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A PRACTICAL MANUAL FOR DETERMINING SETTLING RATES OF OCEAN DISPOSED SEWAGE SLUDGE

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I. INTRODUCTION

The purpose of this manual is to describe a practical approach to the measurement of the size and settling velocity characteristics of sewage sludge particles in seawater. Three measurement sequences are required for complete characterization, but each can be performed separately according to the goals or needs of the user. The first measurement sequence provides percent total solids and some size characterization of the material. This involves standard techniques and the work can be accomplished quickly and easily. The second set of measurements results in a settling velocity distribution of the larger (coarser) fraction of the sludge. These procedures require slightly more time and skill, but they are standard and straightforward methods. The third sequence of measurements is intended to provide a settling velocity distribution for the fine-fraction of sludge particulates. These measurements are not standard and require considerable effort, special apparatus, and the availability of a Coulter Counter or similar electronic particle counter.

The user of this manual must first decide how much information about his samples is needed. The fine-fraction settling velocity distribution will not always be needed because experiments have shown that this fraction tends to settle with velocities less than 1×10^{-3} cm/s. If slowly settling material is of no concern, only the first two measurements sequences need to be considered. If only a basic characterization is required, the user need read only the sample procurement and basic sludge characterization sections of this manual. If the coarse faction settling velocity distribution is required, additional sections of coarse fraction settling should be read. The alternative is to adopt as universal the shape of the coarse fraction settling distributions coming from measurements on four treatment plants described in Lavelle *et al.* (1987) and use the basic sludge characterizations procedures to quantify the masses involved.

If the fine fraction settling is the primary interest, a full experiment, the description of which occupies most of this manual, must be employed. The cause of the complexity is that laboratory procedures must be guided by considerations of how fine sludge particles flocculate and settle in seawater. Two aspects of the experiment must be given special attention: the floccule particle formation stage and the low concentration, flocculation-free settling stage. The reasons for this attention follow.

During ocean discharge, the sludges are continually diluted with seawater. The presence of ions in the water cause small sludge particles to begin forming flocs. While flocs are forming, further mixing of ambient water into the waste plume occurs; this further dilution reduces local particle concentrations, thereby affecting floc formation rates and ultimately the floc size distribution. Laboratory experiments on sludge should attempt to replicate the conditions of floc formation in the field if realistic settling velocities are to be ascertained.

Secondly, once representative flocs are formed their settling velocities must be measured in an environment which prevents further particle flocculation. Continual flocculation during the settling period is suspected in the early experiments on sludges (Brooks, 1956; Myers, 1974; Faisst, 1980). Flocculation during the measurement interval can be suppressed if particle concentrations are kept low ($\leq$10 mg/ℓ). This concentration level is typical of that found in sludge dumping areas several tens of minutes past the time of dump. However, this concentration is much less than is normally suitable for settling experiments. To employ such concentrations in the context of a conventional settling column analysis requires a particle counting device that can measure small volumes of low particle concentrations. This report details the application of a Coulter Counter to

the settling velocity analysis of sludges, but other, similar, instruments could be substituted.

This manual goes through the procedures of analyzing sludges subject to ocean disposal in a step-by-step fashion, detailing procedures developed and tested in the laboratory. The end result of a sludge analysis using these three separate measuring sequences will be the determination of solid content, bulk size fractionation, and settling spectrum of both coarse and fine fractions of sludges as disposed in the ocean. Examples will be taken from our own analysis of sludges from the Hyperion outfall in Los Angeles.

II. SUMMARY OF THE APPROACH AND METHOD

Sludge samples are taken in 1 ℓ plastic bottles into which 1 g of sodium azide is gently mixed. They should be taken as near as possible to the point where sludge enters the discharge pipe or dumping vessel. The samples are then refrigerated (about 4°C), either directly or by being in an insulated chest together with blue ice during shipment to the laboratory. Azide is used to retard growth during shipment and storage; its concentration is limited to prevent flocculation in the samples.

In the laboratory, the samples are split with a Folsom splitter (See Sec. III), one part to be used for bulk analysis and the other for settling. Accurate splitting of the sample is important because rapid settling of large particles may leave them at the bottom of the sample container. The bulk analysis fraction is separated by sieving into four size fractions: >250 μm, 250-125 μm, 125-64 μm, and <63 μm. Gravimetric analysis of each fraction provides a rough size classification of the entire sludge sample.

Coarse Fraction Analysis

The settling fraction is separated into coarse (>63 μm) and fine fractions by a gentle wash through a 63 μm sieve with azide-treated deionized, filtered, distilled water. The suspended fine fraction is then split with the Folsom splitter to obtain a ~100 ml subsample, and that subsample in turn is split into 10 subsamples with a Walker splitter (see Section III). The resulting 10 ml samples of sludge fine fraction in fresh water are then capped and refrigerated to await settling analysis.

Settling analysis of the >63 μm fraction is performed in fresh water using a 1 ℓ glass cylinder. Approximately 200 mg of material is suspended, thoroughly mixed with a perforated disk-type stirring rod (Galehouse, 1971), and sampled by pipette at appropriate times. The resulting aliquots are filtered through preweighed 0.4 μm polycarbonate filters, dried in a desiccator, and the retained mass weighed.

Fine Fraction Analysis

Measurements on the fine fraction are designed to acquire settling velocity spectra for the material in the state that might be found after discharge into the ocean. The sludge is therefore mixed into seawater under increasing dilution and low shear conditions. The fine fraction is introduced to the bottom of a dry settling tube to which a bottom intake port had been inserted. Filtered seawater is introduced at a controlled rate while the contents of the tube are carefully stirred. The dilution rate, controlled by flow meter, is adjusted over a 30 min dilution period so that a concentration of 500 mg/l is reached within 2 min, 100 mg/l is reached in 10 min, and 10 mg/l is reached in 30 min. This dilution schedule is chosen to be analogous to both diffuser and barge discharges (Koh, 1983).

After sample mixing, the settling column is lowered into a water bath to insulate the column from changes in room temperature. Because temperature equilibration of the settling column and the tank takes several hours to achieve, the column should be left overnight in the bath with a magnetic stirrer moving just above stall speed to keep the column mixed. Maintaining a constant temperature bath is critical. Backlighting the settling tube shows that convective current cells can occur in the settling column when the temperature of the bath varies as little as ±0.3°C. Heat exchange through the walls of the settling tube apparently results in temperature gradients between the core and the near-wall fluid within the tube, and the result is convective mixing symmetrically about the tube axis. Accordingly, a temperature controller/circulator with a ±0.02°C range is used in the bath, in conjunction with enough aquarium pumps to thoroughly circulate the water. The bath temperature should be kept slightly higher than maximum room temperature.

Heat flux directly into the upper, open end of the settling tube may also cause some convection. The tube should thus be closed with a cap insulated with styrofoam, through which a pipette sampling hole has been drilled. The foam extends very near the column fluid surface, which is maintained at the same level as the bath fluid, so that no heat flux through the sides of the tube exposed to air can occur. Convective currents are all but eliminated by these means.

The settling experiment for the fine fraction, given this apparatus, proceeds in the conventional way (Krumbein and Pettijohn, 1938). Samples are taken at an initial time and at a schedule that resembles a near doubling time scale up to 8 hours at a column depth of 15 cm. A 24 hour sample is also taken.

The resulting aliquots have a volume of 25 ml and a concentration of ~1 to 10 mg/l. This small volume and low concentration dictates that the particle content be measured by electronic particle counting rather than gravimetry, though optical measurements might also be used for this purpose (Bradley and Krone, 1971; Hunt and Pandya, 1984; Gibbs, 1985). Aliquots are diluted with filtered seawater to a volume of ~175 ml to eliminate counting coincidence and counted with 70 and 200 μm Coulter aperture tubes. To insure good overlap of the size distributions measured, essential to estimating total volume content, samples must be counted on both apertures on the same day. Tests show that the size distribution of unagitated samples stored overnight shift noticeably to the coarse side. Because total volume and not size distribution is the primary interest, samples can be stored overnight as along as the sample is counted on both apertures at times as close together as practical (~4 hours). In fact, the number of samples taken during each settling experiment (~20) will usually prohibit their being all counted on a single day, and some samples will normally be stored under refrigeration overnight.

Total volume concentrations for each sample are calculated for the size range 2.1-80.0 μm diameters after the 70 and 200 μm data are combined. Total volume measurements of the samples are then used to construct a time history of volume loss at the sampling depth. That electronic particle counters can accurately measure total volume in this way is shown in experiments of Sheldon and Parsons (1967). They used quartz samples, measured volume by Coulter Counter and mass gravimetrically, and found that the results were linearly related with a regression coefficient exactly equal to the density of quartz. A similar experiment conducted in our laboratory with glass beads pointed to the same result.

III. APPARATUS, EQUIPMENT AND SPECIAL CONSIDERATIONS

Safety. Digested sludge is toxic and contains pathogenic microbes. To prevent biologically-mediated chemical and physical changes from altering the samples during laboratory storage, the sludge is poisoned with sodium azide which is also poisonous to humans. The azide does not kill all the pathogens but it dramatically reduces metabolic activity and does not detoxify the sludge. Gloves, lab coats, eye protection and respirators should be used; they are available from safety and scientific supply distributors.

Particle Free Water. The "clean" water used in this study was distilled water which had passed through a Millipore, Inc., Milli-Q Water System. Water is sufficiently clean when the particle concentration of blanks as determined by particle counting is acceptable (acceptability of blanks is specified in the Methods section pertaining to Coulter analysis).

Clean Apparatus. All apparatus which comes in contact with a sample must be cleaned and rinsed in a manner such that they do not elevate blanks to unacceptable levels. Normally a hot soapy wash followed by a clean water rinse will suffice.

Filtered Sea Water. Large volumes of filtered sea water are required. A continuous filtration system using a 3.0 µm prefilter and a 0.2 µm secondary filter should produce acceptably clean seawater. A Coulter Electronics Filtration System II was used by our laboratory.

Gravimetric Analyses. Gravimetric determinations are made using preweighed 0.45 µm pore, 47 mm and 142 mm diameter, polycarbonate membranes. Samples are filtered under about 20 inches of vacuum, rinsed thoroughly with clean water, placed in a petrie slide, dried in a desiccator, and reweighed.

Pipettes and Pipetting. Pipettes with large diameter tips are required to pipette sludge coarse fractions. Those recommended have a 2 mm tip I.D.,

10 ml capacity (TD) and are disposable (Falcon 7504). The cotton plug in the top of the pipettes is removed prior to use to avoid particulate contamination. Obtaining a representative sample of a coarse suspension is difficult because of fractionation by sinking in the pipette. Samples must be drawn and transferred quickly to avoid fractionation. Overfilling and ejecting to the mark, or taking a lengthy time to fill exactly to the mark, will lead to erroneous results. A better method is to withdraw less than 10 ml (8-10 ml), read, and transfer.

For pipetting sludge fine fractions, it is necessary to have 20-25 precleaned pipettes or to have convenient facilities for recleaning as the experiment progresses. The settling tube volume dictates to some extent the pipette volume. This study used a 3.5 ℓ settling tube and standard 25 ml class A volumetric pipette (tip opening 0.8-0.9 mm).

Sampling Depth. During the settling rate determinations each sample is withdrawn from exactly the same distance below the water level. The distance should be greater than or equal to 5 cm from the surface and greater than 10 cm from the bottom. It is necessary to mark the pipette stems to indicate the required distance of the tip from the surface.

Noting Exact Volumes. In the procedural text that follows it is often indicated to use "approximately" some volume. This means that if approximately 200 ml is required it is necessary to note the exact volume which presumably will be about 200 ml.

Sample Splitting and Splitters. In several instances it will be necessary to divide a sludge sample into representative subsamples. In general, stirring the sludge, then pouring it into several containers, will not result in representative subsamples.

We recommend the use of a Folsom-type splitter (e.g., Galehouse, 1971) to produce large volume (greater than 50 ml) subsamples. This type of splitter divides one sample into two representative samples while halving the volume of the original sample. To produce smaller volumes it may be necessary to fabricate a small version of the Folsom splitter. During this study a Walker splitter (designed at our laboratory) was used for small volume splitting. It consists of a central chamber mounted above a magnetic stirrer with ten ducts leading from the central chamber to small receptacles. A pressure system holds the sample in the central chamber while it is stirred; switching a valve directs the pressure to the top of the central chamber forcing the suspension through the ducts and into the receptacles.

Sodium Hexametaphosphate Usage. This solution may require filtering prior to use.

Sludge Sieving. A polyethylene pump-pressurized garden sprayer was used to wash large-batch sludges through various sieves to obtain representative size fractions. Lesser size batches were washed with ordinary laboratory squirt bottles.

Water Bath. A conventional rectangular aquarium is sufficient. The bath should be deep enough to fully immerse the settling tubes and voluminous enough to accommodate a water heater and circulator (Fig. 1).

Heater and Auxiliary Circulators. An immersion heater capable of maintaining the bath temperature constant to ±0.02°C is required. Aquarium-type immersible circulators are needed to ensure temperature uniformity throughout the bath. A strip chart recorder should be used to monitor the bath temperature.

Settling Tube. Cast acrylic pipe bonded to ¼" acrylic plate was used to fabricate settling tubes 15" tall with an ID of 4.25". It is usually

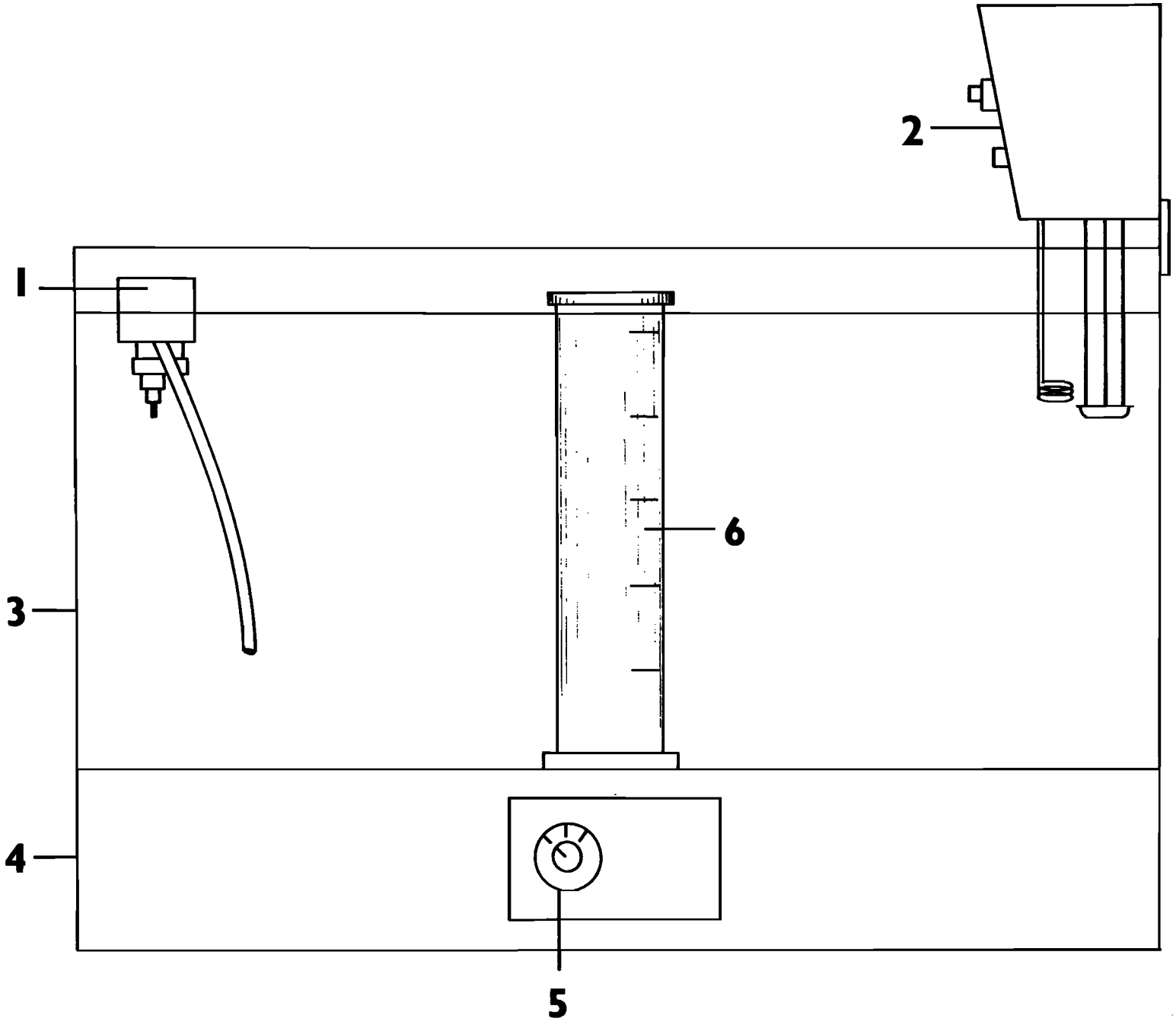

Figure 1.--Schematic drawings of apparatus: (1) Auxilliary aquarium type circulator (2) temperature controller/circulator (3) water bath (4) plywood platform to accommodate (5) magnetic stirrer (6) settling tube.

necessary to accommodate about 3 ℓ to 4 ℓ of suspension. Volume graduations should be inscribed with an indelible marker every 50 ml for the first liter then at 0.5 ℓ increments. The tube should be weighted sufficiently so that it does not become buoyant when the suspension volume is decreased by sample removal.

Pipettor. Any standard pipettor will suffice.

Sample containers. Eight ounce, clear, plastic containers with lids are used during both acquisition and particle counting, thus eliminating the need for sample transfer. The lids were polypropylene with F217 polyethylene (tri-seal) liners. During the early stages of our own work very high particle counts were obtained on blanks. An investigation showed that the cap liners of the sample vessels were the source of the problem. Use of the F217 liners eliminated the problem.

Standard Sieves. Sieves with 250 μm, 125 μm and 63 μm sieve openings are needed.

Electronic particle counter. Particle counting and sizing in our laboratory is performed on a Coulter model ZBI counter and a C-1000 channelizer. A practical description of the use of the Coulter Counter is given by Sheldon and Parsons (1967).

IV. METHODS - PRELIMINARY FINE FRACTION EXPERIMENTS

As a check on the proposed methods and apparatus several standard settling experiments may be performed using single density (ρ = 2.414 g·cm^{-3}) glass spheres (NBS standard reference material 1003a) which have been sieved to obtain sizes ranging from 8 to 38 μm in diameter (NBS beads range from 8 to 58 μm). Since our preliminary tests indicated that the beads clumped after being suspended, they should be sonified (Bransonic 12) for 30 minutes in a

glass beaker containing a 0.5°/₀₀ solution of sodium hexametaphosphate in filtered seawater. An identical solution is used for the settling medium.

Earlier tests also demonstrated that constancy of the water bath temperature was critical when using particles less than 63 μm in diameter because of the existence of convection cells when the bath temperature variability exceeded 0.3°C. To eliminate vertical particle advection, a circulating heater should be used to regulate the bath temperature to ±0.02°C.

Procedures:

- Bring water bath to a temperature slightly above maximum room temperature.
- Clean and rinse all apparatus with particle-free water.
- Weigh out approximately 40 $mg \cdot \ell^{-1}$ of the glass beads and wash through a 38 μm sieve with the hexametaphosphate solution, collecting beads and solution in a clean glass beaker. The wash solution, which should be kept to a final volume of less than 200 ml, is administered easily with a standard laboratory squirt bottle.
- Sonify (~80 watts) the beaker of beads for 30 minutes, making sure that the water level in the sonifier is at about the same level as the water in the beaker.
- Fill a settling tube with the hexametaphosphate solution and withdraw a 25 ml blank. Add the bead suspension, replace settling tube cap, and place in the water bath until the temperature equilibrates (at least 3 hours). Place a flashlight behind center of the settling tube from without the water bath and view the tube into the oncoming light. Do not proceed to the next step until no movement can be seen within the settling tube (temperature equilibrium).

- Stir tube contents ~12 times with a perforated disk stirrer and withdraw a 10 ml sample from a specified depth within 10 seconds after withdrawing stirrer. Use only large tip (2 mm) pipettes. Start a stop watch when stirring is stopped.
- Withdraw additional samples at 1, 2, 3, 4, 5, 10, 15, and 25 minutes after stirring. Take replicates as required.

V. DIGESTED SLUDGE EXPERIMENTS

Sample Procurement

Obtain approximately 1 ℓ of digested sludge (not dewatered) in a suitable clean container containing 1 g of sodium azide. Agitate gently to distribute the azide. Keep sample refrigerated (~4°C) until ready to use.

Basic Sludge Characterization

Several characterization procedures should be performed on each new batch of digested sludge prior to the settling experiments.

Percent Solids in Total Sample:

Several 2 ml aliquots of the sample should be filtered through 0.4 µm preweighed polycarbonate membranes (47 mm diameter), rinsed thoroughly with clean water, dried, and reweighed. Since it is difficult to obtain representative aliquots by pipetting, several replicates should be processed and averaged. For example:

Aliquot	Volume	Net wt (mg) dry	$\bar{x}$ dry wt (mg)	g/ℓ	% solids
1	2 ml	141.8	132.7	66.5	6.65
2	2 ml	123.3			

Size Fractionation:

Approximately 100 ml of digested sludge should be rinsed through a series of sieves (250 μm, 125 μm, 63 μm), each fraction being filtered and weighed. Using this sequence of sieve sizes makes the sieving for coarse material (d>63 μm) easier. The results are, for example:

Size (μm)	Dry wt (mg)	% of total
>250	62.7	5.6
125-250	53.6	4.8
63-125	59.4	5.3
<63	939.0	84.2

Coarse Sludge (>63 μm) Settling Velocity Determination

The coarse sludge is settled separately from the fine material because the settling rates are sufficiently different to require different sampling techniques. A water bath is not required for this determination because the rapidly settling heavy material are not affected by slight fluid motions within the settling tube.

Procedures:

- Use clean water to rinse approximately 500 ml of digested sludge through an 8-inch 63 μm sieve. This sample has been split from the whole sample using the Folsom splitter. Collect the fine fraction in a suitable container (capacity ~4 ℓ).

- Rinse the coarse fraction off the sieve and into a container. Cap and refrigerate until ready to use.
- Add the coarse fraction (about 200 mg is an appropriate amount) to a settling tube nearly full of clean water, fill, and stir with the perforated disk stirrer until well mixed.
- Withdraw samples (about 10 ml each) from a specified depth immediately upon cessation of mixing. Continue sampling at intervals of 10, 20, 30, 40, 50, and 60 seconds, 2, 3, 5, 10, and 15 minutes. Because of rapid sampling during the first minute, practicality requires that the column be stirred before each sample up to and including the 60 second sample. It is a good practice to replicate at least the initial, 60 second, 5 minute and 15 minute samples.
- Filter the samples, dry and weigh. For example:

Los Angeles (Hyperion Pipeline)

Time	Dry wt (mg)	Vol. (ml)	mg/ℓ
0 sec	1.068	9.2	117
0	1.240	9.6	129
10	.997	9.3	107
20	1.024	9.7	106
30	.967	9.3	104
40	.980	9.2	107
50	1.048	9.4	112
60	.963	9.1	106
60	1.012	9.3	109
2 min	.864	9.9	87
3	.748	9.9	76
5	.654	9.7	67
10	.480	9.8	49
15	.360	9.3	39

Fine Fraction Settling Velocity Determination

Addressing the problems of evaluating the settling and dispersion of fine particulates in water bears some special considerations.

Flocculation ensues with slight elevations of dissolved salts (>2-3°/₀₀), increased fluid shear, and increased particle concentrations. The amount of flocculation which occurs at a marine discharge site will be governed to a large extent by these factors. It is with these phenomena in mind that the procedures below are presented for evaluating the settling of the fine (<63 μm) sludge fraction. The propensity for flocculation is inversely related to particle size but the relationship is not clear cut when a particle population is multicompositional, i.e., the surface charge of particles greatly influences the propensity for flocculation.

To approximate oceanic dilution in the laboratory sludge, is diluted to approximately 500 mg/ℓ within 2 minutes, 100 mg/ℓ within 10 minutes, and to 10 mg/ℓ within 30 minutes. It is anticipated that fluid shear is small for most of this initial dilution period and stirring using a magnetic bar at slow speed can be used until the settling tube is about half full (1-1.5 ℓ); continue gentle stirring with a perforated disk type stirrer.

The volume of <63 μm suspension generated from the separation of coarse and fine fractions will be much more than the amount required. To obtain a representative subsample, one or more of the sample splitters (Folsom or Walker splitters) are employed. Since the experimental concentration (filled settling tube concentration) is required to be ~10 mg/1 (35 mg in a 3.5 ℓ settling tube), it is necessary to determine the mass concentration of the suspension prior to reducing its volume. The suspension should also be poisoned with azide prior to reducing to bring azide levels back up to 1 gm/ℓ. The volume of the suspension is then adjusted such that the

subsamples obtained upon splitting are of the appropriate concentrations and volumes. Although final subsample volumes of ~5-10 ml each are recommended, it is somewhat immaterial as long as the dilution rates discussed above can be closely approximated and final concentrations are near 10 mg/ℓ. Subsamples should be capped and refrigerated; after the suspension settles for 4 hrs decant the fraction of clear supernate that represents sieve wash water.

Preparation:

Prior to the outset of the settling determination make the following preparations:

- Fill a five-gallon carboy (Fig. 2) with filtered sea water containing reagent-grade sodium azide (1g/ℓ) and let it flow through the system up to the point where the tubing joins the settling tube inlet. While the water is flowing set the initially required flow rate with the flow meter. Once set, shut the water flow off with a gate valve or pinch clamp without moving the flow meter valve.
- Stabilize the water bath to proper temperature,
- Set the settling tube with a stir bar inside (~2 inch) on a magnetic stirrer and connect the filled dilution supply line to the settling tube inlet.
- Clean and label 20 to 30 sample containers

Seawater Dilution and Settling

Procedures:

- Pour an entire subsample (~10 ml) of the fine sludge suspension slowly and gently to the bottom of the settling tube without undue splashing. Do not rinse the sample container or the settling tube side walls.

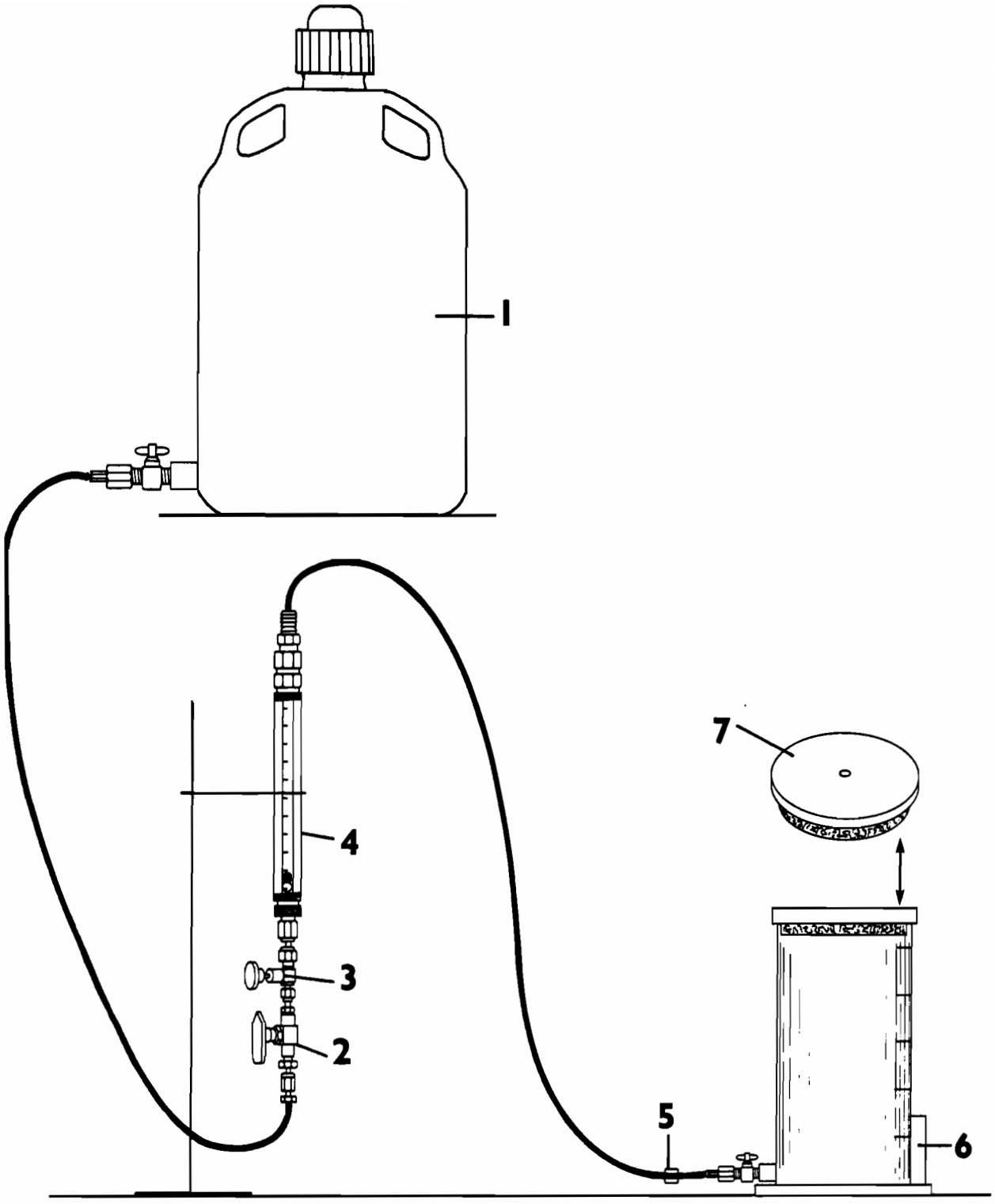

Figure 2.--Schematic drawings of apparatus: (1) seawater reservoir (2) on/off valve (3) metering valve (4) flow meter (5) tubing pinch clamp (6) weight (7) cap drilled for pipette with styrofoam glued to bottom side.

- Open the gate valve or pinch clamp of the dilution line, start a timer, and turn on the magnetic stirrer (to its lowest functional setting). The particulate mass of the initial sample and dilution flow rates are chosen to achieve a final volume of 3.5 ℓ and the scheduled concentrations.
- After 2 minutes adjust the flow meter to the next required setting - the rate required to achieve a concentration of 100 mg/ℓ in 10 minutes total elapsed time.
- After an elapsed time of 10 minutes reset the flow meter to achieve a concentration of 10 mg/ℓ in 30 minutes.
- Turn off the magnetic stirrer when the settling tube is $\frac{1}{3}$ full and begin mixing with the perforated disk stirrer. Move the disk very slowly (1 cycle/5 sec) throughout the water column, being careful not to break the surface.
- The settling tube should fill to the required volume in about 30 minutes. Stop the flow when the required volume is reached. Since flow meters will deviate slightly flow meter settings may have to be reevaluated in subsequent determinations.
- Cap and weight the settling tube and place it into the water bath being careful to keep the tube contents from splashing onto the cap. Leave the settling tube in the bath over a slowly rotating magnetic stirrer overnight (Fig. 1).
- Turn off the magnetic stirrer and gently remix the tube contents with the disk stirrer about 12 times. When the disk is removed draw replicate samples and start a timer or stopwatch. Do not stir the contents after this. Insert and withdraw the pipette with as little lateral motion as possible. Take 10 seconds to insert it and 10 seconds to withdraw it. It is easier to sample the initial (t = 0) through 8-minute samples by

leaving the lid off the settling tube. After the 8-minute sample, replace the lid and insert the pipette through the hole in the cap (Fig. 2).

- A typical sampling schedule is 0, 2, 4, 8, 16, and 32 minutes; 1, 2, 4, 6, 8, and 24 hours.
- Best results are obtained if the capped and labeled samples are kept in a refrigerated room on an oscillating shaker table until they are counted.

Coulter Analysis

Particle volume concentrations of each sample from the fine fraction settling experiments can be measured with a Coulter counter (model ZBI) and a 1000-channel channelizer (model C-1000) equipped with a Log Range Expander or equivalent equipment. Samples are analyzed with 70 µm and 200 µm apertures. The two apertures make it possible to measure total volume concentrations of particles ranging from 2.1 - 80.0 µm diameter.

Seawater for dilution should be filtered until it contains less than 10 counts/0.5 ml when measured with the 70 µm aperture or less than 10 counts/2.0 ml when measured with the 200 µm aperture. Preliminary experiments had determined the volume of sample from the settling column (25 ml) and the amount of dilution (~175 ml) which was needed to (a) achieve a sample with optimum particle numbers for counting (too many particles causes errors due to coincidence; too few particles requires too much time and sample volume to efficiently count); and (b) to increase the sample volume so that it could be counted with two apertures.

Samples were diluted with filtered seawater immediately before being counted with the first aperture and were returned to cold storage while apertures were being changed, instruments re-calibrated, etc.

Attention should be given to the following instrument considerations.

Instrument Calibration. The instruments should be calibrated each time the aperture tube is removed or changed. Calibration procedures and calculations are outlined in the Coulter instrument manuals. Calibration beads should be used to check for and correct instrument drift before counting samples each day.

Blank Samples. Filtered seawater blanks should be run periodically as a check on electronic noise and aperture cleanliness.

Aperture Cleaning and Storage. Clean apertures between use by soaking several hours (or overnight) in Clorox brand bleach (other brands were found to contain impurities which contaminated rather than cleaned the orifice). Rinse the aperture thoroughly with tap water, then clean with a general purpose laboratory detergent (ie. Liquinox) and a soft brush. Rinse thoroughly with tap water then use clean water for the final rinses. Apertures were stored in clean fresh water when not in use. The apertures had to be cleaned frequently during this experiment, possibly due to the highly organic nature of the sludge material being tested.

Coincidence Errors. Coincidence errors are caused when more than one particle is in the orifice of the counter at the same time. Coincidence errors can be kept below 2% when particle number concentrations are less than 5000 counts/2.0 ml with the 200 μm aperture and less than 25,000 counts/0.5 ml for the 70 μm aperture.

Procedures:

- Draw samples from the settling column and place in a container which is suitable for use on the Coulter counter (described above). The containers used in these experiments were large enough to hold enough volume to be counted with both apertures, eliminating the need for subsequent sample transfers.
- Dilute the samples with approximately 175 ml of filtered seawater just before counting with the first aperture. Note that the exact volumes of the undiluted and diluted sample must be recorded for later calculations.
- Place samples on the sample stand and let stand for 30 seconds to remove bubbles. Activate the stirring motor for 1.5 minutes at low speed, then begin the counting sequence. Repeat the counting sequence 6 to 8 times, stirring for 30 seconds between each count. Record the average total count value. Size distributions were accumulated until approximately 2000 counts were stored in the peak channel. Remove the sample and rinse the aperture tube, the external electrode, and stirring paddle with clean water.
- The particle number concentration for the original, undiluted sample (Cn) is calculated by

$$Cn = \frac{\text{Total counts}}{\text{Manometer volume}} \times \frac{\text{Diluted sample volume}}{\text{Original sample volume}}$$

Channel sizes and volumes are calculated by the method described in the Coulter instrument manuals. Size distributions from the two apertures are combined by normalizing each data set to one milliliter then averaging across ten channels in the most closely overlapping size range (9-13 μm diameter). Total volume concentrations are the sum of the

volumes measured in all size channels. All particle diameters are reported as the diameter of a sphere with the equivalent volume, or the equivalent spherical diameter. For example:

Los Angeles (Hyperion Pipeline)

Time	Volume Concentration $(c,^3/L) \times 10^{-3}$
10 s	12.1
20 s	12.5
2 min	12.0
4 min	11.0
8 min	11.4
16 min	12.1
32 min	11.5
1 hr	9.0
1 hr	8.1
2 hr	3.2
4 hr	1.8
6 hr	2.7
8 hr	0.97
24 hr	1.4
26 hr	1.3

Conversion of Concentration vs. Time to Settling Velocities

Total volume concentration determined by Coulter Counter for each sample or from gravimetric analysis should be plotted against elapsed time (Figs. 3 & 4). Smooth monotonically decreasing curves should then be drawn through the data points. In the cases shown in Fig. 4, the curves were drawn based on calculations using the Coulter size distribution measured at t = 0. Particles of each size class were assumed to settle according to Stokes Law with all sizes having uniform, but assumed, density. Volume from individual size classes is lost from the total (initial) volume when that size class can settle the distance of the sampling depth (15 cm) in the indicated elapsed time. The assumed affective density of the particles is chosen to best represent the data (Fig. 3). A hand-drawn smooth curve is also valid (e.g. Fig. 3).

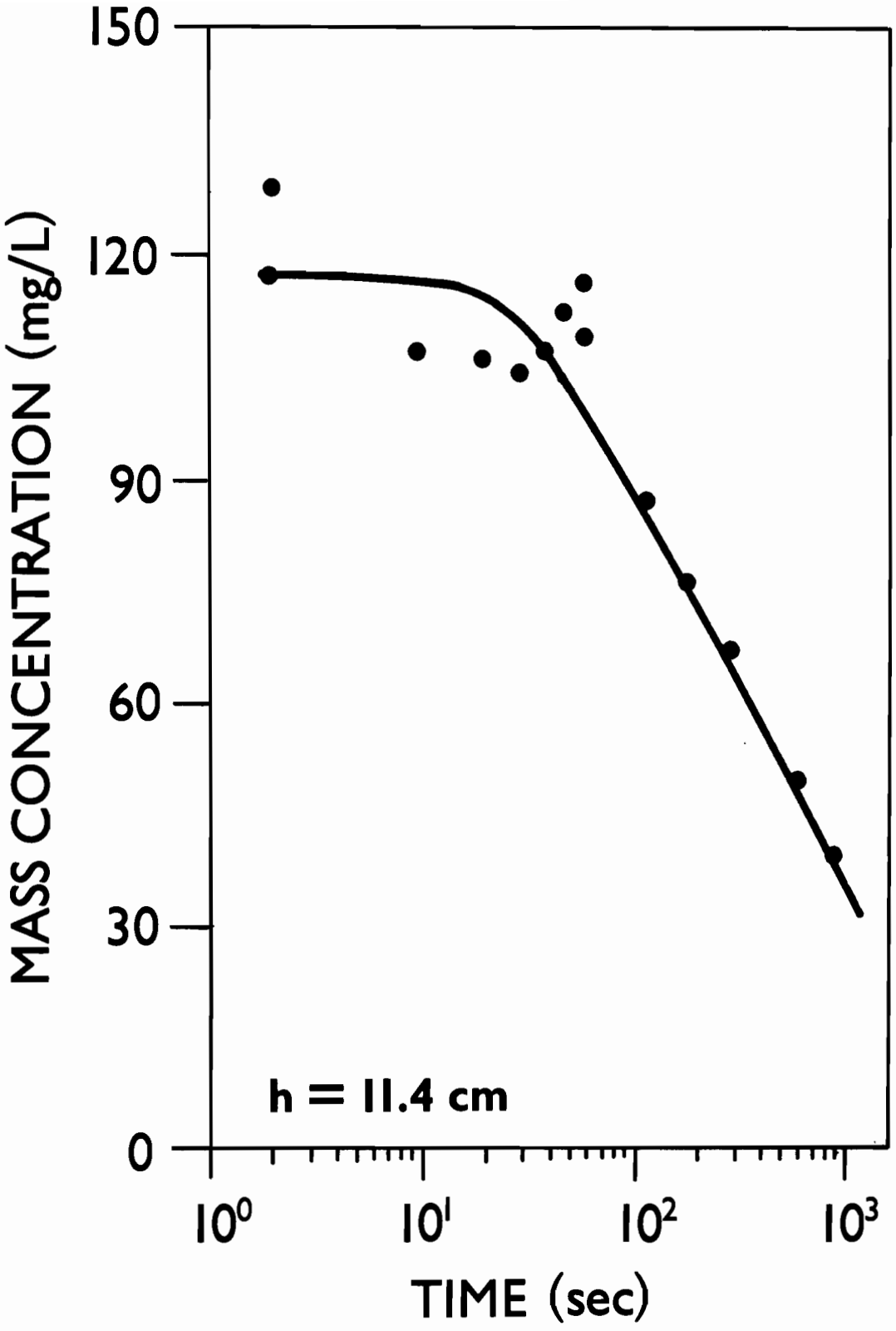

Figure 3.--Changes in mass concentration versus time while settling for the coarse fraction (>63 μm) of sludge from Hyperion.

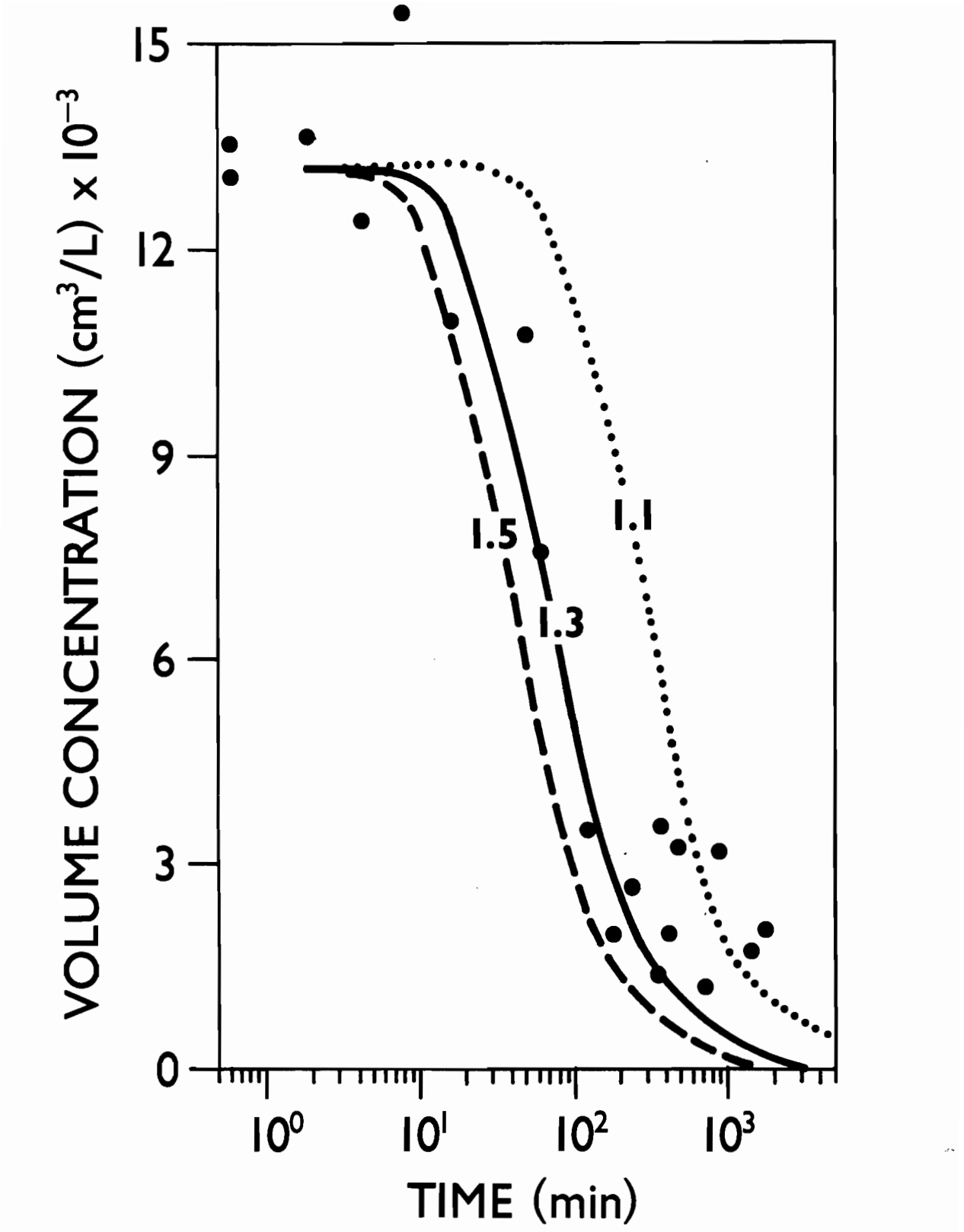

Figure 4.--Changes in volume concentration versus time while settling for the fine fraction (<63 μm) of sludge from Hyperion after flocculation in seawater.

Cumulative settling velocity curves from Lavelle *et al.* (1987) (Figs. 5 & 6) are constructed from the smooth settling loss curves (Figs. 3 & 4) in the following way. The concentration at t = 0 is set at 100% and concentrations at later times are determined in proportions to that value. The settling velocity for each resulting concentration percentage (0-100%) along the curve is found by dividing the settling time into the sampling depth (15 cm). The cumulative settling velocity curve will have the appearance of Figs. 5 & 6.

VI. ACKNOWLEDGMENTS

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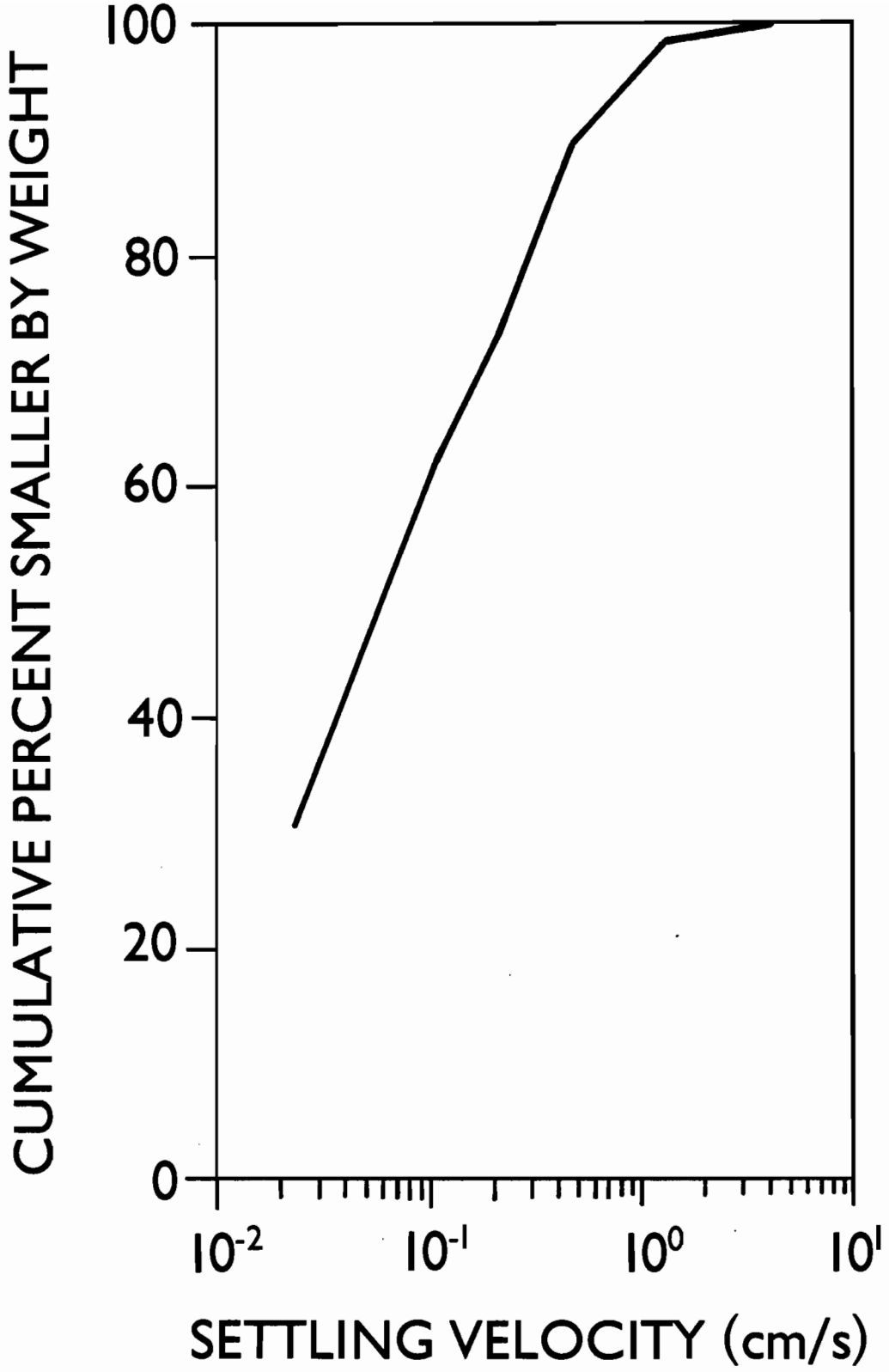

Figure 5.--Cumulative settling velocity curve by mass for the coarse fraction (>63 μm) of sludge from Hyperion.

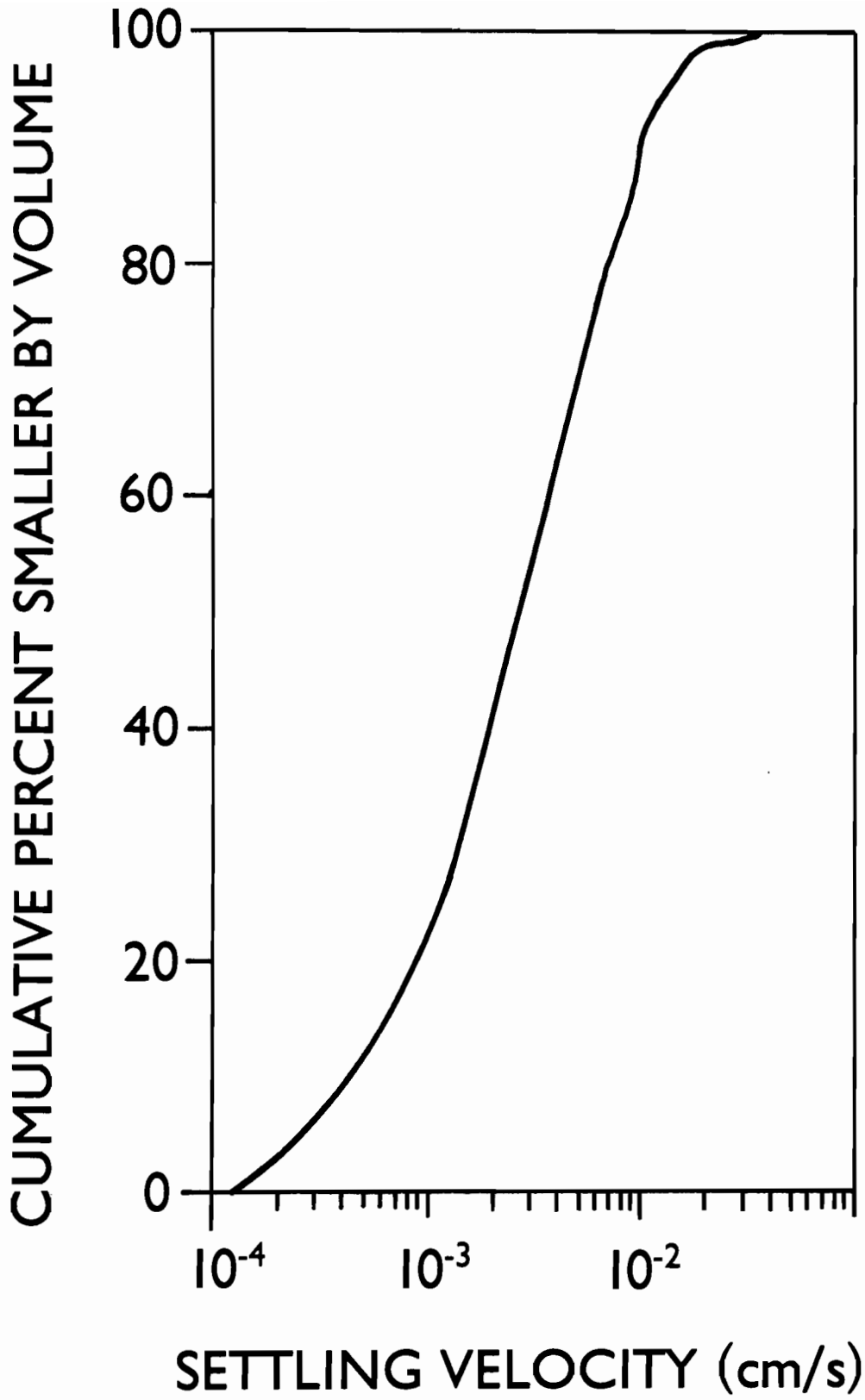

Figure 6.--Cumulative settling velocity curve by volume for the fine fraction (<63 μm) of sludge from Hyperion.

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