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Use of a microdrill for high stratigraphical resolution calcareous nannofossil analysis, and a modified filtration method

Allan Gil S. Fernando*, Hisatake Okada, Hiroshi Nishi, Reishi Takashima

Division of Earth and Planetary Sciences, Graduate School of Science, Hokkaido University, N10 W8 Sapporo 060-0810, Japan; *braaru-dosphaera@yahoo.com

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Abstract A tool capable of obtaining sediment samples at ≤1mm intervals is introduced in this short paper. The microdrill, as the tool is called, was developed in preparation for a high-resolution calcareous nannofossil analysis of Cretaceous black shale sequences.

Keywords Microdrill, high-resolution analysis, calcareous nannofossils, Cretaceous, black shales

1. Introduction

Obtaining sediments for calcareous nannofossil analysis has never been a real problem, since only a very small amount of sediment is needed for any type of qualitative or quantitative nannofossil study. In an attempt to study nannofossil distributions through intervals at high resolution, however, obtaining adequate amounts of sample material becomes a serious problem, especially when coupled with limited amounts of available sample, for example from Ocean Drilling Program (ODP) cores. To address this problem, and in preparation for the quantitative analysis and characterisation of nannofossil assemblages between laminated and non-laminated black shale intervals from the Vocontian Basin of SE France, we conceptualised and developed a tool capable of obtaining samples at ≤1mm intervals. The microdrill, as the tool is called, can be equipped with drill-bits of 0.1 to 1.0mm in diameter (Figure 1).

2. Methodology

2.1 Microdrilling

A sample is placed on the stage/platform and is fixed by a clamp (Figure 1a). The sample should preferably have a planar surface so that the waxed paper (~12cm^2), upon which the powdered sample is to be collected, can be easily attached. If the sample does not have a flat surface, some of the sediment powder produced from the drilling will go into the space between the waxed paper and the sample. A circular hole (~6mm in diameter) is made in the waxed paper with a hole-puncher. The hole is covered with adhesive tape (magic tape is recommended), and, with the sticky side facing the sample, attached to the surface of the sample/core prior to drilling (Figure 1f). The hole should be centred on the area of interest, where the drilling will take place. The use of waxed paper prevents contamination with previously drilled holes by keeping the powdered sample out of contact with the sample surface. The adhesive tape, on the other hand, ensures efficient recovery of the powdered sample by keeping the area around the drill-hole fixed, enabling the powdered sample to be collected above the waxed paper during drilling. After each hole is drilled, the contents of the waxed paper are transferred to another piece of waxed paper, where it is weighed (for the filter method, discussed below) or collected to make smear-slides. Since multiple drillings are sometimes necessary per level, several holes can be made in the waxed paper so it can be used again after transferring each sample. The risk of contamination is further avoided by cleaning the surface of the sample with an air-duster after each sample is taken and cleaning the drill bits ultrasonically after each use.

The microdrill (manufactured by Minitor Company Ltd., Japan) is equipped with a digital ruler to ensure that holes are drilled at the same level, in the case where multiple drillings are necessary per level, and that the intervals between samples are regular (Figure 1d). The diameter of the drill, the number of holes per level, and the amount of sample required ultimately depends on how much sample is available, and on the purpose of the study. Experimental holes drilled in several samples (limestones and black shales) have shown that the amount of sediment powder produced is proportional to the drill-bit diameter. The average amount of sediment produced per hole ranges from 0.9-2.4mg (0.8mm drill) to 4.2mg (1.0mm drill).

In our study, the 0.8mm drill was chosen so as to be able to obtain samples at 1mm intervals with negligible overlap between successive samples. Two to three holes were made per interval, with enough sediment produced (2.7-4.5mg) to make slides for biostratigraphic and semiquantitative palaeoenvironmental nannofossil analyses (smear-slide method of, *e.g.*, Bown & Young, 1998). Although counted data from smear-slides are considered to be semiquantitative, results from this type of analysis have been found to be comparable with quantitative results obtained from a filtering technique (Backman &

Shackleton, 1983). Currently available quantitative methods (*i.e.*, Andruleit, 1996; Flores & Sierro, 1997; Geisen *et al.*, 1999; Okada, 2000) require greater amounts of sediment, which even the microdrill method would have difficulty providing, especially with limited sample material available. In such cases, smear-slide analysis is the usual option *via* which to collect data, either as relative abundance values or, as Backman & Shackleton (1983) have suggested, as a measure/approximation of nannofossil abundance (no. coccoliths/mm^2).

2.2 Filtration

One problem which is usually associated with smear-slides, however, especially if not done properly, is the tendency of large forms (such as nannoconids) to concentrate or be more common in areas with thick sediment smear and/or along the slide edges. To minimise this problem in size fractionation, we employed a filtering technique (Okada, 2000), modified to suit our study. This technique allows us to make quantitative analysis of calcareous nannofossil assemblages even with a small amount of sediment. The method is outlined below.

The sediment powder produced from drilling is weighed. One to 1.5mg of sediment is suggested to avoid crowding the filter. The weighed sample is placed in a test tube, mixed with 10ml of buffered, distilled water, agitated in an ultrasonicator for ~10 seconds, and homogenised against a tube-mixer for several seconds. After homogenisation, the contents of the test-tube are poured into a small beaker containing 150-200ml of buffered, distilled water. The contents of the beaker are then filtered through a 0.45μm *Millipore* filter. The filter can either be air- or oven-dried. Okada (2000) suggested drying the filter in an oven for 10-30 minutes at a temperature of 50°C. After drying the filter, a portion (~1.2cm^2) is cut out, placed on a glass slide, rendered transparent with a drop of immersion oil and covered with a coverslip whose edges are fixed with nail polish. To obtain nannofossil abundance data (number of coccoliths/g of sediment), the total number of specimens counted, and observed fields of view (FOV) are recorded, so that:

$$\text{nannofossil abundance} = \frac{(\text{specimens counted}) \times (\text{area of filtered portion [mm}^2\text{]}) \times (\text{g}^{-1} \text{ of sediment used})}{\#\text{FOV (area of FOV in mm}^2\text{)}}$$

Aside from the number of coccoliths per unit weight of sediment, which can be calculated from the formula given above, the data obtained can also be reported as relative abundance values and number of nannofossils per unit area, following the suggestion of Backman & Shackleton (1983).

3. Conclusions

The described methodology makes it possible to obtain high-resolution samples from consolidated materials. The method, at present, is applicable only to calcareous nannofossils, since other microfossils (*i.e.*, foraminifera and radiolarians) are large enough to suffer considerable damage from the technique. Initial investigation of smear-slides prepared from the powdered samples has shown moderately- to well-preserved nannofossils. We believe that etching observed in some of the specimens is due to poor preservation within the black shale intervals rather than mechanical breakage during drilling. This is supported by scanning electron microscopy of several sections from the core (F. Giraud, pers. comm., 2005). A modified filtering technique (Okada, 2000) is suggested to take into consideration the small sample size and to eliminate the problem of size fractionation.

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References

Andruleit, H. 1996. A filtration technique for quantitative studies of coccoliths. *Micropaleontology*, **42**(4): 403-406.

Backman, J. & Shackleton, N.J. 1983. Quantitative biochronology of Pliocene and early Pleistocene calcareous nannofossils from the Atlantic, Indian and Pacific Oceans. *Marine Micropaleontology*, **8**: 141-170.

Bown, P.R. & Young, J.R. 1998. Techniques. *In*: P.R. Bown (Ed.). *Calcareous Nannofossil Biostratigraphy*. British Micropalaeontological Society Series. Chapman & Hall/Kluwer Academic Press, London: 16-28.

Flores, J.A. & Sierro, F.J. 1997. Revised technique for calculation of calcareous nannofossil accumulation rates. *Micropaleontology*, **43**(3): 321-324.

Geisen, M., Bollmann, J., Herrle, J.O., Mutterlose, J. & Young, J.R. 1999. Calibration of the random settling technique for calculation of absolute abundances of calcareous nannoplankton. *Micropaleontology*, **45**(4): 437-442.

Okada, H. 2000. An improved filtering technique for calculation of calcareous nannofossil accumulation rates. *Journal of Nannoplankton Research*, **22**(3): 203-204.

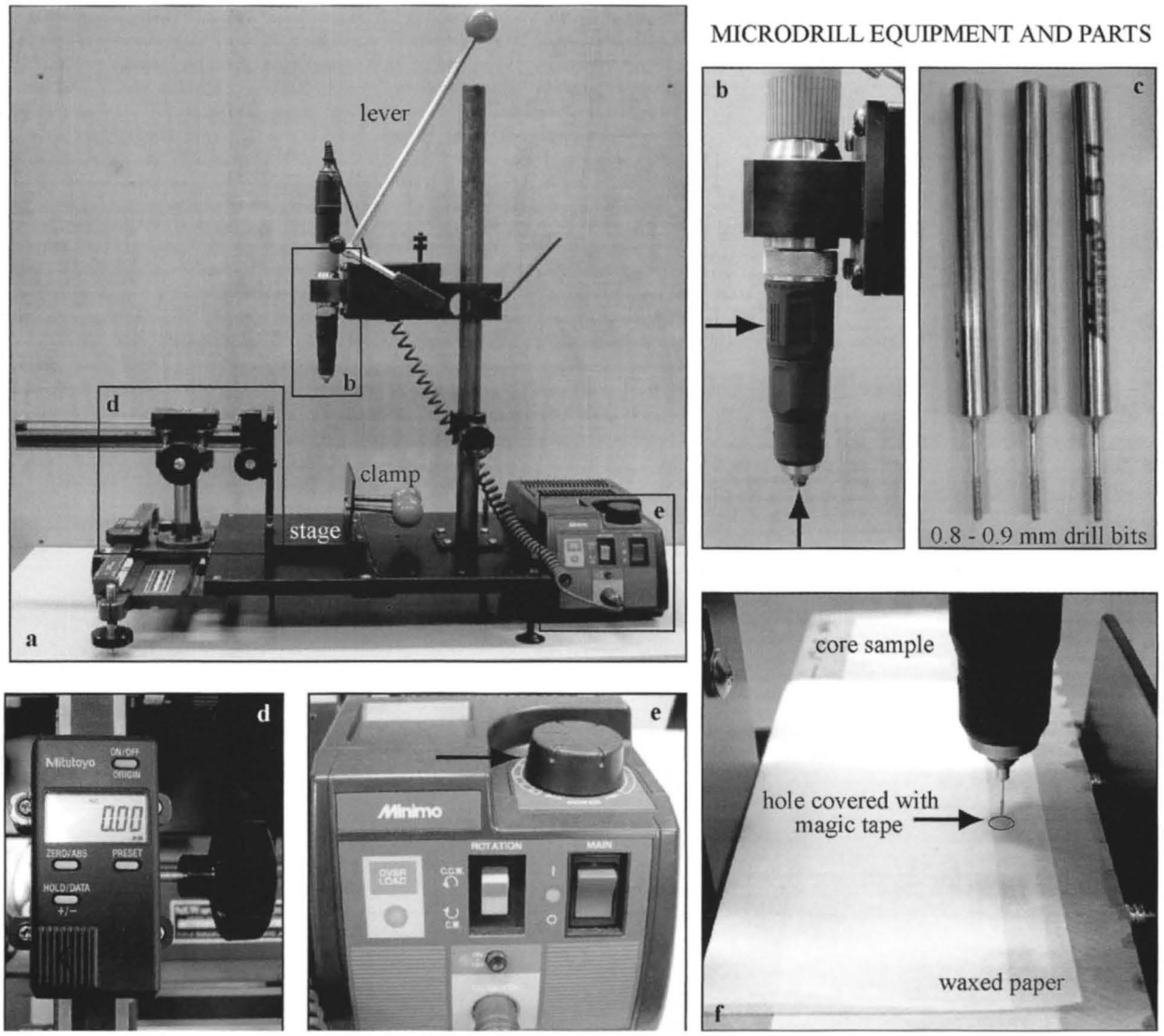

Figure 1: **a**) Microdrill equipment set-up and parts. The sample is placed on the stage/platform and is fixed by a clamp. **b**) Drill apparatus. Vertical arrow indicates the aperture where the drill-bits, (**c**), are inserted, while horizontal arrow indicates the position of the lock to keep the drill-bits in place. Depending on the purpose of the study and amount of sediment needed, drill-bits with diameters of 0.1 to 1.0mm can be used. **d**) One of two digital rulers attached to the microdrill (top view). The digital rulers indicate the position of the drill-bits and are capable of measuring lateral and vertical movements (with respect to the core/sample) at 0.01mm intervals. **e**) Power-source and rotation speed and direction controller. Horizontal arrow indicates the rotation speed controller. **f**) Drilling set-up as described in the methodology section