	Mineral Acid				Sodium hexametaphosphate				Difference	Difference
#	P	M	D	V	P	M	D	V	in number	%
1	6297	1007	3	5289	**10721**	**1613**	**27**	**9081**	**-4424**	**26,00%**
2	**2797**	370	…	**2427**	2164	**415**	…	1749	+633	12,76%
3	**6004**	940	…	**5064**	5444	**1070**	**8**	4366	+560	4,89%
4	6228	2710	4	**3514**	**6529**	**3941**	**43**	2545	**-301**	**2,36%**
5	**3185**	**938**	…	**2247**	2011	918	**7**	1086	+1174	22,59%
6	**9047**	2769	4	**6274**	8995	**4088**	**32**	4875	+52	0,29%
7	4803	1105	3	3695	**8046**	**4106**	**11**	**3929**	**-3243**	**25,24%**
8	**5240**	**693**	…	**4547**	4376	613	**3**	3760	+864	8,99%
9	32	32	1	…	**53**	**40**	**2**	**11**	**-21**	**24,71%**
10	**505**	**321**	**10**	**174**	126	93	…	33	+379	60,06%
11	24	21	…	3	**133**	**105**	**2**	**26**	**-109**	**69,43%**
12	2	2	…	…	**67**	**52**	…	**15**	**-65**	**94,20%**
13	5	4	…	1	**33**	**18**	…	**15**	**-28**	**73,68%**
14	5	5	…	…	**11**	**6**	…	**5**	**-6**	**37,50%**

disparity in productivity are the relative organic richness of the Whitby Mudstone Formation and the overwhelming abundance of the prasinophyte species *Halosphaeropsis liassica* in this unit. This taxon dominates the miscellaneous microplankton counts in samples 8 to 1 (Table 1) and the dominance of *Halosphaeropsis liassica* is typical of the early Toarcian of north-west Europe (Bucefalo Palliani & Riding, 2000). Other miscellaneous microplankton and dinoflagellate cysts are also more diverse and prominent than those from the underlying succession. Samples 8 to 1 are also rich in amorphous organic material, which is a reflection of the oceanic anoxic event (Bucefalo Palliani & Riding, 1999; Bucefalo Palliani *et al.*, 2002). Miospores are present throughout and are the most diverse palynomorph group. They include undifferentiated bisaccate pollen, *Cerebropollenites macroverrucosus, Chasmatosporites* spp., *Cibotiumspora juriensis, Classopollis classoides, Classopollis meyeriana, Concavissimisporites verrucosus, Contignisporites* spp., *Coronatispora valdensis, Cyathidites* spp., *Dictyophyllidites* spp., *Ischyosporites variegatus, Osmundacidites wellmanii, Perinopollenites elatoides, Retitriletes austroclavatidites* and *Todisporites* spp. These associations are typical of the late Pliensbachian-early Toarcian interval (Srivastava, 1984). Dinoflagellate cysts are largely confined to the Whitby Mudstone Formation and include *Mancodinium semitabulatum*, *Nannoceratopsis deflandrei* subsp. *deflandrei*, *Nannoceratopsis deflandrei* subsp. *senex, Nannoceratopsis gracilis, Pareodinia* sp. and *Scriniocassis weberi*. Other prasinophytes comprise species of *Cymatiosphaera* and *Tasmanites*. These associations are also characteristic of the late Pliensbachian-early Toarcian interval (Bucefalo Palliani & Riding, 2000). For example, the co-occurrences of the dinoflagellate cysts *Mancodinium semitabulatum*, *Nannoceratopsis deflandrei* subsp. *deflandrei*, *Nannoceratopsis deflandrei* subsp. *senex* and *Nannoceratopsis gracilis* are indicative of the earliest Toarcian (Riding *et al.*, 1991; Riding & Thomas, 1992). The range bases of the dinoflagellate cyst *Nannoceratopsis gracilis* and the pteridophyte spore *Ischyosporites variegatus* are also reliable early Toarcian markers (Riding *et al.*, 1999). It is unsurprising that the Whitby Mudstone Formation samples prepared using $(NaPO_3)_6$ yielded similar associations as those prepared using the traditional acid procedure. This unit comprises relatively soft shales and mudstones and $(NaPO_3)_6$ is known to be a highly efficient disaggregant of clays.

The two preparation methods for these samples generally produced comparable palynomorph associations in terms of species spectra, diversities and absolute numbers (Table 1; Figures 3A-3H). Furthermore, the kerogen associations are also similar and preservation levels are comparable (Figures 4A, 4B). When comparing the palynological yields for each method, it should be borne in mind that the data in Table 1 are not quantitative. The numbers are simply counts per microscope slide and several factors during the preparation process may have biased these yields. These include the mass of rock processed, the concentration of palynomorphs in the final liquid residue and the amount of residue placed on the coverslip. However, it is anticipated that any significant trends will be clearly manifested. It is difficult to generalise about the palynological yields from the entire succession. For the Dyrham and Marlstone Rock formations (samples 14 to 9), the subsample set prepared using $(NaPO_3)_6$ proved to be generally the most palynologically productive. Here, all the samples except sample 10 produced more palynomorphs than the acid method, and in samples 12, 11 and 9, this difference is significant (Table 1). This trend is not evident in sample 10, which produced significantly more palynomorphs using the acid method than the $(NaPO_3)_6$ procedure. Samples 14 and 13 proved to be primarily sparse in palynomorphs. The eight samples from the Whitby Mudstone Formation all produced abundant palynofloras for both preparation methods. Five of these were more productive using mineral acids (Table 1). The differences vary in magnitude, but are generally relatively low. The productivity of Sample 6 is virtually identical using both methods. However, sample 5 proved much more productive using mineral acids. For the three samples which were palynologically richer using $(NaPO_3)_6$, numbers 7 and 1 were markedly different and sample 4 was virtually identical.

Comparisons of the data in Table 1 indicate that palynomorph taxa only observed in the acid preparation slides are extremely rare. There may, however, be certain palynomorph groups best suited to one or other of the preparation methods. For example, four out of the five samples that yielded foraminiferal test linings were those prepared using acid maceration. It is possible that these forms were lost during decantation. The pollen grain *Perinopollenites elatoides* and the spores *Contignisporites* spp. and *Todisporites minor* are present more often in samples prepared using mineral acids. Additionally, dinoflagellate cysts are more diverse in samples 11 and 5 prepared using acid, as compared to those samples which were treated with $(NaPO_3)_6$. These differences are, however, relatively minor and are deemed to be well within the range of the known natural variability of Toarcian dinoflagellate cyst associations (Bucefalo Palliani & Riding, 2003). The results from the Holwell Quarry samples indicate that the $(NaPO_3)_6$ preparation method appears to work well with both organic-lean, hard limestones and soft, organic-rich shales.

2. The Lower Cretaceous of the Dover Harbour (P000) Borehole, Kent, UK (both preparation methods, quantitative)

Ten samples of the Gault Formation (Middle and Upper Albian), from the Dover Harbour (P000) Borehole, Kent, south-east England, were studied (Figure 2). The Gault Formation is a shallow water claystone deposit (Owen *et al.*, 1996). All the samples are of mid grey, calcareous mudstones. This borehole was drilled as part of the site investigations for the Channel Tunnel and was mentioned by Shephard-Thorn (1988) and Hart (1993). The samples were prepared quantitatively using both the traditional mineral acid method and $(NaPO_3)_6$. The quantitative method was used for both preparation methods in order to test whether the two procedures produce similar numbers of palynomorphs per unit mass of rock in a well-lithified mudstone succession.

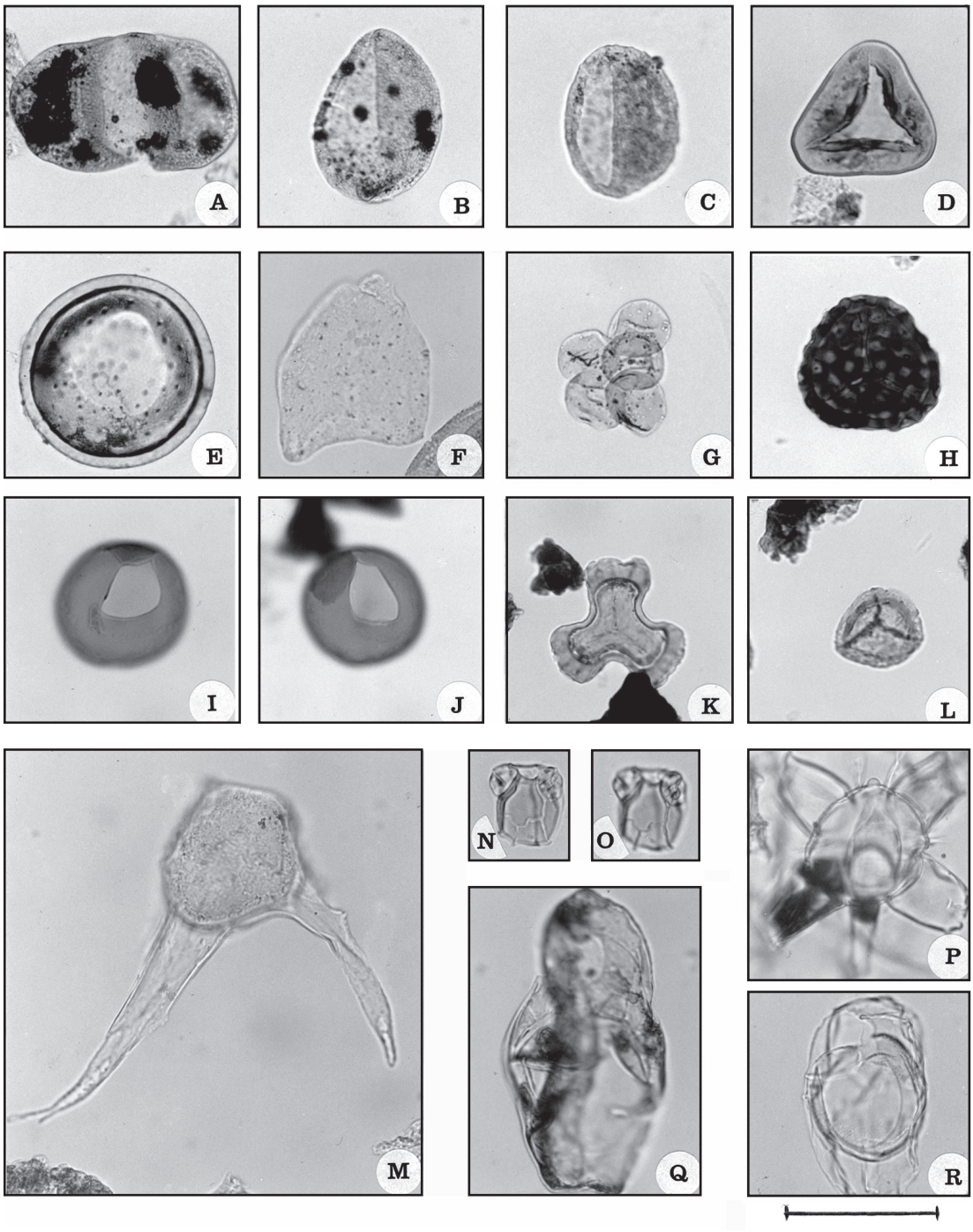

Figure 3. Examples of palynomorphs that were prepared using the sodium hexametaphosphate procedure from the case studies described herein. Scale bar = 50 µm. **A.** *Alisporites* sp. Brown's Hill Quarry, Holwell, Whitby Mudstone Formation, Lower Toarcian. Sample 4, specimen MPK 12769. **B, C.** *Chasmatosporites* spp. Brown's Hill Quarry, Holwell, Marlstone Rock Formation, Lower Toarcian. B - sample 11, specimen MPK 12770. C - sample 10, specimen MPK 12771. **D.** *Cyathidites australis,* location/stratigraphical details as A. Sample 6, specimen MPK 12772. **E.** *Tasmanites newtoni*, location/stratigraphical details as A. Sample 3, specimen MPK 12773. **F.** *Nannoceratopsis deflandrei deflandrei*, location/stratigraphical details as A. Sample 4, specimen MPK 12774. **G.** *Halosphaeropsis liassica, location/* stratigraphical details as A. Sample 4, specimen MPK 12775. **H.** *Ischyosporites variegatus*, location/stratigraphical details as A. Sample 8, specimen MPK 12776. **I, J.** *Brigantedinium simplex*, BGS offshore borehole 56-10/249 CS, Quaternary brown/black clay. I - sample 40, specimen MPK 12777. J – sample 40, specimen MPK 12778. **K.** *Tripartites vetustus* (reworked), location/stratigraphical details as I, J. Sample 40, specimen MPK 12779. **L.** *Lycospora pusilla* (reworked) Commonwealth Games Stadium site, Manchester, Upper Pleistocene Till. Sample 36, specimen MPK 12780. **M.** *Odontochitina operculata* (reworked), BGS Britons Lane Borehole, Quaternary diamicton. Sample 39, specimen MPK 12781. **N, O.** *Gillinia hymenophora* (reworked), N- median focus; O- low focus. location/stratigraphical details as M. Sample 38, specimen MPK 12782. **P.** *Neoeurysphaeridium glabrum* (reworked), location/stratigraphical details as M. Sample 39, specimen MPK 12783. **Q.** *Isabelidinium* sp. cf. *I. pellucidum,* Bodman Point, Seymour Island, Antarctica, López de Bertodano Formation. Sample 35, specimen MPK 12784. **R.** *Hystrichosphaeropsis quasicribrata* (reworked), location/stratigraphical details as M. Sample 38, specimen MPK 12785.

The samples are listed in Appendix 1 and the results tabulated in Table 2.

The samples proved variably palynologically productive and are normally dominated by dark wood phytoclasts and dinoflagellate cysts; other plant tissues, amorphous organic material and miospores are generally less common. Prominent dinoflagellate cysts include *Cribroperidinium*? *edwardsii, Cribroperidinium sepimentum, Cyclonephelium compactum, Odontochitina operculata, Oligosphaeridium* spp., *Spiniferites ramosus* and *Spiniferites* spp. Lower proportions of *Chlamydophorella nyei, Coronifera oceanica, Florentinia mantellii, Fromea amphora, Hystrichodinium* spp., *Ovoidinium scabrosum, Protoellipsodinium spinocristatum, Scriniodinium campanula, Stephodinium coronatum, Subtilisphaera perlucida* and *Wrevittia cassidata* were also observed. Prominent miospores include bisaccate pollen and pteridophtic spores. The palynomorph assemblages were comparable to those described from the Albian of north-west Europe by Cookson & Hughes (1964), Kemp (1970), Davey & Verdier (1971; 1973) and Nøhr-Hansen (1993). The palynomorph assemblages are broadly similar in assemblage compositions and relative proportions throughout the succession studied, indicating it lies within a single genetic sequence.

The different preparation styles of these ten samples normally produced comparable organic residues and palynofloras (Figures 4C, 4D). In five samples, the differences between the numbers of palynomorphs per gram produced by the two procedures accounts for less than 10% of the total palynomorph sum (Table 2). In sample 17, for example, this difference is only 2.77% and the two preparations are virtually indistinguishable. The two methods of preparing sample 16 also gave extremely similar organic residues. Samples 24, 23, 22, 20, 19 and 18 prepared using $(NaPO_3)_6$ produced cleaner palynofloras which were easier to study than when prepared using acids. Specifically, these $(NaPO_3)_6$ preparations produced less amorphous organic material and resistant mineral grains than when prepared with acid. Furthermore, in six of the samples, the $(NaPO_3)_6$ preparations produced more palynomorphs per gram than when the samples were prepared using acids (Table 2). Samples 23, 19 and 18 prepared using $(NaPO_3)_6$ were significantly richer than when prepared using acid. In the case of sample 18, the mineral acid preparation is dominated by dark amorphous organic material, which is not present in the $(NaPO_3)_6$ preparation. However, in sample 21, the mineral acid procedure produced a far richer association than the $(NaPO_3)_6$ preparation. This disparity is largely due to a much reduced level of dinoflagellate cysts in the $(NaPO_3)_6$ slides (Table 2). Sample 15 also produced considerably more palynomorphs per gram using mineral acid than when prepared using $(NaPO_3)_6$ (Table 2). The reasons for these differences are not clear; there were no discernible lithological difference between all ten samples.

Because miospores, dinoflagellate cysts and various microplankton are normally present in similar relative proportions, the two preparation methods appear to lack bias for or against any single palynomorph group. The ten samples from the Dover Harbour Borehole strongly suggest that the $(NaPO_3)_6$ preparation method is suitable for use on successions of moderately lithified mudrocks. In most cases, the palynomorph associations are richer and cleaner than samples prepared using the traditional mineral acid method. This may be due to the loss of some of the buoyant palynomorphs during decantation in the acid procedure.

3. The Upper Cretaceous of the BGS Trunch Borehole, Norfolk, UK (both preparation methods, quantitative)

Five samples of the White Chalk Subgroup, from the Trunch stratigraphical borehole in north Norfolk, south-east

Table 2. Numbers of palynomorphs per gram from samples 24 to 15 from the Dover Harbour (P000) Borehole, Kent, prepared quantitatively using both methods. The largest within the respective pairs of figures are emboldened. The two right hand columns are a comparison between the two methods: these comprise the number of grains per slide from the acid method, minus this figure for the $(NaPO_3)_6$ procedure (Difference in number), and the percentage of this difference with respect to the overall count (Difference in %) for both methods respectively. Where the $(NaPO_3)_6$ preparation method has produced a higher number of palynomorphs per gram, these figures are emboldened. **Abbreviations**: D, dinoflagellate cysts; M, miospores; P, palynomorphs; V, various microplankton.

Sample	Mineral Acid method				Sodium hexametaphosphate method				Difference	Difference
#	P	M	D	V	P	M	D	V	in number	in %
15	**4887**	353	**4405**	129	3867	**397**	3331	**139**	+1020	11,65%
16	1249	173	1043	33	**1451**	**225**	**1150**	**76**	**-202**	**7,48%**
17	**2429**	281	**2031**	**117**	2298	**346**	1876	76	+131	2,77%
18	1202	169	1001	32	**2081**	**210**	**1827**	**44**	**-879**	**26,77%**
19	1637	174	1337	**126**	**2236**	**250**	**1881**	105	**-599**	**15,46%**
20	4036	348	3601	**137**	**4766**	**483**	**4162**	121	**-730**	**8,29%**
21	**4627**	**436**	**4067**	**124**	1199	319	805	75	+3428	58,83%
22	4375	523	3753	99	**4847**	**779**	**3910**	**158**	**-472**	**5,12%**
23	4499	435	3847	217	**8377**	**889**	**7185**	**303**	**-3878**	**30,12%**
24	**5163**	**755**	**4148**	**260**	4312	656	3434	222	+851	8,98%

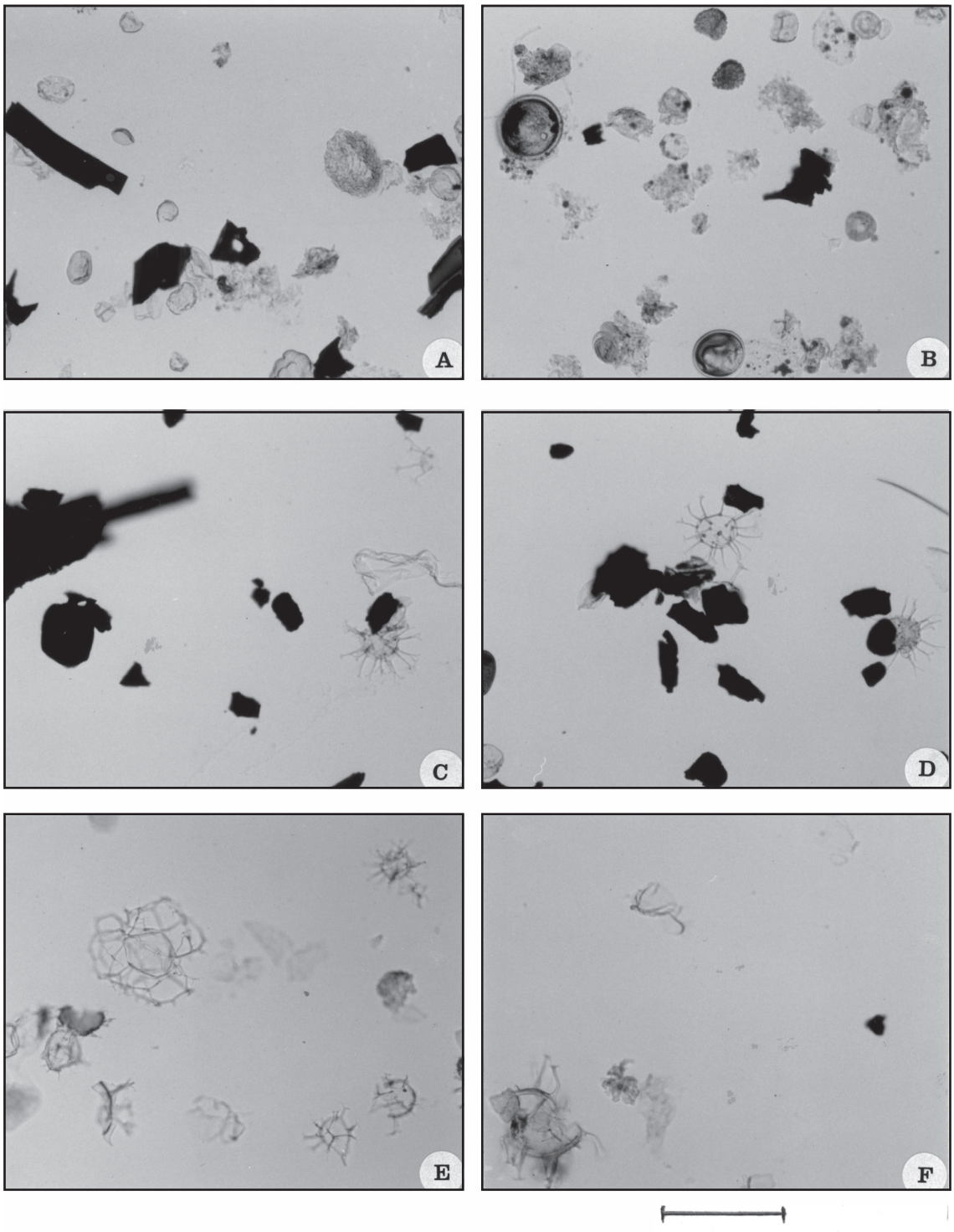

Figure 4. Direct comparisons of the mineral acid preparation technique (A, C, E) with the sodium hexametaphosphate ('hexa') procedure (B, D, F). Scale bar = 100 µm. **A, B.** Brown's Hill Quarry, Holwell, Whitby Mudstone Formation, Lower Toarcian. **A.** mineral acid preparation. Sample 4, 'acid' slide MPA 50794/2, Q51. **B.** sodium hexametaphosphate procedure. Sample details as A, 'hexa' slide MPA 50794/2, O52/2. Note the overall similarities of these non-quantitative preparations. There is a specimen of *Cerebropollenites macroverrucosus* in photomicrograph A and two specimens of *Tasmanites* in photomicrograph B. **C, D.** Dover Harbour Borehole, Lower Gault Formation, Middle Albian. **C.** mineral acid preparation. Sample 22, 'acid' slide MPA 50936/2, R50/4. **D.** sodium hexametaphosphate procedure. Sample details as C, 'hexa' slide MPA 50936/2, N54/3. The preparations are both quantitative; note the overall similarities of the two residues and the preponderance of chorate (spine-bearing) dinoflagellate cysts. **E, F.** BGS Trunch Borehole, Paramoudra Chalk, Upper Campanian. **E.** hydrochloric acid preparation. Sample 29, 'acid' slide MPA 50898/2, K54/2. **F.** sodium hexametaphosphate procedure. Sample details as E, 'hexa' slide MPA 50898/2, O56/1. The preparations are both quantitative; note the significantly better palynological productivity in the acid preparation; the sodium hexametaphosphate preparation is relatively palynologically sparse. Note the specimen of *Cannosphaeropsis utinensis* in E.

England (Figure 2), were examined. The samples are all of greyish white to white chalk with minor wispy marl flasers and which is occasionally burrowed. The Chalk Group is a marine micritic limestone, largely comprising calcareous nannofossils and deposited in a warm palaeoclimate in relatively deep waters (Hancock, 1975). Modern accounts of the stratigraphy of the Chalk Group of southern England were given by Bristow *et al.* (1997) and Rawson *et al.* (2001), although the lithostratigraphy of this unit in north Norfolk remains to be revised. These samples of Campanian-Maastrichtian chalk were all prepared quantitatively using both a modified mineral acid technique and the $(NaPO_3)_6$ procedure. The acid procedure used on these samples omitted the use of HF. Both authors have found that the treatment of HCl to large, non-marly Chalk Group samples effectively releases the palynomorphs from the rock matrix, thereby rendering the use of HF entirely superfluous. The quantitative method was used here to test if these two techniques yield comparable quantities of palynomorphs per unit mass of rock in a relatively organic-lean limestone. The samples are listed in Appendix 1 and the results tabulated in Table 3.

The samples proved moderately palynologically productive and are dominated by well-preserved dinoflagellate cysts. Other palynomorphs are rare and wood fragments are consistently present. Other plant tissues, amorphous organic material and resistant mineral grains are also present in variable proportions. Low numbers of reworked Carboniferous spores (*Densosporites* spp. and *Lycospora pusilla*) were noted in most of the samples. The Chalk Group is a pure coccolith-rich limestone and consequently profoundly organic-lean; this is reflected in the relatively low numbers of palynomorphs per gram of rock (Table 3). Prominent dinoflagellate cysts include *Alisogymnium euclaense, Cannosphaeropsis utinensis, Circulodinium distinctum, Cladopyxidium paucireticulatum, Cladopyxidium* spp., *Dinogymnium* spp., *Exochosphaeridium bifidum, Exochosphaeridium phragmites, Exochosphaeridium* spp., *Gillinia hymenophora, Hystrichosphaeridium tubiferum, Hystrichosphaeropsis quasicribrata, Isabelidinium* spp., *Membranilarnacia angustivela* (= '*Samlandia solida*' of Wilson, 1974), *Microdinium bensonii, Microdinium granocarinatum, Microdinium* spp., *Neoerysphaeridium glabrum, Odontochitina operculata, Palaeoperidinium pyrophorum, Palaeotetradinium maastrichtiense, Rottnestia wetzelii, Spiniferites ramosus, Spiniferites* spp., *Spongodinium delitiense, Trithyrodinium* spp., *Xenascus ceratioides, Xenikoon* sp. and *Xiphophoridium alatum*. Several of these forms have restricted stratigraphical ranges and are entirely consistent with the late Campanian and early Maastrichtian age of the samples. The floras are similar to coeval associations described from northern Europe (Wilson, 1974; Foucher, 1979; Robaszynski *et al.*, 1985; Herngreen *et al.*, 1986; Schiøler & Wilson, 1993; Slimani, 1994, 1996).

Counts of palynomorphs per gram for the two methods (Table 3) indicate that the HCl preparation technique largely yielded richer palynofloras than those prepared using $(NaPO_3)_6$ (see also Figures 4E, 4F). Only the uppermost sample, number 25, produced less palynomorphs using the acid method. Samples 29 to 26 produced relatively abundant palynofloras using HCl; the palynomorph yield using $(NaPO_3)_6$ was significantly lower. This disparity was most marked in samples 27 and 26 (Table 3). Furthermore, the palynomorph preservation of the acid method samples was consistently significantly better than those prepared using $(NaPO_3)_6$ (Figures 4E, 4F). The acid preparations of samples 29, 27 and 26 proved more conducive to microscopical study as they have considerably less extraneous material than is in the $(NaPO_3)_6$ preparations. The relative proportions of the three palynomorph groups are somewhat variable. Because of the overwhelming dominance of dinoflagellate cysts, these palynomorphs consistently reflect the differences in overall numbers between the two preparation methods. However, the concentrations of miscellaneous microplankton (acritarchs, foraminiferal test linings, prasinophytes etc.) are largely similar for each preparation method (Table 3). Only in sample 29 are the miscellaneous microplankton proportional to the overall palynomorph concentrations. In samples 29, 26 and 25, the miospore concentrations are also proportional to the entire palynoflora. Samples 28 and 27, however, are

Table 3. Numbers of palynomorphs per gram from samples 29 to 25 from the Trunch Borehole, Norfolk, prepared quantitatively using both methods. The largest within the respective pairs of figures are emboldened. The two right hand columns are a comparison between the two methods: these comprise the number of grains per gram from the acid method, minus this figure for the $(NaPO_3)_6$ procedure (Difference in number), and the percentage of this difference with respect to the overall count (Difference in %) for both methods respectively. Where the $(NaPO_3)_6$ preparation method has produced a higher number of palynomorphs per gram, these figures are emboldened. **Abbreviations**: D, dinoflagellate cysts; M, miospores; P, palynomorphs; V, various microplankton.

Sample	Mineral Acid (HCl)				Sodium hexametaphosphate				Difference	Difference
#	P	M	D	V	P	M	D	V	in number	in %
25	259	11	243	5	**392**	**14**	**371**	**7**	**- 133**	**20,43%**
26	**431**	**17**	**408**	**6**	113	6	103	4	+ 318	58,46%
27	**732**	**15**	**707**	**10**	228	14	207	7	+ 504	52,50%
28	**425**	7	**407**	**11**	258	**13**	235	10	+ 167	24,45%
29	**480**	**38**	**433**	**9**	190	8	177	5	+ 290	43,28%

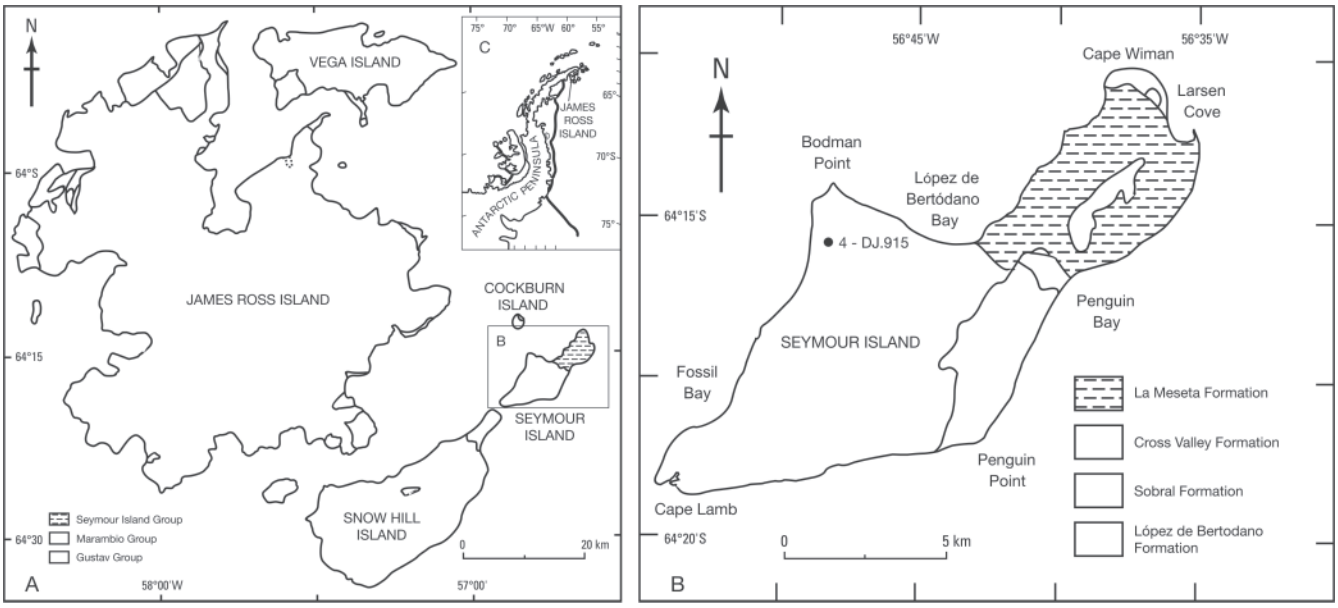

Figure 5. Location map of sample set 4, from south of Bodman Point, Seymour Island, Antarctica (adapted from Pirrie *et al.*, 1998). **A.** The James Ross Island archipelago, northern Antarctic Peninsula. **B.** Detail of Seymour Island, illustrating the location of sample set 4. **C.** The Antarctic Peninsula region.

significantly anomalous. In sample 28, the mineral acid method produced considerably more palynomorphs than the $(NaPO_3)_6$ procedure; paradoxically, the latter preparation method produced almost double the concentrations of miospores in comparison to the acid technique. Furthermore, the levels of miospores in sample 27 are virtually identical for both preparation methods (Table 3). These anomalies are not considered to be statistically significant and thus do not indicate that the two preparation methods may introduce marked preservational biases within the different palynomorph groups. This is because the levels of miospores and miscellaneous microplankton in the Chalk Group are extremely low. These samples indicate that the HCl digestion technique is significantly more effective than the $(NaPO_3)_6$ method for the palynological preparation of samples from the Chalk Group (Figures 4E, 4F). The reason for this phenomenon is that the $(NaPO_3)_6$ is not an effective disaggregant of pure limestones. Phosphates specifically disaggregate and deflocculate clays and other colloids. The results here suggest that calcite is more resistant to $(NaPO_3)_6$. This is confirmed by workers on calcareous microfaunas, who have found that the use of white spirit on finely milled chalk samples for releasing foraminifera from the Chalk Group is far more effective than the use of disaggregants such as $(NaPO_3)_6$ (I. P. Wilkinson, personal communication, 2002).

4. The Upper Cretaceous of Bodman Point, Seymour Island, Antarctica (both preparation methods, quantitative)

Six samples of the Upper Cretaceous part of the López de Bertodano Formation (Marambio Group) from Bodman Point, Seymour Island, Antarctica were studied (Figure 5). All the samples were prepared quantitatively using the mineral acid procedure and $(NaPO_3)_6$. The quantitative method was used in order to test whether the two preparation techniques yield the same levels of palynomorphs per gram of rock in an unlithified, siliciclastic succcession. The samples are listed in Appendix 1 and the results tabulated as Table 4. The samples studied are of Maastrichtian age on the basis of strontium isotope stratigraphy and evidence from belemnites and palynomorphs. The López de Bertodano Formation is a thick succession of largely unlithified silty sands and clays, representing a range of shallow marine shelf environments (Macellari, 1988; Zinsmeister *et al.*, 1989; Pirrie *et al.*, 1991; Crame *et al.*, 1991). The James Ross Basin has not been buried, hence the extensive Cretaceous and Palaeogene back arc basin succession is largely unlithified (Crame *et al.*, 1991).

The samples all proved organically productive, with prominent wood fragments, plant tissues and palynomorphs. The palynomorph assemblages proved to be similar in terms of both species and relative proportions of species, the conservative nature of the floras indicating deposition in a stable marine palaeoenvironment within a single genetic sedimentary sequence. Pollen grains are the dominant element, being more abundant than spores, dinoflagellate cysts and acritarchs. The pollen associations include bisaccate grains, *Microcachryidites antarcticus*, *Nothofagidites* spp., *Peninsulapollis gillii*, *Peninsulapollis truswelliae*, *Phyllocladidites mawsonii*, *Polycolpites langstonii*, *Propylipollis* sp. and *Proteacidites* spp. Cryptogam spores include *Ceratosporites equalis*, *Cyathidites* spp., *Grapnelispora* sp., *Laevigatosporites* spp., *Perotriletes majus* and *Retitriletes austroclavatidites*. The dinoflagellate cysts recovered include *Alterbidinium acutulum*, *Batiacasphaera* spp., *Cribroperidinium* spp., *Exochosphaeridium* sp., *Impagidinium* sp., *Isabelidinium* spp. (Figure 3Q). *Manumiella seymourensis*, *Manumiella* spp.,

Octodinium askiniae and *Palaeocystodinium* spp. The dinoflagellate cyst assemblage indicates an early-mid Maastrichtian age (Askin, 1988).

The two styles of preparation of all six samples produced generally similar organic residues and palynofloras (Table 4). For example, the numbers of palynomorphs per gram in sample 34 are extremely close (Table 4). It is clear, however, that the $(NaPO_3)_6$ preparations are normally significantly palynologically richer than their counterparts prepared using acids. The mineral acid method only produced more palynomorphs per gram in sample 32. The difference in the two yields are highly significant at this horizon; the $(NaPO_3)_6$ preparation yielded only 55.9% of the palynomorphs produced using the acid method (Table 4). The two preparations of sample 31 appear visually to be virtually identical, although the $(NaPO_3)_6$ preparation is clearly richer (Table 4). This situation also pertains to the remaining samples 35, 34, 33 and 30, and this difference in richness is clear from even a cursory examination of these slides. In samples 34 and 33, the acid preparations have more extraneous material than the $(NaPO_3)_6$ slides. The three palynomorph groups differentiated in Table 4, miospores, dinoflagellate cysts and various microplankton, are consistently present in similar proportions. This strongly suggests that neither of the two preparation methods affect one palynomorph group more than another. These samples prove that the $(NaPO_3)_6$ preparation method is suitable for samples of unlithified clays, silts and sands. Furthermore, in most cases, the $(NaPO_3)_6$ preparations are richer and cleaner than samples prepared using mineral acids.

5. The Quaternary of the UK

Because of the generally unlithified nature of Quaternary sediments, the $(NaPO_3)_6$ preparation method has proved extremely effective on samples of this age. Three examples from the Quaternary of the UK are described in this section.

5.1 Devensian till from the Commonwealth Games Stadium site, Manchester (both preparation methods, quantitative).Two samples of Devensian till from the site of the 2002 Commonwealth Games Stadium in Manchester, north-west England (Figure 2), were prepared quantitatively using both mineral acids and $(NaPO_3)_6$. This sediment is a brown, clay-rich till with abundant erratic clasts; similar sediment blankets much of the Pennine Plateau of northern England. Both methods produced closely similar organic residues and palynomorph associations. The two till samples yielded residues dominated by dark brown/black wood fragments and well-preserved Late Carboniferous (Westphalian) spore assemblages. Other plant tissues are also present, but in lower proportions. Pollen and spores of Quaternary aspect are relatively sparse. *Polypodium* spores are present persistently and arboreal pollen is, by contrast, rare; a single specimen of *Tilia* was recorded from sample 36. Minor levels of the freshwater/brackish alga *Botryococcus braunii* were also noted. Resistant mineral grains and amorphous organic material are absent. *Densosporites* spp. and *Lycospora pusilla* (Figure 3L) overwhelmingly dominate the Carboniferous spore associations. Associated with these are forms such as *Cirratriradites saturni, Crassispora kosankei, Cristatisporites indignabundus, Dictyotriletes bireticulatus, Endosporites globiformis, Florinites* spp., *Knoxisporites* spp., *Potonieisporites* spp., *Radiizonates aligerens, Raistrickia fulva, Savitrisporites* spp., *Triquitrites* spp and *Vestispora* sp. This flora is indicative of the Westphalian Series (Smith & Butterworth, 1967; Clayton *et al.*, 1977). The dominance of Westphalian spores indicates that much of the material incorporated into the till was derived from the nearby Lancashire and Yorkshire coalfields.

The preparations were closely comparable in terms of preservation levels, relative proportions, and diversities of both palynomorphs and kerogen macerals (Figure 6A). Counts of palynomorphs per gram, however, indicate that palynomorph recovery is markedly higher in the samples prepared using $(NaPO_3)_6$ (Table 5). The samples prepared using mineral acid digestion proved remarkably similar in terms of numbers of palynomorphs per gram of sediment. In the acid preparations, the proportion of Carboniferous spores is

Table 4. Numbers of palynomorphs per gram from samples 35 to 30 of the López de Bertodano Formation, Seymour Island, Antarctica, prepared quantitatively using both methods. The largest within the respective pairs of figures are emboldened. The two right hand columns are a comparison between the two methods: these comprise the number of grains per slide from the acid method, minus this figure for the $(NaPO_3)_6$ procedure (Difference in number), and the percentage of this difference with respect to the overall count (Difference in %) for both methods respectively. Where the $(NaPO_3)_6$ preparation method has produced a higher number of palynomorphs per gram, these figures are emboldened. **Abbreviations**: D, dinoflagellate cysts; M, miospores; P, Palynomorphs; V, various microplankton.

Sample	Mineral Acid preparation				Sodium hexametaphosphate				Difference	Difference
#	P	M	D	V	P	M	D	V	in number	in %
30	4577	4087	469	**121**	**5941**	**5345**	**543**	53	**-1364**	**12,97%**
31	3167	2419	**412**	**336**	**3849**	**3255**	356	238	**-682**	**9,72%**
32	**4409**	**3723**	**411**	**275**	2465	2173	169	123	+1944	28,28%
33	3837	3241	336	**260**	**4186**	**3658**	**368**	160	**-349**	**4,35%**
34	3981	**3369**	358	**254**	**4027**	3325	**518**	184	**-46**	**0,57%**
35	3387	2869	320	198	**3923**	**3347**	**353**	**223**	**-536**	**7,33%**

Table 5. Numbers of palynomorphs per gram from samples 37 and 36 from the Pennine Plateau near Manchester, which were prepared quantitatively using both methods. The largest within the respective pairs of figures are emboldened. The two right hand columns are a comparison between the two methods: these comprise the number of grains per gram from the acid method, minus this figure for the $(NaPO_3)_6$ procedure (Difference in number), and the percentage of this difference with respect to the overall count (Difference in %) for both methods respectively. Where the $(NaPO_3)_6$ preparation method has produced a higher number of palynomorphs per gram, these figures are emboldened. **Abbreviations**: B, *Botryococcus*; C, Carboniferous miospores; P, Palynomorphs; Q, Quaternary pollen and spores.

Sample	Mineral Acid				Sodium hexametaphosphate				Difference	Difference
#	P	C	Q	B	P	C	Q	B	in number	in %
36	4795	4527	251	**17**	**6549**	**6129**	**409**	11	**- 1754**	**15,46%**
37	4782	4688	87	7	**8765**	**8613**	**139**	**13**	**- 3983**	**29,40%**

slightly higher in sample 37. The concentration of *Botryococcus braunii* in sample 36 is higher in the acid preparation. This is the only category where the acid technique gives a higher proportion than the $(NaPO_3)_6$ method. Sample 37 prepared using $(NaPO_3)_6$ proved significantly richer than sample 36. The major part of this difference represents more Carboniferous spores, however sample 36 has fewer Quaternary pollen and spores than sample 37 (Table 5). These results confirm that the $(NaPO_3)_6$ method is ideal for the preparation of clay-rich, unconsolidated, glaciogenic sediments.

5.2 Chalky diamicton from the BGS Britons Lane Borehole, north Norfolk (sodium hexametaphosphate method only, non-quantitative). Two samples of highly chalky diamictons from the BGS Britons Lane Borehole, north Norfolk, south-east England (Figure 2) were prepared non-quantitatively using the $(NaPO_3)_6$ method only. This borehole was drilled in a quarry near Beeston Regis, west of Sheringham (TG 168 415); an account of the palynology of the diamicton units penetrated to 32.00 m was given by Riding (2002b). Samples 39 and 38 at 22.50-22.95 m and 16.90-17.00 m respectively, both produced extremely rich palynofloras that, unsurprisingly, are dominated by diverse and well-preserved Late Cretaceous dinoflagellate cysts (Figures 3M-3P, 3R; 6B, 6D). This clearly is a reflection of the obvious chalk-rich nature of this unit and the palynomorph associations from samples 39 and 38 were found to comprise 99.3% and 98.7% Chalk Group dinoflagellate cysts respectively. The two intervals sampled were prodigiously productive and yielded 21879 and 18813 palynomorphs per microscope slide, with minor levels of Carboniferous, Jurassic and Quaternary palynomorphs. The Chalk Group dinoflagellate cyst flora is interpreted as being latest Campanian to early Maastrichtian in age, based on the occurrences of key taxa such as *Alisocysta circumtabulata, Cannosphaeropsis utinensis, Cladopyxidium paucireticulatum, Gillinia hymenophora, Hystrichosphaeropsis quasicribrata, Isabelidinium cooksoniae, Neoeurysphaeridium glabrum, Neonorthidium perforatum, Palaeotetradinium maastrichtiense, Spongodinium delitiense, Wilsonisphaera petila* and *Xenascus "wetzelii"* of Slimani (1996). This is based on the European stratigraphical accounts of, for example, Wilson (1974), Foucher (1979), Foucher *in* Robaszynski *et al.* (1985), Herngreen *et al.* (1986) and Slimani (1994, 1996).

There appears to have been no discernible mixing of the Chalk Group input into this diamicton and proves that the Chalk Group input is entirely from the White Chalk Subgroup and was therefore locally derived. The dinoflagellate cyst associations are extremely similar in composition and relative proportions to those from the Upper Campanian and Lower Maastrichtian of the Trunch Borehole (see section 3), which supports a local origin for the Chalk in this diamicton. Other chalky diamictons from north Norfolk have yielded similar Campanian and Maastrichtian markers (Riding, 2002c). The abundance, diversity and well-preserved nature of these largely Late Cretaceous palynofloras from the Britons Lane Borehole highly chalky diamictons indicates that the $(NaPO_3)_6$ preparation technique is ideal for calcareous glaciogenic material. It was noted in section 3 that the HCl preparation method was more effective than the $(NaPO_3)_6$ technique for White Chalk Subgroup samples (Figures 6B, 6D). In these two diamicton samples, however, the $(NaPO_3)_6$ method yielded highly abundant, very well-preserved palynomorphs. The $(NaPO_3)_6$ preparations closely resemble those prepared using HCl from the Trunch Borehole. The reason for this apparent anomaly is not known. It is possible that the Chalk here is more marly, or it was partially disaggregated during glaciogenic processes, thereby making it more suitable for the $(NaPO_3)_6$ processing method. This contention should be tested by using both preparation methods quantitatively on these chalky diamictons.

5.3 Quaternary sediments from the Hebridean Slope (sodium hexametaphosphate method only, non-quantitative). Three Quaternary samples of brown/black clay were studied from BGS offshore shallow borehole number 56-10/249 CS, located west of the Hebrides (56° 14.53' N 009° 13.95' W), between 2.41 and 0.50 m (Figure 7). They were prepared non-quantitatively using the $(NaPO_3)_6$ method only. This borehole was drilled as part of an assessment of offshore slope stability and a more comprehensive account of the palynology was given by Riding (2002d). All three samples proved rich in indigenous and reworked palynomorphs. The indigenous Quaternary dinoflagellate cyst floras are overwhelmingly dominated by *Brigantedinium* spp., including *B. simplex* (Figures 3I, 3J; 6C). Other taxa present include *Achomosphaera andalousiensis, Bitectatodinium tepikiense, Operculodinium centrocarpum* and *Spiniferites* spp. These low diversity associations, dominated by protoperidinioid

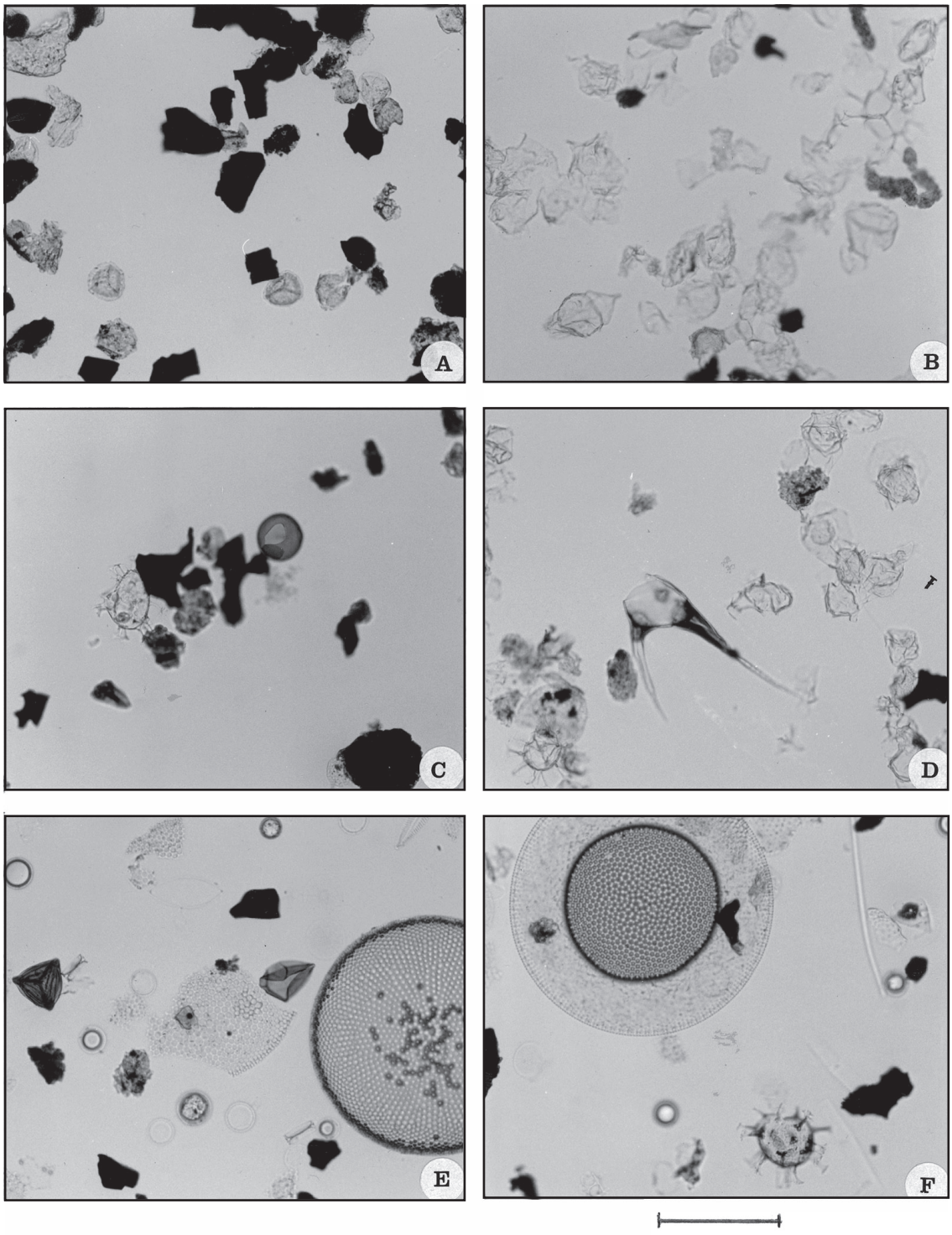

Figure 6. Examples of palynological preparations using the sodium hexametaphosphate ('hexa') procedure. Scale bar = 100 µm. **A.** Commonwealth Games Stadium site, Manchester, Upper Pleistocene Till. Sample 36, 'hexa' slide MPA 51063/2, R52/2. Note the abundant wood fragments and miospores; **B.** BGS Britons Lane Borehole, Quaternary diamicton. Sample 39, 'hexa' slide MPA 50997/2, P56/4. Note the abundant dinoflagellate cysts; **C.** BGS offshore borehole 56-10/249 CS, Quaternary brown/black clay. Sample 40, 'hexa' slide MPA 50819/1, G64. Note the common dinoflagellate cysts; **D.** Location/stratigraphical details as B. Sample 38, 'hexa' slide MPA 50994/1, P47. Note the abundant dinoflagellate cysts; **E, F.** BGS offshore borehole 56-10/255 VE, Quaternary brown/black clay at 3.44 m-3.47 m. Sample MPA 50832, 'hexa' slide 1, J48/4 (E) and S59/2 (F). This borehole cored a similar succession to borehole 56-10/249 CS and is located nearby, at 56° 22.15' N 009° 10.11' W. These two photomicrographs are included in order to demonstrate that the sodium hexametaphosphate preparation technique is suitable for both palynomorphs and silicofossils. Both photomicrographs include both these microfossil types. Note the reworked Cretaceous spore *Cicatricosisporites* in E and the chorate dinoflagellate cyst in F; note also the abundant pennate and centric diatoms in both photomicrographs.

cysts, are indicative of a relatively nearshore, glacial environment with seasonal or permanent sea-ice. The presence of *Brigantedinium simplex* is indicative of a Mid Pleistocene or younger age (Harland, 1992). The palynomorph content of the three samples proved relatively constant and the succession appears to be part of the same genetic sequence. Carboniferous, Jurassic, Cretaceous and Palaeogene reworking was also observed throughout (Figure 3K). Much of the Carboniferous reworking is likely to be Westphalian due to the presence in sample 41 of *Endosporites globiformis* (see Smith & Butterworth, 1967). The typically Bathonian to early Callovian (Mid Jurassic) dinoflagellate cyst *Ctenidodinium combazii* was also present in sample 41. *Nannoceratopsis pellucida* was recovered in sample 42; this Jurassic dinoflagellate cyst is typical of the Bathonian to Oxfordian interval (Riding & Thomas, 1992). The high abundance and well-preserved nature of these indigenous and allochthonous palynofloras from this succession indicates that the $(NaPO_3)_6$ technique is eminently suitable for unconsolidated clay samples (Figure 6C).

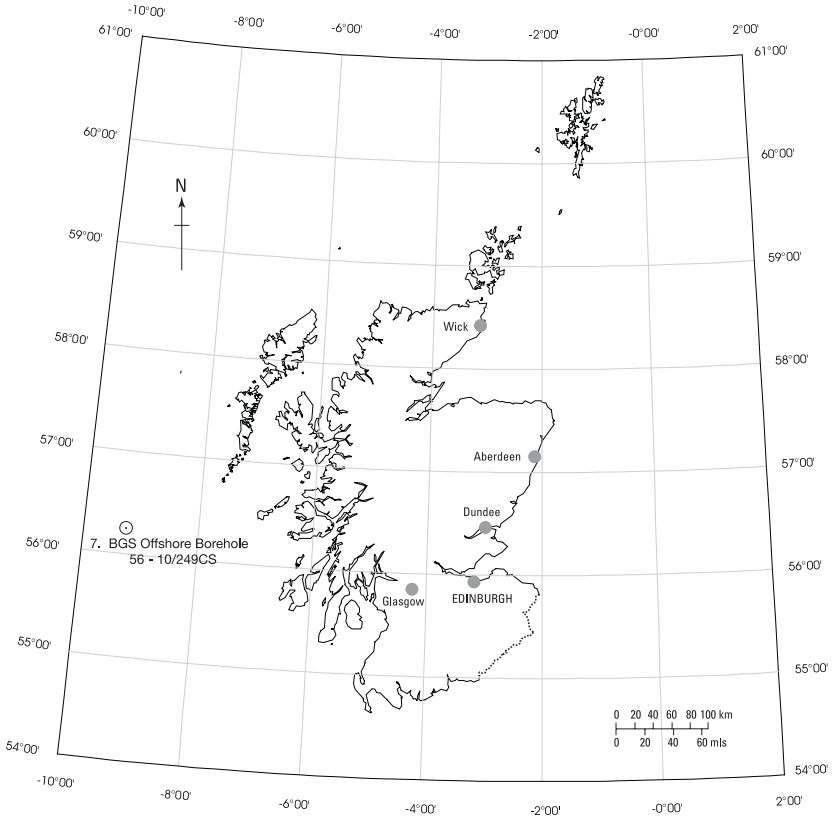

Figure 7. Location map of sample set 7, from BGS offshore borehole 56-10/249 CS, west of the Hebrides.

Marine Quaternary samples may be rich in silicofossils and the $(NaPO_3)_6$ preparation technique is also effective for preparing diatoms, radiolaria and silicoflagellates. A section drilled close to this borehole has yielded Quaternary clays that are rich in both silicofossils and palynomorphs. This is BGS borehole number 56-10/255 VE (56° 22.15' N 009° 10.11' W), which cored a similar succession to borehole 56-10/249 CS. Preparations of these sediments comprise a mixture of palynomorphs and silicofossils (Figures 6E, 6F). The dual function of the $(NaPO_3)_6$ preparation method is another major advantage of this technique.

CONCLUSIONS

The development of the palynological preparation method using $(NaPO_3)_6$, and its demonstration using seven case studies has shown that viable alternatives to the hazardous, time-consuming and expensive mineral acid technique can be developed. It seems clear that sediment/rock disaggregation, coupled with density separation methods, can extract palynomorphs effectively. Furthermore, any procedure that bypasses a complex chemical process using highly aggressive reagents should theoretically yield palynomorph assemblages that are closest to the material preserved. This is especially important as the majority of modern palynological studies are based on quantitative data. The traditional mineral acid procedure involves many stages, any of which could damage palynomorphs. The $(NaPO_3)_6$ technique appears to work on most of the lithologies tested. Of the five case studies where both methods were used, the $(NaPO_3)_6$ method was not as effective as the acid procedure in only one situation, the Chalk Group of north Norfolk. More studies should be done using $(NaPO_3)_6$ to prove its effectiveness in other lithologies and further develop this technique. For example, the $(NaPO_3)_6$ technique needs testing on pre-Jurassic samples, sandstones, highly indurated mudstones and more organic-rich sedimentary rocks. It is hoped that this contribution will stimulate further experimentation on palynological techniques, leading to the discovery of additional safer preparation procedures.

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APPENDIX 1. LISTING OF THE SAMPLES EXAMINED IN THIS STUDY

The 42 samples studied from seven localities in this work are listed here with all relevant geographical and geological information.

1. Brown's Hill Quarry, Holwell, Leicestershire (SK 742 235). Samples were collected by J. B. Riding on 12th December 2001; the bed numbers are those of Hallam (1968). All three formations are within the Lias Group; MRF = Marlstone Rock Formation.

No.	Lithological Unit	Zone/Bed No.	Lithology	Sample position
1	Whitby Mudstone Fm.	Falciferum/9	shale	316 cm from top MRF
2	Whitby Mudstone Fm.	Falciferum/9	shale	216 cm from top MRF
3	Whitby Mudstone Fm.	Falciferum/9	shale	136 cm from top MRF
4	Whitby Mudstone Fm.	Falciferum/9	clay	95 cm from top MRF
5	Whitby Mudstone Fm.	Falciferum/9	limestone	95 cm from top MRF
6	Whitby Mudstone Fm.	Falciferum/9	mudstone	70 cm from top MRF
7	Whitby Mudstone Fm.	Falciferum/9	mudstone	40 cm from top MRF
8	Whitby Mudstone Fm.	Falciferum/9	mudstone	20 cm from top MRF
9	Marlstone Rock Fm.	Tenuiserr./7	limestone	Uppermost part of MRF
10	Marlstone Rock Fm.	Tenuiserr./7	limestone	116 cm below top MRF
11	Marlstone Rock Fm.	Tenuiserr./7	limestone	230 cm below top MRF
12	Marlstone Rock Fm.	?Tenuiserr./7	limestone	90 cm above base MRF
13	Marlstone Rock Fm.	Spinatum/7	limestone	Lowermost 0.75 cm MRF
14	Dyrham Formation	Spinatum/5	sandstone	20 cm below base MRF

2. Dover Harbour Borehole (P000), Kent (TR 3341 4138). Samples were collected by J. E. Kyffin-Hughes. All are from the Gault Formation (Middle and Upper Albian). The Lower Gault (Middle Albian) and the Upper Gault (Upper Albian) are informal lithostratigraphical subdivisions.

No.	Lithostrat.	Bed No.	lithology	Zone	Depth (m)
15	Upper Gault	Bed 6a	grey mudstone	Dispar	108.00
16	Upper Gault	Bed X1	grey mudstone	Inflatum	112.00
17	Upper Gault	Bed X1	grey mudstone	Inflatum	116.00
18	Upper Gault	Bed X1	grey mudstone	Inflatum	120.00
19	Upper Gault	Bed X	grey mudstone	Inflatum	124.00
20	Lower Gault	Bed V11	grey mudstone	Lautus	128.60
21	Lower Gault	Bed 111	grey mudstone	Loricatus	132.70-133.00
22	Lower Gault	Bed 11	grey mudstone	Loricatus	136.70
23	Lower Gault		grey mudstone	Dentatus	140.00
24	Lower Gault		dark grey mudstone	?Dentatus	144.00

3. BGS Trunch Borehole, Norfolk, UK (TG 2933 3455). Samples were collected by J. E. Kyffin-Hughes and are from the White Chalk Subgroup. The sampled interval incorporates the Campanian-Maastrichtian transition. Samples 1 and 2 are soft, marly chalks from the Lower Maastrichtian (Lanceolata Zone). The remaining samples, 3-5, are from the massive white Paramoudra Chalk that is latest Campanian in age (Mucronata Zone). This borehole was drilled in order to give a reference section of the most complete Chalk Group development in the UK and discovered an especially well developed White Chalk Subgroup succession. A preliminary log was given by Gallois & Morter *in* Institute of Geological Sciences (1976, pp. 8-10). The sample details here, however, are taken from the revised log (BGS, unpublished).

No.	BGS Reg. No.	Lithostrat. Unit	Zone	Substage	Depth (m)
25	MPA 50894	Sidestrand Chalk	Lanceolata	L. Maastricht.	57.60
26	MPA 50895	Sidestrand Chalk	Lanceolata	L. Maastricht.	60.14 – 60.50
27	MPA 50896	Paramoudra Chalk	Mucronata	U. Campanian	67.51 – 71.32
28	MPA 50897	Paramoudra Chalk	Mucronata	U. Campanian	80.40 – 80.60
29	MPA 50898	Paramoudra Chalk	Mucronata	U. Campanian	81.00 – 81.20

4. Bodman Point, Seymour Island, Antarctica (64° 15' 24" S; 56° 48' 67"W). Samples were collected by D. J. Cantrill and J. A. Crame (British Antarctic Survey) during the Austral summer of 2000/2001. All samples are from the López de Bertodano Formation (Marambio Group).

No.	BAS Reg. Number	BGS Reg. Number	Height from base of section (m)
30	DJ.915.16	MPA 49924	90.00
31	DJ.915.15	MPA 49923	84.00
32	DJ.915.11	MPA 49919	60.00
33	DJ.915.8	MPA 49916	42.00
34	DJ.915.6	MPA 49914	30.00
35	DJ.915.2	MPA 49910	6.00

5. Commonwealth Games Stadium Site, Manchester, Lancashire, UK (SJ 387100 398500). These samples are of brown, clay- and erratic-rich Devensian (Upper Pleistocene) Till.

Sample Number	BGS Registration Number	Depth (m)
36	MPA 51062	1.00
37	MPA 51063	4.00

6. BGS Britons Lane Borehole, North Norfolk, UK (TG 168 415). This borehole was drilled at Britons Lane Quarry, Beeston Regis, near Sheringham and cored grey, brown and highly chalky diamicton units to 32.00 m (Riding, 2002b). Both samples here are highly chalky diamictons.

Sample Number	BGS Registration Number	Depth (m)
38	MPA 50994	16.90-17.00
39	MPA 50997	22.50-22.95

7. BGS Offshore Borehole 56-10/249 CS. This shallow offshore is located west of the Hebrides at 56° 14.53' N 009° 13.95' W.

Number	BGS Registration No.	Depth (m)	Lithology
40	MPA 50819	0.50-0.53	dark brown/black clay
41	MPA 50820	1.80-1.83	dark brown clay
42	MPA 50821	2.38-2.41	brown/black clay

APPENDIX 2. FULL DESCRIPTION OF THE PALYNOLOGICAL PROCESSING TECHNIQUE USING SODIUM HEXAMETAPHOSPHATE.

This method was developed iteratively in a working palynological laboratory (see also Figure 1).

1. Place approximately 100 g of cleaned rock sample, which has been crushed to *c*. pea-sized fragments, into a large glass/pyrex beaker.
2. Add *c*. 400 ml of hot/warm distilled/pure water and *c*.1% of strong detergent (e.g. *Teepol*), stir thoroughly and leave overnight.
3. Place the beaker on a magnetic hot plate set at a moderate heat level with a plastic-coated magnetic stirrer and agitate the mixture thoroughly.
4. Add *c*. 40 ml of sodium hexametaphosphate [$(NaPO_3)_6$] flakes to the sample vessel on the stirred hotplate. The substance mass in a specific volume of flakes is clearly variable, depending upon factors such as packing and the size/shape of the flakes. However, within reasonable limits, the amount of $(NaPO_3)_6$ is not critical in this technique.
5. Stir the mixture magnetically for *c*. 15-20 minutes.
6. Sieve the >500 μm fraction off and retain.
7. Sieve the sample mixture at 10 μm. It is anticipated that the <10 μm fraction will dominantly be deflocculated clay particles; these may be retained in order to determine if palynomorphs are present in this fraction or discarded. It is unlikely, however, that significant numbers of palynomorphs will be present in the <10 μm fraction. Ensure that all the $(NaPO_3)_6$ is washed out of the residue and retain the residue.
8. Treat the >500 μm fraction with hydrogen peroxide (H_2O_2) for *c*. 15-20 minutes in order to determine if these rock fragments break down.
9. Sieve this mixture at 10 μm to remove the 'fines' and retain the residue. Ensure that all of the H_2O_2 is washed out of the residue.
10. Should any of the >500 μm fraction remain, repeat steps 8 and 9 repeatedly until all the rock sample has broken down using H_2O_2.
11. Mix together the post-$(NaPO_3)_6$ and H_2O_2 residues. Should significant levels of resistant mineral grains be present, the majority can be removed by 'swirling' the residue in a large watch glass. This step should only be performed carefully because there is a chance that palynomorphs can become physically incorporated into the mineral fraction.
12. Centrifuge the residue with heavy liquid to separate any remaining resistant mineral grains and the organic fraction. This type of heavy liquid separation with *c*. 2.0 specific gravity zinc bromide is part of the normal palynological preparatory procedure. Ensure that all remaining zinc bromide is washed out of the residue.
13. Mount the concentrated organic residue, after appropriate staining if required, on microscope slides.

APPENDIX 3. LISTING OF PALYNOMORPH SPECIES.

All validly published palynomorph taxa that are mentioned in this work are listed here. A full bibliography on dinoflagellate c yst taxonomy was given by Williams *et al.* (1998).

Dinoflagellate cysts

Achomosphaera andalousiensis Jan du Chêne 1977

Alisocysta circumtabulata (Drugg 1967) Stover & Evitt 1978

Alisogymnium euclaense (Cookson & Eisenack 1970) Lentin & Vozzhennikova 1990

Alterbidinium acutulum (Wilson 1967) Lentin & Williams 1985

Bitectatodinium tepikiense Wilson 1973
Brigantedinium simplex Wall 1965 ex Lentin & Williams 1993
Cannosphaeropsis utinensis O. Wetzel 1933
Chlamydophorella nyei Cookson & Eisenack 1958
Circulodinium distinctum (Deflandre & Cookson 1955) Jansonius 1986
Cladopyxidium paucireticulatum Slimani 1994
Coronifera oceanica Cookson & Eisenack 1958
Cribroperidinium? *edwardsii* (Cookson & Eisenack 1958) Davey 1969
Cribroperidinium sepimentum Neale & Sarjeant 1962
Ctenidodinium combazii Dupin 1968
Cyclonephelium compactum Deflandre & Cookson 1955
Exochosphaeridium bifidum (Clarke & Verdier 1967) Clarke et al. 1968
Exochosphaeridium phragmites Davey et al. 1966
Florentinia mantellii (Davey & Williams 1966) Davey & Verdier 1973
Fromea amphora Cookson & Eisenack 1958
Gillinia hymenophora Cookson & Eisenack 1960
Hystrichosphaeridium tubiferum (Ehrenberg 1838) Deflandre 1937
Hystrichosphaeropsis quasicribrata (O. Wetzel 1961) Gocht 1976
Isabelidinium cooksoniae (Alberti 1959) Lentin & Williams 1977
Isabelidinium pellucidum (Deflandre & Cookson 1955) Lentin & Williams 1977
Mancodinium semitabulatum Morgenroth 1970
Manumiella seelandica (Lange 1969) Bujak & Davies 1983
Manumiella seymourensis Askin 1999
Membranilarnacia angustivela (Deflandre & Cookson 1955) McMinn 1988
Microdinium bensonii Slimani 1994
Microdinium granocarinatum (Below 1987) Lentin & Williams 1989
Nannoceratopsis deflandrei Evitt 1961 subsp. *deflandrei* (autonym)
Nannoceratopsis deflandrei Evitt 1961 subsp. *senex* (van Helden 1977) Ilyina *in* Ilyina et al. 1994
Nannoceratopsis gracilis Alberti 1961
Nannoceratopsis pellucida Deflandre 1939
Neoeurysphaeridium glabrum Slimani 1994
Neonorthidium perforatum Marheinecke 1992
Octodinium askiniae Wrenn & Hart 1988
Odontochitina operculata (O. Wetzel 1933) Deflandre & Cookson 1955
Operculodinium centrocarpum (Deflandre & Cookson 1955) Wall 1967
Ovoidinium scabrosum (Cookson & Hughes 1964) Davey 1970
Palaeoperidinium pyrophorum (Ehrenberg 1838 ex O. Wetzel 1933) Sarjeant 1967
Palaeotetradinium maastrichtiense Herngreen et al. 1986
Protoellipsodinium spinocristatum Davey & Verdier 1971
Rottnestia wetzelii (Deflandre 1937) Slimani 1994
Scriniocassis weberi Gocht 1964
Scriniodinium campanula Gocht 1959
Spiniferites ramosus (Ehrenberg 1838) Mantell 1854
Spongodinium delitiense (Ehrenberg 1838) Deflandre 1936
Stephodinium coronatum Deflandre 1936
Subtilisphaera perlucida (Alberti 1959) Jain & Millepied 1973
Tabularium senarium Dodekova 1990
Wilsonisphaera petila (Corradini 1973) Slimani 1994
Wrevittia cassidata (Eisenack & Cookson 1960) Helenes & Lucas-Clark 1997
Xenascus ceratioides (Deflandre 1937) Lentin & Williams 1973
Xenascus "wetzelii" of Slimani (1996)
Xiphophoridium alatum (Cookson & Eisenack 1962) Sarjeant 1966

Miscellaneous microplankton

Botryococcus braunii Kützing 1849
Halosphaeropsis liassica Mädler 1963
Tasmanites newtoni Wall 1965

Spores and pollen

Ceratosporites equalis Cookson & Dettmann 1958
Cerebropollenites macroverrucosus (Thiergart 1949) Schulz 1967
Cibotiumspora juriensis (Balme 1957) Filatoff 1975
Cirratriradites saturni (Ibrahim 1932) Schopf et al. 1944
Classopollis classoides (Pflug 1953) Pocock & Jansonius 1961
Classopollis meyeriana Klaus 1960
Concavissimisporites verrucosus Delcourt & Sprumont 1955
Coronatispora valdensis (Couper 1958) Dettmann 1963
Crassispora kosankei (Potonié and Kremp 1955) Bharadwaj 1957
Cristatisporites indignabundus (Loose *in* Potonié et al. 1932) Staplin & Jansonius 1964
Cyathidites australis Couper 1953
Dictyotriletes bireticulatus (Ibrahim *in* Potonié et al. 1932) Smith & Butterworth 1967
Endosporites globiformis (Ibrahim in Potonié et al. 1932) Schopf et al. 1944
Ischyosporites variegatus (Couper 1958) Schulz 1967
Lycospora pusilla (Ibrahim *in* Potonié et al. 1932) Schopf et al. 1944
Microcachryidites antarcticus Cookson 1947
Osmundacidites wellmanii Couper 1958
Peninsulapollis gillii (Cookson 1957) Dettmann & Jarzen 1988
Peninsulapollis truswelliae Dettmann & Jarzen 1988
Perinopollenites elatoides Couper 1958
Perotriletes majus (Cookson & Dettmann 1958) Evans 1970
Phyllocladidites mawsonii Cookson 1947
Polycolpites langstonii Stover in Stover & Partridge 1973
Radiizonates aligerens (Knox 1950) Staplin & Jansonius 1964
Raistrickia fulva Artüz 1957
Retitriletes austroclavatidites (Cookson 1953) Döring et al. 1963
Tripartites vetustus Schemel 1950