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Handbook for Sampling and Sample Preservation of Water and Wastewater

CHAPTER 1

INTRODUCTION

Obtaining representative samples and maintaining their integrity are critical parts of any monitoring or enforcement program. Analytical methods have been standardized but the results of analyses are only as good as the sampling and the sample preservation methods. The purpose of this handbook is to present the best techniques currently available for sampling and sample preservation. The recommendations were developed after an extensive literature review and survey of current laboratory and field practices. The handbook will allow personnel to determine the most effective procedures for their specific applications.

In sampling, the objective is to remove a small portion of an environment that is representative of the entire body. Once the sample is taken, the constituents of the sample must stay in the same condition as when collected. The length of time that these constituents will remain stable is related to their character and the preservation method used.

The sampling technique is determined by the type of water or wastewater to be sampled. This handbook primarily addresses the water and wastewater types shown below and addresses in a limited way, sampling of oceans and estuaries..

1. Municipal wastewaters
2. Industrial wastewaters
3. Surface waters and sediments
4. Agricultural run-off
5. Wastewater sludges
6. Ground water
7. Drinking water

General information on automatic samplers, flow monitoring and statistical methods used to determine number of samples, frequency of sampling, location of sampling, and parameters to be measured are included.

Special consideration is given to sampling for suspended solids, trace organics and radioactive substances.

Since preservation methods relate to the parameters to be analyzed, these techniques are classified by parameter.

CHAPTER 11

SUSPENDED SOLIDS SAMPLING

Suspended solids are a key water quality parameter since they impact such activities as the design of wastewater treatment plants, turbidity removal in drinking water, sediment control in streams, and disinfection. The concentration of other water quality parameters is related to suspended solids, since the solid structure may contain biochemical and chemical oxygen demand materials, trace metals, nutrients, pesticides and toxic or hazardous materials adsorbed on the surface.

11.1 REPRESENTATIVE SAMPLING THEORY

For solids distributed uniformly within a given system and containing the same chemical and physical properties, any sample taken shall be representative. However, most systems in practice contain suspended solids varying in physical and/or chemical properties; in practice, the degree of non-uniformity ranges from slight to large and subsequently causes problems in obtaining a representative sample.

11.1.1 Sampling Error

The error in sampling suspended solids in the field or subsampling from a previously collected sample is attributed to two factors: 1) solid segregation effects; and 2) random distribution of solids:

a) Segregation Effects - Error in sampling due to significant differences between solid particles in specific gravity, size, and shape.
b) Random Solid Distribution - Error due to imperfect sampling or homogenization procedures. For example, a mixture of 1,000 green beads and 5,000 yellow beads, color being the only difference, is homogenized as completely as possible. However, a sample of 24 beads will not always contain four green beads but may vary from zero to eight. The magnitude of this type of error depends on the size of the sample being withdrawn.

Segregation effects are more pronounced in field sampling since solids are difficult to mix throughly or process through devices that eliminate solid segregation. Random effects are more pronounced in the laboratory since segregation effects can be minimized by homogenization of the wastewater sample.

11.2 SEGREGATION SAMPLING ERROR

Typical waters/wastewaters contain solid particles which vary in size, shape, and specific gravity. These properties influence the particle settling rate which must be exceeded to keep the solid suspended and prevent segregation of solids within the water/wastewater system being sampled. The theoretical settling rate of a spherical solid in a quiescent aqueous medium is given by Stokes' Law:

$$V_s = \frac{D^2(S_s - S_w)g}{18\ \nu}$$

Where: V_s = settling velocity

D = sphere diameter

S_s = specific gravity of solid

S_w = specific gravity of water

ν = kinematic viscosity of water

g = acceleration of gravity

11.2.1 Particle Size

Stokes' Law indicates that the settling velocity increases with increasing particle diameter. The size of solids found in water/wastewater varies as shown in Figure 11.1. Approximately 90% of all solids are less than 1 mm in size.

11.2.2 Specific Gravity of Solids

Stokes' Law also indicates that the settling rate increases with increasing specific gravity of the solid. The specific gravity of suspended solids found in waters/wastewaters varies from 0.8 to 3.5, examples are shown below:

Material	Specific Gravity
Oils, other organics	0.95
Flocculated mud particles with 95% water	1.03
Municipal	
a) Effluents	1.15
b) Influent	0.8 - 1.6
c) Grit	1.2 - 1.7
Aluminum Floc	1.18
Iron Floc	1.34
Sand	2.65
Calcium Carbonate Precipitate	2.70

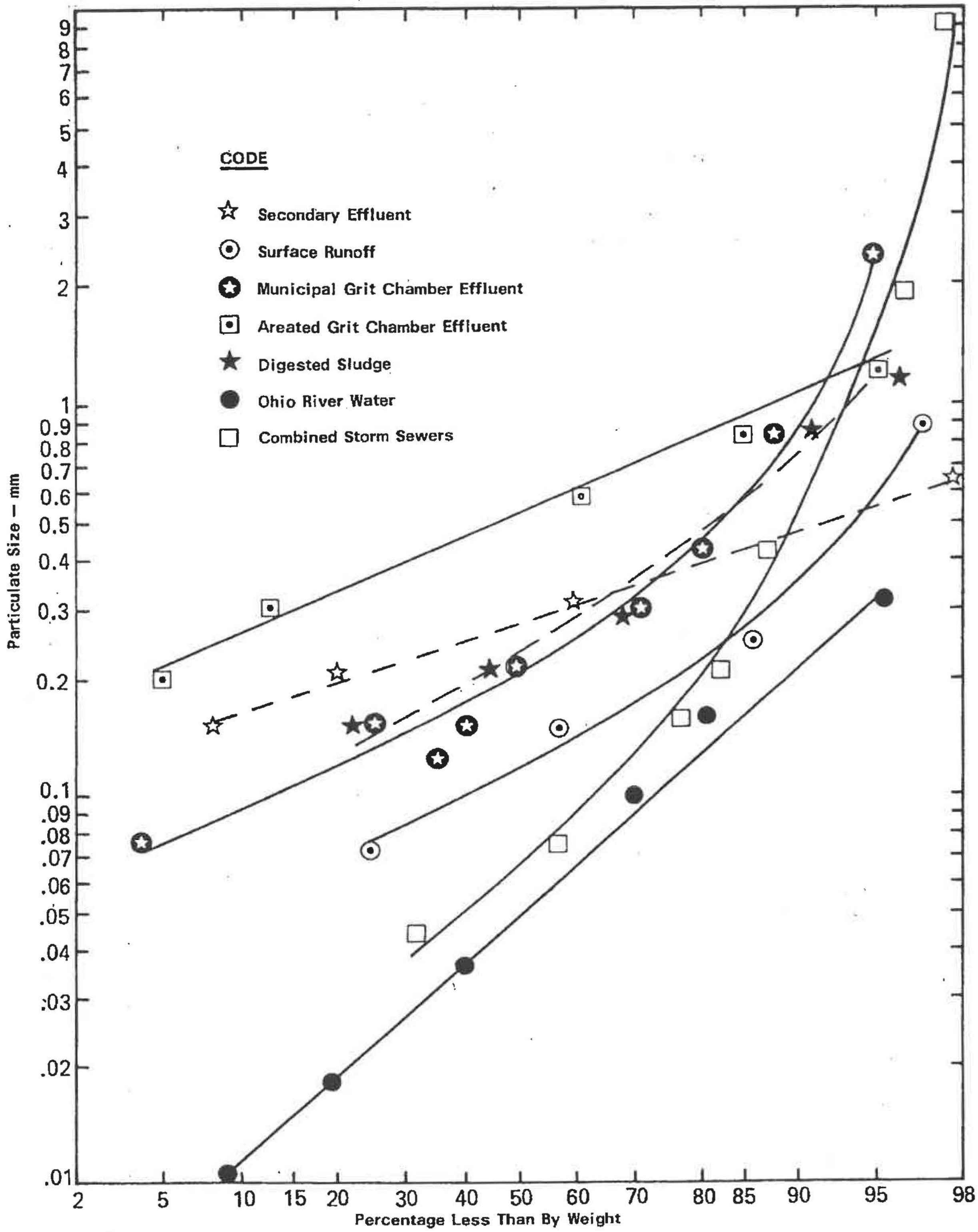


Figure 11.1 Suspended solid particle sizes in various waters/waste waters (1).

11.2.3 Shape of Solids

The settling velocity formula of Stokes applies to spherical particles, however, most waters/wastewaters contain solids of non-spherical shape. In general solids with irregular shapes settle at lower rates than spherical particles of the same specific gravity.(2) Shapes encountered in waters/wastewaters include:

Type	Shape
a) Microbiological and paper scraps	Placoid
b) Sand grains	Angular
c) Plastic monomers	Spherical
d) Fibers - wood, rayon, nylon	Cylindrical-stringy

11.2.4 Settling Velocities

Experimentally determined settling velocities (1) for various solid types are:

a) Erosion soil run-off - Ranges from .015 - 10.1 cm/sec (.0005 - 0.33 ft/sec).
b) Grit chamber effluent - Mean of 0.54 cm/sec (.0017 ft/sec).
c) Primary clarifier design for settable solids removal - .028 - .043 cm/sec (.0009 - .0014 ft/sec).

11.2.5 Scouring Velocity

Sampling of horizontal flowing open channels and pipes for suspended solids must be conducted at velocities which assures adequate mixing. Stratification or segregation of solids are classified as follows:

a) Bed load - Solids that move by saltation, rolling, or sliding along or near the bottom surface.
b) Suspended solids or suspended load - solids that are supported by the upward components of turbulent currents and that they stay in suspension for appreciable amounts of time. The equation for estimating the velocity (3) to transport solids is:

$$V_s = \frac{8B}{f} (g) (S - 1) Dg = \frac{1.486}{n} R^{1/6} B (S - 1) Dg$$

Where:

V_s = Scouring velocity

S = Specific gravity of the particle
Dg = Diameter of particle
B = 0.04 to start scouring and 0.8 for scouring
f = Friction factor - .03 for concrete
n = Manning roughness factor - See Table 11.1

R = Hydraulic Radius - See Table 11.2

g = 32.2 ft/sec^2.

TABLE 11.1 VALUES OF MANNING'S ROUGHNESS COEFFICIENT n

Glass, plastic, machined metal	0.010
Dressed timber, joints flush	0.011
Sawn timber, joints uneven	0.014
Cement plaster	0.011
Concrete, steel troweled	0.012
Concrete, timber forms, unfinished	0.014
Untreated gunite	0.015 - 0.017
Brick work or dressed masonry	0.014
Rubble set in cement	0.017
Earth, smooth, no weeds	0.020
Earth, some stones and weeds	0.025
Natural river channels:	
Clean and straight	0.025 - 0.030
Winding, with pools and shoals	0.033 - 0.040
Very weedy, winding and overgrown	0.074 - 0.150
Clean straight alluvial channels	$0.031d^{1/6}$ d D-75 size in ft.

TABLE 11.2 VALUES OF HYDRAULIC RADIUS R_H FOR VARIOUS CROSS SECTIONS

$$R_H = \frac{\text{area of stream cross section}}{\text{wetted perimeter}}; \quad \text{"equivalent diameter"} = 4R_H$$

Shape of Cross Section	R_H
Pipes and ducts, running full:	
Circle, diam. = D	$\frac{D}{4}$
Annulus, inner diam. = d. outer diam. = D	$\frac{(D - d)}{4}$
Square, side = D	$\frac{D}{4}$

(continued)

TABLE 11.2 (continued)

Shape of Cross Section	R_H
Rectangle, sides a,b	$\frac{ab}{2(a + b)}$
Ellipse, major axis = 2a, minor axis = 2b	$\frac{ab}{K(a + b)*}$
Open channels or partly filled ducts:	
Rectangle, depth = y, width = b	$\frac{by}{b + 2y}$
Semicircle, free surface on a diam. D	$\frac{D}{4}$
Wide shallow stream on flat plate, depth =	y
Triangular trough, = 90^o, bisector vertical, depth = y, slant depth = d	$\frac{d}{4} = \frac{y}{2\sqrt{2}}$
Trapezoid (depth = y, bottom width = b): Side slope 60^o from horizontal	$\frac{yb + y/\sqrt{3}}{b + 4y/\sqrt{3}}$
Slide slope 45^o	$\frac{Y^b + y^2}{b + 2\sqrt{2y}}$

* Values of K. If S = (a - b)/(a + b),

S = 0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
K = 1.010	1.023	1.040	1.064	1.092	1.127	1.168	1.216	1.273

11.3 FIELD SAMPLING

Collection of suspended solids in the field can be performed manually or automatically, however significant differences in results can be expected when sampling non-homogeneous systems such as raw municipal wastewaters as shown in Table 11.3.(4) In addition, automatic samplers with high intake velocities, of 2-10 ft/sec. will capture about one and a half to two times more solids than manual flow proportional or manual grab sampling methods. However, as the system becomes more homogeneous with respect to solids, intake velocities or sampling method becomes less important in obtaining comparable results as indicated by the final effluent values in Table 11.3.

Intake velocities above or below stream velocities for suspended sediment solids (specific gravity 2.65) within Stokes' Law, for example, Reynolds' number less than 1.0, do not result in any significant error as shown in Figure 11.2.(5) However, as the particle size increases, significant error occurs when the intake/stream velocity ratio varies from 1.0. This relationship (Figure 11.3) between the Relative Sampling Rate Ratio as error in concentration has a negative slope. When the intake velocity is less than the stream velocity, more solids will be collected and when the intake velocity exceeds the stream velocity, less solids shall be collected.

The rationale for this inverse relationship is illustrated in Figure 11.4. Therefore, in order to insure representative sampling, the intake/stream velocity ratio should be unity (isokinetic flow).

TABLE 11.3 RICHARDS-GEBAUR SEWAGE TREATMENT PLANT NON-FILTERED SOLIDS COMPARISON RATIO OF SAMPLING METHOD VALUE TO MANUAL FLOW VALUE

Station	Sample Method	Date May 21	May 22	May 23	Average	Intake Velocity ft/sec.
Influent	QCEC	2.099	1.155	1.755	1.669	2-5
	ISCO	0.991	0.431	1.046	0.942	2
	Manual Flow	1.0	1.0	2.0	1.0	--
	Manual Grab	1.223	0.697	0.820	0.907	--
Primary Effluent	Hants	3.141	1.537	1.449	2.042	2.5
	Sigmamotor	0.783	0.700	0.968	0.817	0.25
	Manual Flow	1.0	1.0	1.0	1.0	--
	Manual Grab	0.981	0.975	1.170	1.042	--
Final Effluent	Hants	1.354	0.743	1.387	1.161	2.5
	Brailsford	0.822	0.769	1.225	0.939	.02
	Manual Flow	1.0	1.0	1.0	1.0	--
	Manual Grab	0.951	0.794	1.209	0.985	--

11.4 LABORATORY SUBSAMPLING

Subsampling from previously collected field samples may be subject to error resulting from segregation effects, such as particle size and specific gravity. As shown in Figure 11.5, the shake and pour technique achieves 93% recovery of solids with specific gravities in the range of 2.2-2.6 and particle sizes less than 50 microns; magnetic stirring improves percent recoveries.

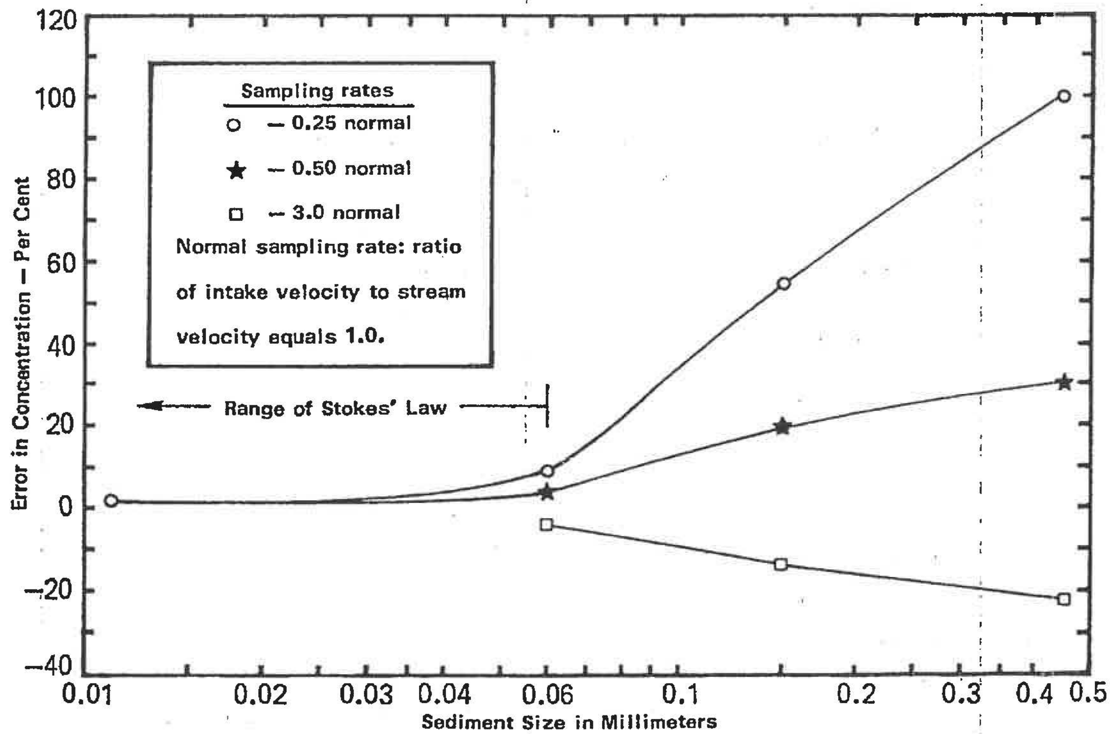


Figure 11.2 Relation of sediment size to errors in sediment concentration.

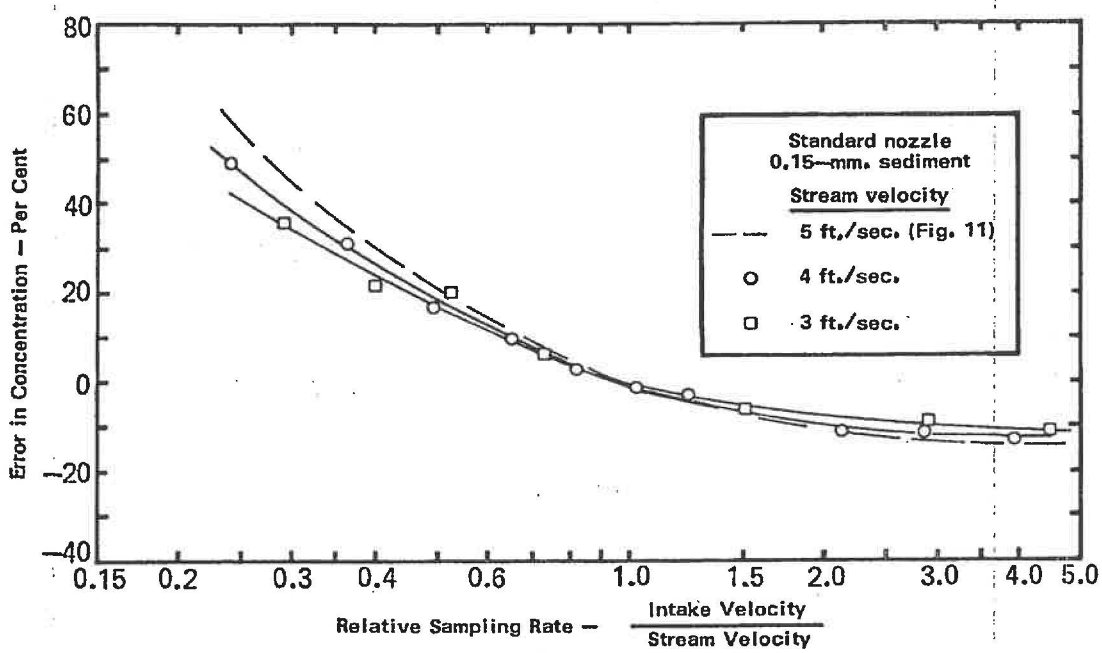
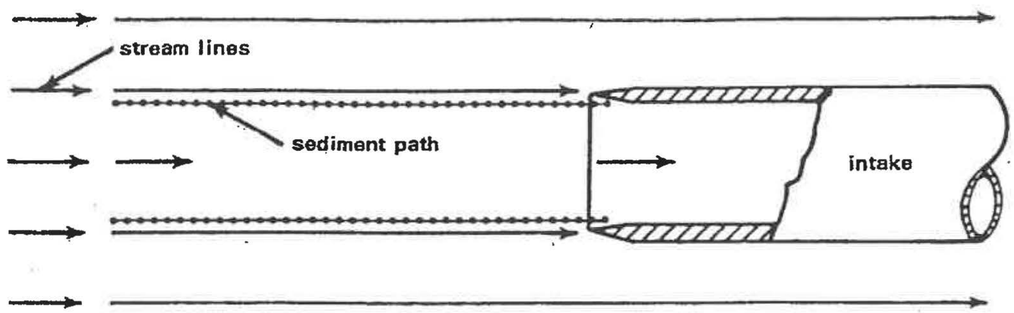
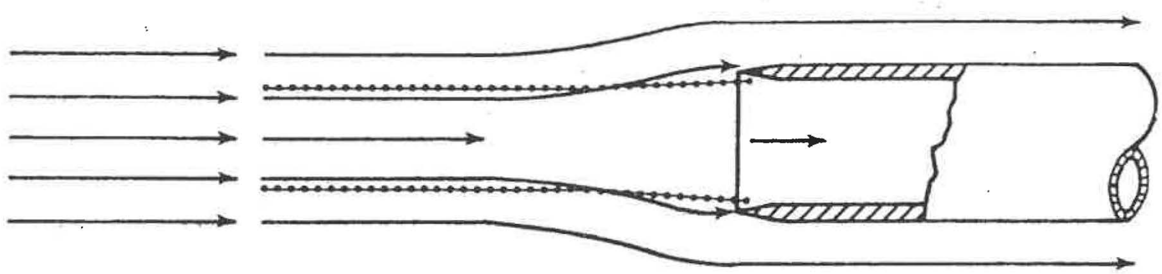


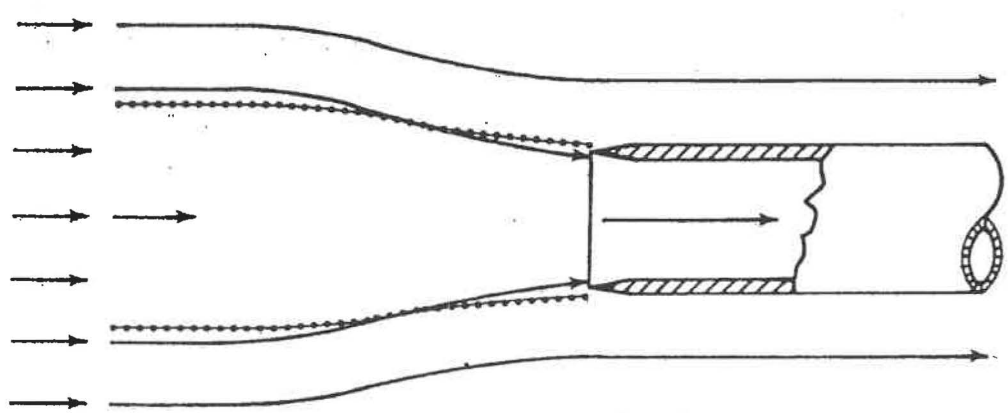
Figure 11.3 Effect of stream velocity on errors in sediment concentration.



a. Normal sampling rate — intake velocity equal to stream velocity.



b. Sampling rate below normal — as illustrated, ratio of intake velocity to stream velocity approximately 1/3.



c. Sampling rate above normal — as illustrated, ratio of intake velocity approximately 3.

Figure 11. 4 Flow patterns at mouth of sampler intake.

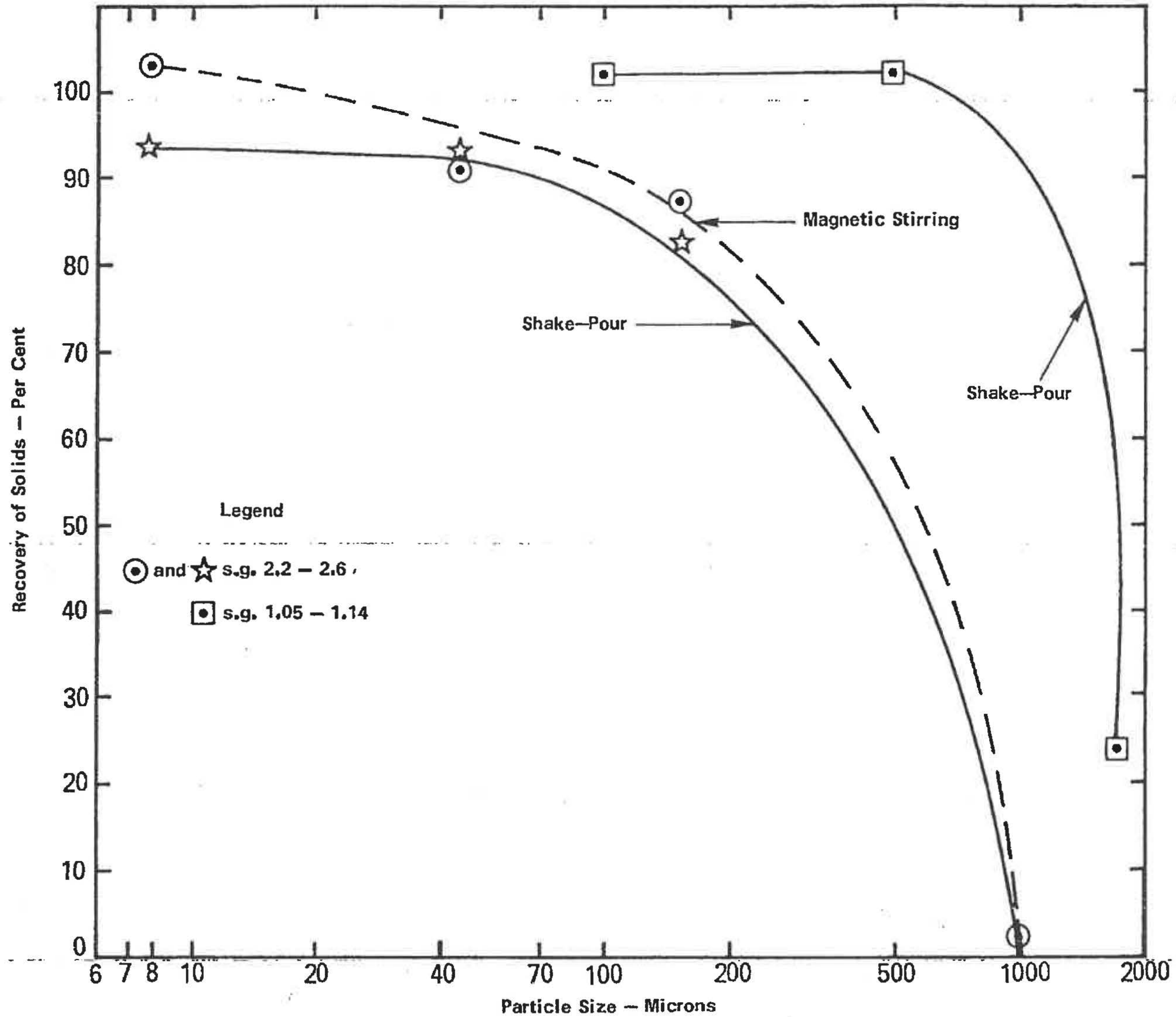


Figure 11.5 Percent recovery vs particle size during subsampling with different mixing techniques

Subsampling recoveries of 100% for solids having specific gravities ranging from 1.05-1.14 can be expected up to 500 microns. Therefore, to insure representative subsampling, the entire sample should be thoroughly blended and as large an aliquot used as possible.

11.5 GUIDELINES FOR SAMPLING OF SUSPENDED SOLIDS

Minimize sampling errors caused by segregation effects by sampling in a well mixed or turbulent zone.

Minimize random sampling errors in the laboratory by homogenizing the sample and using as large a sample aliquot as possible.

Maintain the flow rate in the sample lines to effectively transport suspended solids. For horizontal runs, the velocity must exceed the scouring velocity and in vertical runs, the velocity must exceed the settling velocity of the particle.

For solids falling within the range of Stokes' Law, consistant representative samples can be obtained at intake/stream ratio either greater or less than 1.0. For solids falling outside Stokes' Law, an intake/stream ratio of 1.0 is recommended.

The geometry of the intake has little effect upon the representativeness of the sample, however, the intake should face into the stream at no more than 20 degrees from the direction of stream flow.

11.6 REFERENCES

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4. Harris, D.J., W.J. Keffer. Wastewater Sampling Methodologies and Flow Measurement Techniques. EPA 907/9-74-005, 1974.

5. Interagency Committee on Water Resources, A Study of Methods Used in the Measurement of Analysis of Sediment Loads in Streams: Laboratory Investigation of Suspended Sediment Samplers, Report No. 5, 1940.