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A gravimetric method for determining suspended matter in sea water using Millipore®* filters

K. BANSE, C. P. FALLS and L. A. HOBSON†
University of Washington, Department of Oceanography, Seattle 5, Washington

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Abstract—A modification of earlier methods for the determination of the weight of suspended matter in 0·5 to 5 l. of sea water is presented. Filter pore size can be selected to meet the requirements of the experiment. The precision is ± 0·15 mg at the 95 per cent level. The material can be subjected to subsequent analysis if the presence of the filter does not interfere.

INTRODUCTION

IN 1950, KREY published a method for determining the weight of suspended matter ('seston') in sea water. Samples of about one litre were filtered on board ship through weighed paper filters of 1·3 μ pore diameter. After filtration, the filters were oven-dried, stored, and weighed in the laboratory. To compensate for changes in weight due to changes in humidity of the air, control filters were treated the same way except that no water had been filtered through them. Paper filters were chosen because of economy and because the pore size was thought to be small enough to retain all living material. Because there is no naturally limited size range of suspended matter in the sea, the choice of pore size is arbitrary and depends on the purpose of the investigation. The precision, ± 0·2 mg, corresponded to about 10 per cent of weight of the suspended matter. Employing a better balance, the precision was later improved to at least ± 0·1 mg (KREY, 1953), but the confidence level was not indicated.

Seston weights from the southwestern North Sea were directly proportional to extinction coefficients found from concurrent beam transmission measurements using red light (JOSEPH, 1953). The slope of the regression line varied for different regions (and in the same region, for different years, BANSE, 1956) because of varying particle sizes. In the regions studied, the regressions crossed the abscissa at values of about − 0·6 to − 1·3 mg/l seston, indicating that this was, on the average, the amount of material passed by the filters, a small part of the total seston. However, paper filters may pass more than 50 per cent of the seston in the open ocean, judging from the size ranges of particles reported by LISITZIN (1961). Because material passed by the filters may be of interest in optical or geological studies,

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a variation of KREY's method employing Millipore® filters is proposed. The use of 'molecular' filters for seston determinations has been suggested by GOLDBERG *et al.* (1952). Probably because of the electrostatic charge on the filters, GOLDBERG *et al.* found that weights of the filters were uncertain by ± 0·2 mg (see also LISITZIN, 1956), a finding in agreement with our results when using an ordinary analytical balance. Because controls must also be weighed when using Millipore® filters, the final precision of seston weights would be about ± 0·6 mg at the 95 per cent level of significance.

We worked out the following procedure for 47 mm HA Millipore® filters (0·45 μ ± 0·02 μ pore size) which we use for chlorophyll determination as well. The error due to electrostatic charges is eliminated by weighing filters over alpha-particle emitting polonium with a CAHN *Gram* Electrobalance*, and a precision of final weights of ± 0·15 mg at the 95 per cent level can be achieved. The procedure differs from that recommended by the Millipore Filter Corporation (Unpublished Manuscripts, 1960 and 1962) for determination of particulate matter in hydraulic fluids and in fuels, by using more than one control filter.

After having worked for ten years with seston, the senior author is convinced after a thorough trial of the method described below involving considerable original work that a report is justified at this time. The results should save much time that might be spent by others devising as accurate a procedure.

PROPOSED PROCEDURE

Factory-cut Millipore® filters of 47 mm, or smaller diameter, are numbered on the margin with a ballpoint pen, soaked for a few minutes in distilled water, put on aluminium foil and dried for one hour at 70–80°C. After cooling in a desiccator, they are weighed, and five filters of each 50 are set aside as controls. Subsequently, the controls are treated exactly like the test filters except that no water is filtered through them.

Before inserting the test filter in the filter holder, a small slip of aluminium foil is placed under the margin of the filter disc to facilitate removal of the wet filter. A known volume of water is filtered with a negative pressure not above 2/3 of an atmosphere (500 mm of mercury) to avoid fragmentation of fragile organisms (G. C. ANDERSON, personal communication). The filter is washed two or three times with 5–10 ml distilled water, the vacuum is released and the funnel removed. Vacuum is applied again and the margin of the filter is then washed two or three times. After releasing the vacuum, the filter is removed. Test filters are then desiccated with the control filters. They can be stored for an indefinite period. Before reweighing, the filters may be dried in an oven as before.

The weight of suspended matter is the difference between initial and final weights of the test filters, plus or minus the average change of weight of the control filters.

REMARKS ON THE PROCEDURE

Weights of 47 mm HA filters vary between 60 and 100 mg but are reasonably constant within a lot. In order to show reproducibility of filter weights, ten filters

*Cahn Instrument Company, Paramount, California. Ainsworth & Sons Inc., Mettler Instrument Corporation, and Sartorius-Werke AG. produce instruments for the same purpose.

were washed, air-dried and weighed for seven successive times (a), and eleven filters were washed, oven-dried and weighed for eleven successive times (b). The mean weights of both oven- and air-dried filters fluctuated as much as 0·6 mg from day to day, presumably due to changes in humidity. The mean initial weights of the two groups of filters were used as controls. The day to day means were subtracted from the initial mean and the differences were added or subtracted to the respective daily individual weights. Thus all corrected weights for each filter could be considered as replicates. Standard deviations were calculated for all filters and were pooled to estimate the standard deviation. The results were : (a) $\pm$ 0·057 mg and (b) $\pm$ 0·054 mg, with a 't' value of 0·46. It is concluded that air- or oven-drying produces the same results, because at the 95 per cent level (a) and (b) must be considered as equal, and weight changes can be accounted for by weighing control filters. To keep the error of the control filter small, five control filters should be used. The final result is the difference between two test filter weights, corrected by the difference between the weight of five control filters. At the 95 per cent level, the probable error of the result is twice $\pm \sqrt{0{\cdot}05^2 + 0{\cdot}05^2 + \frac{0{\cdot}05^2}{5} + \frac{0{\cdot}05^2}{5}}$, or about $\pm$ 0·15 mg. Even in the open ocean, the amount of suspended matter is not much below 1 mg/l, and several litres may be filtered to reduce the percentage error.

Following an initial weight loss of a few tenths of a percent upon soaking in water for two to three minutes, no further weight change occurs upon soaking for one hour, or upon filtering 100 ml of distilled water. A set of six filters was soaked for 16 successive one-minute periods, and another set of six was soaked for 16 successive one-hour periods. Following each soaking period, three of each set were oven-dried, three desiccated and weighed. These treatments did not change the weight when compared with control filters. To test the effect of sea water on the filters, prefiltered sea water was passed for about one minute through eight filters, which were then washed properly with distilled water. A decrease in the mean weight of 0·03 mg $\pm$ 0·02 mg was observed, but this change is not significant, in view of the error in the control weight.

NOTES

1. If the lots of filters vary too much (more than 10 mg for a 47 mm filter), five control filters from each 25 should be used. It is always necessary to weigh the controls before and after the lot has been weighed.

2. Filters should be handled only with flat-bladed forceps without serrated tips. Filters dried on glass or enamel may stick, but aluminium foil is satisfactory. Oven-drying at 70°C is generally the fastest and safest procedure. The choice of oven- or air-drying depends on the material. Drying at higher temperatures makes filters brittle and difficult to handle but 105°C can be used.

3. Filtration on board ship is facilitated by an apparatus similar to that shown by KREY (1950).

4. A crucial part of the procedure is washing the sea salt out of the margin of the filters. A 47 mm filter, even though not fully wet at the margin, and sucked 'dry' in the filter holder, can contain about 5 mg salt. This salt cannot be entirely removed by washing with the funnel in place. Five filters were found to have gained 0·4 mg $\pm$ 0·1 mg dry weight due to salt retention.

5. If the living fraction of the seston is to be analyzed subsequently, or if the plankton is supposed to make up a sizeable part of the seston, lysis may be prevented by washing with isotonic ammonium formate solution instead of distilled water (PARSONS *et al.*, 1961). The ammonium formate is volatilized in two hours at 105°C.

REFERENCES

BANSE, K. (1956) Produktionsbiologische Serienbestimmungen im südlichen Teil der Nordsee im März 1955. *Kieler Meeresforsch.* **12** (2) : 166–179.

GOLDBERG, E. D., BAKER, M. and FOX, D. L. (1952) Microfiltration in oceanographic research. I. Marine sampling with the molecular filter. *J. Mar. Res.* **11** (2) : 194–204.

JOSEPH, J. (1953) Die Trübungsverhältnisse in der südwestlichen Nordsee während der 'Gauss'-Fahrt im Februar/März 1952. *Ber. Dtsch. Komm. Meeresforsch.* **13** (2) : 93–103.

KREY, J. (1950) Eine neue Methode zur quantitativen Bestimmung des Planktons. *Kieler Meeresforsch.* **7** (1) : 58–75.

KREY, J. (1953) Plankton- und Sestonuntersuchungen in der südwestlichen Nordsee auf der Fahrt der 'Gauss' Februar/März 1952. *Ber. Dtsch. Komm. Meeresforsch.* **13** (2) : 136–153.

LISITZIN, A. P. (1956) Methods for collecting and studying suspended matter for geological purposes (In Russian). *Trud. Inst. Okeanol. Akad. Nauk SSSR* **19** : 204–231.

LISITZIN, A. P. (1961) Distribution and content of suspended matter in the waters of the Indian Ocean (In Russian). *Mezhd. Kom. Mezhd. Geofiz. Goda, Prezidiume Akad. Nauk, SSSR, Okeanol. Issled.*, (3) 52–89.

MILLIPORE FILTER CORPORATION, ANON. (Unpubl. Manuscript, 1960) Proposed method of test for the determination of particulate contamination in hydraulic fluids by the *control filter* gravimetric procedure. 4 pp.

MILLIPORE FILTER CORPORATION, ANON. (Unpubl. Manuscript, 1962) A test for the determination of particulate contamination in fuels by the *control filter* gravimetric procedure. 6 pp.

PARSONS, T. R., STEPHENS, K. and STRICKLAND, J. D. H. (1961) On the chemical composition of eleven species of marine phytoplankton. *J. Fish. Res. Bd. Canad.* **18** (6) : 1001–1016.