# A NEW PREPARATION TECHNIQUE FOR CALCAREOUS NANNOFOSSILS FROM ORGANIC-RICH ARGILLACEOUS SEDIMENTS

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## INTRODUCTION

The technique described below was developed to extract nannofossils from the Senonian, organic-rich, micaceous, often phosphatic, silty shale ("Hot Shales") sequences in well sections drilled offshore West Africa. The technique may be applied to similar sediments of different ages with appropriate modifications.

The "Hot Shales" either contain very etched assemblages of calcareous nannofossils, dominated by the solution-resistent species *Micula staurophora*, or are occasionally barren. The assemblages recovered by conventional techniques (smear and short centrifuge as described by Taylor and Hamilton, 1982) contain moderately abundant specimens and show very low species diversity. Age determinations based on these nannofossil assemblages and other microfossils (foraminifera and palynomorphs) are essentially broad. This has prompted research into increasing the nannofossil concentration in order to observe more taxa and thus provide more detailed age determinations. These aims were achieved mainly by removing large proportions of the *non*-nannofossil particles, firstly by sieving (to remove particles >10 μm), secondly by centrifuging (to remove particles <3 μm), and thirdly by floatation (to remove kerogen) and then settling (to remove heavy minerals). As nannofossil concentration increases through these stages, a slide may be prepared after each stage until an age diagnostic assemblage is obtained, thus avoiding unnecessary treatments.

## DISCUSSION

The organic matter in sediments is either soluble in organic solvent (oil and bitumen) or insoluble (kerogen, sensu stricto). Removing soluble organic matter from apparently barren Upper Liassic bituminous rocks has been proved to liberate nannofossils, resulting in rich assemblages being obtained (Herold-Vieuxblé, 1979). The soluble fraction (oil) in the present material is negligible (less than 0.5% by weight) and the removal of this does not appear to increase the abundance of nannofossils, although it produces cleaner specimens. The oil was extracted by a mixture of dichloromethane and methanol (95:5 by volume) in Soxhlet apparatus, which was run for seven hours. This extraction was described in detail by Herold-Vieuxblé (1979). The present technique was carried out on oil-free samples.

Kerogen and heavy minerals are known to have respectively lower and higher densities than calcite (specific gravity 2.715), of which nannofossils are composed, and thus a heavy liquid is needed to separate them. A mixture of tetrabromoethane (TBE) and acetone is the liquid chosen because it is a neutral solution (pH = approximately 7), easy to handle and can be prepared to give the wide range of densities necessary to separate both kerogen and heavy minerals. The density of the liquid varies between approximately 0.8 and 3.0 depending on the proportions of TBE and acetone used, as shown in Fig. 1. In order to determine the optimum densities of the liquid at which

kerogen and heavy minerals could be separated from nannofossils, the abundance of nannofossils (number of specimens in 100 fields of view) was recorded in both the float and sink fractions in the liquid at different densities. The relative abundances (in percentages) were calculated and presented in Fig. 2. Almost all the specimens (mainly *M. staurophora*) float at specific gravity 2.2 and sink at 2.9.

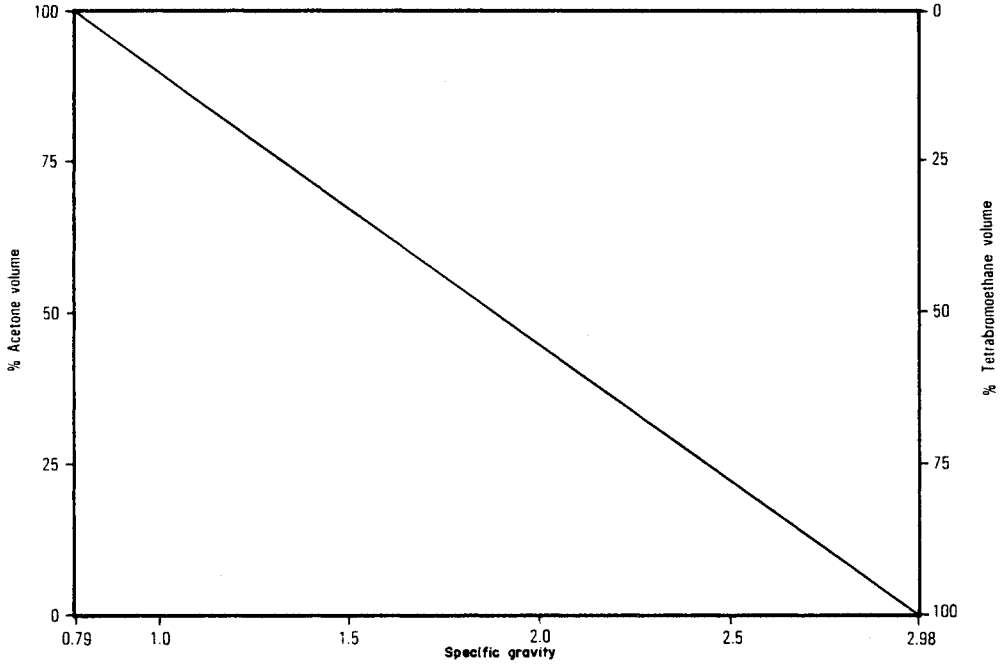


FIG. 1. Diagram showing the range of specific gravity of tetrabromoethane/ acetone at 20°C.

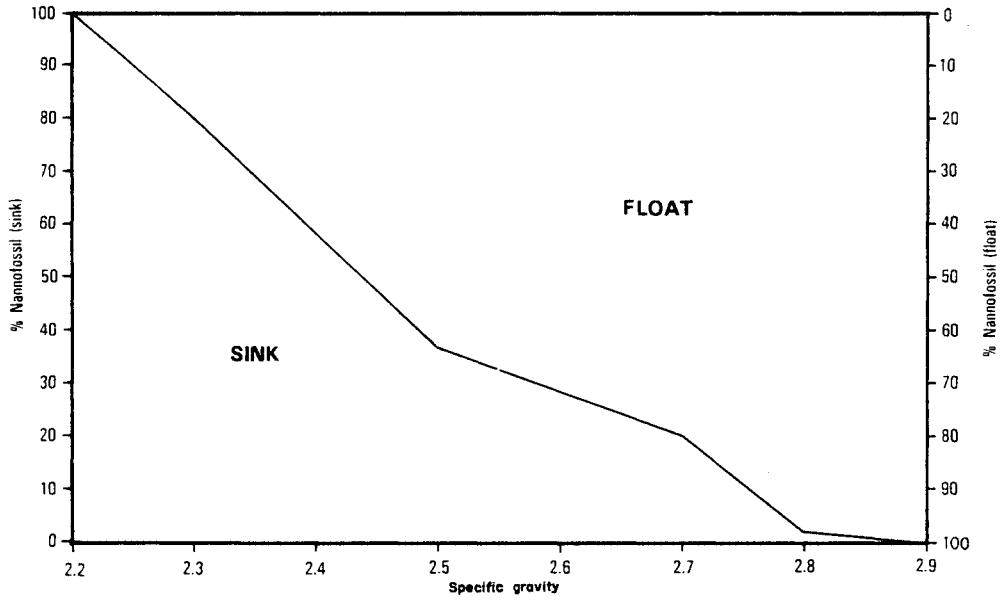


FIG. 2 Diagram showing relative abundances (in percentages) of nannofossils in the float and sink fractions at different densities.

## METHODS

(1) Sieving: about $2cm^3$ of oil-free sample was disintegrated in distilled water overnight, placed in ultrasonic vibration for a few seconds, shaken well and then passed through a 10 µm nylon sieve. Five to ten minutes are usually required to collect enough filtrate. A narrow jet of distilled water may be used to accelerate the process and ensure efficient sieving. The presence of a few 11-13 µm particles was noted in the filtrate, due to the deviation of the sieve's pores from circular shape and/or to pressure applied on the sieve. Considerable quantities of kerogen were removed by this sieving process.

(2) Centrifuging: the filtrate (<10 μm fraction) was centrifuged at 1000 r.p.m. for 104 seconds and decelerated using a brake. The supernatant containing particles <3 μm was discarded and the precipitate retained. The process was repeated on the precipitate until the supernatant was nearly clear. The speed and time are calculated for an Heraeus Christ model Labofuge 6000. The calculations to derive the required times and speeds for particle sizing are outlined in Katz (1978).

(3) Floatation: the dry residue from stage (2) was suspended in TBE/acetone of 2.2 sp. gr. and centrifuged at 3000 r.p.m. for 10 minutes to accelerate the separation. The floating kerogen and most of the TBE/acetone was pipetted off. The residue was resuspended in acetone and then centrifuged at 3000 r.p.m. for 10 minutes to settle the suspension. Acetone was then pipetted off and the residue was dried.

(4) Settling: the dry residue from (3) was suspended in TBE/acetone of 2.9 sp. gr. and centrifuged at high speed as in (3) to accelerate the separation. The float containing nannofossils was collected by a pipette and sufficient acetone was added to lower the specific gravity to at least 2.0. This mixture was shaken well, centrifuged at high speed and the supernatant was decanted off. This treatment on the float fraction was repeated three times to remove the TBE completely. The supernatant in the last stage was pipetted off, and the residue was dried and resuspended in distilled water ready for mounting on slides.

RESULTS

The abundance and species diversity of the nannofloral assemblages recovered by the present technique are considerably greater than that achieved by conventional preparations. The concentration is 4 to 6 times greater and diversity is 1.5 to 5 times higher. Among the age-restricted indicators which were observed only after the present technique had been employed are *Quadrum trifidum*, *Broinsonia parca constricta* and many specimens of the *Arkhangelskiella cymbiformis* group, sufficient for the determination of the group's mean length. This mean length was found to be important stratigraphically (Girgis, 1985).

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