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*A Device for Collecting In-Situ Samples of Suspended Sediment for Microscopic Analysis*¹

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ABSTRACT

An in-situ sampler for collecting small samples of suspended sediment for microscopic analysis has been built and tested. The device rapidly freezes a thin layer of water entrapping all of the suspended particles in it; when the sampler is recovered, the disc of ice is placed on a suitable substrate and freeze-dried. The particles can then be examined in an undisturbed state with a light microscope or with an electron microscope.

Introduction. A sample of suspended sediment to be analyzed microscopically for size should be an undisturbed sample mounted on a substrate that is suitable for microscopic examination. The ideal sample is a single-particle layer, with no particle touching another. If the particles are in contact or are piled on top of one another it is impossible to assess the degree of agglomeration or to determine the in-situ size distribution.

Suspended-sediment samples have been collected by means of in-situ filtering devices; but, more commonly, suspended particles are removed by shipboard filtration from portions of water samples collected with water bottles. The filtration procedure involves a number of operations, some of which may alter the natural size distribution of the particles. From the time a water bottle closes, the conditions for particle agglomeration are enhanced. Settling increases the concentration of the particles in the lower part of a sample, thereby increasing the frequency of particle collisions and the chance for agglomeration.

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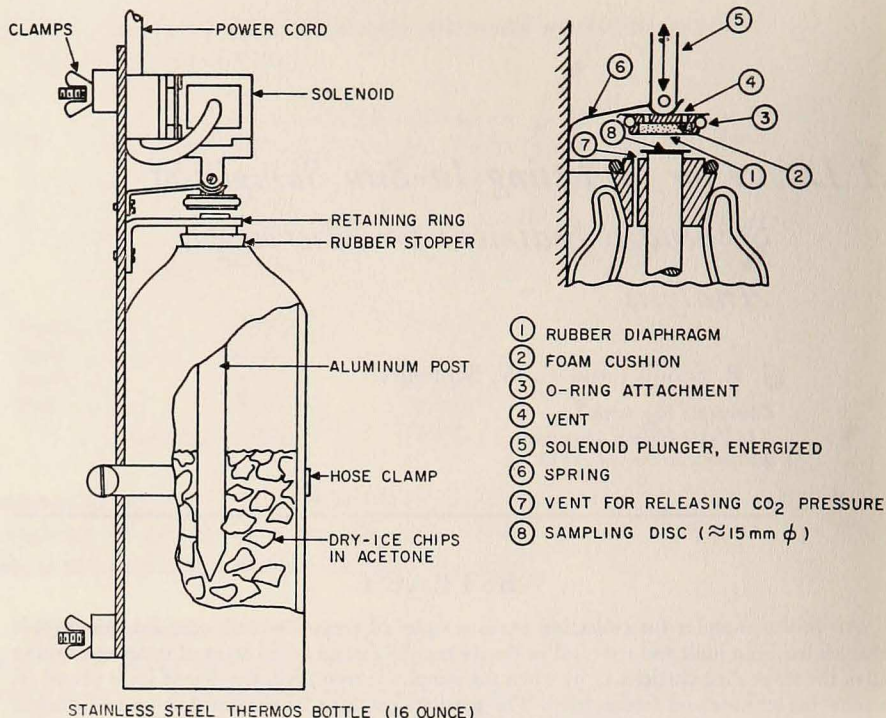


Figure 1. The Sampler.

The sample bottle, when recovered, must be agitated to produce a uniform suspension. While the agitation must be sufficient to resuspend all of the particles and to produce a uniform suspension, it must be gentle enough to prevent the destruction of naturally occurring agglomerates. While in-situ filtering devices eliminate, or at least diminish, these drawbacks, changes in the natural particle-size distribution may also occur during the actual filtration process. Weakly bound composite particles may be produced by agglomeration because of the increase in particle concentration that occurs during filtration. On the other hand, weakly bound agglomerates may be flattened and disrupted when they impinge upon the filter, particularly if the filtration is done under a high vacuum.

The ideal sampler would be an in-situ device that would rapidly and gently remove or isolate all of the particles suspended in a thin layer of water. Then the particle-particle interactions that occur during the sampling process would be minimized.

We have had success in collecting samples of suspended sediment for microscopic analysis with a sampler that rapidly freezes a thin layer of water, entrapping all of its suspended particles. Following recovery of the sampler,



Figure 2. Scanning electron micrographs of samples of suspended matter collected in the upper Chesapeake Bay with the sampler. All samples were collected, freeze-dried, and photographed on silver-membrane filters. Note the good preservation of weakly bound composite particles (agglomerates) and fragile organisms. See also Fig. 3.

the disc of ice is placed on a suitable substrate and freeze-dried. The particles can then be examined with a light microscope or with an electron microscope.

The Sampler. The sampler (Fig. 1) consists of a stainless-steel 16-ounce thermos bottle, a stopper and rod assembly, and a solenoid triggering mechanism for exposing and covering the sample support disc that is attached to the exposed end of the rod.

The thermos bottle is partially filled with a mixture of dry-ice chips and acetone. A thin metal disc, 10–20 mm in diameter, is attached to the aluminum rod with silicone grease, and the rod is inserted into the bottle until the stopper is properly seated. The stopper is vented to prevent CO_2 pressure from building up in the bottle. The triggering mechanism and its supporting bracket are then attached to the thermos bottle with a hose clamp and adjusted so that the retaining ring holds the rubber stopper firmly in place. In this position the rubber diaphragm covers the metal sampling disc. The rubber diaphragm is attached with an O-ring to a vented brass cap that is loosely coupled to the solenoid

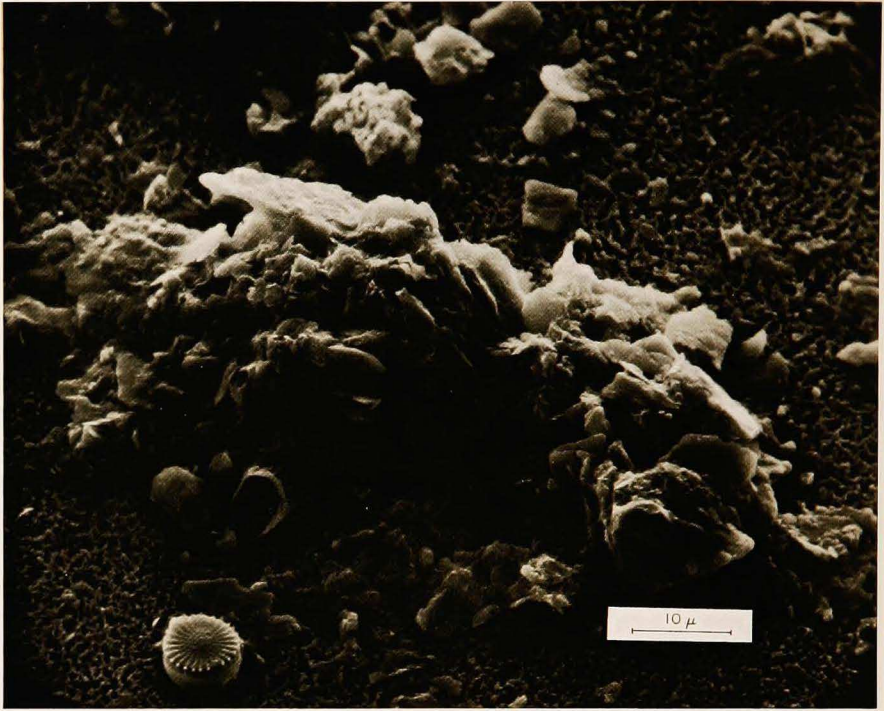


Figure 3. See Fig. 2 for legend.

plunger. The loose connection permits self alignment for sealing. The rubber diaphragm is backed up with an open-cell urethane foam pad that is saturated with anti-freeze to preserve the sealing properties of the cap at rod-end temperatures. Since the foam pad is flooded freely through the vents in the cap, its shape is preserved under pressure.

The sampler, when in use, is attached to a hydrographic wire and lowered to the desired depth. When the solenoid is energized, the rubber diaphragm is raised off the metal disc, now at a temperature of about -70°C , and a thin layer of water is rapidly frozen. The solenoid is then de-energized, and the rubber diaphragm is returned by the cantilever spring to cover the ice and prevent further freezing. The required sampling interval is only about two seconds.

After the sampler is recovered, the ice and its back-up disc are removed and stored in one of a number of individual cups machined into a brass or aluminum plate. The metal plate rests in a tray over a layer of dry ice at the bottom of a small ice chest.

Before the samples to be examined in transmitted light are freeze-dried, the metal support discs are replaced with either Millipore filters or with glass

slides. Samples for scanning electron microscopy are either left on the metal discs or are transferred to Nuclepore filters or glass cover slips. Membrane filters are the most desirable supporting medium for samples collected in salt or brackish water because the salts that remain after the freeze-drying may have to be removed. If the samples are mounted on filters, the salts can be removed either by floating the membranes on distilled water or by gently flooding the filters with a little distilled water and filtering it off under low vacuum.

Extensive examination of samples, with both a light microscope and a scanning electron microscope, suggests that, with these techniques of collecting and preparing a sample, the naturally occurring size distribution of the particles is preserved. An unequivocal evaluation of the technique is, of course, not possible, since there are no accepted standard methods of collecting samples or performing a size analysis of fine-grained particles. Perhaps one of the best indications that our techniques do not destroy the in-situ size distribution is the preservation of delicate organisms and weakly-bound composite particles (Figs. 2, 3). Although we were initially concerned that the freezing would disrupt such particles, experience has shown that agglomerate particles collected in this way are less disturbed than when they are collected by filtration. Another indication that our methods do not destroy the in-situ size distribution of particles is found in comparing microscopic size analyses of samples that were collected with our sampler with similar analyses of samples collected with an in-situ filtering device. A *t*-test for a number of such pairs of samples failed to show any significant differences at the 0.1% level.

The sampler was designed for sampling well-mixed suspensions of fine-grained silt and clay particles, such as those found in Chesapeake Bay and in many other estuaries. To avoid particle selection as a result of an "umbrella" effect of the solenoid mechanism on particles with large settling velocities, the sampler should be used only for populations of particles whose settling velocities are small relative to the horizontal component of the water velocity. In our sampling, the maximum settling velocities of the suspended particles were at least three orders of magnitude smaller than the mean horizontal water velocity.

The sampler is particularly useful in areas where the concentration of suspended sediment is relatively high—greater than about 3 mg/l. With very low concentrations there are not enough particles for meaningful analysis. The number of particles can easily be increased by freezing a disc with a larger area, but the only way to increase the number of particles per unit area on the supporting substrate is to increase the thickness of the layer of ice by increasing the sampling time. This is undesirable, however, because the decrease in the rate of freezing may lead to particle selection.

The sampler can be used in either an upright or an inverted position. For use in a horizontal position, the clamping arrangement must be changed. The sampler has been used successfully in a number of areas in Chesapeake Bay.