EVALUATION OF SELECTED INFORMATION ON SPLITTING DEVICES FOR WATER SAMPLES

By Paul D. Capel and Steven J. Larson

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Conversion Factors

Multiply	By	To obtain
gram (g)	0.03527	ounce, avoirdupois
gram (g)	0.002205	pound, avoirdupois
liter (L)	0.2642	gallon
millimeter (mm)	0.03937	inch

Temperature is given in degrees Celsius (°C), which can be converted to degrees Fahrenheit (°F) by the following equation:

$$°F=1.8(°C)+32$$

Vertical Datum

Sea Level: In this report, "sea level" refers to the National Geodetic Vertical Datum of 1929 (NGVD of 1929)—a geodetic datum derived from a general adjustment of the first-order level nets of both the United States and Canada, formerly called Sea Level Datum of 1929.

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EVALUATION OF SELECTED INFORMATION ON SPLITTING DEVICES FOR WATER SAMPLES

By Paul D. Capel *and* Steven J. Larson

Abstract

Four devices for splitting water samples into representative aliquots are used by the U.S. Geological Survey's Water Resources Division. A thorough evaluation of these devices (14-liter churn, 8-liter churn, plastic cone, and Teflon cone) encompasses a wide variety of concerns, based on both chemical and physical considerations. This report surveys the existing data (as of April 1994) on cleaning efficiency and splitting capability of these devices and presents the data in a systematic framework for evaluation. From the existing data, some of these concerns are adequately or partially addressed, but the majority of concerns could not be addressed because of the lack of data. In general, the existing cleaning and transport protocols are adequate at the milligram per liter level, but the adequacy is largely unknown for trace elements and organic chemicals at lower concentrations. The existing data indicate that better results are obtained when the splitters are cleaned in the laboratory rather than in the field. Two conclusions that can be reached on the splitting capability of solids are that more work must be done with all four devices to characterize and quantify their limitations and range of usefulness, and that the 14-liter churn (and by association, the 8-liter churn) is not useful in obtaining representative splits of sand-sized particles.

INTRODUCTION

During the last few decades, as the number of analytes has increased and detection limits have decreased, water-quality studies have become increasingly sophisticated. These advancements have resulted in the introduction of new field sample processing devices to accommodate the needs of field and laboratory personnel. Due to chemical constraints (loss or contamination) of some analytes and the logistical needs of multiple laboratories, numerous sample bottles (perhaps as many as 20) are often required for water quality analyses at a sampling site. To accommodate the large number of samplings, a series of devices for splitting water samples into representative aliquots has been devised and incorporated into routine use.

Early attempts at sample splitting (before 1976) called for compositing the water sample into a large, clean, jug or bottle, shaking it to assure uniform mixing, then withdrawing the required number of water samples (Office of Water Branch technical memorandum 76.17, appendix A). In 1976, the 14-L churn splitter (fig. 1) was introduced "to facilitate the withdrawal of a representative subsample of a water sediment mixture...Theoretically, any subsample withdrawn from the churn should be equal in chemical quality and sediment concentration to any other subsample from the churn" (Office of Water Branch technical memorandum 78.03, appendix A). By 1978, an 8-L churn splitter was introduced to accommodate smaller water samples. Both of these devices are made out of plastics (polyethylene and polypropylene). In 1979, the need arose for a splitting device that could be used in an automatic sampler for small volume water samples. This device, termed the cone splitter (fig. 2), was designed by the U.S. Geological Survey and introduced for wide scale use in 1980. The original cone splitter was constructed from Lucite and they continued to be made from plastic materials. In general, the churn splitters have found widespread use within the Water Resources Division of the U.S. Geological Survey. Use of the cone splitter has been more limited, but has filled important niches in the needs for water sample processing. Since both types of splitting devices were constructed of plastic, they

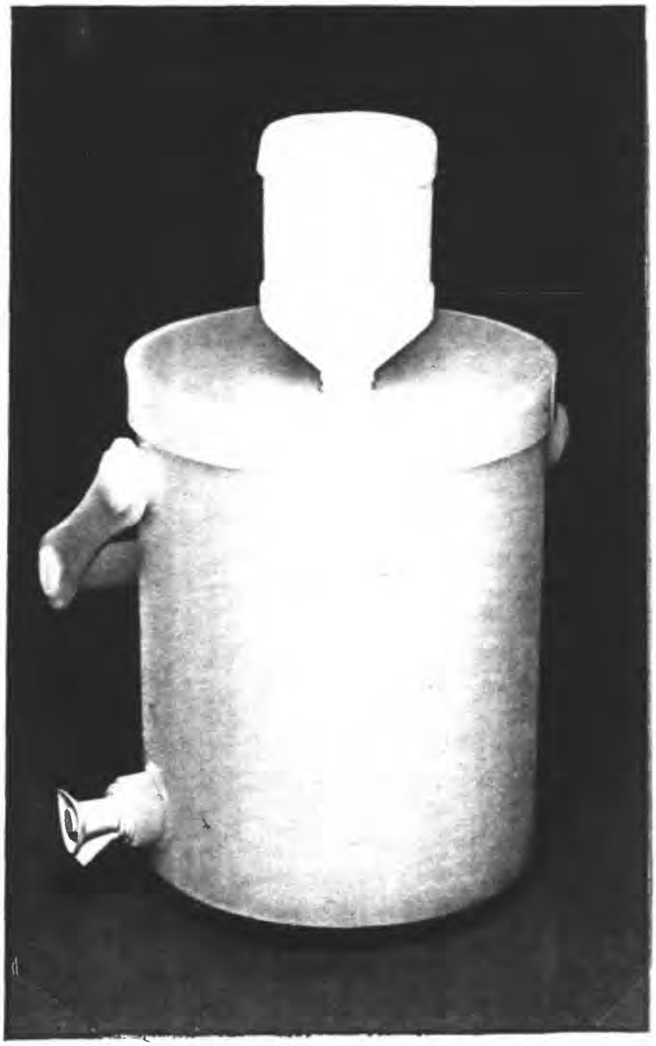

Figure 1. Churn splitter.

were not appropriate for use with water samples that were to be analyzed for organic chemicals. In the mid-1980's, the requirement to split water samples for analysis of trace-level organic chemicals created a need to develop a cone splitter constructed of Teflon. This device was immediately incorporated into many sample processing protocols for organic chemicals. Many variations of these splitting devices have been custom made (e.g., stainless steel cone splitters, Teflon-coated stainless steel churn splitters, and larger churn splitters), but these devices have not been made available commercially and have not been widely used within the WRD. This report, the product of a study undertaken in support of water-quality data collection activities of the U.S. Geological Survey's Office of Water Quality (OWQ) and National Water Quality Assessment Program (NAWQA), considers the four most common splitting devices used within the WRD: 14-L churn, 8-L churn, plastic cone, and Teflon cone.

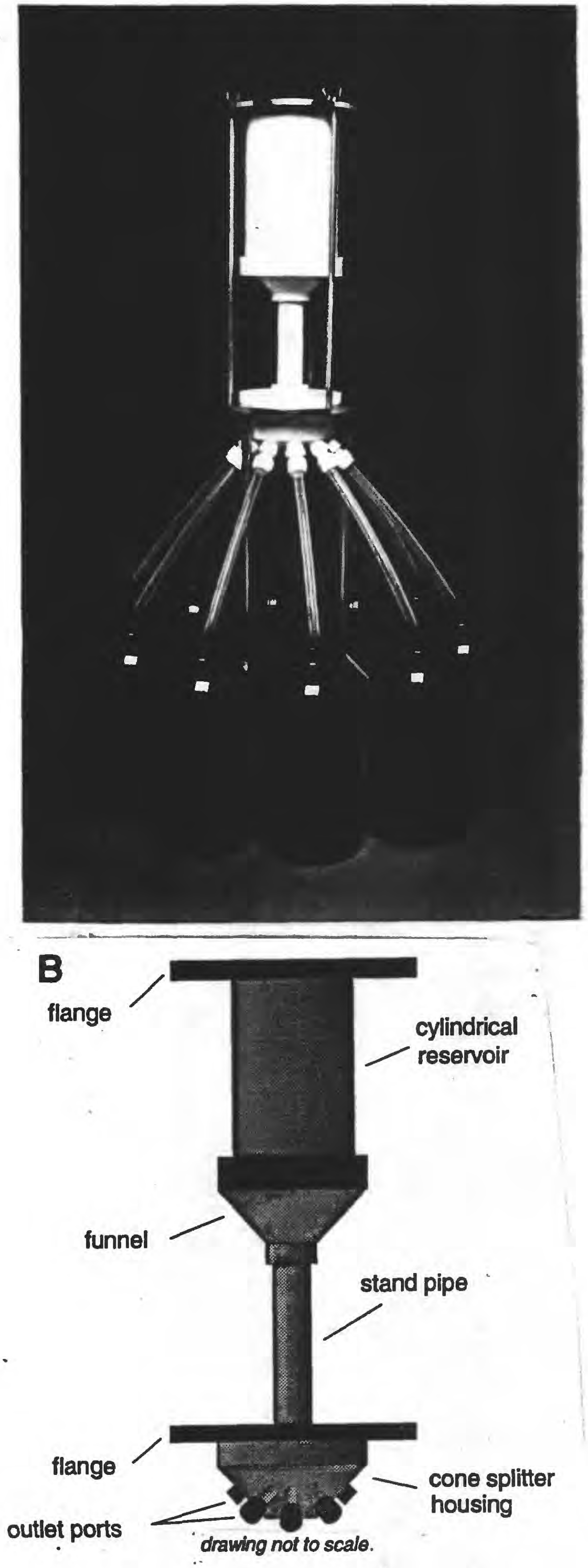

Figure 2. (A), Cone splitter; (B), diagram of cone splitter.

Scope and Purpose

This report surveys the available existing knowledge (as of April 1994) on the cleaning efficiency and splitting capabilities of four devices used for splitting composited water samples: 14-L churn splitter, 8-L churn splitter, plastic-cone splitter, and Teflon-cone splitter. The report fulfills three objectives. First, it provides a reference for those using these devices in their water quality studies. Second, it provides a basis for decision making on the limitations of and continued use of these devices. Third, it serves as a long-term planning guide for additional tests needed for further evaluation of these devices.

Data Sources

The data on which this report is based have been compiled from many sources. Technical memoranda from the OWQ, its predecessor the Quality of Water Branch (QWB), and the Office of Surface Water have been perused (these memoranda are reproduced in appendix A). Published and unpublished data and reports from a variety of sources have also been examined. Unpublished data that have been used in this report are summarized in appendix B. There are additional data on splitting devices that were not available for inclusion in this report.

USES AND LIMITATIONS OF SPLITTERS

The WRD policy on the use and limitations of the splitting devices has been published and printed in numerous protocols and reports (Knapton, 1985; Ward and Harr, 1990), but has most of its origins in the early technical memoranda from the QWB and OWQ. Perhaps the most current and extensive listing of the uses and limitations of splitting devices can be found in Ward and Harr (1990).

From the perspective of Federal WRD programs, the National Stream Accounting Network uses the churn splitter in its standard water sample processing (Ward and Harr, 1990; Horowitz and others, 1994), whereas NAWQA investigators use the Teflon cone splitter exclusively, or as the first step in series with the churn (Shelton, 1994). In 1994, NAWQA added a two–millimeter Teflon screen at the bottom of the reservoir of the cone to aid in distributing the coarse-grained particles and to prevent large debris, such as leaves and twigs, from entering the cone chamber. All of the tests for the Teflon cone splitter cited in this report were conducted without the use of such a screen. The traditional WRD method of suspended sediment collection uses neither splitting device, but collects water samples directly into the sample bottle (Edwards and Glysson, 1988).

SUMMARY OF EXISTING DATA

Tables 1 and 2 summarize the existing data on the adequacy of cleaning protocols and on the splitting capability of the four splitting devices. These matrices describe the complete set of data that should exist ideally, and indicate what types of tests still need to be performed for a full evaluation of the devices. Table 1 focuses on the cleaning efficiency, as described in the current protocols (Ward and Harr, 1990; Horowitz and others, 1994; Shelton, 1994) for the devices. Table 2 focuses on the splitting capability of the devices. Each entry in the tables contains information on one of the four splitting devices. The entries are completed in the following manner. An entry indicated by "n/a" signifies that the information is not needed or that the splitting device inherently cannot be used for this type of analyte. An entry indicated by "--" signifies that no information exists, often because the device cannot be used for the specified purpose, and that results from existing studies cannot be extrapolated to provide the information. The values (3,2,1,or 0) signify the authors' level of confidence in the conclusions from the data considered in this study. A value of "3" signifies a high level of confidence based on agreement of multiple studies. A value of "2" signifies a weaker confidence level based only on one well planned and conducted study. A value of "1" signifies minimal confidence based on one partial study, or on disagreement between two or more studies. A value of "0" signifies that no actual data exist, but that the authors feel that other data can be extrapolated to cover the case being considered. The values are assigned either a "+" or a "–", signifying either a positive result for the splitting device (cleaning procedure is adequate or splitting capability is effective) or a negative result (cleaning procedure is inadequate or splitting capability is ineffective).

Table 1. Evaluation of the effectiveness of cleaning and transport protocols for splitting devices

[Explanation of table entries: n/a - information not needed or the device inherently cannot be used for this type of analyte. "--" indicates that no information exists and cannot be extrapolated. Numbers indicate authors' level of confidence in conclusions from existing data: 3 - high level, based on agreement of multiple studies; 2 - medium level, based on one well-planned study; 1 - minimal level, based on one partial study or disagreement between multiple studies; 0 - no actual data exist, but other existing data may be extrapolated to cover case being considered. "+" or "-" indicates whether conclusions drawn are positive or negative.]

	14-liter churn	8-liter churn	Plastic cone	Teflon cone
1. Effectiveness of laboratory cleaning and transport protocols				
Major cations	+2	+0	+0	+1
Major anions	+0	+0	+0	+1
Sediment mass	--	--	--	--
Dissolved organic carbon	n/a	n/a	n/a	--[1]
Particluate organic carbon	n/a	n/a	n/a	--
Nutrients, dissolved	+0	+0	--	+1
Nutrients, total	--	--	--	--
Trace elements, dissolved	+2	+0	--	+1
Trace elements, total	--	--	--	--
Semi-volatile organic chemicals, dissolved	n/a	n/a	n/a	+3
Semi-volatile organic chemicals, total	n/a	n/a	n/a	--
Chorophyll	--	--	--	--
Bacteria	n/a	n/a	--	--
Radiochemicals, dissolved	+0	+0	+0	+0
Radiochemicals, total	--	--	--	--
Alkalinity	+0	+0	+0	+0
2. Effectiveness of field cleaning protocols				
Major cations	+2	+0	+0	+0
Major anions	+0	+0	+0	+0
Sediment mass	--	--	--	--
Dissolved organic carbon	n/a	n/a	n/a	-3[1]
Particluate organic carbon	n/a	n/a	n/a	--
Nutrients, dissolved	+0	+0	--	+0
Nutrients, total	--	--	--	--
Trace elements, dissolved	+2	+0	--	+0
Trace elements, total	--	--	--	--
Semi-volatile organic chemicals, dissolved	n/a	n/a	n/a	--
Semi-volatile organic chemicals, total	n/a	n/a	n/a	--
Chorophyll	--	--	--	--
Bacteria	n/a	n/a	n/a	--
Radiochemicals, dissolved	+0	+0	+0	+0
Radiochemicals, total	--	--	--	--
Alkalinity	+0	+0	+0	+0

[1] Samples for dissolved organic chemicals are not normally processed with the Teflon cone, due to possible contamination from methanol used in the cleaning procedure.

Table 2. Evaluation of performance of splitting devices based on existing data

[A: accuracy; P: precision. Explanation of table entries: n/a - information not needed or the device inherently cannot be used for this type of analyte. "--" indicates that no information exists and cannot be extrapolated. Numbers indicate authors' level of confidence in conclusions from existing data: 3 - high level, based on agreement of multiple studies; 2 - medium level, based on one well-planned study; 1 - minimal level, based on one partial study or disagreement between multiple studies; 0 - no actual data exist, but other existing data may be extrapolated to cover case being considered. "+" or "-" indicates whether conclusions drawn are positive or negative.]

	Teflon cone		Plastic cone		8-liter churn		14-liter churn	
	A	P	A	P	A	P	A	P
A. Concerns related to splitting capability								
1. Splitting capability of total suspended sediment	+1	+3	+1	+3	--	--	+/-1	+/-1
2. Splitting capability as a function of particle size								
Sand-sized particles	+1	+3	+1	+3	--	--	-1	-1
Silt- and clay-sized particles	+1	+3	+1	+3	+1	+1	+1	+1
3. Splitting capability as a function of particle density	--	--	--	--	--	--	--	--
4. Splitting capability as a function of water volume	+3	+3	+3	+3	n/a	n/a	n/a	n/a
5. Splitting capability as a function of particluate and "total" chemical concentration								
Nutrients	--	--	--	--	--	--	--	--
Trace elements	--	--	--	--	--	--	--	--
Semi-volatile organic chemicals	--	--	n/a	n/a	n/a	n/a	n/a	n/a
Chlorophyll/phytoplankton	--	--	--	--	--	--	--	--
Bacteria	--	--	--	--	--	--	--	--
Particulate organic carbon	--	--	--	--	--	--	--	--
Radiochemicals	--	--	--	--	--	--	--	--
6. Splitting capability as a function of Dissolved chemical concentrations								
Major cations	+0	+0	+0	+0	+0	+0	+0	+0
Major anions	+0	+0	+0	+0	+0	+0	+0	+0
Dissolved organic carbon	+0[1]	+0[1]	n/a	n/a	n/a	n/a	n/a	n/a
Nutrients, dissolved	+0	+0	+0	+0	+0	+0	+0	+0
Trace elements, dissolved	+0	+0	+0	+0	+0	+0	+0	+0
Semi-volatile organic chemicals, dissolved	+0	+0	--	--	--	--	--	--
Radiochemicals, dissolved	+0	+0	+0	+0	+0	+0	+0	+0
pH	+0	+0	+0	+0	+0	+0	+0	+0
Conductivity	+0	+0	+0	+0	+0	+0	+0	+0
Alkalinity	+0	+0	+0	+0	+0	+0	+0	+0
B. Concerns related to splitter operation								
1. Use of multiple splitters of the same type by the same operator	+1	+1	+1	+1	--	--	-1	-1
2. Use of the identical splitter by different operators	--	--	--	--	--	--	--	--
3. Leveling effects on cone splitter	+1	+1	+2	+2	n/a	n/a	n/a	n/a
4. Effects of total processed water volume	+2	+2	+2	+2	--	--	--	--
6. Effects of sequential aliquots	--	--	--	--	--	--	--	--
from churn splitter	n/a	n/a	n/a	n/a	-1	-1	-1	-1
7. Effects of combining ports from the cone splitter	+2	+2	+0	+0	n/a	n/a	n/a	n/a
C. Concerns related to inherent creation of chemical artifacts								
1. Creation of artifacts in particle size distribution	--	--	--	--	--	--	--	--
2. Gain/loss of chemical due to air/water transfer	--	--	--	--	--	--	--	--

[1] Samples for dissolved organic chemicals are not normally processed with the Teflon cone, due to possible contamination from methanol used in the cleaning procedure.

EFFECTIVENESS OF PROTOCOLS FOR CLEANING AND TRANSPORT OF SPLITTERS

Laboratory

Ideally, the two protocols, laboratory and transport, should be evaluated independently, but almost all of the existing data on equipment blanks integrates both protocols. Three types of studies have been conducted to evaluate the effectiveness of the cleaning protocols: blank water (deionized or "organic-free"), laboratory solutions of target analytes, and natural water solutions. These studies will be discussed for each of the devices.

14-liter Churn

The 14-L churn is the primary device for sample splitting of inorganic analytes. Those who introduced the device advised that it not be used for water samples requiring analysis for organic carbon, organic chemicals, or bacteria (QWB technical memoranda 76.24-T and 77.01). The equipment blanks generated over the years by various WRD investigations suggest that the cleaning protocols outlined in Ward and Harr (1990) are sufficient for processing water with inorganic constituents and solids in the concentration range of mg/L (milligrams per liter). Of greater concern is the capability of the cleaning and transport protocols to provide a splitter able to process water without contaminating samples with trace elements or nutrients at the µg/L (micrograms per liter) level.

Horowitz and others (1994) describe the protocols for the cleaning, transport, and field use of the churn splitter for water samples to be analyzed for dissolved inorganic constituents at the µg/L level. OWQ technical memorandum 94.13 (appendix A) presents data on a series of experiments designed to certify that these procedures are adequate for samples to be analyzed for dissolved major cations and trace elements at µg/L levels. In one study the churn was cleaned in the laboratory with deionized water processed through the sampler. Although one low-level concentration of copper was detected, the results suggested that the laboratory cleaning procedures were sufficient. The adequacy of the cleaning protocol for analysis of major ions (at the mg/L level), dissolved nutrients, dissolved radiochemicals, and alkalinity can be inferred from these results as well. There have been no studies found that examined the adequacy of the cleaning protocols for analysis of particle-associated or "total" inorganic constituents.

8-liter Churn

The cleaning tests mentioned above were not performed using the 8-L churn splitter. However, it is reasonable to assume that the cleaning procedures are adequate for both sizes of the churns. Contamination problems are not expected for dissolved, inorganic analytes if the same cleaning protocols (OQW technical memorandum 94.13) are followed.

Plastic Cone

There have been no studies found that examined the adequacy of the laboratory cleaning protocols for dissolved, particle-associated, or "total" inorganic constituents. The routine field equipment blanks generated by various studies within the WRD suggest that the cone splitter can be sufficiently cleaned in the field with the protocols outlined by Ward and Harr (1990) for inorganic constituents at the mg/L level. This plastic splitting device should not be used to collect water samples for analysis of organic carbon or organic chemicals.

Teflon Cone

The Teflon cone is the only splitting device that has potential application with the complete suite of water-quality analytes, including low-level trace elements and organic chemicals. Because of this, the NAWQA program has incorporated the use of this device in their field protocols for splitting water samples to be analyzed for both trace elements and organic chemicals (Shelton, 1994).

An initial study to examine the laboratory cleaning protocols was conducted using a 200 µg/L solution of chloride or nitrate salts of barium, calcium, cadmium, cobalt, copper, potassium, magnesium, tin, and zinc in deionized water. This solution was passed through the cone. The cone was then cleaned with the standard protocols (Shelton, 1994) and packed for transport. The packed cone was opened and one L of deionized water was passed through it. Samples were collected from three ports and analyzed for trace elements, major cations, major anions, and nutrients. The data are summarized in appendix B (Shelton and Capel, 1994). Although very low-level concentrations of aluminum and zinc (<1 µg/L), calcium and magnesium (< 0.003 mg/L), and chloride (<0.04 mg/L) were detected (all less than their reporting limits), the results suggest that this laboratory cleaning procedure is adequate.

Zorgorski, Sandstrom, and Capel (1990, appendices A and B) conducted a study on the adequacy of laboratory cleaning of sample processing equipment, including the Teflon cone splitter for organic chemicals. The target analytes included the chlorophenoxy acid herbicides, carbamate insecticides, triazine and other nitrogen-containing herbicides, and the organochlorine and organophophorus insecticides. The sample processing equipment was cleaned with laboratory soap and water, rinsed with "organic-free" water until all soap bubbles disappeared, rinsed with methanol, air dried, and wrapped in aluminum foil for transport. The equipment was taken to the field site and opened. "Organic-free" water was processed as a sample. This procedure was performed at three sites. All target analytes were below the detection limit at two of the three sites. At the third site, low-levels of 2,4-D, 2,4,5-T, silvex, picloram, dicamba, and carbaryl were quantified, but these low-level detections occurred in one of the four laboratory schedules used (except carbaryl) and may have been an artifact of the laboratory processing. Overall, the cleaning procedure appeared adequate.

During a study of pesticides in the Mississippi River basin, in which the Teflon cone splitter was used, approximately 68 field equipment blanks were collected. The equipment was cleaned and rinsed as described above. The equipment blanks were analyzed for up to 46 pesticides by gas chromatography-mass spectrometry. Only one equipment blank had detections (low-levels of atrazine and metolachlor), which were attributed to laboratory contamination (Coupe and others, 1994). The contamination problem that did arise during this study was caused by dissolved organic carbon (DOC). The DOC concentrations were randomly elevated in many samples. The problems were traced back to residual methanol in the processing equipment. After this observation was made, DOC samples were no longer collected in the study. To avoid this problem in the NAWQA program, a separate DOC sample is obtained directly from the stream and is not processed through the methanol rinsed cone splitter (Shelton, 1994).

Field

14-liter Churn

The field equipment blanks generated over the years in various WRD investigations suggest that the field cleaning protocols outlined in Ward and Harr (1990) are sufficient for processing water with inorganic constituents and solids in the concentration range of mg/L. As with the laboratory cleaning protocols, the greater concern is the capability of the field cleaning and transport protocols to provide a splitter to process water without contaminating samples with trace elements or nutrients at the µg/L level. Horowitz and others (1994) describe the protocols for field cleaning and use of the churn splitter for water samples to be analyzed for dissolved inorganic constituents at the µg/L level. OWQ technical memorandum 94.13 (appendix A) presents data on a series of experiments designed to certify that these procedures are adequate.

In one experiment, the churn was cleaned in the field, and deionized water was then processed through the churn. In another experiment, numerous natural waters with known elevated presence of trace elements were composited in the churn. The churn was then field cleaned. Then deionized water was added, processed, removed as a normal environmental sample, and analyzed. Although low-level concentrations of numerous trace elements were detected, the OWQ technical memorandum 94.13 suggests that the field cleaning procedures are adequate. The adequacy of this cleaning protocol for the 14-L churn probably can be extrapolated to the major ions (at the mg/L level), dissolved nutrients, dissolved radiochemicals, and alkalinity. There have been no studies found that examined the adequacy of the cleaning protocols for particle-associated or "total" inorganic constituents.

8-liter Churn

Although the field cleaning tests mentioned above were not done using the 8-L churn splitter, it is reasonable to assume that the cleaning procedures are adequate for both sizes of the churn. Contamination problems in the 8-L churn splitter are not expected for dissolved, inorganic analytes if the field cleaning protocols (OWQ technical memorandum 94.13) are followed.

Plastic Cone

No studies have been found that examined the adequacy of the field cleaning protocols for the plastic cone splitter for dissolved, particle-associated, or "total" inorganic constituents. The routine field equipment blanks generated by various studies throughout the WRD suggest that the cone splitter can be sufficiently cleaned in the field by the protocols outlined by Ward and Harr (1990) for inorganic constituents at the mg/L level.

Teflon Cone

No studies have been found that specifically examined the adequacy of the field cleaning protocols for the Teflon cone splitter for dissolved, particle-associated, or "total" inorganic or organic constituents. The equipment blank data generated by the NAWQA program will give an indication of the adequacy of cleaning procedures for the whole sample processing scheme, which includes processing with the Teflon cone splitter.

CONCERNS RELATED TO SPLITTING CAPABILITY

To put in perspective the following data on the capabilities of the splitting devices, some knowledge of the other errors involved in quantifying suspended sediment concentrations is useful. Potential errors in measuring the suspended sediment concentration can arise from sample collection, sample processing, and final analytical measurements. Moody and Meade (1992) discuss the analytical procedures and show that the analytical error for fine material (<63 µm) is typically 1 mg/L or about one percent for water with a suspended sediment concentration of 100 mg/L. For water with very low suspended sediment, the error will increase significantly. As an example, for water with a suspended sediment concentration of 2 mg/L, the analytical error may be on the order of 50 percent. For sand-sized material (>63 µm), the analytical errors were much less. They found a two percent error for concentrations of 0.1 mg/L to less than 0.1 percent error for concentrations of 100 mg/L. The errors in sample collection are significantly greater. Moody and Meade (1992) collected duplicate suspended sediment samples at 44 sites on the Mississippi River and some of its tributaries. At each site, they collected between 14 and 40 verticals and alternately designated the vertical "Sample A" or "Sample B." The sand-sized material was sieved and composited, whereas the finer material was processed through the churn splitter. They found that there was a mean difference in the absolute values of the duplicate sand concentrations of 9, 10, 6, and 18 percent on four cruises, whereas the absolute values of the silt/clay concentrations had a mean difference of 3, 2, 2, and 2 percent for the four cruises. This suggests that the natural variability of the suspended sediment concentration, especially the sand-sized fraction, is real and at times large. These natural variations should be kept in mind as the errors associated with the splitting devices are discussed below.

Splitting Capability of Total Suspended Sediment

14-liter Churn

The original information on the splitting capability of the churn was released in QWB Technical memorandum 76.24-T (appendix A). This document, without any details, cites a study that found that a water sample with 5,000 mg/L of sand-sized solids can be split within ±10 percent of the true concentration. Although the original data could not be found, an unfinished manuscript by Delaney and Ong (personal communication) records that the solids used in the study were in the range of 62 to 500 µm and had a median diameter of 150 µm. A water sample with a suspended sediment concentration of 362 mg/L from the Rio Grande at Albuquerque, New Mexico was also processed in the tests. For the laboratory studies using sand-sized particles, it was reported that all subsamples obtained from the churn were within ± 15 percent of the original concentration and that the one river sample was within ± 5 percent of its original concentration. All of these tests started with a volume of a little less than 14 L and withdrew subsamples of one L. The investigators concluded that the last four L from the churn should not be used for analysis that involves the particulate phase.

QWB technical memorandum 77.01 further describes the churn splitter. It states that "the churn splitter is designed and tested especially for use in streams transporting sand-sized sediments...and they are the only acceptable means of splitting samples containing sand-sized materials in the field." The 1978 QWB technical memorandum (78.03) described the limitations of the churn splitter. Without presenting any data, the memorandum states that "tests of the churn splitter using sand-sized particles (>62 µm) have indicated that when relatively high concentrations of such particles are present, subsamples for suspended-sediment concentrations or particle-size determinations should not be taken from the churn...When essentially all particles are silt-size or smaller (<62 µm), sediment-concentration and particle-size subsamples may be taken directly from the churn." The data supporting these conflicting observations were not available to these authors.

The Branch of Quality Assurance (BQA; 1990, appendix B) evaluated the capability of the 14-L churn to split water samples containing coarse and fine particles. Two mixtures were used, each containing a total suspended sediment concentration of 20,000 mg/L. Sediment in solution "A" was composed of 70 percent fine particles (<63 µm) and 30 percent coarse particles (>63 µm). Sediment in solution "B" was composed of 95 percent fine particles and 5 percent coarse particles. In six separate trials, using two different churns, 10 L of solution were placed in the churn and six 1-L subsamples were withdrawn while raising and lowering the churn-paddle at a rate of nine inches per second. The subsamples and the four L of solution remaining in the churn were analyzed for total suspended sediment concentration and particle-size distribution. The results for total suspended sediment, and fine and coarse particle concentration are shown in figure 3. Several points can be made from the two plots in this figure. First, deviations from the "true" concentrations were substantial in some cases, due primarily to variability in coarse particle concentrations. The range of deviations, and the overall mean and median deviations for all six trials are given in table 3. For total suspended sediment, the range of the deviations for all 18 subsamples was approximately ±15 percent. The mean and median deviations ranged from 5 to 7.5 percent of the expected concentration, with the deviations primarily due to the coarse particles [see section on "Splitting Capability as a Function of Particle Size (14-liter Churn)]. The results in figure 3 indicate a certain amount of unpredictability in the churn's capability to split suspended sediments equally, with differences between churns (see section on "Use of Multiple Splitters of the Same Type by the Same Operator") and effects of subsample-withdrawal order (see section on "Effects of Sequential Aliquots from Churn Splitter") both evident. The errors due to the operator are imbedded in these overall errors.

8-liter Churn

No available data addressed this topic specifically for the 8-L churn splitter. Because of the differences in geometry, any extrapolations from the 14-L churn are tenuous.

Plastic Cone

The original documentation that introduced the plastic cone to the WRD (QWB technical memorandum 80.17, appendix A) contains solids splitting data from the prototype. Six tests with a suspended sediment concentration of about 2,400 mg/L were conducted. Each sample contained the following mass of solids in three particle-size ranges in 2,486 mL of water: 1.0 g of 62-125 µm, 4.5 g of 125-250 µm, and 0.5 g of 250-500 µm. This was considered a worst case scenario because the finer particles were assumed to yield better results. The coefficients of variation between subsamples in each test (for the six tests) were all in the range of 1.7 to 3.5 percent. In the test with the poorest results, the deviation from the mean was in the range of -4.4 to +5.6 percent.

Durham and McKenzie (1985, appendix B) conducted an early WRD-sponsored study to evaluate the plastic cone. They used a sand-sized particle mixture with a concentration of 1,000 mg/L. The mixtures had the following particle size distribution (from a visual accumulation analysis): 100 percent finer than 350 µm, 99 percent finer than 250 µm, 91 percent finer than 175 µm, 51 percent finer than 125 µm, 7 percent finer than 88 µm, 0 percent finer than 62 µm. The coefficients of variation for the suspended sediment concentrations between the ten subsamples were in the range of 2.0 to 6.7 percent for five tests. The largest absolute deviation from the mean concentration (949 mg/L) was -101 to +76 mg/L (-10.6 to +8.0 percent). The absolute deviations in concentration for the other four tests were considerably smaller.

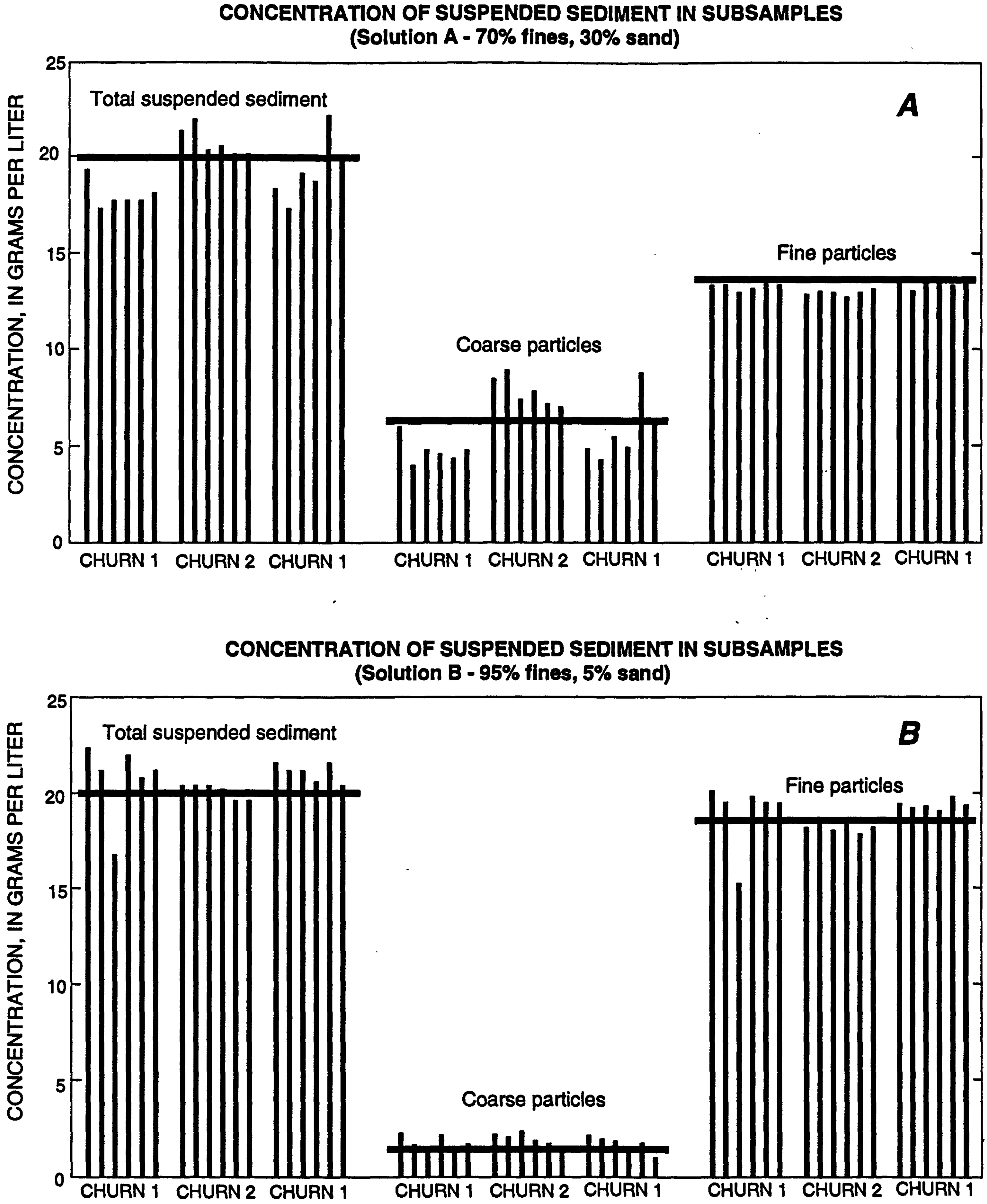

Figure 3. Concentrations of total suspended sediment, coarse particles, and fine particles in subsamples from the 14-liter churn splitter. Solid horizontal lines indicate the "true" concentration. Subsamples within each group are arranged in order of withdrawal from the churn. Data from Branch of Quality Assurance (1990, appendix B).

Table 3. Range, mean, and median percent deviations from the known (true) suspended sediment concentrations for subsamples from the 14-L churn (data from appendix B, Branch of Quality Assurance)

		Range of percent deviation from true value	Mean percent deviation from true value	Median percent deviation from true value
Total suspended sediment	Solution A (30% sand)	-13 to +10	6.9	7.5
	Solution B (5% sand)	-16 to +12	5.4	5
Fines (<0.063 mm)	Solution A (30% sand)	-6.8 to +1.1	3.2	2.9
	Solution B (5% sand)	-18 to +8.3	4.9	4.3
Coarse (>0.063 mm)	Solution A (30% sand)	-37 to +42	23	24
	Solution B (5% sand)	-28 to +65	31	29

In the same study, the investigators tested the precision of the plastic cone with a real environmental water sample (urban runoff). They conducted three tests that used 1,800 mL of water with a suspended sediment concentration of about 1,700 mg/L. They reported coefficients of variation for the suspended sediment concentrations between the ten subsamples were in the range of 0.9 to 1.3 percent for the total suspended sediment concentration. The largest absolute deviation from the mean concentration (1,711 mg/L) was -51 to +17 mg/L (-3 to +1 percent).

Skinner and Szalona (1980, appendix B) evaluated the cone splitter with a laboratory mixture of coarse-grained material (sieved between 8 and 0.25 mm). They created two mixtures: a high concentration mixture of about 239,000 mg/L and a low concentration mixture of about 127 mg/L. The high concentration mixture was split with a coefficient of variation of 0.078 percent, whereas the low concentration mixture was slightly less precise at 4.3 percent. The researchers suggested that this difference in error may not be due the splitter's performance, but rather to limitations of the analytical balance. Each mass was read to the nearest mg and the individual readings of mass with the low-concentration mixture were about 20 mg.

Gray and Ferguson (1990, appendix B) evaluated the cone splitter with one test of 9 L of natural water with suspended sediment that was mostly <63 µm. The coefficient of variation for solids concentrations in the subsamples was 3.2 percent.

The Branch of Quality Assurance (1992, appendix B) conducted a series of tests on one plastic cone splitter to evaluate its splitting capability. With one cone, they ran duplicate samples of suspended sediments (about 1,000 mg/L) that were all either < 63 µm, or 20 percent >63 µm and 80 percent <63 µm. They found that the coefficients of variation for the fine materials (1.8 and 4.4 percent) were similar to the mixture of coarse and fine materials (2.2 and 3.2 percent). They also observed that the variations of sediment concentration within any one test were between 69 and 143 mg/L, which is equivalent to variations between 7 and 15 percent of the mean solids concentration.

All of these studies on the plastic cone splitter are summarized in table 4.

Table 4. Summary of five studies for the splitting capability of the plastic cone for solids

[mg/L, milligram per liter]

Study	Approximate suspended sediment concentration (mg/L)	Range of coefficient of variation (percent)
Quality of Water Branch technical memorandum 80.17	2,400	1.7-3.5
Skinner and Szalona, 1980	127 239,000	4.3 .078
Durham and McKenzie, 1985	1,000 1,700	2.0-6.7 0.9-1.3
Gray and Ferguson, 1990	260	3.2
Branch of Quality Assurance, 1992	1,000	1.8-4.4

Teflon Cone

The Teflon cone was evaluated for its capability of splitting various size fractions of solids under controlled conditions in the laboratory (Capel and Nacionales, 1995). Nine discreet inorganic particle-size ranges from clay to very coarse sand were mixed with deionized water and processed through the cone at concentrations of 50 and 200 mg/L. Deviations from the known sediment concentration were generally in the range of ± 6 percent. For particles smaller than coarse sand, the coefficient of variation between splits was < 7 percent. For larger particles, the cone splitter was somewhat less precise (fig. 4).

Martin (1993, appendix B) collected a sample of river water with artificially high sand content and processed it through the Teflon cone splitter, analyzing each split for suspended sediment concentration. The sample contained sandy bottom material

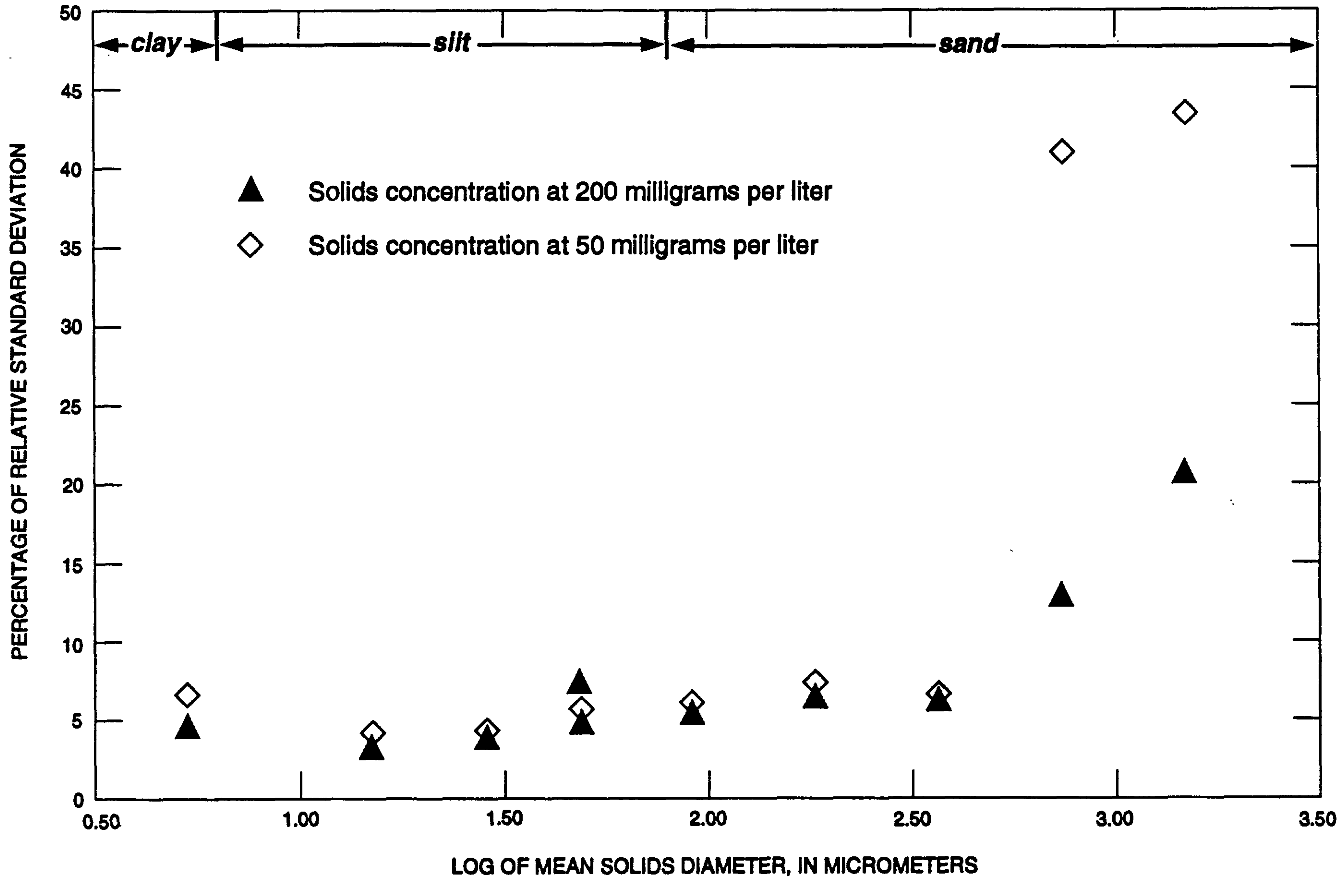

Figure 4. Effect of particle size and concentration of suspended sediment on the precision of the Teflon cone. The coefficient of variation (percent) is the standard deviation of the suspended sediment concentrations in the subsamples divided by the mean suspended sediment concentration in the subsamples, times 100. Data from Capel and Nacionales, 1995.

purposely mixed with river water, so that the suspended sediment was 88 percent coarse material (>63 µm). Concentrations in each of the 10 splits, and the "true" concentration, are shown in figure 5. The mean deviation from the true concentration was ± 5.8 percent (median: ± 5.2 percent, standard deviation: 3.4 percent).

Between 1991 and 1993, the White River Basin NAWQA study unit processed 71 river samples with the Teflon cone and analyzed two subsamples from each for suspended sediment concentration. Total sediment concentrations ranged from 8 to 890 mg/L, and the fine material (<63 µm) ranged from 4.0 to 98 percent (White River Basin NAWQA, 1993, appendix B). The agreement between splits is shown in figure 6*A* (note that the axes in these plots are logarithmic and the line is the line of exact agreement). For the 71 duplicates, the mean percent difference between splits was 11 percent (median: 4.2 percent) for total suspended solids concentration. The pre-cone processing of the samples differed in 1993 compared to 1991 and 1992. In the early years, the water was composited in a 20 gallon stainless steel milk can before it was poured through the cone splitter. In 1993, the water was poured directly from the 3-L D-77 sample bottle through the cone. The capability of the cone to precisely split the solids was different for the two different pre-processing steps. When the milk can was used, the mean percent difference between splits was 7.0 percent (median: 3.8 percent, fig. 6*B*) compared to 29 percent (median: 18 percent, fig. 6*C*) when the milk can was not used. The difference may be due to those solids (particularly sand-size materials) that are retained in the milk can and do not enter the cone. The present NAWQA sample processing protocols do not employ the milk can.

Only one data set was found in which suspended sediment concentrations can be directly compared in samples collected using the traditional sampling method and samples processed through a cone splitter. The South Platte NAWQA study unit (1993, appendix B) collected concurrent water samples, one or two using the traditional method with a D-74 sampler (Edwards and Glysson, 1988) and one collected with a D-77 sampler and processed with the Teflon cone splitter as specified in the NAWQA field protocols (Shelton, 1994). Total suspended solids concentrations ranged from 3 to 2,300 mg/L . The agreement between suspended sediment concentrations from the cone splitter samples and the traditional samples, and between duplicate traditional samples is shown in figure 7 (note that the axes in these plots are logarithmic and the line is the line of exact agreement). Agreement between the cone-processed and traditional samples was generally quite good, especially for suspended sediment concentrations above 50 mg/L. Agreement was more variable for concentrations less than 50 mg/L. The mean and median percent differences were 12 and 9 percent, respectively, for suspended sediment concentrations greater than 50 mg/L. Agreement between the two traditional samples was somewhat better, with a mean and median percent difference between samples of 7.5 and 5.2 percent, respectively, for concentrations greater than 50 mg/L. There was no apparent bias between the two methods using the Wilcoxon signed-rank test on the differences between paired data points (p-value = 0.33, which implies no significant difference between the two sets of measurements). These data imply that the accuracy of total suspended solids concentrations in samples processed with the Teflon cone splitter is comparable to concentrations obtained from the traditional method over a wide range of concentrations.

Splitting Capability as a Function of Particle Size

14-liter Churn

One approach that has been used to minimize the effects of non-homogeneous mixing of sand-sized particles is to remove them from the water before they enter the churn (Meade, 1985; Meade and Stevens, 1990; Moody and Meade, 1992; Moody and Meade, 1993). Meade (1985) observed that the "churn splitter does not distribute sand grains evenly because the stirring action does not entirely overcome the tendency for sand grains to settle. This sets up a gradient in the splitter where sand is more concentrated near the bottom (where the spigot is) than near the top." Because of these concerns, these investigators processed water through a 63 µm nickel sieve before the water entered the churn. Meade (1985) used a 295 mg/L mixture of mostly coarse silt (100 percent finer than 63 µm, 27 percent finer than 31 µm, and 14 percent finer than 16 µm) to evaluate the 14-L churn splitter. They found that five subsamples deviated slightly from the known concentration (average 301 mg/L, range 299 to 302 mg/L), but were replicated with errors of <±10 percent.

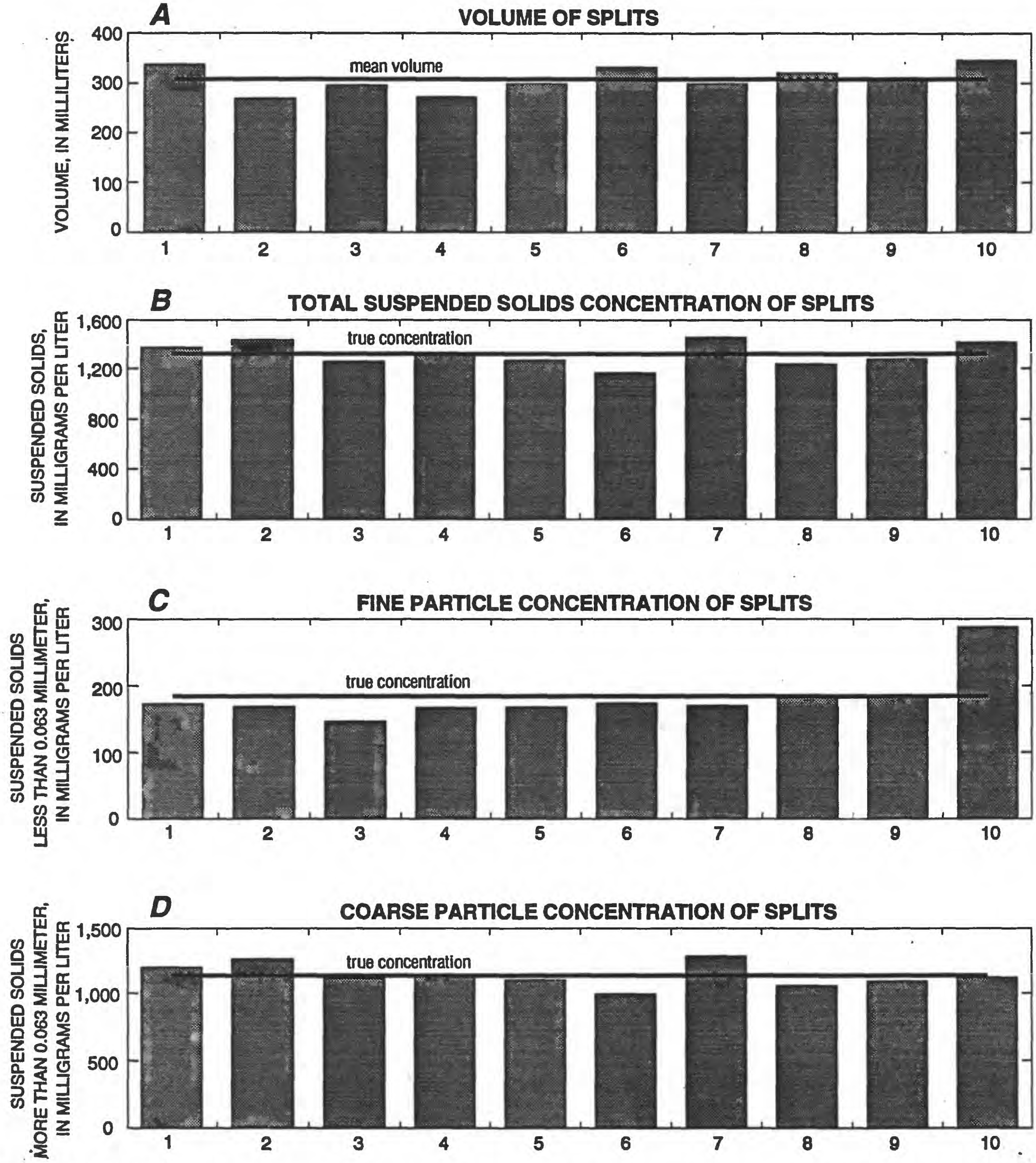

Figure 5. Precision and accuracy of water and solids splitting of one water sample by the Teflon cone.

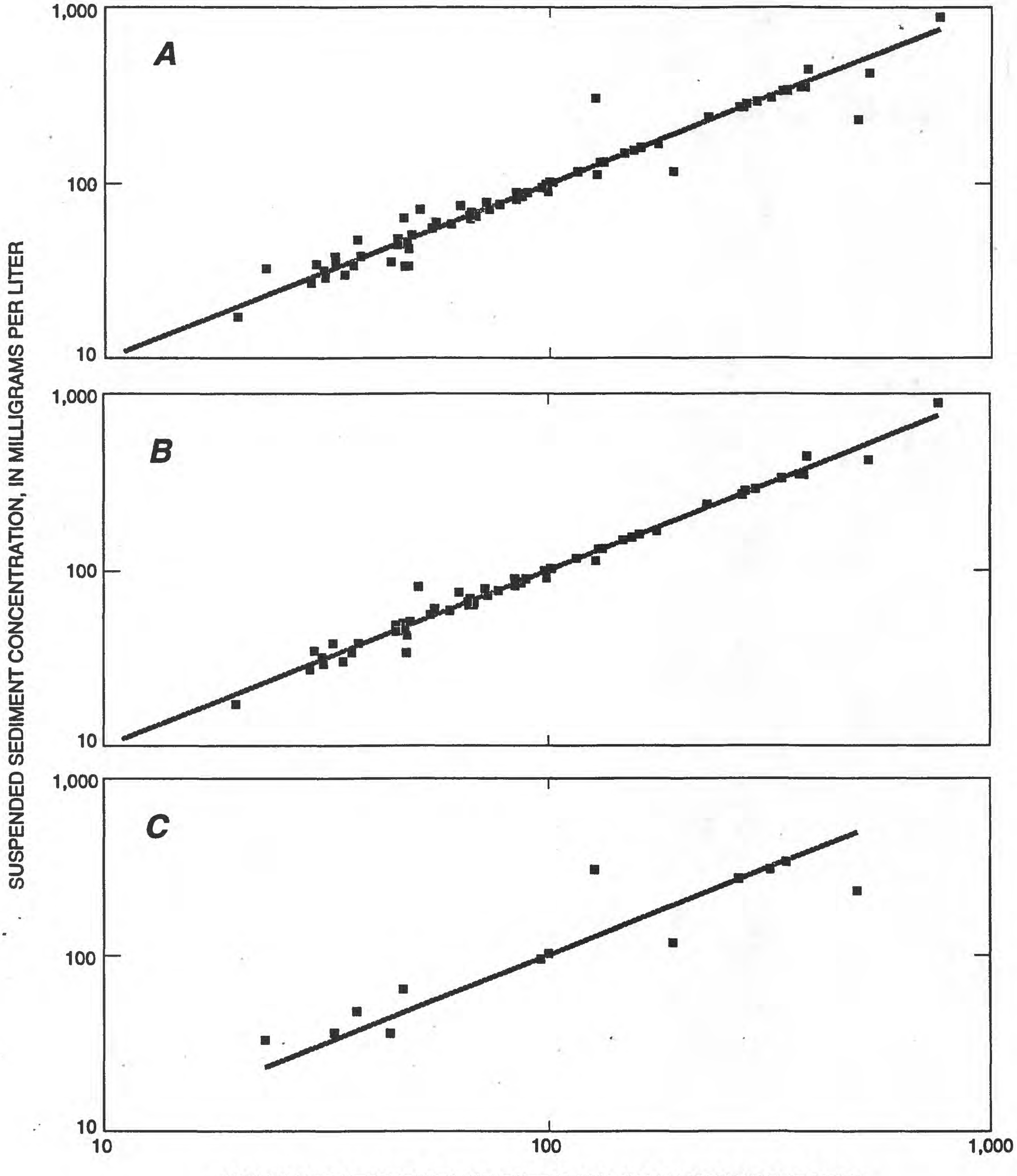

Figure 6. Agreement between suspended sediment concentrations in pairs of subsamples from the Teflon cone, from the White River NAWQA study unit (data in appendix B). ***A,*** Complete set of 71 pairs of subsamples. ***B,*** Data from samples composited in a milk can before pouring through the cone splitter. ***C,*** Data from samples poured directly from sampling bottle through the cone splitter. Note that the lines are not regression lines, but rather lines of what would be exact agreement.

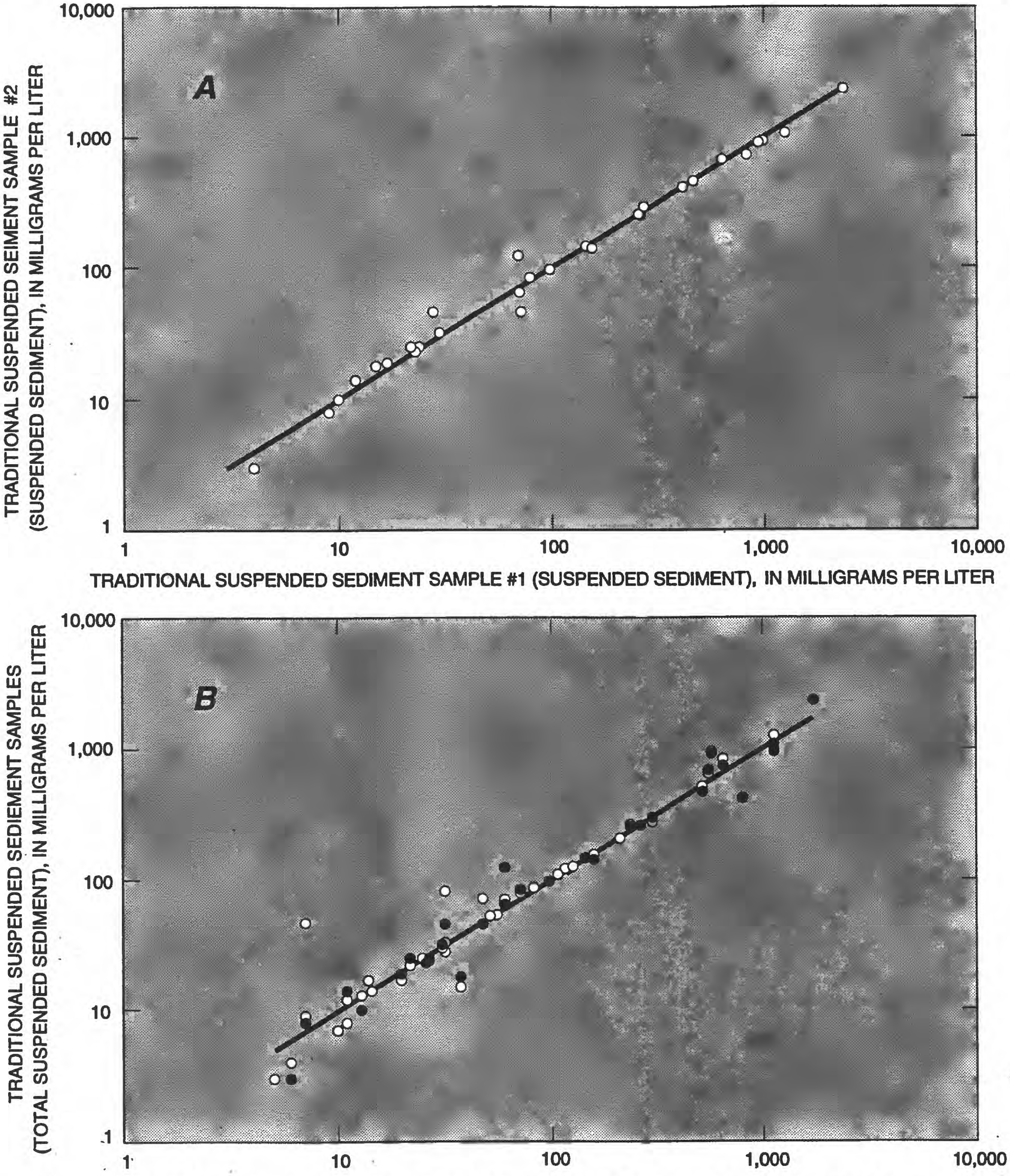

Figure 7. Agreement between suspended sediment concentrations in replicate water samples from the South Platte NAWQA study unit (data in appendix B). ***A,*** Replicate samples collected using the traditional method. ***B,*** Cone-processed samples versus samples collected at the same time by the traditional method. The symbols, • and ∘, represent the agreement with each of the replicate samples collected using the traditional method. Note that the lines are not regression lines, but rather lines of what would be exact agreement.

The Branch of Quality Assurance (1990, appendix B), as discussed in section "Splitting Capability of Total Suspended Sediment" for the 14-L churn above, evaluated the capability of the 14-L churn to split water samples containing sand-sized and finer particles. The results for total suspended sediment, and fine and coarse particle concentration are shown in figure 3. Deviations from the "true" concentrations were substantial in some cases, due primarily to variability in coarse particle concentrations, since the concentration of fine particles was nearly constant in subsamples from both mixtures. The range of deviations, and the overall mean and median deviations for all six trials are given in table 3. For total suspended sediment and the fine particles, the mean and median deviations were considerably lower (3 to 8 percent) than for coarse particles (20 to 30 percent). High concentrations of suspended sediment were used in these tests (20,000 mg/L). For the five percent sand mixture, a difference of one percent in the concentration of coarse particles in a subsample translates to a difference in mass of approximately 10 mg, which is easily measured on an analytical balance. Analytical measurement error should, therefore, not be a factor in the differences observed in the deviations of coarse and fine particles.

8-liter Churn

No data has been found that addresses this topic specifically for the 8-L churn splitter. Because of the differences in geometry, any extrapolations from the 14-L churn are tenuous.

Plastic Cone

The original documentation that introduced the plastic cone to the WRD (QWB technical memorandum 80.17, appendix A) contains solids–splitting data from the prototype. Six tests with sand-sized solids at a concentration of about 2,400 mg/L were conducted. In one of these tests, the distribution of the 125 to 250 μm size-fraction was evaluated. Of the solids in the test mixture, 75 percent were in this range. After splitting, the average was 72.2 percent (range 70.0 to 74.6) in this range. Although some particles appeared to be lost (probably an analytical error), the agreement between the 10 outlets was quite good (-3.0 to +3.4 percent deviation from the mean).

Durham and McKenzie (1985, appendix B) conducted an early WRD-sponsored study to evaluate the plastic cone with a real environmental water sample (urban runoff). They conducted three tests that used 1,800 mL of this runoff water with a suspended sediment concentration of about 1,700 mg/L. The water contained solids that were 13 percent >63 μm and 87 percent <63 μm. The plastic cone split the fine and coarse particles with coefficients of variation in the range of 1.3 to 2.0 percent and 4.9 to 11.2 percent, respectively. The largest absolute deviation from the mean concentration of the finer particles (1,468 mg/L) was -57 to +34 mg/L (-4 to +2 percent). The largest absolute deviation from the mean concentration of the coarser particles (232 mg/L) was -34 to +58 mg/L (-15 to +25 percent). Apparently, errors in the fine particle fraction partially offset the errors in the coarse particle fraction because the results obtained on total suspended sediment were better than either of the size fractions.

The Branch of Quality Assurance ran a series of tests on one plastic cone splitter to evaluate its splitting capability (Branch of Quality Assurance, 1992, appendix B). With one cone, they ran duplicate samples of suspended sediments (about 1,000 mg/L) that were all <63 μm or 20 percent >63 μm and 80 percent <63 μm. They found that the coefficients of variation between splits of the mixture of all fine materials (1.8 and 4.4) were similar to those of the mixture of coarse and fine materials (2.2 and 3.2).

Teflon Cone

In a laboratory evaluation, Capel and Nacionales (1995) examined the effect of particle size on the capability of the Teflon cone to reproducibly split the particles. They used nine discreet particle-size ranges at two concentrations. Figure 4 is reproduced from their study. Particles smaller than coarse sand were split with coefficients of variation <7 percent, but for the larger particles the splitting is much less precise. The errors with the larger particle sizes were attributed to one or a few grains of sand not being split correctly.

Martin (1993, appendix B) collected a sample of river water with artificially high sand content and processed it through the Teflon cone splitter, analyzing each split for suspended sediment concentration. The sample contained sandy bottom material purposely mixed with river water, so that the suspended sediment was 88 percent coarse material (>63 μm). Concentrations of fine and coarse suspended sediment in each of the 10 splits, and the "true" concentrations, are shown in figures *5C* and *5D*. The mean percent deviation from the true concentration was ± 12.5 percent (median: ± 8.0 percent) for the fine material and ± 5.9 percent (median: ± 4.6 percent) for the coarse material. These results show good precision for the coarse material. The reason for the elevated concentration of fine material in the sample from one of the ports is unknown.

The White River basin data described in section "Concerns Related to Splitting Capability, Splitting Capability of Total Suspended Sediment, Teflon Cone", (1993, appendix B) can also be used to evaluate the precision of the Teflon cone splitter with respect to particle size, using natural water samples. Of the 71 split samples, 29 pairs were analyzed for particle size distribution (sand/silt break) as well as for total suspended sediment concentration. The concentrations of fine (<63 μm) and coarse (>63 μm) particles in the splits are compared in figure 8 (note that the axes in these plots have logarithmic scales and that the line is the line of exact agreement). For fine particle concentrations, the mean percent difference between splits was 26 percent (median: 13 percent). For coarse particle concentrations, the mean percent difference between splits was 82 percent (median: 25 percent). These errors are greater than the errors reported for the laboratory studies.

The South Platte NAWQA data described in section "Concerns Related to Splitting Capability, Splitting Capability of Total Suspended Sediment, Teflon Cone". (1993, appendix B) can be used to evaluate the capability of the Teflon cone splitter to reproduce the size distribution of suspended sediments accurately in splits of real natural water samples. In the 47 pairs of samples processed by both the traditional method and with the cone splitter, the fine particles (<63 μm) ranged from 20 to 99 percent. In figure 9, concentrations of fine (<63 μm) and coarse (>63 μm) particles are compared for the traditionally collected and cone-processed samples, and for duplicates of the traditional samples. The mean and median percent difference between cone-processed and traditional samples were 19 and 10 percent, respectively, for the entire range of concentrations. Agreement between the cone processed and traditional samples was generally good for the fine particles in the range of 25 to 300 mg/L (fig. 9*A*). Agreement was better between the duplicate traditional samples (fig. 9*B*), with a mean and median percent difference of 5 and 3 percent, respectively. Agreement on concentrations of coarse particles was not as good (fig. 9*C*). Mean and median percent differences between cone-processed and traditional samples were 80 and 33 percent, respectively. Agreement between duplicate traditional samples (fig. 9*D*) was also not as good for the coarse particles, with mean and median percent differences of 33 and 22 percent, respectively. There was no apparent bias between these two methods for fine particles ($p>0.1$, signed-rank test), with a median difference in concentration between paired samples of + 1.2 mg/L. For coarse particles, there was a slight bias toward lower concentrations using the cone splitter ($p = 0.005$ to 0.01, signed-rank test), although the median concentration difference was only -2.0 mg/L. These data suggest that use of the D-77 sampler and Teflon cone splitter to process natural water samples will not appreciably change the concentration of fine particles, compared to the results obtained from the traditional suspended sediment method employing a D-74 sampler, although it does appear that more variability may be introduced. Some of this variability may be in the collection procedure itself and not related to the cone. For coarse particles, there does appear to be a slight bias toward lower concentrations and more variability using the cone splitter. Duplicates of traditionally collected samples also show more variability for coarse materials.

Splitting Capability as a Function of Particle Density

There have been no studies found that examined the ability of the devices to split particles as a function of density. Essentially all of the studies that have been conducted have used inorganic particles that have densities far greater than water. Organic particles, such as algae and detritus that have densities close to water, may act differently when they are processed through the splitting devices. The less dense organic particles would be more likely to flow with the water and to split more accurately and precisely than the heavier inorganic particles, but this has not been confirmed. One concern for the organic particles is their potential retention on the coarse screen in the cone splitters. Numerous field personnel have observed that the screens can turn green during spring sampling. This

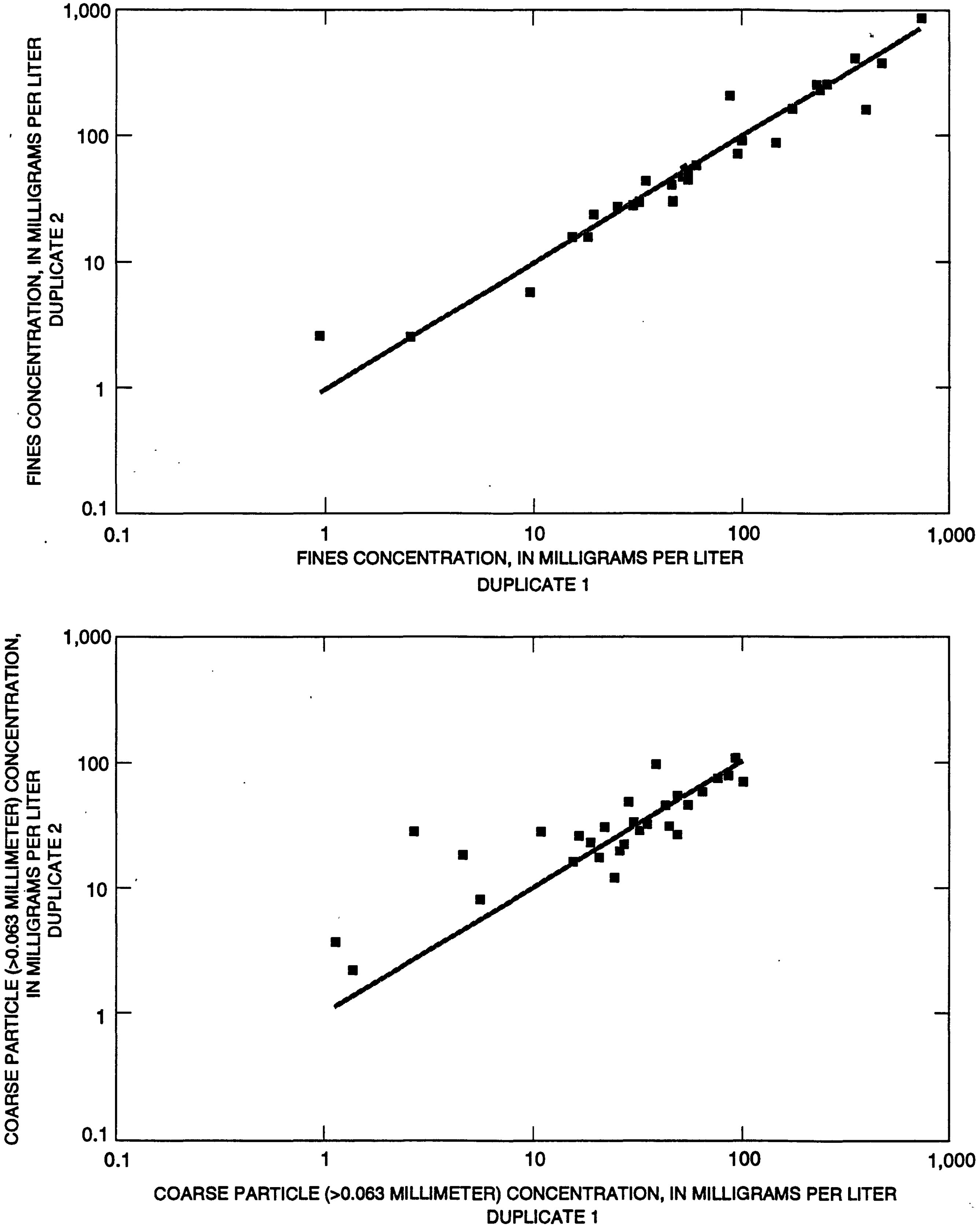

Figure 8. Agreement between concentrations of fine (<0.063 mm) and coarse (>0.063 mm) particles in pairs of subsamples from the Teflon cone, from the White River NAWQA study unit (data in appendix B).

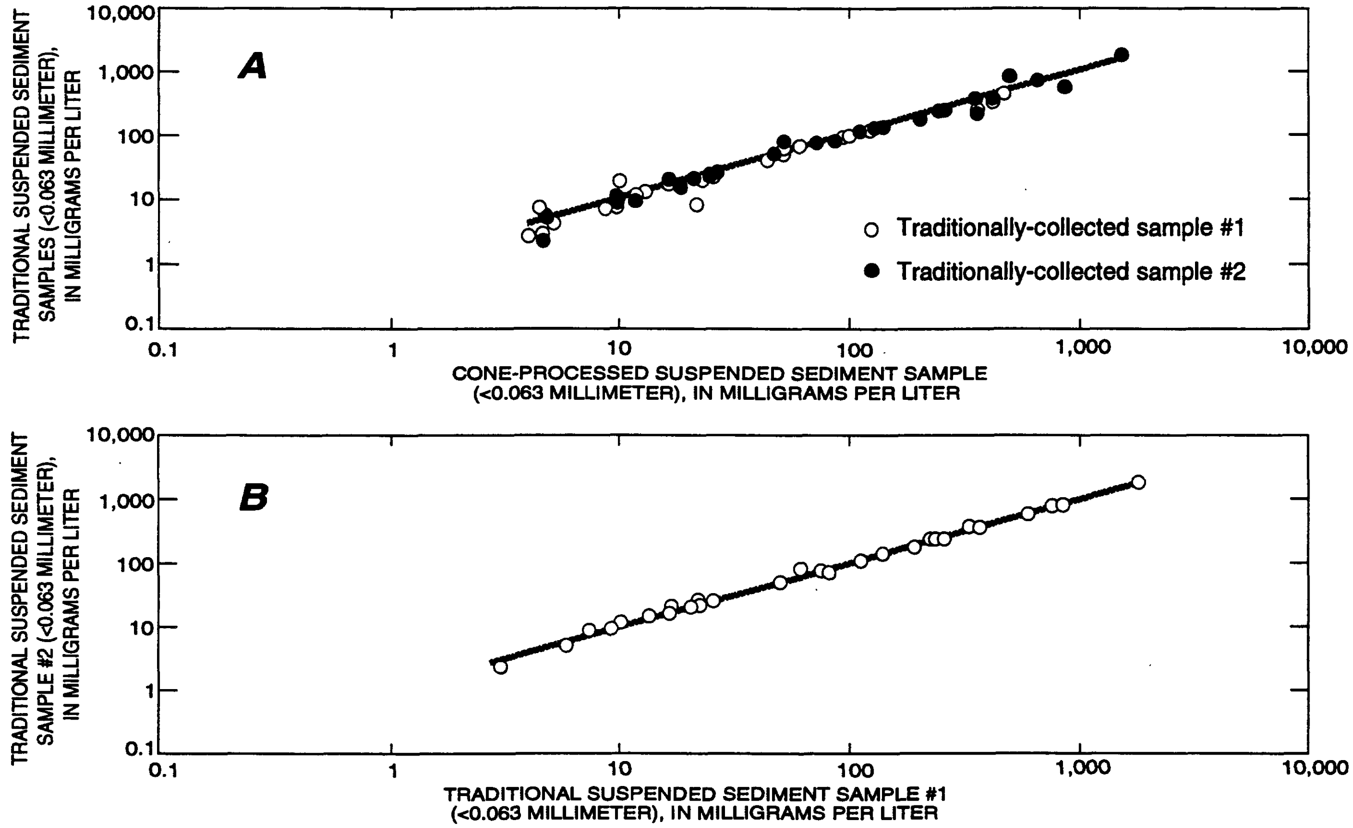

Figure 9. Agreement between fine and coarse suspended sediment concentrations in replicate water samples from the South Platte NAWQA study unit (appendix B). ***A,*** Fine (<0.063 mm) particle concentrations in cone-processed samples versus samples collected at the same time using the traditional method. ***B,*** Fine particle concentrations in replicate samples collected using the traditional method. ***C,*** Coarse (>0.063 mm) particle concentrations in cone-processed samples versus samples collected at the same time using the traditional method. ***D,*** Coarse particle concentrations in replicate samples collected using the traditional method. The symbols, • and ◦, in plots ***A*** and ***C*** represent the agreement with each of the replicate samples collected using the traditional method. Note that the lines are not regression lines, but rather lines of what would be exact agreement.

is due to retention of filamentous algae. The effect of this retention on the concentration of suspended sediments would be negligible, but it would cause lower results for particulate and/or total carbon, nitrogen, and phosphorus.

Splitting Capability for Volume of Water

Plastic Cone

The original documentation that introduced the plastic cone to the WRD (QWB technical memorandum 80.17, appendix A) contains water splitting data from the prototype. Six tests, each using about 2.5 L were conducted. The investigators demonstrated that the coefficients of variation for the six tests ranged from 0.92 to 1.12 percent. They also observed that 1.2 to 2.8 mL of water was retained by the plastic cone. They noted a very small systematic bias in the splitting of water by certain outlets and suggested that this was due to slight variations in the cone's fabrication process.

Durham and McKenzie (1985, appendix B) conducted an early WRD-sponsored study to evaluate the plastic cone. These investigators observed that about 3 mL of water was retained by the cone and accumulated mostly at the joint above the splitting chamber. Using distilled water, they conducted eleven tests with 2,500 mL, six tests with 250 mL, and four tests with 100 mL. The tests using the 2,500 and 250 mL sampler of water yielded similar results. All of the coefficients of

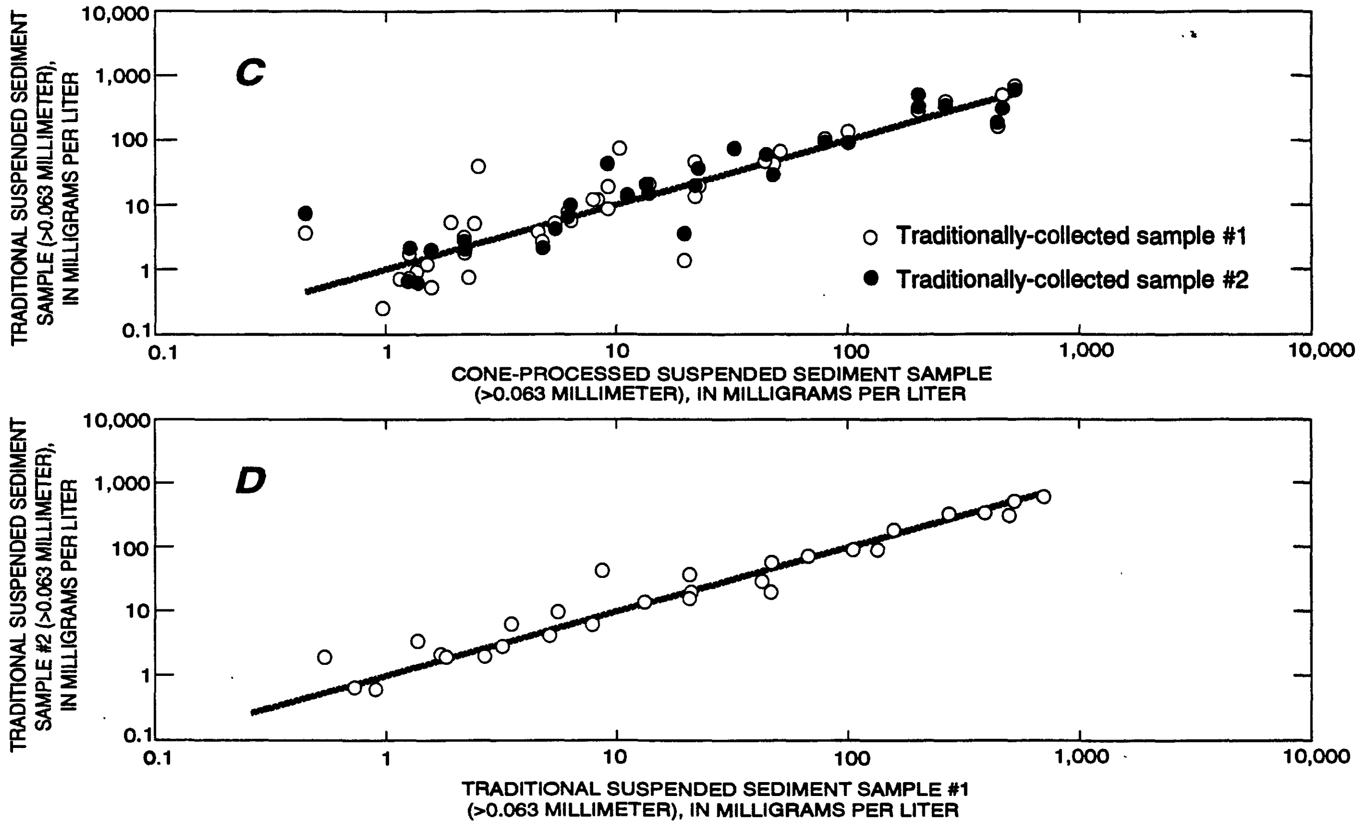

Figure 9.--*Continued.*

variation were in the range of 1.1 to 4.5 percent. In this series of tests, temperature was considered as potentially affecting precision, but there was no difference in the results of tests conducted at 4 and 24 °C. Precision was much lower for the tests conducted with a volume of 100 mL. The coefficients of variation between splits in the four tests were 2.2, 2.6, 25.9, and 29.0 percent. This would suggest that small water volumes are not split as evenly in the cone as larger water volumes (>250 mL). Three tests (about 1,800 mL each) with urban runoff water were also conducted. There was no difference in the precision of these results (coefficients of variation: 1.2, 1.9, 2.3 percent) compared to tests using distilled water.

Gray and deVries (1984) incorporated the cone section of the cone splitter in an autosampler and evaluated its splitting capability. They performed 10 tests on three different cones. They found that the volume of water in subsamples from the three cones was between +3.5 and -4.2 percent of the expected volume. They concluded that there was no statistically significant difference in the capability of the three splitters.

Skinner and Szalona (1980, appendix B) tested the water splitting capability of one plastic cone splitter with two tests using about 2.5 L of water in each. They found that the coefficients of variation between the water volumes in splits were 4.2 and 4.9 percent.

In another study, Gray and Ferguson (1990, appendix B) evaluated a plastic cone splitter for its effectiveness in splitting water and solids evenly. They found that the water was split with an accuracy of -5 to +6 percent of the mean. They also found that the errors in water volume splitting were greater in a dry cone then in one that was pre-wet. They concluded that the cone should be pre-wet before use. They also observed that the deviations from the mean water volume showed a consistent pattern. All of the outlets with a negative bias were located adjacent to each other and all of the outlets with a positive bias were located adjacent to each other on the other side of the cone. This will be discussed further in the section on the effects of leveling on cone splitter performance.

The Branch of Quality Assurance ran a series of tests on the plastic cone splitter (1992, appendix B). They ran a total of five tests on two cones in which they added about two L of water and measured the volume of water from each outlet. The two cones did not yield comparable results in terms of precision. One cone yielded coefficients of variation of 2.6, 2.8, and 3.0 percent, whereas the other cone yielded results of 7.2 and 6.0 percent. The reason for these differences are is known. They could be due to leveling, different operators, or inherent differences between the two cones. The investigators in this study also noted that one outlet from each cone almost always yielded the maximum volume.

Teflon Cone

Capel and Nacionales (1995) examined the capability of the Teflon cone to reproducibly split water. They processed replicates of 0.6, 0.8, 1, 4, 6, 8, and 9 L of water through the cone splitter (see section "Concerns Related to Splitter Operation; Teflon Cone"). They combined the outlets into various configurations and processed replicate water samples (see section "Effects of Combining Ports from the Cone Splitter; Teflon Cone", below). Replicate samples of water were poured at one fixed location on the cone and from various sides of the cone. These variables had no significant effect on the cone's capability to split the water evenly. For all of these replicate experiments, they observed that the mean of the coefficients of variation ranged from 2.5 to 5.2. This is the same range observed for the plastic cone splitter.

Martin (1993, appendix B) found that the volumes of subsamples split in the field were somewhat less precise (fig. 3*A*). In 10 splits of a water sample with high sand content, the deviations from the mean volume averaged ± 6.8 percent with a coefficient of variation of 8.5 percent.

Splitting Capability as a Function of Particulate and "Total" Chemical Concentrations

There has been essentially no work performed to measure the capability of the various devices to split particulate or "total" chemical and biological constituents effectively. This issue is critical in describing their overall effectiveness. It is well known that many particle-associated chemical constituents, particularly trace elements and hydrophobic organic chemicals, are associated with the finer particles in aquatic systems. Also, many biological particles (living and dead) and, therefore, particulate carbon, nitrogen, and phosphorous are <63 µm. Since both types of splitters seem to split the finer particles effectively, it appears that the splitting of particulate-associated chemicals is probably just as effective. This has yet to be documented by experiment, however.

Splitting Capability as a Function of "Dissolved" Chemical Concentrations

There has been essentially no work done to measure the capability of the various devices to split dissolved constituents effectively, but of all of the factors listed in table 2, this is probably the least significant. From fundamental chemical principles, the splitting of a solution should not affect the concentration of dissolved constituents, other than the concerns specifically addressed in the last sections on creation of chemical artifacts. Supporting anecdotal data can be gleaned from Patton and Triutt (in press). In a preservation study of dissolved nutrients, they split samples 10 ways with a plastic cone splitter and found no statistical difference in the sample concentrations in splits held for various periods of time. This suggests that the splitting of the sample had no effect on the dissolved concentrations.

CONCERNS RELATED TO SPLITTER OPERATION

Use of Multiple Splitters of the Same Type by Same Operator

14-liter Churn

The Branch of Quality Assurance (1990, appendix B) evaluated the effectiveness of two different 14-L churns to split water samples containing sand-sized and fine particles. The results for total suspended sediment and fine and coarse particle concentration are shown in figure 3. With solution A (30 percent sand), there was a marked difference between the two churns, for both total and coarse particle concentrations. Concentrations in subsamples from churn 1 were nearly all below the expected concentration, while subsamples from churn 2 were all above. The deviations were due almost entirely to the deviations of the coarse particle concentrations, as the concentrations of the fine particles were nearly constant regardless of which churn was used (although lower than the expected concentration). A difference between the churns was not obvious with subsamples of solution B, in which the sand content of the sediment was only five percent. Unfortunately, records did not indicate whether the churns were operated by more than one person. Thus, it is not possible to rule out human factors as a possible explanation for the variation observed (which would be an indication of a separate problem).

8-liter Churn

No data has been found that addresses this topic specifically for the 8-L churn splitter.

Plastic Cone

Gray and deVries (1984) incorporated the cone section of the cone splitter in an autosampler and evaluated its splitting capability. They performed 10 tests on three different cones. They found that the volumes of water in subsamples from all three cones were between +3.5 and -4.2 percent of the true volume of water. They concluded that there was no statistically significant difference in the results from the three splitters.

The Branch of Quality Assurance (1992, appendix B) ran a series of tests on the plastic cone splitter. The Branch ran a total of five tests on two cones in which they added approximately two L of water, then measured the volume of water from each outlet. The two cones did not yield comparable results in terms of precision. One cone yielded coefficients of variation between splits of 2.6, 2.8, and 3.0 percent, whereas the other cone yielded results of 7.2 and 6.0 percent. The reason for these differences are not known. They could be due to leveling, different operator handling of the cone, or minor physical differences between the two cones. The investigators in this study also noted that one outlet from each cone almost always yielded a maximum volume compared with the other outlets.

Teflon Cone

In the study by Capel and Nacionales (1995), two Teflon cones were compared at one range of particle size (38 to 63 μm) and a particle concentration of 200 mg/L. The deviation from the mean suspended sediment concentration ranged from -6.8 to 7.7 percent for the first splitter and -9.5 to 10.4 percent for the second splitter. The coefficients of variation were 5.2 and 7.7 percent, respectively. The authors concluded that there was little systematic difference in the precision of the two splitters. They also observed no difference between the coefficients of variation for one splitter tested with solutions of silt and clay particles in three size ranges, and for another splitter tested with six silt and sand particle sizes. The results from these two cones agreed well. However, since these devices are individually machined, resulting in possible physical differences between individual cones, the degree to which this observation can be generalized for all Teflon cones is unknown.

Use of the Identical Splitter by Different Operators

Churn Splitters

Although there is no data found that specifically addresses this issue, it is worth noting that the capability of the churn to consistently split solids with equal results is very operator dependent. In early work on the development of the churn, Delaney and Ong (personal communication) write, "The sample is mixed with a rate of one round trip stroke per second when the tank volume is 10 - 14 L. As the tank volume decreases, the stroke rate should increase so that the paddle velocity remains the same. The paddle should touch the bottom of the tank on every stroke. Stroke length should be as long as possible without breaking the water surface. IMPORTANT: Failure to use a full stroke while mixing the sample can produce very erroneous results in the representative subsamples." Apparently from this work, the QWB technical memorandum 78.03 (appendix A) states that "tests have indicated that it is very important that a churning rate of about nine inches per second be established and maintained during the sample withdrawal procedure. When faster or slower churning rates were used, maximum errors of about ± 45 to ± 65 percent were observed, as compared with maximum errors of about ± 8 to ± 15 percent when using the nine in/sec rate." Such observations and warnings suggest that the results obtained from the churn could be extremely operator dependent.

Cone Splitters

No data has been found that addresses this topic specifically for the cone splitters.

Leveling Effects on Cone Splitter

Capel and Nacionales (1995) stated that "equal splitting of the water and solids is based on the premise that whatever falls on the center point of the cone has an equal chance of making it to any of the 10 outlet ports. If the cone splitter is not leveled, then the chance of the water or solids exiting from the down-side ports will be greater than the up-side ports and the cone will not work effectively." Some of the systematic variations from adjacent ports observed in the water and solids splitting capability may be due to artifacts created by a non-level cone splitter (QWB technical memorandum 80.17). Figure 10 illustrates the results of Gray and Ferguson (1990, appendix B) from tests on the water splitting capability of the plastic cone. It can be seen that the deviations from the mean volume show a consistent pattern, with all of the outlets showing a negative bias located adjacently and all of the ports showing a positive bias located adjacently. The quantitative effects of leveling on the splitting of water, chemicals, and solids need to be determined and written into future protocols on the use of the cone splitter.

Effects of Total Processed Water Volume

14-liter Churn

No data has been found that addresses this topic specifically for the 14-L churn splitter. It is common practice within the WRD to sample only several L and allow at least 4 L to remain in the churn unused.

8-liter Churn

No data has been found that addresses this topic specifically for the 8-L churn splitter.

Plastic Cone

One of the original motivations in the development of the cone splitter was the need to split small volumes of water (QWB technical memorandum 80.17, appendix A). Although no data were presented, it was assumed that the cone would yield precise results over a wide range of water volumes.

Durham and McKenzie (1985, appendix B) conducted a study to evaluate the plastic cone. Using distilled water, they conducted eleven tests with 2,500 mL, six tests with 250 mL, and four tests with 100 mL. The tests with 2,500 and 250 mL of water yielded similar results. All of the coefficients of variation between splits were in the range of 1.1 to 4.5 percent. Precision was much lower for the tests conducted with a volume of 100 mL. The coefficients of variation of these four tests were 2.2, 2.6, 25.9, and 29.0 percent. This would suggest that small volumes of water are not split as effectively in the cone as larger volumes (>250 mL).

Teflon Cone

Capel and Nacionales (1995) examined the effect of total water volume on the capability of the device to split water consistently with similar results. They processed replicates of 0.6, 0.8, 1, 4, 6, 8, and 9 L of water through the cone splitter and found that the mean coefficient of variation for all of the volumes were very similar (3.2 to 4.9 percent).

Effects of Sequential Aliquots from Churn Splitter

The Branch of Quality Assurance (1990, appendix B) evaluated the effectiveness of two 14-L churns to split water samples containing sand-sized and fine particles. The results for total suspended sediment, and fine and coarse particle concentration are shown in figure 3. Concentrations showed a statistically significant ($\alpha = 0.1$) negative trend with subsample withdrawal order in several of the trials, using both parametric regression and the non-parametric Kendall's tau test. For churn 2, both the total mass and the mass of coarse particles decreased with successive withdrawals for both sediment mixtures used. For churn 1, concentrations of coarse particles in subsamples of solution

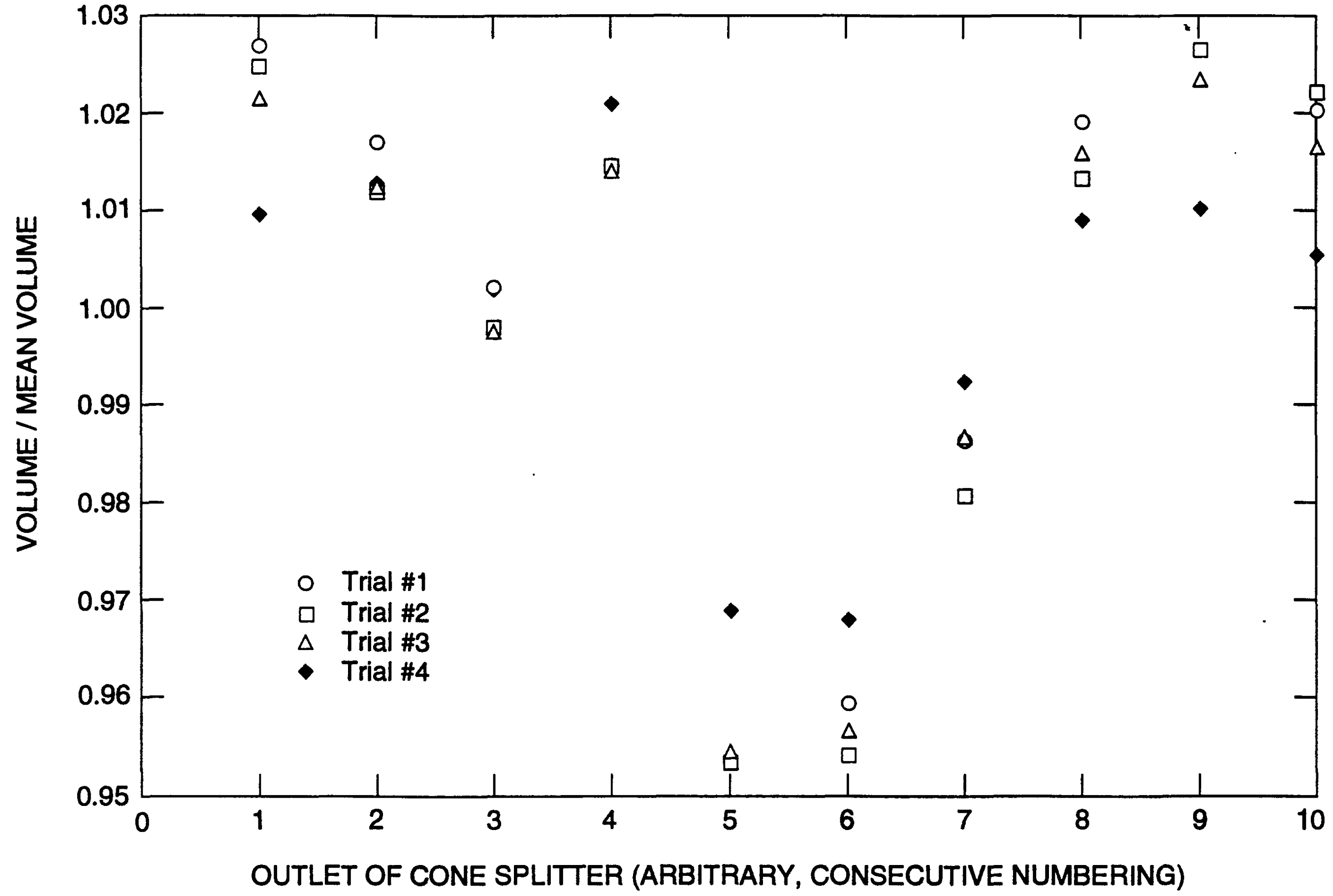

Figure 10. Relative volumes of water between the ten splits from a plastic cone for four trial runs.

B (five percent sand) showed a significant negative trend with withdrawal order in one of two trials. To confound matters, subsamples from churn 1, solution A, had increased concentrations of both total and coarse particles, although the trend was not quite statistically significant ($p = 0.13$). There was no noticeable trend in fine particle concentrations with either churn, implying that the observed trends in total concentrations were primarily due to coarse particles.

Effects of Combining Ports from the Cone Splitter

The idea that tubes from more than one port may be directed to one sample bottle was originally suggested in the first QWB technical memorandum (80.17) on the cone splitter. This procedure has been subsequently repeated in a WRD field protocol manual (Ward and Harr, 1990). Although no early data has been found to verify this practice, the precaution that there must be no additional back-pressure resulting from restriction of flow is always given.

Plastic Cone

No data was found that examined the effect of combining the ports on the plastic cone splitter, but from the data discussed in the following section for the Teflon cone, it can be assumed to have little effect on the volume of splits.

Teflon Cone

Capel and Nacionales (1995) examined the effect of combining ports. They combined the 10 ports to produce an eight-way split (combined two sets of two ports), a five–way split (combined five sets of two ports), and a three-way split (combined two sets of three ports and one set of four ports.) They reported that the coefficients of variation between volumes of water in the splits were no different when ports were combined compared to when ports were not combined. The effect of combined ports on the splitting of solids was not examined.

CONCERNS RELATED TO INHERENT CREATION OF CHEMICAL ARTIFACTS

Creation of Artifacts in Particle Size Distribution

Particles often flocculate and coagulate in natural waters. The turbulence within the splitters, especially the churn splitters, could disrupt these aggregates and create an artificial particle size distribution. This has not been addressed in any studies reviewed here. From the study by the South Platte NAWQA (1993, appendix B, figure 7), the agreement between the traditional- and cone-processed samples suggests that this may not be a significant problem from mass considerations. The major implications of the disruption of natural flocs would be reflected in the potential changes in water chemistry, particularly in the "dissolved" and "particulate" distributions of chemical constituents.

Gain/Loss of Chemical Due to Air/Water Transfer

In all of the devices, the water sample is mixed and exposed to the atmosphere. From the early documents (QWB technical memorandum 78.03, appendix A), it was advised that dissolved oxygen measurements should not be made on a churned sample because of the possible addition of oxygen to the water. There has also been an undocumented rule that water samples that are to be analyzed for volatile organic chemicals should not be processed through cone or churn splitters to avoid loss of the chemicals from the water. The potential for loss of organic chemicals from splitter-processed water may also apply to semi-volatile compounds with relatively high Henry's Law constants ($> 10^{-5}$ atmosphere-meter^3/mole; Thomas, 1990). This group would include many of the polychlorinated biphenyl congeners and some of the polyaromatic hydrocarbons, as well as elemental mercury. This potential problem has not been previously reported.

SUMMARY OF THE INTERPRETATION OF EXISTING DATA

14-liter Churn

The existing information on the 14-L churn splitter does not provide a firm understanding of any aspect of its evaluation. There is a strong indication that it can be adequately cleaned in the laboratory for µg/L concentrations of dissolved inorganic constituents. There is an even stronger indication that it will not adequately process sand-sized particles to yield representative subsamples, but it seems to be able to process finer particles with much less error.

Many questions remain unanswered on the overall usefulness of the 14-L churn splitter. The capability to be cleaned adequately and consistently in the field and the capability to be cleaned for particulate and "total" chemical constituents must be addressed further. In terms of splitting capability, tests need to be done with environmentally reasonable particle concentrations for a number of different churns by a number of different operators. The questions regarding introduction of chemical artifacts during processing also remain unanswered.

The 14-L churn has gained widespread popularity in the WRD due to ease of use and versatility. It appears, from the available data, that this churn is capable of processing fine (silt and clay) particles and, most likely, dissolved constituents to yield representative subsamples.

Limitations on the usefulness of the 14-L churn include its lack of producing equal results during splitting and its inadequacy in processing sand-sized particles, including chemical constituents associated with these particles. Due to its construction material, it is limited to processing water to be analyzed for inorganic constituents. It cannot be used for water that will be analyzed for organic chemicals, including dissolved and particulate organic carbon. The size of the 14-L churn makes it difficult to sterilize, limiting its use for bacterial analysis as well. The size of the churn also limits the composited sample that can be collected for water quality analyses to about 8 to 10 L.

8-liter Churn

The 8-L churn, like the 14-L churn, has gained widespread popularity in the WRD due to ease of use and versatility. There were no studies reviewed in this report that evaluated the 8-L churn. All information presented here is extrapolated from findings on the 14-L churn. For some aspects, such as cleaning efficiency, this approach is probably adequate. For concerns about the solids processing capabilities, however, extrapolation would be very tenuous due to the different geometries of the two devices. All aspects of the capabilities of the 8-L churn seem to be wide open to questions. The limitations on the use of the 8-L churn include all of the items mentioned for the 14-L churn and probably other items that are presently unidentified due to lack of data.

Plastic Cone

The existing information on the plastic cone splitter gives a better understanding of its range of usefulness than does existing information on the churn splitters. Many tests by numerous investigators discussed earlier indicate that the plastic cone, when used in a laboratory setting, can produce precise and accurate subsamples in terms of water volume and suspended solids concentration. The plastic cone works about as well for sand-sized particles as it does for finer particles.

Many questions remain unanswered and must be addressed before an overall evaluation can be made. There have been no studies that evaluated the cleaning of the plastic cone for low-level (μg/L) concentrations of trace elements and nutrients. Field tests of the plastic cone are also lacking. Since leveling of the cone is an important factor in its overall performance, the issue of leveling in the field must be thoroughly addressed.

The plastic cone has gained only limited popularity in the WRD due to its awkwardness in the field. In general, however, the plastic cone seems to yield superior subsamples over a broad range of particle sizes, compared to the churn splitters.

Limitations of the plastic cone include its awkwardness when used in the field and its unsuitability for water samples that are analyzed for organic chemical constituents. From the data reviewed in this report, it appears that the plastic cone has the same splitting capability as its Teflon counterpart, but with less versatility with respect to the number of analytes for which it can be used.

Teflon Cone

The existing information on the Teflon cone is perhaps the most extensive of the devices reviewed here. When used in a laboratory setting, the Teflon cone can yield precise and accurate subsamples in terms of water volume and suspended solids concentrations. The Teflon cone seems to work well for particles ranging in size from very fine clay and silt (1 to 10 μm) to medium coarse sand (125 μm), but it loses its precision when used with larger particles. Early results indicate that it can be adequately cleaned in the laboratory for both trace organic chemicals and trace elements. However, many important questions remain unanswered, both in field use (see the previous section on the plastic cone) and laboratory and field cleaning.

The Teflon cone has gained wide popularity within the WRD for research that involves analysis of water samples for organic chemicals. Because of its construction material, the Teflon cone is the only one of the four splitting devices that is able to process water samples for the complete suite of chemical constituents (except volatile organic chemicals). In all other aspects, the Teflon cone appears to yield results comparable to those of the plastic cone.

Limitations on the usefulness of the Teflon cone are largely due to its awkwardness when used in the field. To obtain numerous water samples of varying sizes from one site, sampling personnel often composite water samples in a vessel first, or repeatedly pour samples through the cone. Both of these procedures are potential sources of chemical and biological contamination and may introduce errors in the splitting of solids. The details of the field cleaning procedure and use of the Teflon cone must be examined and a more precise protocol written and adhered to before this device can be fully evaluated.

CONCLUSIONS

The proper evaluation of the water sample splitting devices encompasses a wide variety of concerns, both chemical and physical. This report attempts to survey and interpret existing data and present it in a systematic framework. From the existing data, a few of the concerns are adequately addressed, a few more are partially addressed, many more can be estimated by extrapolation, but the majority of the concerns cannot be addressed in this report. The conclusions in this report summarize and review the existing information in a useable manner. Based on this review, however, it appears that new studies are definitely required to fully evaluate the splitting capability (water, solids, and chemicals) of the four devices and to address the issues of contamination and cleaning.

In general, the existing cleaning and transport protocols are adequate at the mg/L level, but the adequacy is largely unknown for trace elements and organic chemicals at lower concentrations. The existing data suggests that better results are obtained when the splitters are cleaned in the laboratory rather than in the field. Preliminary data indicate that the Teflon cone can be adequately cleaned for both trace elements and organic chemicals.

The splitting capability for solids is ultimately the most important concern from both a physical and chemical (particulate and "total") perspective. If natural variability among replicate samples for concentration of suspended sand is about 10 percent, and of suspended finer materials is about 3 percent, then most of the errors observed in the laboratory evaluations for both the churn and cone splitters are encompassed by or close to this natural variation, with the exception of sand in the churn splitter. The errors associated with the field tests are commonly greater than the errors in the controlled laboratory tests. Two conclusions that can be reached on the splitting capability of solids are that more work must be done on all four devices to characterize and quantify their limitations and ranges of usefulness, and that the 14-L churn (and by association, the 8-L churn) is not useful in obtaining representative splits of sand-sized particles.

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APPENDIX

APPENDIX A: U.S. GEOLOGICAL SURVEY TECHNICAL MEMORANDA (QWB, QUALITY OF WATER BRANCH; OSW, OFFICE OF SURFACE WATER; OWQ, OFFICE OF WATER QUALITY) AND UNPUBLISHED PROTOCOLS

United States Department of the Interior

GEOLOGICAL SURVEY
RESTON, VIRGINIA 22092

4351 6016
QW Branch

May 12, 1976

QUALITY OF WATER BRANCH TECHNICAL MEMORANDUM NO. 76.17

Subject: WATER QUALITY--Sampling mixtures of water and suspended sediment in streams

As a result of District reviews, the Quality of Water Branch has become more aware that principles and methods for sampling mixtures of water and suspended sediment in streams are not well understood and, therefore, are not always followed properly. Although proper methods generally are used in collecting representative samples for determination of suspended-sediment concentration and particle-size distribution, they often are ignored when it comes to collecting representative samples for chemical analyses of mixtures of water and suspended sediment (so-called whole-water, or unfiltered samples).

Several districts have issued field instructions dealing with methods for collecting samples for analysis of mixtures of water and suspended sediment. The subject will be covered fully in a TWRI chapter on collection of samples for water-quality determinations (now in preparation). In the meantime, I ask that all Districts review their field instructions and practices to see that they conform to the following guidelines.

PRINCIPLE

All samples to be analyzed for determination of "total" concentrations of constituents in the mixture of water and suspended sediment are to be collected in such a manner that they will best represent the water being transported by the stream.

METHODS WHERE SAND-SIZE SEDIMENTS ARE IN TRANSPORT

When turbulence and mean stream velocities are great enough to support the transport of sand in suspension [generally greater than about 2 ft/s (0.6m/s)], sampling for mixtures of water and suspended sediment must be done using appropriate sediment-sampling equipment and techniques. In descending order of accuracy, these methods are:

1. Equal-transit-rate (ETR),
2. Equal-discharge-increment (EDI),
3. Multi-vertical, and
4. Single-vertical.

Equal-transit-rate method.--*This method yields the most accurate sample of the streamflow.* It requires the collection of depth-integrated samples at 10 to 25 verticals in the cross section (F.I.A.S.P., 1963, p. 41). Guy and Norman (1970, p. 40) discuss this method and the conditions under which fewer than ten verticals may be sampled. For example, for smaller streams or when the ratio of velocity squared to depth, V^2/D, is less than about 1.2 ft/s^2 (0.37 m/s^2) as few as three verticals may yield an acceptable sample.

Equal-discharge-increment-method.--The EDI method is more complex, and generally requires a better knowledge of the flow conditions prior to sampling. Two to ten verticals generally are sampled in this method (F.I.A.S.P., 1963, p. 41). Guy and Norman (1970, p. 31-32) describe in detail how centroids of equal-discharge increments of flow may be determined. *Sampling by this method may yield samples equal in accuracy to the ETR method if samples of equal volume are collected at each centroid of flow.*

Multi-vertical method.--The basis for selection of the number of verticals to be sampled is mainly intuition. Generally, two to five depth-integrated samples are collected in the cross section--usually, only three verticals. Discussion and guidelines for this method are found in the manual by Guy and Norman (1970, p. 30) and in F.I.A.S.P. (1963, p. 39-40) Report 14.

Single vertical method.--Next to automatic pumping-type samplers, this method is the least accurate. It is the last resort, but its use may be necessary under extreme conditions such as rapidly changing stage. Guy and Norman (1970, p. 27-30) discuss this method and its many shortcomings in relation to the more accurate ETR and EDI methods for sampling of streamflow transporting sand-size sediments.

Remember that standard depth-integrating sediment samplers should not be used to sample depths greater than about 15 ft (4.6 m) (Guy and Norman, 1970, p. 24; F.I.A.S.P., 1963, p. 44). Point-integrating samplers should be used to sample depths greater than 15 ft (4.6 m). *All district water-quality specialists should be thoroughly familiar with the two references referred to in this discussion.*

CONDITIONS OF LOW VELOCITY

When mean stream velocities are low [less than about 2 ft/s (0.6m/s)] and the flow is tranqil, generally only fine silt- and clay-sized particles are in suspension, and sediment concentrations do not vary greatly either vertically or laterally. Furthermore, standard suspended-sediment samplers do not fill properly at velocities less than about 1.5 ft/s (0.5m/s) (OWDC, p. III-18). When such conditions are documented at a site by discharge measurements, it usually is acceptable to collect depth-integrated samples using open-mouth bottles. The open-mouth bottle commonly used is a narrow-mouth bottle, usually one litre or more in size. It should be weighted so that it will sink readily to the bottom, taking in sample on the trip from the surface to near the bottom and back to the surface (Brown and others, 1970, fig. 2; Beam, 1973). There still is a need for a more suitable sampler for deep, slowly moving rivers.

Open-mouth bottles used as samplers should be filled by lowering and raising at several verticals in the cross-section in order to best sample the vertical and lateral variations in water quality that frequently exist in slowly moving waters. Here again, the number of verticals sampled is largely a matter of intuition, realizing that large variations in the water quality in the cross section will require sampling at more verticals than if little variation exists.

COMPOSITING

Samples from several verticals should be composited and then split into fractions for various types of field treatment and for different laboratory analyses. This is easily accomplished using a large clean jug or bottle. *Care should be taken to try to assure uniform mixing and withdrawal of representative aliquots.* A new churn-type sample splitter has been developed, tested, and ordered; a limited number will be available to field offices within the next few months. *Samples for bacterial determination or for analysis of sediment concentration and particle size analyses should* <u>*not*</u> *be composited,* but should be left in the original collecting bottles. Obviously, samples for chemical analyses should not be composited if separate analyses at different points or verticals in the cross-section are desired.

SAMPLING FOR DETERMINATION OF PESTICIDES

Special 1-pint, borosilicate, screw-cap, glass bottles must be used to collect samples of mixtures of water and suspended sediment for determination of pesticides and organics. These bottles are streamlined versions of the standard sediment (milk) bottles. *Use only freshly cleaned bottles that have been supplied by the Central Laboratory or EPA pesticide laboratory.* These bottles are to be used in standard depth-integrating or point-integrating suspended-sediment samplers fitted with metal or teflon/nylon nozzles and silicone rubber gaskets. Bottles are to be filled to the shoulders (standard sediment practice; Guy and Norman, 1970, p. 28-29) *and are not to be composited or transferred.* The bottles should be labeled to indicate the vertical (station) in the cross-section where the sample was collected. For low-velocity streams where open-mouth sampling can be conducted, samples may be collected as described in the earlier section of this memorandum on "conditions of low velocity." Care should be taken to avoid touching the lip of the sample container, and to collect the sample upstream from the body when wading.

A NOTE REGARDING FILTERED SAMPLES

Samples for determinations of "dissolved" constituents are filtered in the field. Usually it is assumed that solutes are well mixed throughout a cross-section, and therefore, require somewhat less care in sampling than do mixtures of water and suspended sediment. However, non-uniform chemical quality frequently exists in places such as estuaries, slack-water pools, below dams, and below tributaries, and *an assumption of a well mixed system should be used with caution.* As a general rule, unless one has measurements to show that chemical quality of the constituents being sampled is relatively uniform (range in variation of specific conductance no more than 10 percent) throughout a cross-section for flow conditions experienced, it is best to use caution and apply the same practices that are recommended for mixtures of water and suspended sediment.

CHOICES OF CONTAINER MATERIALS

Instructions issued by the Central Laboratories ("Central Laboratory Parameter List") specify the types of containers to be used for the shipment of samples to the laboratories. The types of container materials specified are intended to prevent contamination of the samples by their shipping containers. In general, specifications call for the use of containers made of materials different from those of the particular constituent for which the lab is to analyze. For example, plastics are used for inorganic samples (special acid rinsing for trace metals), and glass is used for organics.

These same principles of selection of non-contaminating materials apply also to every piece of equipment that touches the sample, including the sampler nozzle and gasket, and the compositing container. Plastic (teflon or nylon) nozzles should be used when sampling for determination of inorganics; metal nozzles are preferable for determination of organics, although contamination from teflon or nylon probably is minimal if the nozzles have been carefully cleaned. Care should be taken to use silicone rubber gaskets when sampling for determination of trace metals or organics. Samples collected for *inorganic analyses* should be composited in *clean plastic containers* (acid-rinsed for trace metals). Samples collected for *organic analyses* should be composited in *clean glass containers*.

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COMMENTS INVITED

I hope that every District will review the practices being used by all of its field people, and will bring this memorandum to the attention of every field person. Your comments regarding the recommendations and instructions contained in this memorandum are invited. Please contact your Regional Water Quality Specialist or write to me through him.

Ronald J. Pickering Jr.

R. J. Pickering
Chief, Quality of Water Branch

WRD Distribution: A,B,S,FO,PO

United States Department of the Interior
GEOLOGICAL SURVEY
RESTON, VIRGINIA 22092

4351-6016
QW Branch

August 16, 1976

QUALITY OF WATER BRANCH TECHNICAL MEMORANDUM NO. 76.24-T

Subject: EQUIPMENT & SUPPLIES: Sample Splitter for Water-Sediment Samples

Within the next few months, all offices that are involved in the collection of water-quality samples under the NASQAN program will be receiving a new type of sample splitter to be used on site to split samples into the various containers required by the Central Laboratories system. The attachment to this memorandum describes the USGS Churn Splitter and presents procedures to be followed in splitting composited samples.

Research and development of this splitter was carried out for the Quality of Water Branch by Jack Dewey, assisted by Bruce Delaney and Kim Ong of the New Mexico District. Experiments have shown that if proper procedures are followed splits of samples containing up to 5000 mg/l sand (coarser than 0.062 mm) can be obtained to within ± 10% of the true concentration.

Splitters are being furnished at this time for use at all NASQAN sites; however, we recommend that they also be used whenever water-sediment sample splitting of large composit samples is required in the field or laboratory. We plan to have smaller versions (2 gallon or smaller) available within the next few months.

Those who receive these splitters will find a copy of the instructions inside. After delivery of the instructions to the manufacturer, however, we revised the 2nd paragraph of page 2 and the spelling of "liter."

Because this is the first model, we will welcome comments regarding its use and/or improvement.

for R. J. Pickering
Chief, Quality of Water Branch

Attachment

WRD Distribution: A, B, FO, PO

Procedures for Subsampling Water-Sediment Mixtures
(14-Liter Churn Splitter)

General - The water-quality laboratory may require 4 to 16 subsamples of a representative cross-section sample of the water-sediment mixture (streamflow) for water-quality analyses. The cross-section sample is collected in 1-pint or 1-quart bottles using suspended-sediment samplers at no fewer than three and preferably eight to ten verticals (ETR or EDI techniques). These samples are composited into one single representative cross-section sample of the streamflow. This composited sample can then be split, using the churn splitter, into the required 4 to 16 representative subsamples as exaplined under Procedure. *Samples collected for organic analyses (e.g. organic carbon, pesticides) should not be composited in this container becuase of the possibility of contamination from the plastic.*

14-Liter churn splitter - The churn splitter is a 1/4 inch thick polyethylene cylinder, 10 inches in diameter and 12 inches deep with a lid. It has been manufactured for the Survey by a commercial manufacturer. The valve and spout are polypropylene. The stirring disc is a 3/8 inch thick polyethylene disk, 9-15/16 inches in diameter with 16 holes, 8 as scallops in the outer edge and 8 in an inner circle. The handle, a 1-inch diameter by 18-inch long polyethylene rod, is welded perpendicular to the center of the disk and supported by four ribs. A small "lip" on the disc aligns with the valve, and a guide notch and rib are provided to maintain the correct alignment. Replacement valves and spouts are available from the Quality of Water Branch.

Procedure - This procedure requires a total sample volume of 8 to 14 liters, of which 4 to 10 liters are suitable for water-sediment mixture subsamples. The remaining 4 or more liters may be used for filtered subsamples if required by the analytical schedule. If not, they may be discarded. *This size churn splitter does not reliably produce representative water-sediment mixture subsamples when it contains less than about 4 liters.*

Before starting to collect the representative sample of the streamflow, label all the subsample containers to be used and determine the total sample volume needed. Add to this sample volume at least 10% to cover filter losses and spillage. It is less frustating to throw away a small amount of sample than to have to go back and collect another cross-section sample.

Collect approximately one liter of water and thoroughly rinse the churn splitter.

Representative samples of the streamflow are collected by using standard EDI or ETR sampling techniques as described in "Field Methods for Measurement of Fluvial Sediment" TRI Book 3, Chapter C2. Specific sample volumes cannot be obtained with sediment samplers, but properly collected pint bottles (approximately two-thirds full) will yield about 1/3 liter each. Only one sediment sample bottle is used over and over again in collecting the cross-section samples in order to minimize the amount of sediment lost in transferring samples from the bottles to the churn splitter. Each time the bottle is filled, the sample is poured into the splitter and the bottle is used again so that each succeeding sample washes the sediment left from the previous one into the splitter. Remember that the volume to be used for water-sediment mixture subsamples must be "on top of" the 4 liters of sample in the tank from which representative water-sediment mixture subsamples cannot be obtained.

Suspended-sediment concentration should always be determined whenever a sample is analyzed for total concentrations of chemical constituents. The sample for determination of suspended-sediment concentration can be collected (1) as a separate cross-section sample as if no other sampling were required, or (2) it may be obtained as a single-bottle subsample from the churn splitter if the amount of sediment in a single bottle appears to be sufficient for the lab to obtain accurate weights of both the fine and sand fractions. The fieldman can decide whether (1) or (2) will be used by looking at the first bottle collected to see whether an appreciable amount of sand settles to the bottom of the bottle within 20 to 30 seconds; if so, (2) can be used. If in doubt, always use alternative (1).

When the required volume plus 10% for waste is in the churn splitter, place all water-sediment mixture subsample containers within easy reach, so that once started the stirring can be continuous. The sample should be stirred *at a uniform rate of approximately nine (9) inches per second.* As the volume of sample in the tank decreases the round trip frequency should increase so that the churning disc velocity remains the same. The *disc should touch the bottom of the tank on every stroke,* and the stroke length should be as long as possible *without breaking the water surface.* Before using the sample splitter for first time, practice this stroke using tap water. Observe that, as the stroke length and/or disc velocity is increased beyond the recommended rate, there is a sudden change of sound and churning effort which is accompanied by the introduction of excessive air into the mixture. The introduction of excessive air into the sample is undesirable because it may tend to change the dissolved gases, bicarbonate, pH, and other characteristics. On the other hand, inadequate stirring may result in non-representative subsamples.

The sample in the splitter should be stirred at the uniform churning rate for about 10 strokes prior to the first withdrawal to establish the desired stirring rate of nine (9) inches per second and to assure uniform dispersion of the suspended matter. The churning must be continuous during the withdrawals; therefore, if a break in withdrawals is necessary, the stirring rate must be reestablished before continuing the withdrawals.

When all of the required water-sediment mixture subsamples have been obtained, the remaining portion of the sample is used as necessary for the filtered samples. It will be advantageous to allow the sediment to settle out in the mixing tank for a few minutes before pouring the sample into the filter apparatus. When all of the necessary filtered subsamples have been obtained, the mixing tank, churning disc and filter apparatus should be cleaned thoroughly with deionized water. If deionized water will not remove all of the residue, clean by using a small amount of a detergent such as Alconox, rinse with a weak acid solution (4 ml of nitric acid per liter of water), rinse repeatedly with tap water, and then rinse with deionized water.

Equipment not furnished - 1. A stand to support the mixing tank that will allow the subsample containers to be placed under the spout.
2. Small supports to hold various sizes of subsample containers such that the top or opening is at or near the mixing tank spout.

NOTE: When used in compositing samples collected from a bridge or roadway the lid should be kept on at all times except when pouring sample, in order to protect sample from dust contamination.

United States Department of the Interior

GEOLOGICAL SURVEY
RESTON, VIRGINIA 22092

4351-6016
QW Branch

December 13, 1976

QUALITY OF WATER BRANCH TECHNICAL MEMORANDUM NO. 77.01

Subject: EQUIPMENT AND SUPPLIES: Sample Splitter for Water-Sediment Samples

Recently, new USGS Churn Splitters were distributed to WRD field offices on the basis of one splitter to each office that operates NASQAN stations. Unfortunately, we underestimated the count and were about six short; the shorted offices will be supplied as soon as more splitters are received. Districts requiring additional splitters should contact the Branch.

INSTRUCTIONS FOR USE OF SPLITTERS

A set of "Procedures" was included with each splitter. These are the same instructions that were distributed to all field offices with QW Branch Memo 76.24-T on August 16, 1976. Also, please see QW Branch Memo 76.17 (May 12, 1976) for general instructions on sampling mixtures of water and suspended sediment. The QW Branch can provide additional copies of both memos if they are needed.

CLEANING

The Churn Splitter should be washed with a quality laboratory detergent and thoroughly rinsed with tap water followed by distilled water prior to taking it into the field. In addition, churns used for the splitting of trace metal samples should be acid washed--let soak for four hours in a 5% solution of hydrochloric acid, then rinse with tap water and distilled water. Cleaning between station visits can be accomplished by rinsing with distilled water after sampling. Then, rinse with sample water before use.

WHEN TO USE CHURN SPLITTERS

Use of the Churn Splitter is not limited to samples collected at NASQAN sites. All samples to be used for analysis of "total" constituents are to be collected from multiple verticals to ensure representativeness of the flow in the cross section (QW Branch Memo 76.17). Also, samples for "dissolved" analyses should be collected by the same means unless cross-sectional uniformity is documented (QW Branch Memo 76.17, p. 4). The Churn Splitter is designed and tested especially for use in streams transporting sand-size sediments. Other compositing containers such as jugs can be used satisfactorily for waters containing only fine materials; however, *it is recommended that only the Churn Splitters be used because they can be cleaned much more easily than can jugs or other similarly shaped containers.* Samples may be taken from the Churn Splitter for analysis of all dissolved and suspended inorganic constituents, including trace metals, and phytoplankton analysis.

EXCEPTIONS

A statement on pages 4 and 5 of QW Branch Memo 76.17 deals with the choice of container materials. The following point is emphasized in the instructions for use of the Churn Splitter. *THE CHURN SPLITTERS ARE MADE OF PLASTIC, AND THEREFORE, SHOULD NOT BE USED IN COMPOSITING SAMPLES FOR ANALYSES OF ORGANIC SUBSTANCES.* Bacteria samples are not to be taken from the Churn Splitter because it cannot be sterilized adequately.

SUMMARY

The new USGS Churn Splitter, designed and carefully tested for use in the field, is particularly well suited for splitting large composite samples of water-sediment mixture into subsamples of any desired volume. We believe that they are the best splitters available for these mixtures, and they are the only acceptable means of splitting samples containing sand-size materials in the field. The Jones Ore Splitter is still the most accurate, of course, for use in the laboratory to split a sample or subsample into equal volumes.

R. J. Pickering
Chief, Quality of Water Branch

WRD Distribution: A, B, FO, PO

United States Department of the Interior

GEOLOGICAL SURVEY
RESTON, VIRGINIA 22092

4351 6016
QW Branch

January 17, 1978

QUALITY OF WATER BRANCH TECHNICAL MEMORANDUM NO. 78.03

Subject: EQUIPMENT & SUPPLIES -- Churn Splitters

The USGS churn splitter, first introduced in Quality of Water Branch Technical Memo 76.24T, has been in use by our field offices for about a year. Since its introduction, several questions have arisen about its operation. This memo is intended to answer most of those questions.

Question: What is the purpose of the churn splitter?

The churn splitter was designed to facilitate the withdrawal of a representative subsample from a large composite sample of a water-sediment mixture. For example, samples from several verticals in a stream cross section, differing slightly from each other in chemical quality and sediment concentration, can be placed in the churn and be mixed into a relatively homogenous suspension. Theoretically, any subsample withdrawn from the churn should be equal in chemical quality and sediment concentration to any other subsample from the churn.

Question: When should the churn splitters be used?

The churn splitter was designed to be used for compositing and subsampling of chemical-quality samples that are to be analyzed for "total" or "total recoverable" inorganic constituents. Currently, the Central Laboratory Quality Assurance Section is evaluating the churn splitter to determine if it also can be used for organic carbon, radiochemical, or pesticide residue samples. The results of their evaluation will be announced in a later memorandum. Meanwhile, subsamples should not be taken from the churn splitter for determination of organic carbon, radiochemicals, pesticide residues, oil and grease, bacteria, or other constituents that require special handling.

The use of the churn splitter should not be considered to be limited to NASQAN stations alone. The splitter can and should be used at any chemical-quality sampling site where subsampling of a composite water-sediment sample is required. In addition, the churn splitter can be used for subsampling composited surface-water or ground-water samples that are to be filtered and analyzed for "dissolved" constituents.

Question: Are there any limitations to using the churn splitter?

Yes. Tests of the churn splitter using sand-size particles (>0.062 mm) have indicated that when relatively high concentrations of such particles are present, subsamples for suspended-sediment concentration or particle-size determinations should not be taken from the churn. Those samples should be collected in separate bottles directly from the stream. When essentially all particles are silt-size or smaller (<0.062 mm), sediment concentration and particle size subsamples may be taken directly from the churn.

Question: How critical are the operational procedures?

Tests have indicated that it is very important that a churning rate of about 9 inches per second be established and maintained during the sample withdrawal procedure. When faster or slower churning rates were used, maximum errors of about ±45 to ±65% were observed, as compared with errors of about ±8 to ±15% when using the 9 in/sec rate. It is also important that the churn disc not break the water surface during mixing of the sample because this would aerate the sample and could cause chemical changes that would result in the sample no longer being representative of the water in the stream.

Question: Can subsamples for measuring pH, bicarbonate and carbonate, dissolved oxygen, specific conductance, and temperature be taken from the churn splitter?

In-stream measurements of these characteristics (except biocarbonate and carbonate) are preferred and recommended. If this is impractical, pH, specific conductance, bicarbonate and carbonate may be measured on a subsample taken from the churn before any other subsamples are withdrawn. However, the proper churning rate must be established before taking the subsample for these measurements in order to assure that a representative mixture remains in the churn for further sampling. Dissolved oxygen and temperature should never be measured on subsamples from the churn splitter.

Question: Is there any preferred order in which subsamples should be withdrawn?

1. Withdraw subsamples for "total", "total recoverable" or "suspended" determinations first.

2. The first subsample withdrawn should be the largest subsample required (usually 1-liter) of the water-sediment mixture.

United States Department of the Interior

GEOLOGICAL SURVEY
RESTON, VA. 22092

In Reply Refer To:
EGS-Mail Stop 412

July 3, 1980

QUALITY OF WATER BRANCH TECHNICAL MEMORANDUM NO. 80.17

Subject: EQUIPMENT AND SUPPLIES--New sample splitter for water-quality samples

Bob Middelburg of the Quality of Water Branch has developed a new sample splitter called a cone splitter. The cone splitter divides a water sample or sample of a water-sediment mixture into ten equal parts. The cone splitter was originally developed for the Urban Hydrology Studies Program for use in splitting samples taken with an automatic pumping sampler for analysis of chemical and physical constituents. In addition, the cone splitter will be quite useful in compositing proper proportions of several samples taken throughout a runoff event into a single discharge-weighted sample that represents the flow event.

Enclosed for your information is an article describing the cone splitter; its accuracy, application, and procedure for use. The differences in application between the cone splitter and the churn splitter are noted in the article. Cone splitters are currently being distributed to all projects that are part of the Urban Hydrology Studies Program. Further information regarding the use or availability of the cone splitter can be obtained by calling Bob Middelburg, Quality of Water Branch, FTS 928-6834.

R. J. Pickering
Chief, Quality of Water Branch

Enclosure

Distribution: A, B, S, FO, PO

Key Words: Water quality, instrumentation, subsampling, sample splitting
Superseded memoranda: None

One Hundred Years of Earth Science in the Public Service

The USGS Cone Splitter

Sampling methods have been developed that produce samples that are representative of flow through a cross-section. These methods frequently conclude with one bulk volume of water-sediment mixture. Unfortunately, preservation techniques and analytical methods do not always allow the submission of one sample in a single container to the laboratory for analysis. The sample must be subdivided, usually within a short time after collection, into a number of subsamples each of which must be virtually equivalent in concentration of suspended and dissolved constituents.

The USGS churn splitter can be used to subsample a very large volume (8-12 liters) sample collected for chemical analysis. It allows obtaining different subsample volumes from the sample while still maintaining the same basic chemical and physical properties of the original sample. The churn splitter has proven to be an invaluable tool for the collection and processing of composited cross-section samples from rivers and streams. The major disadvantages of the churn splitter are 1) sample volumes less than about 6 liters cannot be split, and 2) inorganic sediments coarser than 62 um cannot be split with an accuracy of less than about ± 10-15%.

The recent use of automatic samplers has introduced a problem that makes the use of the churn splitter impractical. Automatic samplers usually collect relatively fixed sample volumes, most of which are at or below the minimum volume of water required for proper operation of the smallest available churn splitter. Most automatic samplers collect only between 0.5 to 3 liters in one sampling cycle, which is not enough volume for proper use of a churn splitter.

The new cone splitter was first developed in December 1979 as a means to reliably subsample samples collected for the Urban Hydrology Studies Program conducted by the U.S. Geological Survey in cooperation with the U.S. Environmental Protection Agency. Tests have shown that the cone splitter can split samples as small as 250 mL volume into 10 equal subsamples, each subsample being with ± 3 percent of the correct volume and sediment concentration.

Description of Cone Splitter

The cone splitter illustrated in Figure 1 is a pour-through device. A funnel-shaped reservoir on the top receives the sample and directs it into the splitting chamber. Located in the reservoir funnel is a 2 mm-mesh screen which retains large debris such as leaves that could clog or interfere with the splitting process. The screen reduces the vortex action of the water leaving the funnel and also helps mix the sample.

Below the funnel is a short section of stand pipe. Its function is to direct water as a steady stream into the splitting chamber which contains a cone-shaped splitting head.

The cone splitter housing is machined from a solid block of Lucite or comparable material. Ten exit ports have been precisely drilled through one common point at a 45-degree angle from the vertical and spaced at 36-degree intervals around the circumference. The resultant configuration in the splitting chamber is a notched cone with 10 equally spaced exit ports about its base. There are no flat walls, benches, or surfaces inside the splitting chamber that can retain material or interfere with the splitting process.

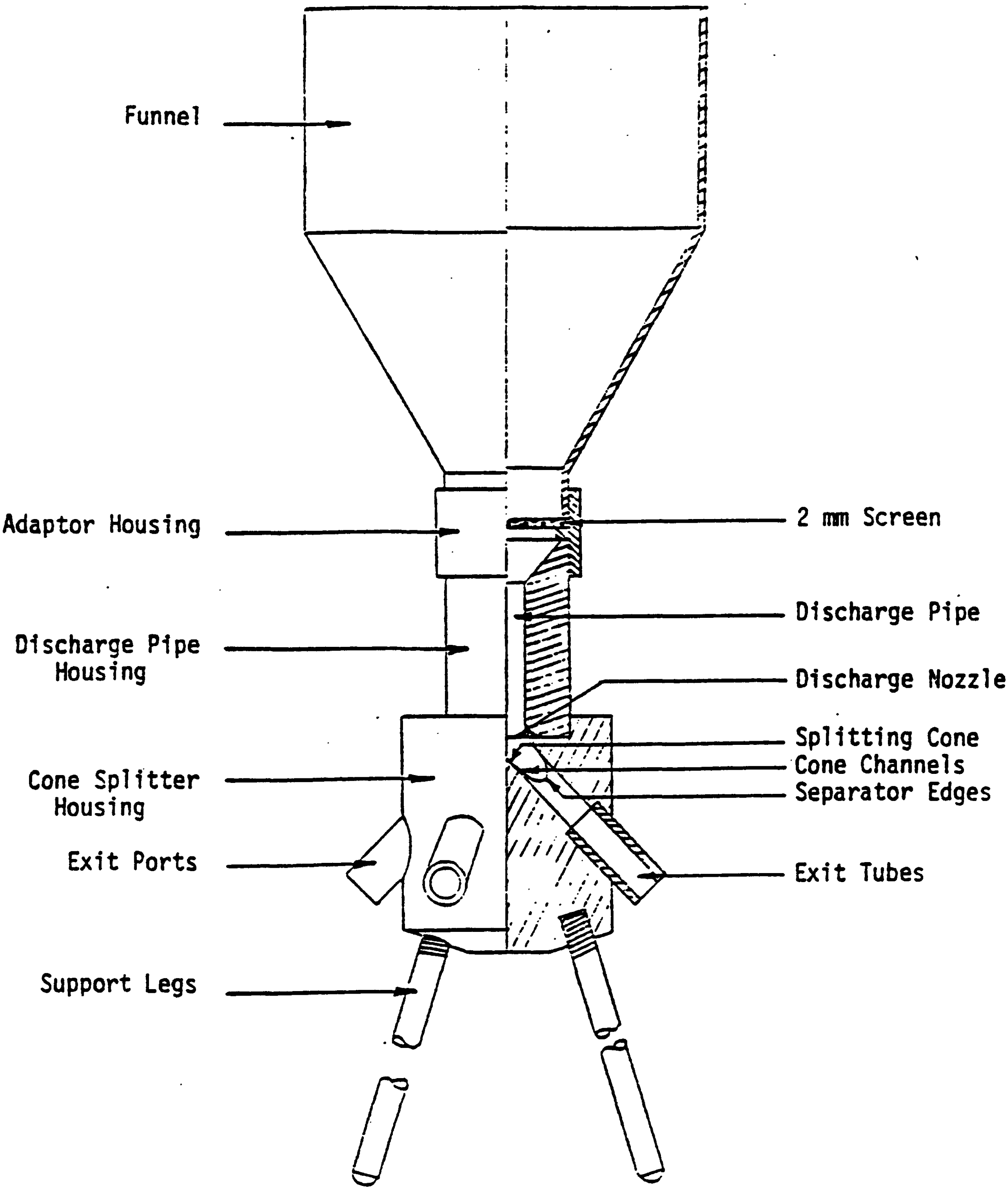

Figure 1. Cone sample splitter with right portion sectioned for detail.

The 10 exit ports direct the individual subsamples into distributer tubes leading to the subsample containers. The tubes are of sufficient size and alignment to prevent any back pressure or restriction of flow from the splitting chamber. They also are kept to a minimum length to prevent submergence of the end in the subsample. Any restriction of flow from an exit port will interfere with the rate of split-sample entry into that port, causing a bias in the splitting.

Evaluating the cone splitter

Two prototype cone splitters were constructed and tested for accuracy and bias. The tests were conducted using both clear water and prepared samples of water and sand-size sediment. In addition, tests were made to determine the effect of tilting the splitter and of pouring the sample into the splitter at different rates and orientations.

To test the accuracy and bias with respect to volume, Bruce M. Delaney of the New Mexico District sediment laboratory prepared six samples of deionized water placed in 1-gallon plastic containers, similar to commercially used milk, juice, and water jugs. The volumes for all observations were determined by weighing to the nearest 0.1 grams (essentially equivalent to 0.1 mL using deionized water). Samples were introduced into the splitter by inverting the sample bottle over the reservoir, allowing it to empty as rapidly as possible. The splitter was allowed to sit for approximately 1 minute after splitting for draindown before the subsamples were removed and weighed for volume tests.

The results of the six volume tests are given in Table 1. After weighing each subsample, it was determined that on the average 2 mL of water was lost during a splitting process due to droplets of water adhering to various parts of the splitter. A small bias in the distribution was observed from outlet to outlet. This was probably due to slight variations during the fabrication process. Table 1 shows that the average discharge from tube No. 8 was consistently high by 1.5 percent, but this is considered well within acceptable limitations. It should be noted that the outlet numbers do not correspond to the sequence that the outlet ports were drilled.

To check the accuracy with respect to volume splits, each subsample was compared to the mean volume for each split. The maximum error observed was +1.9 percent (outlet 8, test 6) and the minimum error was -1.7 percent (outlet 2, test 3). The standard error in percent (standard deviation divided by the mean times 100) for each test was 1.1 percent or less. These observations indicate that the cone splitter is capable of accurately subdividing a sample into 10 equal parts by volume within an arbitrary acceptable error limit of ± 3 percent.

An additional series of tests were made using a water-sediment mixture to test the splitter capability to produce subsamples equivalent in physical composition to the original sample. Six samples were prepared. Each consisted of 1.0 grams of 62- to 125- um sand, 4.5 grams of 125- to 250- um sand, and 0.5 grams of 250- to 500- um sand plus deionized water to bring the total sample weight of 2500.0 grams. Using a suspended-sediment mixture of predominantly sands was considered to be a worst-

Test Number Initial Sample Weight	1 2499.4		2 2499.5		3 2499.5		4 2499.5		5 2499.5		6 2499.4		Average	
	Outlet Volume	Percent	Outlet Volume	Percent	Outlet Volume	Percent	Outlet Volume	Percent	Outlet Volume	Percent	Outlet Volume	Percent	Mean Volume	Percent
Outlet No. 1	248.4	-0.5	249.5	-0.1	247.4	-0.9	248.1	-0.6	247.8	-0.8	249.2	-0.2	248.4	-0.5
2	246.8	-1.1	246.8	-1.2	245.6	-1.7	248.4	-0.5	246.3	-1.4	246.7	-1.2	246.8	-1.2
3	249.4	-0.1	251.0	0.5	250.6	0.4	251.1	0.6	249.8	0.0	248.7	-0.4	250.1	0.1
4	250.7	0.4	252.6	1.1	252.5	1.1	251.3	0.6	251.8	0.8	250.5	0.3	251.6	0.7
5	248.1	-0.6	248.3	-0.6	249.8	0.0	249.3	-0.2	250.2	0.2	248.1	-0.6	249.0	-0.3
6	252.2	1.0	250.3	0.2	252.7	1.2	252.0	0.9	252.7	1.1	250.6	0.4	251.8	0.8
7	245.7	-1.6	246.2	-1.5	246.0	-1.5	246.3	-1.4	246.6	-1.3	245.9	-1.5	246.1	-1.5
8	252.7	1.2	254.2	1.7	252.9	1.3	253.3	1.4	253.1	1.3	254.6	1.9	253.5	1.5
9	248.7	-0.4	247.3	-1.0	247.5	-0.9	247.1	-1.0	248.2	-0.7	249.5	-0.1	248.0	-0.7
10	253.9	1.7	252.1	0.9	251.8	0.8	250.6	0.4	251.7	0.7	253.0	1.3	252.0	0.9
Final sample weight	2496.6		2498.3		2496.8		2497.5		2498.2		2496.8		2497.4	
Sample loss	2.8		1.2		2.7		1.9		1.3		2.6			
Mean weight	249.7		249.8		249.7		249.8		249.8		249.7		249.7	
Standard deviation	2.7		2.7		2.8		2.3		2.5		2.7			
Error percent	1.1		1.1		1.1		0.9		1.0		1.1			

Table 1. Sample Split Tests Using Deionized Water (Measurement In Grams)

case condition test because sands will not easily stay in suspension as compared to silts or clays. Particle sizes finer than sand (< 62 um) should split with an accuracy comparable to the volume-test results. If the cone splitter operates properly, the sediment concentrations of the subsamples should be virtually equivalent for each outlet and should not vary with the variation in volume from outlets.

The results of the water-sediment mixture tests shown in Table 2 indicate that the splitter will subsample samples containing sand-size sediment with a precision of 2.3 percent as calculated by averaging the standard deviations from each test. Test 2 produced both the maximum (+5.6, outlet 10) and the minimum (-4.4, outlet 3) individual subsample errors. Figure 2 shows the plot of the mean, maximum, and minimum volume and concentration for each outlet. There does not appear to be any correlation between the variation in sediment-concentration means and volume means from outlet to outlet. Variability about the mean is greater for concentration than for volume, which is expected because the measurement of sand-size sediment concentration is less precise than measurement of volume alone.

The 10 subsamples obtained from test 2 were further analyzed for particle-size distribution. Summary results given in Table 2 show that the percent by weight of each subsample in the size range 125 to 250 um is well distributed among the subsamples with a maximum deviation of 3 percent from the mean.

The series of sediment-concentration tests do indicate a possible bias in the splitter operation, although the error of the bias appears to be acceptable. When observing the percent variation from the mean concentration, the outlets having a positive differences are grouped together. For example, for test 1, table 2, outlets 10, 1, 2, 3, and 4 are all greater than the concentration average and they all represent one side of the splitter outlet ports. This pattern was observed in the other tests, although it was not always the same group of outlets that contributed an above-average concentration. This phenomenon may be attributed to a slight vortex action associated with the flow through the stand-pipe. The sand leaving the funnel may tend to string out into a ribbon rather than mix. Further tests and changes of stand-pipe and screen designs will be necessary to determine the actual cause. It is believed, however, that such additional tests are not warranted considering that the observed errors are well within an acceptable range and the fact that the tests were conducted using predominantly sands, which represent the worst-case situation.

Testing of new cone splitters

To obtain reliable results, as observed in the series of tests previously discussed, a controlled operating procedure must be followed. Before using a new splitter, operators should familiarize themselves with the individual instrument by running a series of tests to determine any bias that could result from imperfection or operator procedures. The following test procedure should be followed:

1. Inspect the cone splitter housing and outlet ports. They should be smooth and symmetrical without any burrs or chips visible. Make sure the cone splitter is clean and place on a stable platform or bench in a level position. Visual leveling is sufficient.

Test	Test 1		Test 2		Test 3		Test 4		Test 5		Test 6		Summary		Test 2	
Outlet	Concentration mg/L	Percent deviation from mean	Concentration mg/L	Percent deviation from mean	Concentration mg/L	Percent deviation from mean	Concentration mg/L	Percent deviation from mean	Concentration mg/L	Percent deviation from mean	Concentration mg/L	Percent deviation from mean	Average Concentration mg/L	Percent deviation from mean	Percent sand 125-250μm	Percent deviation from mean
No. 1	2368	0.8	2377	0.4	2355	0.5	2339	-0.3	2345	-0.1	2323	-1.1	2351	0.0	73.9	2.4
2	2406	2.4	2297	-2.9	2396	2.2	2357	0.5	2296	-2.1	2376	1.2	2355	0.2	74.0	2.5
3	2390	1.7	2263	-4.4	2410	2.8	2284	-2.6	2264	-3.5	2335	-0.6	2324	-1.1	74.6	3.4
4	2355	0.3	2298	-2.9	2377	1.4	2277	-2.9	2335	-0.5	2318	-1.3	2327	-1.0	73.5	1.8
5	2303	-2.0	2306	-2.6	2333	-0.5	2317	-1.2	2298	-2.1	2285	-2.7	2307	-1.8	72.5	0.5
6	2314	-1.5	2345	-0.9	2288	-2.4	2390	1.9	2341	-0.2	2302	-2.0	2330	-0.9	70.3	-2.6
7	2311	-1.6	2356	-0.5	2278	-2.8	2365	0.8	2356	0.4	2389	1.7	2342	-0.3	70.1	-2.9
8	2344	-0.2	2486	5.0	2307	-1.6	2407	2.7	2439	3.9	2455	4.5	2406	2.4	70.8	-1.9
9	2306	-1.8	2441	3.1	2304	-1.7	2333	-0.5	2367	0.9	2344	-0.2	2349	0.0	72.0	-0.2
10	2393	1.9	2499	5.6	2393	2.1	2386	1.7	2423	3.3	2356	0.3	2408	2.5	70.0	-3.0
Mean mg/L	2349.0		2366.8		2344.1		2345.5		2346.4		2348.3		2350		72.2	
Std. dev. mg/L	39.3		83.0		48.6		44.0		54.6		49.2					
Std. dev. in percent of mean	1.7		3.5		2.1		1.9		2.3		2.1					

Table 2. Delaney tests - Concentration sample split test

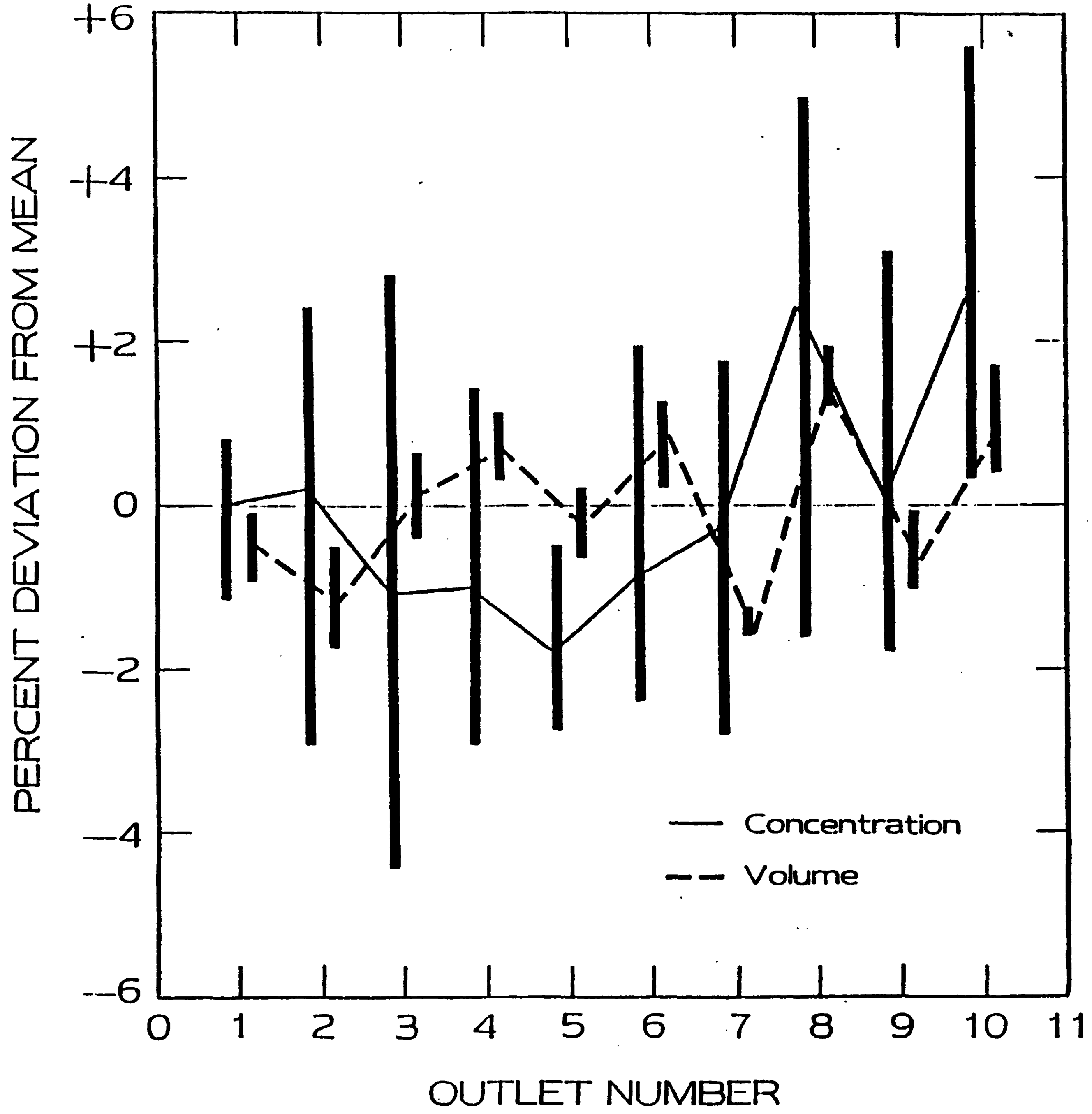

Figure 2. Maximum, minimum, and mean deviations for the volume and concentration tests.

2. Connect 10 discharge tubes to the outlet ports. All tubes must be approximately the same length, and the length should be as short as possible. The tubes need only extend into the receiving containers sufficiently to prevent spillage. They must not extend in so far that the end becomes submerged. Mark the outlets from 1 to 10.

3. Wet the cone splitter by pouring through several liters of clear water. Lightly tap the system to dislodge adhering water drops, then discard the water. Replace an empty container under each outlet.

4. Accurately measure approximately 3 liters of clear water into a 1-gallon narrow-mouth plastic bottle.

5. Rapidly invert the gallon bottle over the reservoir, letting it flow as fast as possible. Rest the inverted bottle on top of the reservoir. The rising water level in the reservoir will regulate the rate that water will leave the gallon bottle once the bottle opening becomes submerged. For proper operation, the stand-pipe must be discharging at its full flowing capacity.

6. After all water has passed through the splitter, tap the assembly several times to dislodge adhering water drops. Check for spills and leaks. If any are observed, discard the test, correct the problem, and repeat the test.

7. Accurately measure the volumes of the 10 subsample within ±1 mL. Record the volumes for each outlet on a form similar to Table 1.

8. Repeat the test two more times for a total of three tests. Use approximately the same initial volume for each test.

Calculating Results

To determine the accuracy of the cone splitter tested, calculate the mean volume of each subsample ($\overline{x}$) and standard deviation (Sx) for each test, by

$$\overline{x} = \frac{\sum Xi}{n}$$

and

$$Sx = \sqrt{\frac{\sum Xi^2 - n\overline{x}^2}{n-1}}$$

calculate the standard deviation in percent (Ex) by the following:

$$Ex = \frac{Sx}{\overline{x}} \times 100$$

also calculate the error for each subsample (Ei) by

$$Ei = \frac{\overline{x} - Xi}{\overline{x}} \times 100$$

where Xi is the measured volume for the individual subsample. Finally, compute the average standard error ($\overline{Ex}$) for the three tests and note the maximum and minimum errors (Ei) for all tests.

A cone splitter is considered acceptable for sample processing if the average standard error ($\overline{Ex}$) for the three tests is 3.0 percent or less, and no individual errors (Ei) exceed ± 5.0 percent. Note the error patterns for individual outlets to determine which outlets show consistant bias and mark them with their average percent bias error.

Using cone splitters

The cone splitter works best when the following procedure is followed. A consistent procedure such as always tapping the assembly at the end of a split and always wetting the system before a split should be practiced to help assure unbiased results.

1. Set up the cone splitter on a flat open area. Check for level and proper tubing lengths. Visually inspect the splitter for broken parts, misalignment or debris.

2. Rinse through one or two liters of deionized water. Discard the water.

3. Place containers under each outlet.

4. Shake the sample for 10 to 15 seconds.

5. Rapidly invert the sample container over the reservoir and rest it on the reservoir top.

6. After the flow has stopped, tap the assembly to dislodge adhering drops.

7. Remove desired subsamples. Repeat as necessary if any of the subsamples need splitting starting with step 3.

8. At completion of all splits for the station being processed, disassemble the splitter and clean before splitting another sample.

All subsamples do not have to be collected in separate bottles. Outlet tubes can be combined to collect various combinations of the original sample. Care must be taken, however, when combining outlet tubes into a single bottle to make sure there is no backpressure resulting from restriction of the flow.

Consider for example, the following subsamples are required from a 3-liter sample:

3-250 mL subsamples for chemical analyses (total).
1-500 mL subsample for chemical analysis (total).
2-250 mL subsamples for chemical analyses (dissolved).
1-500 mL subsample for chemical analysis (dissolved).

The sample then is split by placing a 500-mL bottle under three outlets, two outlet tubes are combined into a 1-L bottle, and the remaining five outlet tubes can be combined into one convenient container for later filtering. The resulting split of the 3-L sample would provide three 500-mL bottles with with 300-mL each and the liter bottle with 600 mL. There would then be 1500 mL left for filtering. These volumes are close enough to the desired amounts for lab analysis.

If a more exact subsample volume is desired the following procedure is used. For example, if 440 mL is required from a sample of 2850 mL the first step is to compute the percentage needed. In this case 450 mL is 16 percent of 2850 mL. The 16-percent split is achieved by first obtaining 10 percent from one tube from the first pass. The remaining 6 percent is obtained by pouring one of the 10-percent splits through the splitter a second time and drawing off 6 tubes or 60-percent. By this procedure a subsample of ±1 percent of the whole sample can be obtained by two passes through the splitter.

Care and maintenance of cone splitter

Cone splitters must be cleaned before being used for processing any samples. It is not necessary to clean it before splitting repetitively from one sample, but between a series of samples from the same station and runoff event, rinse the splitter with several liters of distilled water. Before using a previously cleaned splitter, start by pouring several liters of deionized water through the splitter. After using a splitter, acquiring a new splitter, or before starting to process a sample from a different station, clean the splitter by disassembling it and washing the parts in soap and water using a good quality laboratory detergent. A soft bristle test-tube brush works well for cleaning inside the ports. Rinse thoroughly with tap water followed with deionized water. Store cleaned cone splitters in plastic bags between usages.

The cone splitters should be visually inspected for damage especially the cone splitting chamber. Units that show damage or wear should be retested to check their serviceability. Check discharge tubing frequently for proper length and cleanliness. Replace tubes as conditions warrant.

The cone splitter is built to very close tolerances which are required for accurate and reliable operation. Given proper care and handling and operated according to the approved methods, the cone splitter should produce reliable results for a considerable number of samples.

United States Department of the Interior

GEOLOGICAL SURVEY
RESTON, VA 22092

In Reply Refer To:
Mail Stop 415

October 8, 1992

OFFICE OF SURFACE WATER TECHNICAL MEMORANDUM NO. 93.01

SUBJECT: Summary of Documentation that Describes Instrumentation and Field Methods for Collecting Sediment Data

Office of Surface Water Technical Memorandum No. 92.08 listed all Water Resources Division (WRD), Office of Water Quality (OWQ), and Office of Surface Water (OSW) memorandums issued since 1971 that pertain to sediment activities. The present memorandum builds upon OSW Technical Memorandum No. 92.08 by summarizing documentation that describes instrumentation and field methods recommended for collection of sediment data. Information contained in this memorandum should provide a reference for field personnel involved in collecting sediment data. Subsequent memorandums will be issued that summarize procedures recommended for other aspects of sediment activities.

INSTRUMENTATION

SUSPENDED SEDIMENT

Instrumentation currently available for the collection of suspended sediment is summarized in Edwards and Glysson (1986), which was announced in OSW Technical Memorandum No. 88.17. There are seven depth-integrating, three point-integrating, and two pumping samplers currently available from either the Federal Interagency Sedimentation Project or the Hydrologic Instrumentation Facility (HIF) for collecting suspended sediment samples. Proper use of all samplers is described in Edwards and Glysson (1986). Sampler characteristics are summarized in Edwards and Glysson (1986), Table 1. Tables and text in "Guidelines for the collection, treatment, and analysis of water samples, U.S. Geological Survey Western Region Field Manual, (Written communication, M. A. Sylvester) provide concise, field-oriented information on proper use of suspended-sediment samplers. It should be noted that this field manual has not received Director's approval. Several errors, omissions, and/or inconsistencies have been noted between the manual and WRD policy and within the manual itself (see attached comments on "Guidelines for the Collection, Treatment, and Analysis of Water Samples, U.S. Geological Survey, Western Region, Field Manual"). Official WRD policy should be followed if any inconsistencies are found. The field manual is printed on waterproof paper to maximize usefulness in the field. In addition to the references mentioned above, information on availability and proper use of suspended-sediment samplers is contained in the following U.S. Geological Survey (USGS) memorandums:

WRD Technical Memorandum Nos. 76.07, "New suspended sediment--water quality samplers" and 76.156T, "Suspended-sediment sampler, D-74"--

> Memorandum No. 76.07 announces the availability of the D-74 depth-integrating sampler. Memorandum 76.156T reiterates that the D-49 sampler was replaced by the D-74 sampler and lists advantages of the D-74 over the D-49 sampler.

Quality of Water Branch (QWB) Technical Memorandum 77.03, "DH-75 Suspended-sediment sampler"--

> Announces the availability of the DH-75 suspended-sediment sampler, which was designed to sample under ice. Versions capable of holding both pint or quart plastic bottles are available. A version of this sampler capable of holding a half-gallon bottle was originally available. The half-gallon version, designated DH-75H, is no longer available due to the large unsampled zone associated with it. This problem was alluded to in Memorandum No. 77.03. The DH-75 is generally not recommended for general use. It should only be used under freezing conditions.

WRD Technical Memorandum No. 77.151, "Removal of sediment samplers from controlled property"--

> Announces that sediment samplers costing less than $500 will no longer be considered controlled property.

QWB Technical Memorandum No. 80.03, "P-61 and P-63 point-integrating samplers"--

> This memorandum informs users that the P-61 and P-63 samplers most commonly malfunction because water gets into the head cavity and corrodes the plug and solenoid assembly. The memorandum describes how this can happen and what can be done to avoid it. A summary of operating procedures for the P-61 sampler is also attached to the memorandum.

QWB Technical Memorandum No. 81.02, "Operation and availability, D-77 water-quality sampler"--

> Announces availability of the D-77 sampler. The D-77 sampler uses a relatively large 3-liter plastic bottle that can be autoclaved. The design was chosen to allow a large volume, depth-integrated sample for biological and chemical studies. The memorandum describes the sampler design and recommended uses.

QWB Technical Memorandum No. 81.06, "Automatic pumping samplers--Test of efficiency of Manning and ISCO samplers"--

> Transmitted a brief technical report written by the U.S. Forest Service describing tests done on performance of Manning and ISCO pumping samplers. The report discusses the need to frequently check the efficiency of pumping samplers. The memorandum indicates that sampler efficiency is generally reduced as the concentration of sand-size material increases.

QWB Technical Memorandum No. 80.06, "Samplers: color-coded nozzles for sediment samplers"--

> Announces use of color coded nozzles for sediment samplers. Sediment samplers are now supplied with color-coded nozzles to easily identify what nozzle should be used with a given sampler.

QWB Technical Memorandum No. 80.18, "Samplers--Problems with installation of plastic nozzles on samplers"--

> Warns against overtightening plastic nozzles when installing them in sediment samplers, particularly the DH-48.

QWB Technical Memorandum No. 83.08, "Bag-type suspended-sediment sampler"--

> Announces a modification of the D-77 sampler, termed the bag-type suspended-sediment sampler. The bag-type sampler can collect samples at depths greater than 19 feet, which is generally accepted as the maximum depth at which samples can be collected isokenitically in rigid containers. This is usually referred to as the "compression-depth limit." Memorandum 83.08 suggests four rules that should be followed when using the bag-type sampler. These rules should be followed closely, because the sampler is prone to undersampling.

TRACE ELEMENT DATA

Epoxy coated versions of the D-77 and DH-81 samplers are available for collecting trace metal data. However, recent experiments to identify sources and levels of contamination have brought traditional field methods and sampling equipment into question. Actions being taken by the OWQ to improve trace element sampling and to provide advice to field personnel are described in several recent OWQ memorandums:

OWQ Technical Memorandum No. 91.10, "Dissolved Trace Element Data"--

> Describes the present understanding, ramifications, and issues that require resolution surrounding possible contamination of dissolved trace-element data.

OWQ Technical Memorandum No. 92.03, "Statement Regarding Dissolved Trace-Element Data Production Through Water Year 1991"--

> Provides a statement to be placed in State annual data reports that warns that some trace-element data contained in the reports might reflect sample contamination. This memorandum was replaced by OWQ Technical Memorandum No. 92.04 (see below).

OWQ Technical Memorandum No. 92.04, "Revised Statement Regarding Dissolved Trace-Element Data Production"--

> Revises statement included in OWQ Technical Memorandum No. 92.03.

OWQ Technical Memorandum No. 92.05, "Quality of Existing Dissolved Trace-Element Data"--

> Describes implications of OWQ Technical Memorandum 91.10. Also describes how the Division can deal with issues of uncertainty in the validity and usefulness of existing dissolved trace-element data.

OWQ Technical Memorandum No. 92.12, "Trace Element Concentrations in Deionized Water Processed Through Selected Surface-Water Samplers: Study Results and Implications"--

> Identifies levels of dissolved trace element contamination associated with selected surface-water samplers. Additionally, concludes a cleaning procedure using acid is necessary for all samples to be used in parts-per-billion protocols.

OWQ Technical Memorandum No. 92.13, "Trace Element Concentration: Findings of Studies on the Cleaning of Membrane Filters and Filtration Systems"--

> Summarizes results and conclusions from a series of experiments emphasizing the cleaning of filters and filtration systems. Also, compares dissolved trace element concentrations in sequential rinses of three brands of filters.

PUMPING SAMPLERS

Pumping samplers can be used where frequent samples are needed and conditions make manual collection of samples impractical. In the past the Federal Interagency Sedimentation Project has supplied two samplers, the US PS-69 and US PS-82. Neither sampler is presently in stock. US PS-69 samplers can be special ordered from the U.S. Army Corps of Engineers, Waterways Experiment Station (WES) (contact Wayne O'Neal at 601/634-2624) at 3909 Halls Ferry Road, in Vicksburg, Mississippi. US PS-82 samplers have been phased out. Some parts for both samplers are available from the Federal Interagency Sedimentation Project. Electrical repairs on either the US PS-69 or US PS-82 samplers should be coordinated through the HIF at the Stennis Space Center in Mississippi. Mechanical repairs on either type of sampler should be coordinated through WES. There are a number of automatic pumping-type samplers available commercially. The Manning S-4050 and ISCO 1680 are two that are commonly used by USGS personnel. Edwards and Glysson (1986) list 17 criteria that should be met when operating pumping-type samplers. Edwards and Glysson (1986) also describe installation and use of pumping samplers as well as how the "point" data collected by these samplers should be analyzed.

TURBIDITY

QWB Technical Memorandum No. 73.11 transmits "Falling-stream turbidimeters as a means of measuring sediment concentrations in streams," by H. P. Guy and R. C. Olson. The memorandum and report discuss the use of turbidity as an indicator of suspended-sediment concentration. Records of turbidity should only be used to provide an indication of variations in sediment concentration with time. Any record of turbidity must be accompanied with actual samples of sediment concentration.

BEDLOAD

OSW Technical Memorandum No. 90.08 summarizes WRD policy issues relevant to bedload. As pointed out in Memorandum 90.08, bedload samples can be collected wherever physical conditions permit. OSW Technical Memorandum No. 90.08 lists 10 different samplers available for sampling bedload. Four of those samplers have a nozzle expansion ratio of 3.22. The other six have an expansion ratio of 1.40, which is the ratio accepted by the Technical Committee of the Federal Interagency Sedimentation Subcommittee. As pointed out in OSW Memorandum No. 90.08, samplers with a nozzle expansion ratio of 1.40 are presently recommended, although use of samplers with a 3.22 expansion ratio is acceptable. OSW Technical Memorandum No. 90.08 supercedes OWQ Technical Memorandum No. 76.04, 77.07, 79.17, and 80.07, as well as WRD Technical Memorandum No.77.60.

BED MATERIAL

There are at least 17 different samplers available for sampling bed material. Nearly all of those samplers are useful only for sampling material finer than 16 mm (Edwards and Glysson, 1988). Standard equipment is not available for sampling large bed material. Indirect methods, such as the "pebble count" method used by Wolman (1954), are commonly used to estimate the grain-size distribution of coarse bed material. Edwards and Glysson (1986) and "Guidelines for the collection, treatment, and analysis of water samples, U.S. Geological Survey Western Region Field Manual, (Written communication, M. A. Sylvester) discuss commonly used bed material samplers.

FIELD METHODS

SUSPENDED-SEDIMENT SAMPLING

WRD Technical Memorandum No. 71.73 was issued to inform the Division that techniques adopted by the USGS for the collection and analysis of sediment samples are described in three Techniques of Water-Resources Investigations reports:

Book 3, Chapter C1 - "Fluvial Sediment Concepts" by H. P. Guy

Book 3, Chapter C2 - "Field Methods for Measurement of Fluvial Sediment" by G. Porterfield

Book 3, Chapter C1 - "Laboratory Theory and Methods for Sediment Analysis" by H. P. Guy.

Although there have been no additional Techniques of Water Resources chapters written to supersede any of the chapters mentioned in WRD Technical Memorandum No. 71.73, Open-File Report 86-531 is essentially a replacement for Book 3, Chapter C2. Open-File Report 86-531, which is presently being prepared for release as a Techniques of Water-Resources Investigations report, should now be used as the reference on field methods and measurement of fluvial sediment. Field methods for collecting suspended-sediment samples are summarized in "Guidelines for the collection, treatment, and analysis of water samples, U.S. Geological Survey Western Region Field Manual, (Written communication, M. A. Sylvester)

In addition to the references mentioned above, guidance on collecting suspended-sediment samples can be found in several OWQ, OSW, and WRD memorandums, which are summarized below.

USE OF THE CHURN SPLITTER FOR COLLECTING SUSPENDED-SEDIMENT SAMPLES--

OWQ Technical Memorandum Nos. 76.17, 76.24-T, 77.01, 78.03 and 80.17 contain information on use of the churn splitter for drawing representative subsamples from large composite samples. However, information contained in these memorandums is somewhat vague and confusing. For example, OWQ Technical Memorandum No. 76.17 states that "Samples for bacterial determination or for analysis of sediment concentration and particle size analyses should not be composited." Memorandum 78.03 states that "sediment concentration and particle-size subsamples may be taken directly from the churn" when "essentially all particles are silt-size or smaller." Because it is difficult to tell in the field whether or not samples contain sand-sized particles, it is suggested that all sediment samples be collected in accordance with Water Quality Technical Memorandum No. 76.17, which states that "Samples ... for analysis of sediment concentration and particle size analyses should not be composited." The churn splitter, therefore, should not be used for sediment sampling. If samples must be split, it is best to use the cone splitter (see QWB Technical Memorandum 80.17).

BEDLOAD SAMPLING

Bedload samples can be collected wherever physical conditions permit. OSW Technical Memorandum No. 90.08 summarizes WRD policy on bedload. OSW Memorandum No. 90.08 also provides a background of bedload-related research done in the WRD, supplies guidelines on sampler selection, and summarizes methods used to collect and reduce bedload data. A detailed description of methods used to collect and reduce bedload data is given in Edwards and Glysson (1986). "Guidelines for the collection, treatment, and analysis of water samples, U.S. Geological Survey Western Region Field Manual," (Written communication, M. A. Sylvester) provides a field-oriented summary of methods used to collect bedload data.

GUIDELINES FOR MISCELLANEOUS FIELD METHODS

Memorandums issued since 1971 provide guidelines for several field methods related to several different types of investigations:

QWB Technical Memorandum No. 72.10, "Sediment computations--Conversion of suspended-sediment concentration from parts per million to milligrams per liter"--

> Contains a table for converting suspended-sediment concentrations from parts per million to milligrams per liter. The table attached to Memorandum 72.10 supercedes Table 1 in Techniques of Water Resources Investigations, Book 5, Chapter C1, p. 4.

QWB Technical Memorandum 81.16, "Report--A guide for predicting sheet and rill erosion on forest land, USDA Technical Publication SA-TP 11"--

> Informs Division personnel that demand for the report "A guide for predicting sheet and rill erosion on forest land," USDA Technical Publication SA-TP 11, September, 1980, has exceeded supply of reports at the QWB. The memorandum gives an address for the Forest Service in Atlanta from whom additional copies can be ordered.

WRD Technical Memorandum No. 83.79, "Collection and analysis of samples in connection with investigations of hydrologic effects of hazardous waste sites"--

> Presents WRD's commitment to train employees in the proper use of equipment and techniques to insure safe collection, handling, and analysis of samples in connection with studies of hazardous waste sites. Also, transmits a safety plan for WRD field activities at hazardous waste sites.

OSW Technical Memorandum No. 87.07, which transmits the report "Pilot study for collection of bridge-scour data" and 89.10, which announces the availability of the report, "Use of surface-geophysical methods to assess riverbed scour at bridge piers"--

> This memorandum and related report provides information relative to the study of bridge scour. Memorandum 87.07 transmitted results of "Pilot Study for Collection of Bridge Scour Data" by R. D. Jarrett and J. M. Boyle. The Jarrett and Boyle report provides information on how to collect data on scour around bridge piers. Memorandum 89.10 announced availability of "Use of Surface-Geophysical Methods to Assess Riverbed Scour at Bridge Piers," by S. R. Gorin and F. P. Haeni. The Gorin and Haeni report discusses the performance and characteristics of four geophysical methods to define existing and previous scour holes around bridge piers.

REFERENCES

Edwards, T.K. and Glysson, G.D., 1988, Field methods for measurement of fluvial sediment: U.S. Geological Survey Open-File Report 86-531, 118 p.

Wolman, M.G., 1954, A method of sampling coarse river bed material: American Geophysical Union Transcript, V. 35, no. 6, p. 951.

Charles W Boning

Charles W. Boning
Chief, Office of Surface Water

Attachment

WRD DISTRIBUTION: A, B, FO, PO

Comments on "Guidelines for the Collection, Treatment, and Analysis of Water Samples, U.S. Geological Survey, Western Region, Field Manual."

1) Page 2 First paragraph-It should be mentioned that the OSW sets policy for sediment data-collection activities.

2) Page 10-11 Table 1-Several corrections are needed. For example, the DH-81 can use a 1/8-inch nozzle, the maximum depth for the DH-81 is either 9 feet or 16 feet depending on the bottle size, and the D-74 with a 1/4-inch nozzle and pint bottle has a maximum depth of 9 feet.

3) Page 12 The DH-75 sampler was designed to be used under ice (see QWB Technical Memorandum 77.03). This is not mentioned on page 12.

4) Page 12 The Federal Interagency Sedimentation Project currently only recommends use of 3/16- and 1/4-inch nozzles with the DH-81. Filling rate problems have been identified using the 5/16-inch nozzle. The small opening of the 1/8-inch nozzle becomes frayed reducing sampling efficiency significantly.

5) Page 13 The D-77 has the same problems with the 1/8-inch nozzle mentioned above for the DH-81.

6) Page 13-14 It should be noted that the P-61, P-63, and P-72 samplers are to be used when the stream depth exceeds 15 feet.

7) Page 15 The second paragraph from the bottom should state that a 1/8-inch nozzle should not be used when sand-sized particles are in suspension because a nozzle will start to exclude particles about one-half of its diameter. In the case of the 1/8-inch nozzle, this would include particles about 1.5 mm in size.

8) Page 18 Table 2. The HS-85 bedload sampler is not available.

9) Page 25 It should be noted that the EDI method is only valid if the concentration in the cross section is relatively uniform. If the concentration is not uniform, more than nine verticals may be needed. The EWI method may be more desirable under these conditions. Also, the manual does not mention that samples collected using the EDI method can be composited if the volume of samples from each vertical are nearly the same. This, however, will cause information on lateral distribution of concentration to be lost. Compositing samples also

eliminates the possibility of detecting individual samples, which may not be representative.

10) Page 49 Second paragraph-statement here conflicts with statement on page 33 about use of the churn splitter. See statements under the heading "Use of the churn splitter for collecting suspended-sediment samples" in the main body of this memorandum for guidance in use of sample splitters.

United States Department of the Interior

GEOLOGICAL SURVEY
RESTON, VA 22092

In Reply Refer To:
Mail Stop 412

April 16, 1993

OFFICE OF WATER QUALITY TECHNICAL MEMORANDUM 93.09

Subject: EQUIPMENT--Sample Splitting Devices

Within the Water Resources Division (WRD), composited water samples traditionally have been subsampled for field and laboratory analyses by means of a churn splitter or, less frequently, a cone splitter. Historically, the splitting devices have been constructed of plastic, which has proved adequate for many water-quality parameters including field measurements, major ions, and nutrients.

There are two concerns with using plastic devices for water samples that are to be analyzed for trace elements and semi-volatile trace organic chemicals (pesticides and base/neutral compounds). The first concern is contamination of the water sample by material inherent in the plastic. The second is analyte loss from the water sample due to sorption onto the plastic surfaces. To avoid these two potential problems, an all-Teflon cone splitter has been created, and plans are in progress for fabricating an all-Teflon churn splitter. The probability is that chemicals will have less of a tendency to sorb to a Teflon surface and, if cleaned properly before use, such containers should not contaminate water samples. However, experiments need to be run to test: (a) assumptions concerning contamination and analyte loss for all constituents, and (b) the splitting characteristics of the churn and cone splitters.

Accordingly, the Office of Water Quality (OWQ) is beginning a series of studies aimed at evaluating the usefulness and limitations of splitting devices for water samples. Areas of investigation will include:

1. Contamination potential and proper cleaning procedures--for both polyethylene and Teflon splitters--for the various splitting devices for a wide variety of analytes;

2. Potential for analyte loss for polyethylene and Teflon splitters;

3. Precision and accuracy of solids splitting;

4. Precision and accuracy of dissolved chemical splitting;

5. Reproducibility between different splitting devices;

6. Reproducibility between splitters of the exact same type;
7. Effects of different operators on the reproducibility of splitting.

Paul Capel will coordinate the studies. Suggestions/concerns should be communicated to Paul by telephone (612/471-0438) or FAX (612/471-9070).

The all-Teflon cone splitter has been used successfully within the WRD since 1991 for processing water samples for pesticides. A recent study (Attachment 1) evaluated the precision of the all-Teflon cone splitter for splitting water and solids over a range of particles sizes, sample volumes, and sample introduction techniques. The overall conclusion was that the all-Teflon cone splitter is comparable to the traditional plastic cone splitter.

Results of the additional studies will be forthcoming from OWQ over the next several years.

Dave Rickert

David A. Rickert

Attachment

This memorandum does not supersede any Office of Water Quality Technical Memorandum.

Key Words: Equipment

Distribution: A, B, S, FO, PO

Precision of a Splitting Device for Water Samples

By Paul D. Capel *and* Fernando C. Nacionales

ABSTRACT

Two identical cone splitters, devices designed to split water and its suspended solids into equal aliquots for semi-volatile organic chemical analyses, are evaluated for their precision. The water-splitting evaluations consisted of experiments to test the effect of water volume, the effect of combining outlet ports, and the effect of different techniques of water introduction. The solids-splitting evaluations consisted of experiments to test the effect of particle size (nine different particle diameter ranges from very coarse sand to clay) and suspended-solids concentration. In general, water was equally split with a precision of less than 5% relative standard deviation. The accuracy of splitting the solids was a function of particle size. Clay, silt, and fine and medium sand were split with a precision of less than 7% relative standard deviation, and coarse sand was split with a relative standard deviation between 12 and 45%.

INTRODUCTION

The cone splitter is a device developed to split small to very large volume water samples for suspended solids and other water-quality constituents into equal concentration aliquots. Since its development, the cone splitter commonly has been used in water-sampling techniques. The original plastic design splits water samples as small as 250 mL into 10 subsamples with an accuracy of ±3% (R.S. Pickering, U.S. Geological Survey, oral commun., 1980). In recent years, concerns of contamination and sorption of the plastic cone splitters have lead to the development of an all-Teflon cone splitter for water samples that are analyzed for semi-volatile organic chemicals. The design of the all-Teflon splitter is similar to the design of the plastic devices. This study systematically evaluates the precision of the all-Teflon cone splitter for equally splitting water and suspended solids of various particle-size ranges. This study is part of a comprehensive investigation of pesticides in surface and ground water of the United States. The investigation is part of the National Water Quality Assessment Program (NAWQA) of the U.S. Geological Survey.

EXPERIMENTAL METHODS

Description of Cone Splitter

The all-Teflon cone splitter (Dekaport Splitter, GeoTech Environmental Equipment, Denver, Colorado) consists of a cylindrical reservoir, funnel, stand pipe, and cone splitter housing with 10 outlet ports (Figure 1). The cone splitter housing is precisely machined from one block of Teflon with holes drilled at a 45° angle from the vertical and spaced at 36° intervals around the circumference. The drilled holes converge at the center of the housing creating a single point for splitting. The resultant configuration in the splitting chamber is a notched cone with 10 equally spaced outlet ports (6 mm I.D. opening) about its base. Walls and surfaces inside the splitting chamber are smooth so that water or solids will not be retained and interfere with the splitting process. In the all-Teflon cone splitter, the reservoir is not screened as it is in the plastic cone splitter.

Leveling of the Cone Splitter

Equal splitting of the water and solids is based on the premise that whatever falls on the center point of the cone has an equal chance of making it to any of the 10 outlet ports. If the cone splitter is not leveled, then the chance of the water or solids exiting from the down-side ports will be greater than the up-side ports and the cone will not work effectively. The all-Teflon splitter came from the manufacturer with three support legs that were to be screwed into the bottom of the cone housing. On testing, the support legs were awkward to use, unstable, and extremely difficult to level properly. Because of this, a new support stand was designed (Figure 1).

The cone splitter is leveled by placing it on the stand with a fine bubble level resting on the cone housing flange. The screws in the base plate are adjusted until the bubble stays at the center of the bubble level when the level is turned in various directions.

Evaluation of Water Splitting

De-ionized water volumes of 0.6, 0.8, 1, 4, 8, and 9 L were continuously introduced into the cone splitter, split 10 ways, and collected in pre-weighed, clean, dry 1-L glass bottles. Each bottle was weighed on an analytical balance to determine the water mass and volume. Replicates of each volume of water were tested. Two different all-Teflon splitters were tested, denoted as "A" and "B" throughout this paper.

When using the cone splitter in the field, uneven volumes of replicate water samples commonly are desirable to collect for different laboratory analyses. One way to do this is to combine the outlet ports of the cone splitter. In combining outlet ports, there theoretically should be no additional head loss and the integrity of the splitting should be maintained, as long as the tubing used to combine the outlet ports is of a larger diameter than the tubing coming from the cone housing. To test the effect of combining outlet ports, ports were combined in various ways with large diameter (9 mm O.D.) Tygon tubing. The ports were configured into six single outlets and two double outlet combinations (eight way split) for four tests. The ports were configured into five sets of double outlets (five way split) for five tests. The ports were configured into two sets of three outlets and one set of four outlets (three way split) for three tests. In all these tests, 1-L of deionized water was used.

In another series of tests, duplicate 1-L and 9-L volumes of water were poured into the cone splitter from one location in 50 mL aliquots to evaluate the effect of interrupted water introduction into the splitter. In separate tests, the location of the water pouring around the circumference of the reservoir was changed, but the interrupted pouring technique was held constant.

Evaluation of Solids Splitting

The various particle-size fractions were obtained by sequential wet sieving through a series of brass sieves for particle-size diameters as small as 38 µm and through a series of nylon filters as small as 1 µm. The particle-size classes and diameter ranges used in this study are shown in Table 1. The solids were dried in an oven at 105°C for at least 24 h. A pre-determined mass was weighed on an analytical balance and mixed into a known volume of de-ionized water to create the desired suspended-solids concentration.

For each of the nine different particle-size ranges, test sample suspensions were made at concentrations of 50 and 200 mg/L. Each suspension was split 10 ways using the leveled cone splitter. The water and sediment mixture was collected in 10 pre-weighed, dry, clean 1-L glass bottles and then filtered through pre-weighed, dried paper filter (VWR, Grade 415, diameter: 7.5 cm) in a Buchner funnel for particle-size diameters as small as 38 μm and through pre-weighed, dried nylon membrane filters (Lida, filter diameter: 47 mm, pore opening 0.45 μm) for particle-size diameters from 1 to 38 μm. The total volume of water passed through the filter was collected and weighed to determine its mass. The filter was removed with forceps and transferred to an aluminum pan for drying. The filter was dried at 105°C for at least 24 h then reweighed on an analytical balance. The difference in mass was attributed to the solids present on the filter. The suspended-solids concentration of the water exiting each of the 10 outlet ports was calculated from the mass of solids retained by the filter and the volume of water.

Estimation of Systematic Errors in Measurements

To check the errors in weighing the mass of solids, determining the water volume, and calculating the solids concentration, the 10 resulting water and solids subsamples from the 200 mg/L test of 21 to 38 μm particles were weighed 10 times each. The 10 replicates of the 10 subsamples (100 measurements) of solids mass, water volume, and solids concentrations were used to determine the errors. The calculated average percentage of relative standard deviation (%RSD) of the weighing error for water volume is 0.012%. The calculated average %RSD of the weighing error for the mass of solids is 0.384%. The average %RSD of the measurement and calculation for the suspended-solids concentration is 0.385%. These errors are less than an order of magnitude compared to the errors in the tests with the cone splitter. This suggests that errors reported for the cone splitter are inherent in the cone splitter and not in the laboratory techniques.

RESULTS AND DISCUSSION

Evaluation of Water Splitting

The results of the water splitting evaluation experiments are summarized in Table 2. The %RSD, an indication of the precision of the cone splitter, was calculated for each test. Two different all-Teflon cone splitters of the same model were evaluated using a 0.6 L test volume (A and B in Table 2), providing a comparison of manufacturing consistency.

There were not large differences between the precision of the cone splitter as a function of water volume, the number of outlet ports that were combined, or the manner in which the water was introduced. Combining outlet ports slightly improves the precision as might be expected. There also was no difference between the precision of the two different cone splitters. These data indicate that the all-Teflon cone splitter creates volume splits of a water sample with a precision less than 5%. For actual field use, this implies that sequentially introduced aliquots that have different concentrations of dissolved constituents will be effectively combined into equivalent replicate subsamples.

Evaluation of Solids Splitting

For solids with particle diameters greater than 38 μm, the tests were made with cone splitter "A" from Table 2. For solids with particle diameters less than 38 μm, the tests were made with cone splitter "B" from Table 2. One comparison of the two cone splitters was made (200 mg/L for solids with particle diameters between 38 and 62 μm).

Figure 2 illustrates the results for one particle-size fraction. The water volume, solids mass, and solids concentration are plotted as bars for each of the 10 outlet ports. The mean water volume, mean solids mass, and mean solids concentration are plotted as horizontal lines. The percentage of deviation from the mean for the solids concentration from each port is printed above the respective bar.

A summary of the tests for nine particle-size ranges at two concentrations each are presented in Figure 3. Two important observations can be made from this graph. First, for particles that are smaller than coarse sand, the all-Teflon cone splitter is able to split them with a precision of <7%. For larger particles, the cone splitter is much less precise. The second observation is that there is no systematic difference in the precision of the cone splitter as a function of solids concentration, at least for particle diameters of less than 500 μm. For larger particles, the cone splitter does seem to be less precise for the lower solids concentration.

The precision in the splitting ability of the two different cone splitters can be compared from two tests with 38 to 62 μm particles at 200 mg/L. The range of percent deviation from the mean is -6.8 to 11.7% for "A" and -9.5 to 10.4% for "B". The %RSDs are 5.2 and 7.7%, respectively. There seems to be little systematic difference in the precision of the two cone splitters. The %RSD in Figure 3 for the three smallest particle sizes (data from splitter "B") are in the same range as the next four larger sizes (data from splitter "A"). This also suggests that the two splitters yield similar results.

CONCLUSIONS

The precision of the all-Teflon cone splitter to split water is about the same as the plastic cone splitter. The all-Teflon splitter is able to split a water sample with a %RSD of <5%. The ability of the all-Teflon cone splitter to split solids is a function of particle diameter. Particles smaller than coarse sand are split with a %RSD of <7%, but larger particles have a greater error but improve with increased concentration. The all-Teflon cone splitter potentially can split a water sample for subsequent analysis of semi-volatile organic chemicals without contamination. If the precision of the all-Teflon cone splitter is sufficient for the purposes of a given study, then it would be useful in obtaining water sample splits for analysis of suspended solids, semi-volatile organic chemicals, and other water-quality constituents.

Table 1. Particle-size classes and diameter ranges of the solids used in cone-splitter evaluation

[µm, micrometer]

Size class	Diameter range (µm)	Log diameter range (µm)
Sand		
Very coarse	2000 - 1000	3.30 - 3.00
Coarse	1000 - 500	3.00 - 2.70
Medium	500 - 250	2.70 - 2.40
Fine	250 - 125	2.40 - 2.10
Very fine	125 - 62	2.10 - 1.79
Silt		
Coarse	62 - 38	1.79 - 1.58
Coarse to medium	38 - 21	1.58 - 1.32
Medium to fine	21 - 10	1.32 - 1.00
Silt and clay		
Very fine	10 - 1	1.00 - 0.00

Table 2. Summary of water-splitting test

[%RSD, Percentage of relative standard deviation]

Description of test	Cone splitter	Number of tests	Average %RSD
Effect of Water Volume			
9 L	A	2	3.7
8 L	A	3	4.3
4 L	A	5	3.4
1 L	A	3	3.2
0.8 L	A	3	3.8
0.6 L	A	3	4.9
0.6 L	B	5	4.3
Effect of Combining Outlet Ports			
Eight way split	A	4	2.5
Five way split	A	4	3.5
Three way split	A	3	2.0
Effect of Incremental Sample Introduction			
Rotated while pouring	A	2	5.2
Poured from one location	A	6	4.4

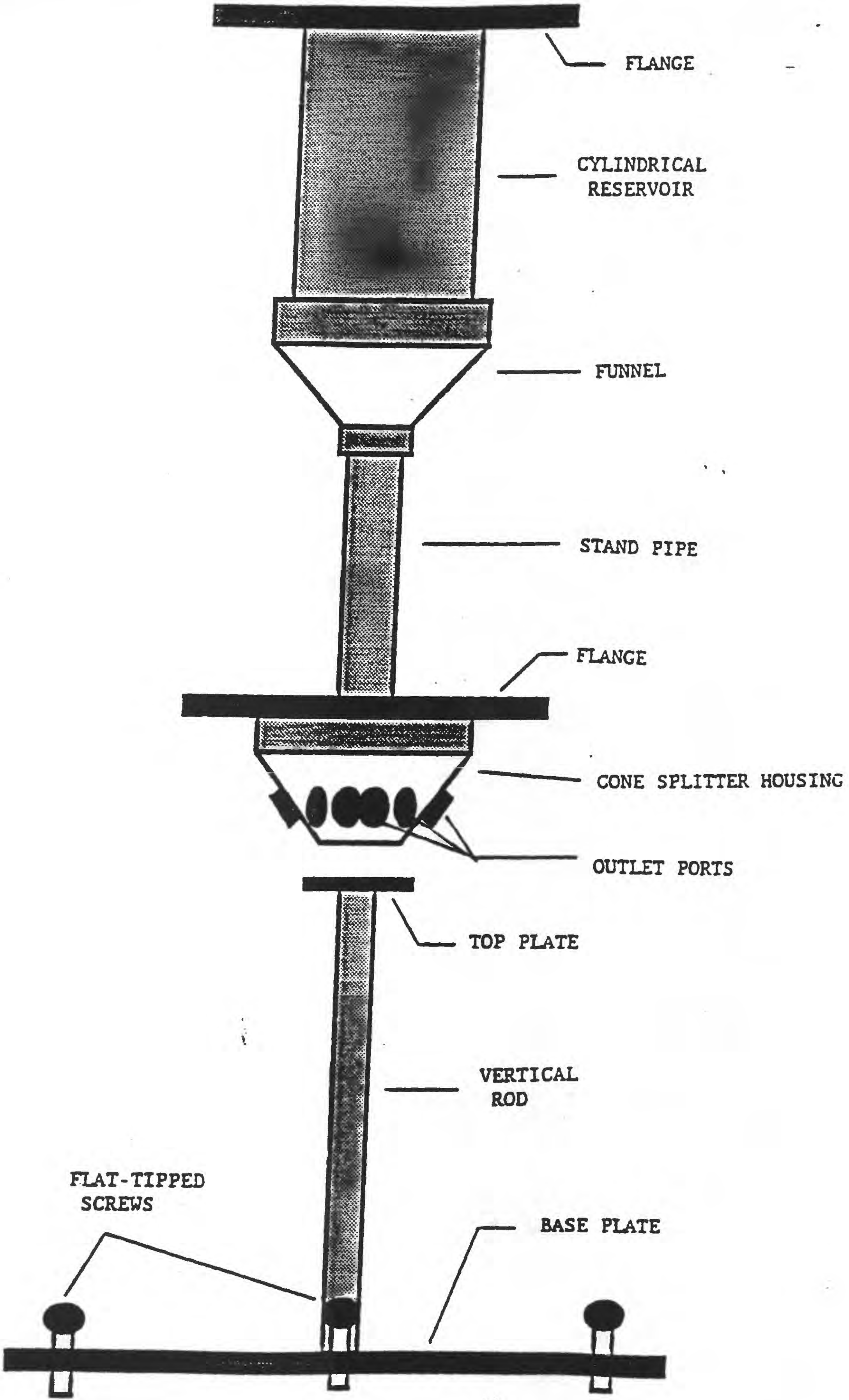

Figure 1. Schematic of the all-Teflon cone splitter and stand. The drawing is not to scale.

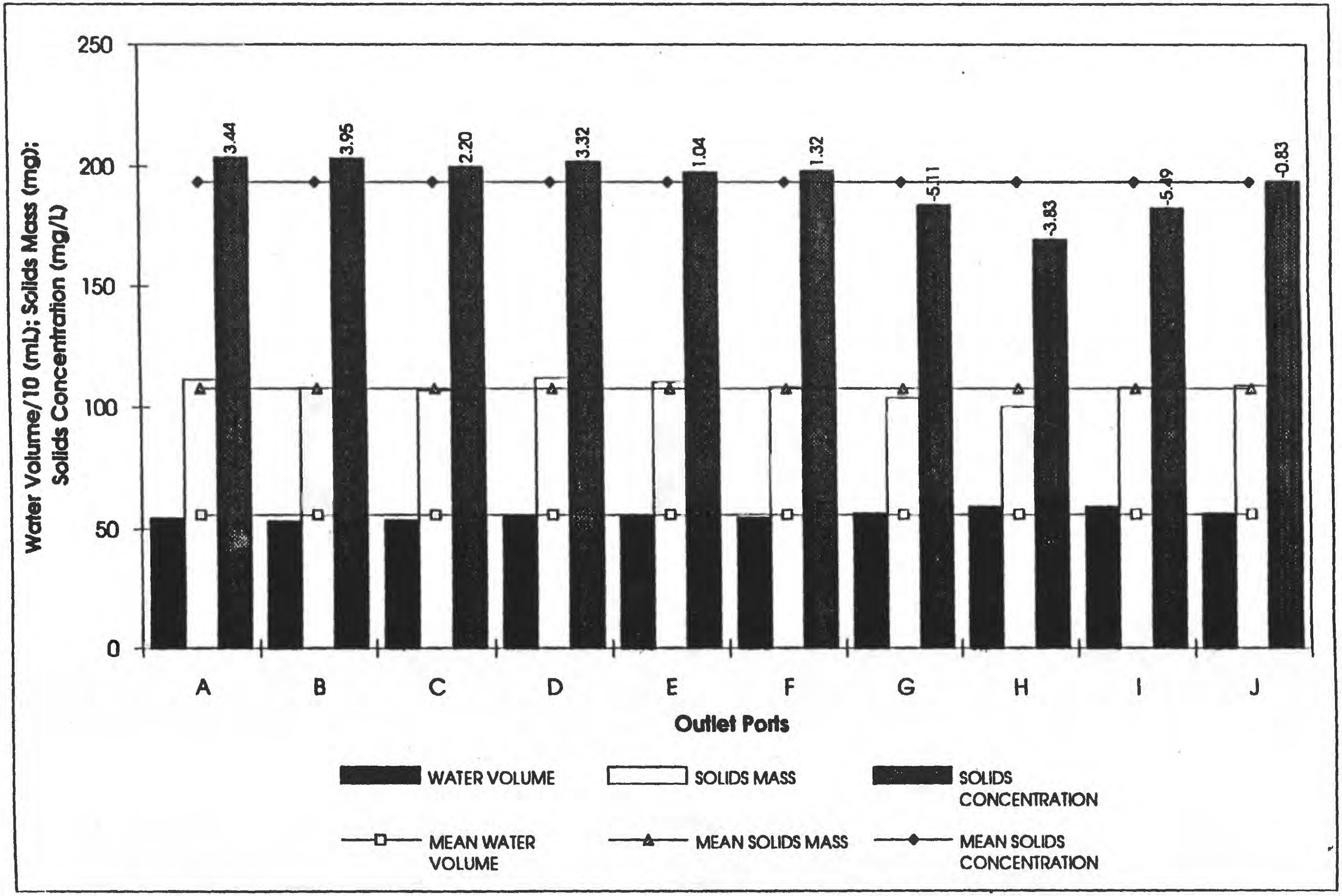

Figure 2. Example results from a split of suspended solids and water. Solids diameter: 21-38 µm; solids concentration 200 mg/L. The percentage of deviation from the mean for the solids concentration from each port is printed above the respective bar.

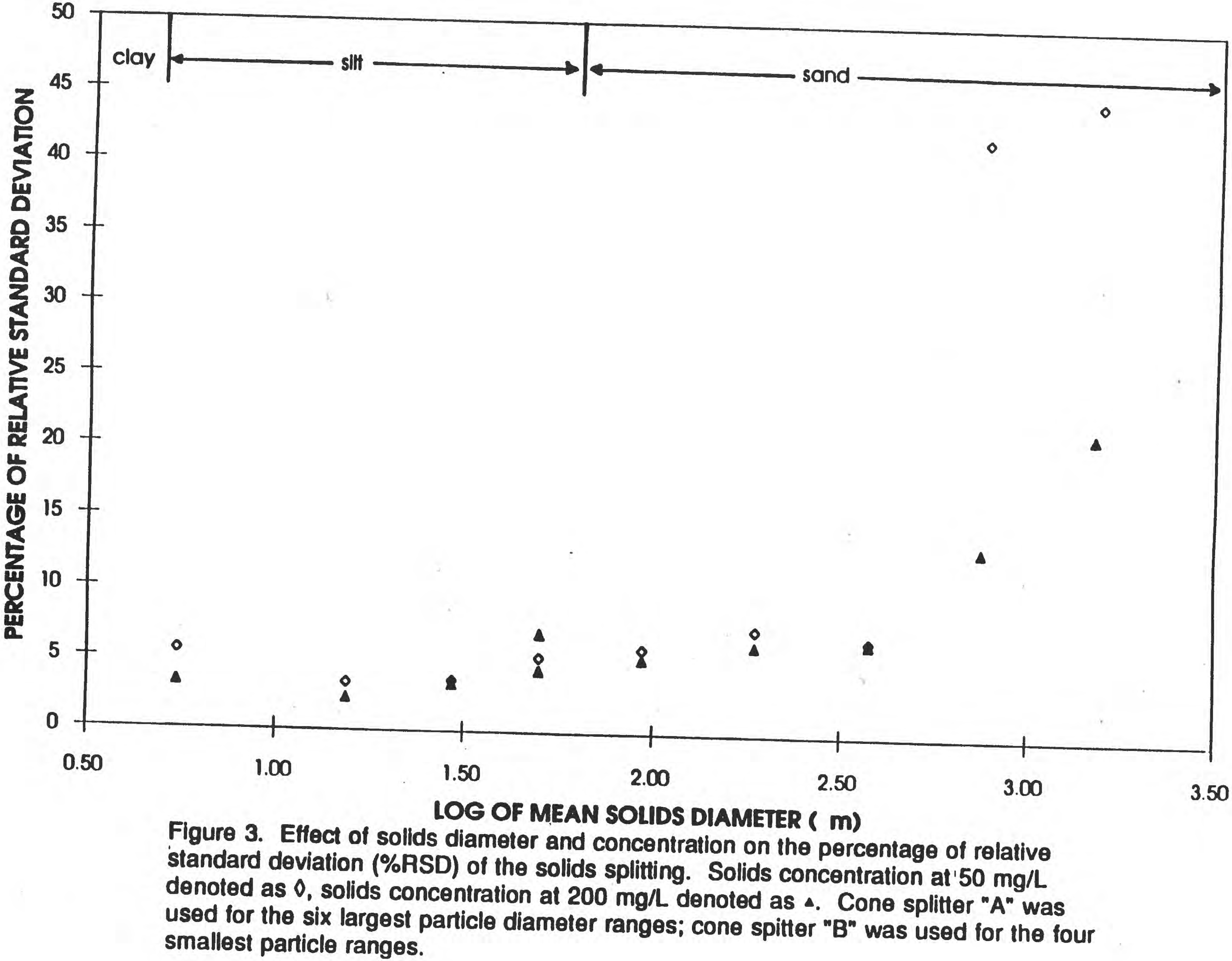

Figure 3. Effect of solids diameter and concentration on the percentage of relative standard deviation (%RSD) of the solids splitting. Solids concentration at 50 mg/L denoted as ◊, solids concentration at 200 mg/L denoted as ▲. Cone splitter "A" was used for the six largest particle diameter ranges; cone spitter "B" was used for the four smallest particle ranges.

United States Department of the Interior

U.S. GEOLOGICAL SURVEY
Reston, Virginia 22092

In Reply Refer To:
Mail Stop 412

April 21, 1994

OFFICE OF WATER QUALITY TECHNICAL MEMORANDUM 94.13

Subject: Evaluation of the Churn Splitter for Inclusion in the Division Protocol for the Collection and Processing of Surface-Water Samples for Subsequent Determination of Trace Elements, Nutrients, and Major Ions in Filtered Water

INTRODUCTION

The new inorganic protocol for filtered water (Office of Water Quality Technical Memorandum 94.09) requires the use of a modified churn splitter for purposes of compositing cross-sectional whole water samples. The new protocol was developed for the constituents and reporting limits listed in Table 1. For the majority of the trace elements, the reporting limit is 1 microgram per liter (µg/L). However, the reporting limit for iron (Fe), aluminum (Al), and zinc (Zn) is 3 µg/L. This limit was raised well beyond current quantification capabilities because these trace elements are commonly present in most field and laboratory working environments and, therefore, are very difficult to eliminate as contaminants.

Sample contamination typically falls into two categories--consistent or erratic. The cleaning, handling, and processing procedures included in the new protocol are designed to limit consistent contamination to less than half the stated reporting limits. The same procedures are also selected because they substantially reduce the chances of erratic contamination. The quality assurance/quality control (QA/QC) procedures and guidelines incorporated in the protocol are intended to provide adequate checks on potential sample contamination. Further, the QC data generated under the protocol are intended to provide defensible environmental data of known quality. Such information is a requisite for data interpretation.

PURPOSE

Since the new protocol was published, questions have arisen concerning the data supporting the use of the churn splitter. The purposes of this memorandum are to provide the justifying data and to certify the churn splitter for use with the protocol.

SCOPE

As is stated in the protocol, use of the churn requires (1) putting a limited-diameter funnel in the lid, (2) enclosing the churn inside two sealable polyethylene or polypropylene bags and placing these inside a churn carrier, and (3) replacing the existing spigot valve with a new one from the Quality of Water Service Unit (Ocala). An appropriate number of field blanks (defined in the protocol) should be run when any samples are collected. Furthermore, because the protocol was developed for filtered samples, an additional field blank should be run (including passing water through the spigot) when unfiltered samples are collected.

RINSING AND CONDITIONING SOLUTION TESTS

During the initial development of the protocol, a series of laboratory tests evaluated the effectiveness of preconditioning the processing equipment with deionized water (DIW) rather than with native water. Samples from five streams were processed and analyzed, and the results compared. Before filtering samples, a blank was run through the entire processing system (churn splitter, pump tubing, 142-millimeter (mm) plate filter, and a 142-mm 0.45-µm MFS filter) to evaluate whether the filtrates might be contaminated from the processing equipment. The equipment was thoroughly cleaned between each processed sample using the procedure provided in the protocol. After all the samples had been run, the system was cleaned one final time, and a final blank was run in the same manner as the first. The results for all the blank samples are in Table 2. The data listed in the rows marked "Conditioning Blank" represent duplicate aliquots of DIW used to condition the processing system. The data listed in the rows marked "Equipment Blank" come from the two separate aliquots of DIW that were actually passed through the processing system. Equipment Blank 1 was run at the beginning of the tests, whereas Equipment Blank 2 was run after all environmental samples had been processed. Little or no contamination was detected in blanks from the processing equipment.

CARRYOVER AND FIELD CLEANING TESTS

During the development of the office- and field-cleaning procedures, questions arose about potential "contaminant" carryover if a sample was obtained at a highly contaminated site followed by collection of a sample at a relatively pristine site. An evaluation of this potential problem, which would also provide a test for the proposed field-cleaning procedures, was designed and implemented. The two test sites were (1) Davis Mill Creek, in Copper Hill, Tennessee, and (2) Broad River at Bell, near Elberton, Georgia. Flow at the Davis Mill Creek site is highly contaminated by acidic discharges from abandoned copper mines, as well as by effluent from a chemical company. The Broad River site is in a rural, agricultural area. Both sites had been used extensively for previous studies on the evaluation of dewatering equipment.

The Davis Mill Creek site was sampled first. All the appropriate sampling and processing equipment had been cleaned and packaged in appropriate noncontaminating plastic containers in the office the previous day. Upon arrival at the site, a series of environmental subsamples was collected using a weighted bottle and composited in a standard 14-L churn splitter. The composite sample was processed using a peristaltic pump, silicon pump tubing, a GeoTech 142-mm nonmetallic filtering system, and a 142-mm 0.45-µm MFS filter. The filtrate was split between two bottles and acidified with Ultrex nitric acid. After the processing was completed, all equipment was thoroughly field cleaned. A new filter was placed in the filter holder, preconditioned with DIW, and a field blank was processed. The equipment was then repackaged in plastic bags for the drive to the Broad River site. Upon arrival at the second site, the procedure used at Davis Mill Creek was repeated. A sample was collected, composited, and processed. The system subsequently was disassembled and field cleaned, and a final field blank was processed.

The data for the actual samples and the subsequent blanks are presented in Table 3. The Davis Mill Creek site contained substantial quantities of "dissolved" iron (Fe), manganese (Mn), cobalt (Co), copper (Cu), and zinc (Zn). The analytical data for the subsequent blank indicate that the field-cleaning was sufficient to remove any traces of the processed sample as indicated by the low and/or "less than" concentrations of Fe, Mn, Co, Cu, and Zn. The source(s) for the measurable silver (Ag) in the blank is unknown. The Broad River sample contained little or no detectable trace elements or major ions, and the subsequent blank was essentially as clean as the one processed at the Davis Mill Creek site. Again, as with the Davis Mill Creek blank, Ag was detected, the source(s) of which is also unknown.

LOUISIANA SIDE-BY-SIDE FILTRATION ARTIFACT TESTS

During the development of the inorganic protocol, results from a series of filtration tests indicated that the type of filter used to process whole-water samples could have a substantial effect on "dissolved" trace-element concentrations (Horowitz and others, 1992). A further evaluation of this phenomenon was planned and carried out at three sites (Mississippi River at St. Francisville, Tangipahoa River at Robert, and Big Creek at Pollack) in Louisiana. The tests entailed the following: (1) all the equipment was cleaned following the procedures detailed in the protocol; (2) upon arrival at the site, a field blank using inorganic blank water (IBW) obtained from the Quality of Water Service Unit (Ocala) was processed and preserved following the procedures outlined in the protocol; and (3) a field sample was collected, processed, and preserved following the procedures outlined in the protocol. The data for the field blanks using capsule filters are provided in Table 4; the data indicate that the office-cleaned equipment and the field-collection and processing procedures used with the IBW are capable of limiting contamination to acceptable levels.

ENVIRONMENT CANADA/USGS/CANADIAN GEOLOGICAL SURVEY SIDE-BY-SIDE FILTRATION ARTIFACT TESTS

Based on mutual interest in further evaluating the effects of filtration artifacts on "dissolved" trace-element concentrations, a series of tests were planned by representatives of the USGS/WRD, Environment Canada, and the Canadian Geological Survey. The actual experimental work was carried out by Environment Canada at their St. Lawrence Center in Montreal. Five different samples were collected for processing: (1) a sample from an acid mine drainage site, (2) a sample from a peat bog, (3) a sample containing a high suspended-sediment concentration from the St. Lawrence River, (4) a sample containing a low suspended-sediment concentration from the St. Lawrence River, and (5) a sample from a near-neutral or alkaline river site. The samples were collected and brought to Montreal for processing. All the processing equipment (churn splitter, pump tubing, and various filters and filter holders) was cleaned per the protocol. Actual processing was carried out inside a laminar flow hood in a laboratory. Before processing each sample, a blank was run through each system. After each environmental sample was processed, the system was recleaned following the field-cleaning procedures described in the protocol. Before running the next sample, a new equipment blank was processed. This continued until all the samples had been processed through a variety of filtration devices. The acid mine drainage sample was processed first, followed by the peat bog sample, followed by the others.

The filtrates resulting from all the processed samples and blanks were split and subsequently analyzed by both the USGS National Water Quality Laboratory (NWQL) and the Canadian Geological Survey (Ottawa). The chemical data from both facilities are comparable. Table 5 contains USGS/NWQL-generated chemical data for both the blanks, as well as the acid mine and peat bog samples (2 of the 5 samples run during the experiment) processed with capsule filters. These two samples were selected because they were run in sequence and represent the worst-case scenario of a sample containing relatively low trace-element concentrations following the processing of one containing relatively high concentrations.

The data indicate that (1) the processing equipment started out at acceptably clean levels, (2) the acid mine drainage sample contained substantial amounts of selected trace elements (Mn, Ni, Cu, Zn, and Cd), (3) there were elevated Al and Zn levels in the acid mine drainage blanks, (4) the cleaning procedure readily removed residues from the acid mine sample before processing the next blank, (5) the peat bog sample did not contain excessive amounts of trace elements, and (6) the residues from the peat bog sample also were readily removed before processing the next blank (Table 5). The source(s) for the elevated Zn concentration in the first equipment blank is unknown; however, based on the results from the other blanks run during this study it should be viewed as erratic rather than consistent contamination (that is, it did not result from problems associated with the actual cleaning procedures because it did not show up in the other blanks). The Al concentration (1.4 µg/L) in the second equipment blank is elevated, but is viewed as being at an acceptable level because it is less than half the Al reporting limit (3 µg/L). The source(s) of the elevated B levels in the blanks and samples is unknown at the present time.

CONCLUSIONS

Data cited in this memorandum indicate that the cleaning procedures (office and field) incorporated in the new protocol limit consistent contamination associated with the churn splitter to concentrations less than one half the reporting limit. The extent to which some trace elements in blank samples were detected is typical of erratic contamination detected during normal quality-control tests. The data support the view that the cleaning procedures outlined in the protocol are appropriate for rendering the churn splitter sufficiently clean for use at the reporting limits listed in Table 1.

REFERENCE CITED

Horowitz, A.J., Elrick, K.A., and Colberg, M.R., 1992, The effect of membrane filtration artifacts on dissolved trace element concentrations: Water Resources, v. 26, p. 753-763.

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Attachment

This memorandum refers to Office of Water Quality Technical Memorandum 94.09.

Key Words: Analysis, protocol, surface water

Distribution: A, B, S, FO, PO, AH

Churn Splitter Data [all concentrations in µg/L except Ca, Mg, Na, and Si (mg/L)]

Table 1: Reporting Limits for the Constituents Covered by the Dissolved Inorganic Protocol

MS	MS	AES	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	AES	AES	AES	AES	AES	AES	AES	
Be	Al	Fe	Cr	Mn	Co	Ni	Cu	Zn	Mo	Ag	Cd	Sb	Ba	Pb	U	B	Li	V	Sr	Ca	Mg	Na	Si
0.5	3.0	3	1	1	1	1	1	3.0	1	1	1	1	1	1	1	1	4	6	0.5	0.02	0.01	0.2	0.01

Table 2: System Tests During Evaluations of Rinsing and Conditioning Solutions (Lab Environment)

	AES	AES	AES	AES	AES	GF	GF	AES	AES	GF	GF	AES	GF	AES	AES	AES	AES	AES	AES	AES
Sample	Be	Fe	Cr	Mn	Co	Ni	Cu	Zn	Mo	Ag	Cd	Ba	Pb	Li	V	Sr	Ca	Mg	Na	Si
Conditioning Blank	<0.5	<3	<5	<1	<3	<1	0.8	<3	<10	<1	<0.1	<1	<1	<4	<6	<0.5	<0.2	<0.1	<0.2	0.01
Conditioning Blank	<0.5	<3	<5	<1	<3	<1	1.2	<3	<10	<1	<0.1	<1	<1	<4	<6	<0.5	<0.2	<0.1	<0.2	0.02
Equipment Blank 1	<0.5	<3	<5	<1	<3	<1	<0.1	<3	<10	<1	<0.1	<1	<1	<4	<6	<0.5	<0.2	<0.1	<0.2	0.02
Equipment Blank 2	<0.5	<3	<5	<1	<3	<1	0.1	<3	<10	<1	<0.1	<1	<1	<4	<6	<0.5	<0.2	<0.1	<0.2	0.02

Table 3: System Tests During Evaluations of Carryover and Field Cleaning Procedures (Field Trials)

	AES	AES	AES	AES	AES	GF	GF	AES	AES	GF	GF	AES	GF	AES	AES	AES	AES	AES	AES	AES
Sample	Be	Fe	Cr	Mn	Co	Ni	Cu	Zn	Mo	Ag	Cd	Ba	Pb	Li	V	Sr	Ca	Mg	Na	Si
Initial Field Blank	<0.5	<3	<5	<1	<3	2	<1	<3	<10	<1	<0.1	<1	<1	<4	<6	0.9	0.16	<0.01	<0.2	0.01
Davis Mill Creek Sample	<0.5	51,000	<5	5400	126	20	315	5500	<10	1	4.1	20	1	8	<6	150	96	13	11.7	9.4
Field Blank After Cleanup	<0.5	4	<5	<1	<3	<1	<1	<3	<10	8	<0.1	<1	<1	<4	<6	<0.5	0.02	<0.01	<0.2	0.01
Broad River at Bell	<0.5	365	<5	16	<3	<1	<1	<3	<10	4	<0.1	11	<1	<4	<6	35	4.3	1.7	4.4	15
Field Blank After Cleanup	<0.5	<3	<5	<1	<3	<1	<1	<3	<10	7	<0.1	<1	<1	<4	<6	<0.5	<0.02	<0.01	<0.2	<0.01

Table 4: Field Blanks from Various Sites in Louisiana

	MS	MS	AES	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	AES	AES	AES	AES	AES	AES	AES	AES
Sample	Be	Al	Fe	Cr	Mn	Co	Ni	Cu	Zn	Mo	Ag	Cd	Sb	Ba	Pb	U	B	Li	V	Sr	Ca	Mg	Na	Si
Miss. R. @ St. Francisville	<0.2	<0.2	<3	<0.2	<0.2	<0.2	<0.2	0.3	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<2	<4	<6	<0.5	<0.02	<0.01	<0.2	0.01
Tangipahoa @ Robert	<0.2	<0.2	<3	<0.2	<0.2	<0.2	<0.2	0.3	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	2	<4	<6	<0.5	<0.02	<0.01	<0.2	0.06
Big Creek @ Pollack	<0.2	<0.2	<3	<0.2	<0.2	<0.2	0.8	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<2	<4	<6	<0.5	<0.02	<0.01	<0.2	0.02
Tangipahoa @ Robert	<0.2	0.3	<3	0.2	0.2	<0.2	<0.5	0.4	1.0	<0.5	<0.3	<0.3	<0.3	<0.4	<0.3	<0.2	<2	<4	<6	<0.5	0.1	<0.01	<0.2	0.04

Table 5: Test Data From the Canadian-USGS Side-by-Side Filtration Artifact Study

	MS	MS	AES	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	AES	AES	AES	AES	AES	AES	AES	AES
Sample	Be	Al	Fe	Cr	Mn	Co	Ni	Cu	Zn	Mo	Ag	Cd	Sb	Ba	Pb	U	B	Li	V	Sr	Ca	Mg	Na	Si
System Blank	<0.2	0.3	<3	<0.2	1.0	<0.2	<0.5	3.4	3.4	<0.2	<0.2	<0.3	<0.2	<0.4	<0.3	<0.2	11.9	<4	<6	0.6	<0.02	<0.01	<0.2	0.01
Acid Mine Drainage Sample	<0.2	7.4	3.0	<0.2	1700	10	65	16	2500	<0.2	<0.2	17	<0.2	21	<0.3	0.23	16.3	5.9	<6	500	130	62	3.6	6.5
System Blank	<0.2	1.4	<3	<0.2	0.1	<0.2	<0.5	0.3	0.6	<0.2	<0.2	<0.3	<0.2	<0.4	<0.3	<0.2	19.0	<4	<6	<0.5	<0.02	<0.01	<0.2	0.02
Peat Bog Sample	<0.2	149	118	<0.2	1.0	<0.2	0.7	1.7	4.8	3.1	<0.2	0.1	<0.2	9.1	1.5	<0.2	9.7	<4	<6	29	4.3	0.4	1.8	6.4
System Blank	<0.2	<0.3	<3	0.5	0.3	0.3	<0.5	<0.3	<0.5	<0.2	<0.2	<0.3	<0.2	<0.4	<0.3	<0.2	<2	<4	<6	<0.5	<0.02	<0.01	<0.2	0.01

"PROCEDURES FOR COLLECTION AND FIELD HANDLING OF SURFACE-WATER AND GROUND-WATER SAMPLES AND EQUIPMENT BLANKS FOR TRACE-ORGANIC ANALYSES"

For the National Blank and Spiking Projects (NBSP) in FY 1990

by

J. Zogorski, M. Sandstrom, and P. Capel

Water Resources Division
U. S. Geological Survey

June, 1990

Cleaning of Field Equipment

Field equipment, new or used, which will come into contact with "organic-free" water (used to obtain the Field Equipment Blank) and surface-water or ground-water samples must be cleaned prior to use. Examples of such equipment include surface-water sampler and sample bottle, submersible pump, milk can, filter apparatus, forceps, cone splitter, and Teflon tubing. The equipment can be cleaned in the District's laboratory or in the field. The equipment should be cleaned and stored in an environment free of organic contaminants (solvents, fumes, paints, etc.). As noted previously, the field equipment must be cleaned at the start of each day's sampling, if sampling is to be conducted over two or more days.

Three solutions will be used in the cleaning of field equipment in the NBSP:

Solution A - Alconox soap (or equivalent) prepared with tap water or distilled water.

Solution B - Tap water or distilled water, which must first be passed through a GAC filter.

Solution C - Methanol, pesticide quality (to be provided by the NWQL's DENSUPPLY).

The authors recognize that: (1) commercially prepared "organic-free" water could be used in solutions A and B; and (2) some tap waters contain low levels of organic contaminants, such as pesticides and VOCs. However, the use of "organic-free" water for solutions A and B may not be practical for routine District sampling, due to the high cost of purchasing large volumes of "organic-free' water commercially, or the need to maintain a QA/QC program for locally prepared "organic-free" water. The approach being taken in the NBSP initially is to specify the use of GAC treated tap water or GAC treated distilled water for solution B only, and to monitor Field Equipment Blanks for contamination. If contamination is found in these blanks, follow-up studies will be conducted to identify the source of the contaminant(s). Then, if appropriate, changes to the make-up of the cleaning solutions will be made. As implied above, regular tap water or distilled water can not be used as a substitute for wash solution "B". These waters must be passed through a GAC filter prior to use as Solution "B".

District personnel should wear rubber gloves when washing and rinsing with solutions A and B, and disposable laboratory gloves and safety glasses during the methanol rinsing of equipment. The prescribed cleaning procedure is as follows:

1. In a sink, large container, or tube (material not specified), soak and wash the equipment with a laboratory soap, such as Alconox. As noted previously, the soap solution can be prepared with tap water or distilled water. The soap solution should be warm and made following the manufacturer's directions. Do not make it too strong, or you will have difficulty removing the residual soap from the equipment. (Note: the manufacturer's recommended solution may be too strong and can be altered, as appropriate). For the ground-water pump and Teflon line, washing can best be done by filling a tube or cylinder (containing the pump), closed at one end, with the soap solution and turning on the pump while continually adding soap solution until there is enough volume to fill the cylinder. Place the open end of the Teflon tubing into the cylinder and allow the soap solution to recirculate. Similarly the filtration apparatus (filter unit, filter pump, Teflon line, etc.) is best cleaned by pumping the soap solution through the fully assembled apparatus.

2. Rinse the equipment, inside and out, twice with GAC treated tap water or GAC treated distilled water, or until soap bubbles are no longer present. Again, this is best done with the pump and filter apparatus fully assembled by pumping the cleaning solution, as described in 1) above.

3. Using a Teflon-squeeze bottle containing pesticide-grade methanol, rinse all of the inside surfaces of equipment. For the ground-water pump and filtration apparatus this is done best by pumping methanol through the apparatus fully assembled. A stainless-steel aluminum, glass, or Teflon cylinder must be used during this step to clean the submersible pump.

4. Excess methanol should be drained or shaken from the equipment. Care should be taken so that the freshly cleaned surfaces, which will come in contact with the water sample, are not touched by hands, plastic, or paper. Freshly cleaned equipment can be placed on a sheet of aluminum foil to air dry. Place the equipment on the dull side of the foil, since the shinny side may contain an organic film.

5. Disassemble the Teflon tubing connecting the filter apparatus and pump. Open parts of the sampling and processing equipment should be covered with heavy-duty aluminum foil. Again, only the dull side of aluminum foil should contact surface which will come into contact with water samples.
6. All equipment should be stored in an environment free of organic contaminants such as solvents, fumes, paints, etc.

It is a common practice to dispose of diluted waste methanol by flushing it down a sink with lots of tap water. However, District personnel must inquire with local officials to ascertain an acceptable disposal plan for the waste methanol. Please note that methanol, unless diluted with considerable water, is flammable.

APPENDIX B: ORIGINAL UNPUBLISHED U.S. GEOLOGICAL SURVEY DATA FROM TESTS OF SPLITTING DEVICES

Cleaning study of Teflon cone for inorganic constituents.

Concentrations (in micrograms per liter) for trace elements, major cations, major anions, and nutrients in de-ionized water before and after it was passed through a cleaned Teflon cone split

	de-ionized water	cone split 1	cone split 2	cone split 3
nitrate (as N)	2	1	1	1
nitrate + nitrite (as N)	<5	<5	<5	<5
ortho-phosphorous (as P)	<1	<1	<1	<1
calcium	<2	2	3	2
magnesium	<1	<1	<1	1
sodium	<25	<25	<25	<25
silica	<20	<20	<20	<20
barium	<0.2	<0.2	<0.2	<0.2
beryllium	<0.2	<0.2	<0.2	<0.2
boron	<20	<20	<20	<20
cadium	<0.3	<0.3	<0.3	<0.3
chromium	<0.2	<0.2	<0.2	<0.2
cobalt	<0.2	<0.2	<0.2	<0.2
copper	<0.2	<0.2	<0.2	<0.2
iron	<3	<3	<3	<3
lead	<0.3	<0.3	<0.3	<0.3
manganese	<0.1	<0.1	<0.1	<0.1
thallium	<0.1	<0.1	<0.1	<0.1
molybedenum	<0.2	<0.2	<0.2	<0.2
nickel	<0.5	<0.5	<0.5	<0.5
silver	<0.2	<0.2	<0.2	<0.2
strontium	<0.1	<0.1	<0.1	<0.1
zinc	<0.5	<0.5	0.96	0.58
antimony	<0.2	<0.2	<0.2	<0.2
aluminum	<0.3	0.56	0.36	0.52
uranium	<0.2	<0.2	<0.2	<0.2
chloride	NA	30	30	40
sulfate	<10	<10	<10	<10
fluoride	<10	<10	<10	<10
bromide	<10	<10	<10	<10

From Shelton, L.R. and Capel, P.D., 1994

Concentrations in micrograms per liter (ug/L), <0.1, less than 0.1

Organic constituent	National Water-Quality Laboratory reporting limit	Constituent concentration in field equipment blank for the following water-quality sampling stations: S1-44B Connecticut 4153510723500501	IRA-21C Iowa 415020092094003	Laguna #2 California 382550121273202
		Chlorophenoxy acid herbicides		
2,4-D	0.01	<0.01	0.06	>0.01
2,4,5-T	0.01	<0.01	0.02	>0.01
silvex	0.01	<0.01	0.02	>0.01
dicamba	0.01	<0.01	0.02	>0.01
		Carbamate insecticides		
carbaryl	0.5	<0.5	0.6	<0.5
propham	0.5	<0.5	<0.5	<0.5
methomyl	0.5	<0.5	<0.5	<0.5
oxamyl	0.5	<0.5	<0.5	<0.5
carbofuran	0.5	<0.5	<0.5	<0.5
aldicarb	0.5	<0.5	<0.5	<0.5
aldicarb sulfoxide	0.5	<0.5	<0.5	<0.5
aldicarb sulfone	0.5	<0.5	<0.5	<0.5
1-naphthol	0.5	<0.5	<0.5	<0.5
3-hydroxycarbofuran	0.5	<0.5	<0.5	<0.5
		Triazines and other nitrogen-containing herbicides		
prometryne	0.1	<0.1	<0.1	---
atrazine	0.1	<0.1	<0.1	---
prometone	0.1	<0.1	<0.2	---
simazine	0.1	<0.1	<0.1	---
simetryne	0.1	<0.1	<0.1	---
propazine	0.1	<0.1	<0.1	---
cyanazine	0.1	<0.1	<0.2	---
ametryne	0.1	<0.1	<0.1	---
alachlor	0.1	<0.1	<0.2	---
trifluralin	0.1	<0.1	<0.1	---
metribuzin	0.1	<0.1	<0.1	---
metolachlor	0.1	<0.1	<0.2	---
		organochlorine and organophorous insecticides		
perthane	0.1	<0.1	<0.1	<0.1
chlordane	0.1	<0.1	<0.1	<0.1
toxaphene	1.0	<1.0	<1.0	<1.0
diazinion	0.01	<0.01	<0.01	<0.1
ethion	0.01	<0.01	<0.01	<0.1
malathion	0.01	<0.01	<0.01	<0.1
methyl parathion	0.01	<0.01	<0.01	<0.1
methyl trithion	0.01	<0.01	<0.01	<0.1
parathion	0.01	<0.01	<0.01	<0.1
trithion	0.01	<0.01	<0.01	<0.1
PCB	0.1	<0.1	<0.1	<0.1
PCN	0.1	<0.1	<0.1	<0.1
methoxychlor	0.01	<0.01	<0.01	<0.01
mirex	0.01	<0.01	<0.01	<0.01
endosulfan	0.001	<0.001	<0.001	<0.001
aldrin	0.001	<0.001	<0.001	<0.01
DDD	0.001	<0.001	<0.001	<0.001
DDE	0.001	<0.001	<0.001	<0.001
DDT	0.001	<0.001	<0.001	<0.001
dieldrin	0.001	<0.001	<0.001	<0.001
endrin	0.001	<0.001	<0.001	<0.001
heptachlor	0.001	<0.001	<0.001	<0.001
heptachlor epoxide	0.001	<0.001	<0.001	<0.01
lindane	0.001	<0.001	<0.001	<0.001
chlorpyrifos	0.01	<0.01	<0.01	<0.1
fonofos	0.01	<0.01	<0.01	<0.1
phorate	0.01	<0.01	<0.01	<0.1
disyston	0.01	<0.01	<0.01	<0.01
DEF	0.01	<0.01	<0.01	<0.1
2,4-DP	0.01	<0.01	<0.01	<0.01

Churn splitter evaluation for solids splitting.

	A	B	C	D	E	F	G
1	Churn splitter results for sand with a total concentration of				20,000 mg/l		
2		14 L churn filled with 10 l of water					
3							
4	Weight added = 60 +2.26% * 140 gm =			63.164	gram Churn 1		
5	Weight added = 60 +2.20% * 140 gm =			63.080	gram Churn 2		
6			30 % sand results		True Conc =	6.3122	Concen gm/l
7		Test 1, Chrn 1, Sample A		Test 2, Chrn 2, Sample B		Test 3, chm 1, sample C	
8	Subsample	Wt in sample	Concen gm/l	Wt in sample	Concen gm/l	Wt in sample	Concen gm/l
9	1 top	6.0085	6.01	8.4846	8.48	4.8818	4.88
10	2	4.0048	4.00	8.948	8.95	4.2968	4.30
11	3	4.8024	4.80	7.3958	7.40	5.4936	5.49
12	4	4.5984	4.60	7.8447	7.84	4.9608	4.96
13	5	4.3741	4.37	7.1751	7.18	8.7976	8.80
14	6 bottom	4.8048	4.80	7.0129	7.01	6.2132	6.21
15	R (4L)	35.3794	8.84	16.6079	4.15	29.4154	7.35
16	Total sand	63.9724		63.469		64.0592	
17	Percent error	1.28		0.62		1.42	
18							
19			5 % sand results				
20	Weight added = 10 +2.31% * 190 gm =			14.389	gram		
21			True conc =	1.4389	conc gm/l		
22		Test 4, Chrn 1, Sample D		Test 5, Chrn 1, Sample E		Test 6, Chrn 1, Sample F	
23	Subsample	Wt in sample	Concen gm/l	Wt in sample	Concen gm/l	Wt in sample	Concen gm/l
24	1 top	2.2891	2.29	2.2292	2.23	2.1565	2.16
25	2	1.6923	1.69	2.0803	2.08	1.9871	1.99
26	3	1.5194	1.52	2.3704	2.37	1.8708	1.87
27	4	2.1838	2.18	1.9007	1.90	1.5402	1.54
28	5	1.3249	1.32	1.7632	1.76	1.7822	1.78
29	6 bottom	1.7332	1.73	1.402	1.40	1.0261	1.03
30	R (4L)	5.0029	1.25	3.7355	0.93	5.2406	1.31
31	Total sand	15.7456		15.4813		15.6035	
32	Percent error	9.43		7.59		8.44	

From Branch of Quality Assurance, 1990

Table 1. Volume data using distilled water

[Direction of pour toward tube number indicated; R, rinse]

Test number and total volume (mL)	Direction of pour	Temperature (°C)	Volume of subsample, in milliliters									
			Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6	Tube 7	Tube 8	Tube 9	Tube 10
1 - 2,500	1	24	250	252	244	243	237	242	246	257	257	258
2 - 2,500	1	23	249	254	248	251	242	245	246	255	254	254
3 - 2,500	1	25	247	253	246	249	244	246	247	256	253	254
4 - 2,500	1	24	259	262	250	245	234	235	237	252	258	265
5 - 2,500	6	26	240	241	236	243	244	254	258	265	256	252
6 - 2,500	6	25	239	239	234	241	244	256	260	268	259	252
7 - 2,500	6	25	245	248	252	252	247	250	249	254	250	251
8 - 2,500	6	23	242	249	247	253	249	253	251	254	249	248
9 - 2,500	6	23	241	248	244	250	247	252	253	256	250	248
1 - 2,500	1	4	251	253	241	248	243	248	247	252	250	260
2 - 2,500	6	4	241	245	243	250	247	252	252	259	255	252
1 - 250	6	21	24.5	25.1	24.7	24.5	24.1	24.5	24.9	25.1	24.7	25.2
2 - 250	1	21	24.6	25.1	24.2	24.4	24.0	24.4	24.5	25.4	24.9	25.5
3 - 250	1	22	24.3	24.9	24.3	24.7	24.7	25.0	24.5	25.3	24.9	24.8
4 - 250	1	22	24.6	25.2	24.7	24.9	25.1	24.5	24.6	25.1	25.0	25.4
5 - 250	1	22	24.7	25.3	24.7	24.7	24.4	24.6	24.6	25.5	25.2	25.3
6 - 250	1	22	24.8	25.3	24.5	24.7	24.4	24.7	24.8	25.7	25.2	25.5
1 - 100	1	23	9.3	9.7	9.3	9.6	9.3	9.7	9.7	10.1	9.7	9.7
2 - 100	1	23	9.2	9.7	9.6	9.9	9.8	9.6	9.7	9.8	9.4	9.5
3 - 100	6	23	11.2	14.9	9.6	9.3	10.7	12.4	8.5	6.8	7.0	7.9
4 - 100	6	23	8.4	8.3	7.9	9.8	12.8	15.0	11.3	8.1	8.2	8.4
1 - ~150 R	6	23	7.9	9.4	12.1	12.1	16.6	15.7	25.7	20.6	9.7	9.0
2 - ~100 R	6	23	9.5	6.8	10.8	14.8	11.0	6.0	11.4	16.7	7.9	5.8
3 - ~125 R	1	23	10.6	9.9	10.7	11.6	14.1	10.0	14.1	14.4	13.1	10.9
4 - ~100 R	1	23	6.0	8.6	10.1	8.8	16.1	9.5	18.9	13.4	7.2	5.6

for plastic cone}

Volume of subsample, in percent, using equation 1										Splitter	Container	Splitter	Container
										Remained			
Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6	Tube 7	Tube 8	Tube 9	Tube 10	mL		Percent	
10.1	10.1	9.8	9.8	9.6	9.7	9.9	10.3	10.3	10.4	--	3	--	--
10.0	10.2	9.9	10.0	9.7	9.8	9.8	10.2	10.2	10.2	--	1	--	--
9.9	10.1	9.9	10.0	9.8	9.9	9.9	10.2	10.1	10.2	--	2	--	--
10.4	10.5	10.0	9.8	9.4	9.4	9.5	10.1	10.3	10.6	--	2	--	--
9.6	9.7	9.5	9.8	9.8	10.2	10.4	10.6	10.3	10.1	--	3	--	--
9.6	9.6	9.4	9.7	9.8	10.3	10.4	10.7	10.4	10.1	--	1	--	--
9.8	9.9	10.1	10.1	9.9	10.0	10.0	10.2	10.0	10.0	--	2	--	--
9.7	10.0	9.9	10.1	10.0	10.1	10.1	10.2	10.0	9.9	--	1	--	--
9.7	10.0	9.8	10.0	9.9	10.1	10.2	10.3	10.0	10.0	--	2	--	--
10.1	10.1	9.7	10.0	9.8	9.9	9.9	10.1	10.0	10.4	--	2	--	--
9.7	9.8	9.7	10.0	9.9	10.1	10.1	10.4	10.2	10.1	--	3	--	--
9.9	10.1	10.0	9.9	9.8	9.9	10.1	10.1	10.0	10.2	--	0	--	--
10.0	10.1	9.8	9.9	9.7	9.9	9.9	10.3	10.1	10.3	--	1	--	--
9.8	10.1	9.8	10.0	10.0	10.1	9.9	10.2	10.1	10.0	--	1	--	--
9.9	10.1	9.9	10.0	10.1	9.8	9.9	10.1	10.0	10.2	--	0	--	--
9.9	10.2	9.9	9.9	9.8	9.9	9.9	10.2	10.1	10.2	--	1	--	--
9.9	10.1	9.9	9.9	9.8	9.9	9.9	10.3	10.1	10.2	--	0	--	--
9.7	10.1	9.7	10.0	9.7	10.1	10.1	10.4	10.1	10.1	--	1	--	--
9.6	10.0	10.0	10.3	10.2	10.0	10.0	10.2	9.8	9.9	--	1	--	--
11.4	15.2	9.8	9.5	10.9	12.6	8.6	6.9	7.1	8.0	--	1	--	--
8.6	8.4	8.0	10.0	13.0	15.3	11.5	8.2	8.4	8.6	--	1	--	--
5.7	6.8	8.7	8.7	12.0	11.3	18.5	14.8	7.0	6.5	--	--	--	--
9.4	6.8	10.7	14.7	10.9	6.0	11.3	16.6	7.8	5.8	--	--	--	--
8.9	8.3	9.0	9.7	11.8	8.4	11.8	12.0	10.9	9.2	--	--	--	--
5.8	8.3	9.7	8.4	15.4	9.1	18.1	12.9	6.9	5.4	--	--	--	--

Table 2. Volume, weight, and concentration data using sand and distilled water mixture

[Direction of pour toward tube number indicated. Original data from C.J. Durham and S.W. McKenzie, 1985.]

Test number and total volume (mL)	Direction of pour	Temperature (°C)	Volume of subsample, in milliliters									
			Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6	Tube 7	Tube 8	Tube 9	Tube 10
1 - 2,500	6	24	229	235	239	252	258	265	264	263	249	240
2 - 2,500	6	24	237	241	239	246	249	257	259	263	254	249
3 - 2,500	1	24	255	261	252	252	241	241	239	248	250	258
4 - 2,500	1	24	259	260	248	243	233	235	239	253	258	264
5 - 2,500	1	24	254	257	245	245	237	240	242	254	257	261
			Weight of subsample, in milligrams									
1 - 2,500	6	24	222.1	223.6	234.3	238.1	236.9	240.8	235.1	235.4	228.9	228.2
2 - 2,500	6	24	201.0	220.8	212.7	219.3	229.0	252.5	263.9	269.7	259.7	243.0
3 - 2,500	1	24	234.6	244.3	243.6	246.0	236.7	234.3	230.2	241.1	241.5	246.3
4 - 2,500	1	24	249.8	247.8	230.8	217.6	208.3	215.1	226.4	245.3	252.3	255.5
5 - 2,500	1	24	259.7	260.6	247.5	243.4	237.2	232.5	229.0	239.7	248.6	263.9
			Concentration of subsample, in milligrams per liter									
1 - 2,500	6	24	970	994	980	945	918	909	890	895	919	951
2 - 2,500	6	24	848	916	890	892	920	982	1,019	1,026	1,022	976
3 - 2,500	1	24	920	936	968	976	982	972	963	972	966	955
4 - 2,500	1	24	964	953	931	896	894	915	947	970	978	968
5 - 2,500	1	24	1,022	1,014	1,011	994	1,001	969	946	944	967	1,011

Volume of subsample, in percent, using equation 1										Splitter	Container	Splitter	Container
										Remained			
Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6	Tube 7	Tube 8	Tube 9	Tube 10	mL		Percent	
9.2	9.4	9.6	10.1	10.3	10.6	10.6	10.6	10.0	9.6	--	2	--	--
9.5	9.7	9.6	9.9	10.0	10.3	10.3	10.5	10.2	10.0	--	2	--	--
10.2	10.4	10.1	10.1	9.7	9.7	9.6	9.9	10.0	10.3	--	2	--	--
10.4	10.4	10.0	9.8	9.3	9.4	9.6	10.2	10.3	10.6	--	2	--	--
10.2	10.3	9.8	9.8	9.5	9.7	9.7	10.2	10.3	10.5	--	2	--	--
Weight of subsample, in percent, using equation 3										mg		Equation 3	
8.90	9.37	9.39	9.55	9.50	9.65	9.42	9.44	9.18	9.15	115.7	45.2	4.64	1.81
8.13	8.93	8.60	8.87	9.26	10.21	10.67	10.91	10.50	9.83	55.9	45.4	2.26	1.83
9.41	9.79	9.77	9.86	9.49	9.39	9.23	9.67	9.68	9.88	89.5	6.1	3.59	.24
9.99	9.91	9.23	8.71	8.33	8.61	9.06	9.81	10.09	10.22	97.6	53.2	3.91	2.13
10.38	10.42	9.90	9.73	9.48	9.30	9.16	9.58	9.94	10.55	35.3	3.9	1.41	.15
Subsample, in percent, using equation 2													
10.35	10.61	10.46	10.08	9.80	9.70	9.50	9.55	9.81	10.14	--	--	--	--
8.94	9.65	9.38	9.40	9.69	10.35	10.74	10.80	10.77	10.28	--	--	--	--
9.57	9.74	10.06	10.16	10.22	10.12	10.02	10.12	10.05	9.94	--	--	--	--
10.24	10.12	9.88	9.51	9.50	9.72	10.06	10.30	10.39	10.28	--	--	--	--
10.35	10.26	10.23	10.06	10.13	9.81	9.58	9.55	9.79	10.24	--	--	--	--

Table 3. Volume, weight, and concentration data using urban storm runoff

[Direction of pour toward tube number indicated. Original data from C.J. Durham and S.W. McKenzie, 1985.]

Test number and total volume (mL)	Direction of pour	Temperature (°C)	Volume of subsample, in milliliters									
			Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6	Tube 7	Tube 8	Tube 9	Tube 10
1 - 1,954	6	23	194	200	195	195	191	190	188	196	197	197
2 - 1,899	6	23	190	193	186	188	182	185	184	193	192	194
3 - 1,817	6	23	177	182	179	182	179	181	181	184	182	182
			Weight of < 62 µm subsample, in milligrams									
1 - 1,954	6	23	279.5	292.5	295.3	296.1	292.3	287.2	286.1	296.7	298.2	296.9
2 - 1,899	6	23	280.2	289.9	277.2	281.9	265.1	270.0	269.8	272.3	283.6	280.0
3 - 1,817	6	23	265.6	277.1	270.9	268.8	263.1	271.3	268.5	275.9	272.5	277.2
			Weight of > 62 µm subsample, in milligrams									
1 - 1,954	6	23	42.5	44.7	39.1	40.4	34.9	38.8	37.7	41.3	42.0	42.0
2 - 1,899	6	23	43.4	37.9	41.3	39.9	47.1	43.0	43.0	56.0	40.8	44.3
3 - 1,817	6	23	42.4	40.2	37.3	41.0	43.6	41.1	44.2	43.0	43.6	43.5
			Concentration of < 62 µm subsample, in milligrams per liter									
1 - 1,954	6	23	1,441	1,462	1,514	1,518	1,530	1,512	1,522	1,514	1,514	1,507
2 - 1,899	6	23	1,475	1,502	1,490	1,500	1,457	1,460	1,466	1,411	1,477	1,443
3 - 1,817	6	23	1,501	1,522	1,513	1,477	1,470	1,499	1,483	1,500	1,497	1,523
			Concentration of < 62 µm subsample, in milligrams per liter									
1 - 1,954	6	23	1,441	1,462	1,514	1,518	1,530	1,512	1,522	1,514	1,514	1,507
2 - 1,899	6	23	1,475	1,502	1,490	1,500	1,457	1,460	1,466	1,411	1,477	1,443
3 - 1,817	6	23	1,501	1,522	1,513	1,477	1,470	1,499	1,483	1,500	1,497	1,523
			Concentration of > 62 µm subsample, in milligrams per liter									
1 - 1,954	6	23	219.1	223.5	200.5	207.2	182.7	204.2	200.5	210.7	213.2	213.2
2 - 1,899	6	23	228.4	196.4	222.0	212.2	258.8	232.4	233.7	290.2	212.5	228.4
3 - 1,817	6	23	239.5	220.9	208.4	225.3	243.6	227.1	244.2	233.7	239.6	239.0
			Concentration of > 62 µm subsample, in milligrams per liter									
1 - 1,954	6	23	219.1	223.5	200.5	207.2	182.7	204.2	200.5	210.7	213.2	213.2
2 - 1,899	6	23	228.4	196.4	222.0	212.2	258.8	232.4	233.7	290.2	212.5	228.4
3 - 1,817	6	23	239.5	220.9	208.4	225.3	243.6	227.1	244.2	233.7	239.6	239.0

Volume of subsample, in percent, using equation 1										Splitter	Container	Splitter	Container
										Remained			
Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6	Tube 7	Tube 8	Tube 9	Tube 10	mL		Percent	
10.0	10.4	10.0	10.0	9.8	9.8	9.7	10.1	10.1	10.1	--	3	--	--
10.1	10.2	9.9	10.0	9.6	9.8	9.7	10.2	10.2	10.3	--	3	--	--
9.7	10.1	9.9	10.1	9.9	10.0	10.0	10.1	10.1	10.1	--	2	--	--
Weight of < 62 μm subsample, in percent, using equation 3										mg		Equation 3	
9.33	9.77	9.86	9.89	9.76	9.59	9.54	9.91	9.96	9.91	63.5	10.8	2.12	.36
9.78	10.12	9.67	9.84	9.25	9.42	9.41	9.50	9.90	9.77	70.3	25.5	2.45	.89
9.53	9.94	9.71	9.64	9.43	9.73	9.63	9.89	9.77	9.94	51.9	25.8	1.86	.93
Weight of < 62 μm subsample, in percent, using equation 3										mg		Equation 3	
9.45	9.94	8.70	9.00	7.70	8.63	8.39	9.19	9.34	9.34	43.4	2.7	9.66	.60
9.23	8.06	8.78	8.48	10.01	9.14	9.14	11.90	8.68	9.42	30.5	3.2	6.48	.68
9.36	8.87	8.24	9.05	9.63	9.06	9.80	9.49	9.63	9.60	29.0	4.0	6.40	.87
Subsample of < 62 μm subsample, in percent, using equation 2													
9.6	9.7	10.1	10.1	10.2	10.0	10.1	10.1	10.1	10.0	--	--	--	--
10.0	10.2	10.2	10.2	9.9	9.9	10.0	9.6	10.1	9.9	--	--	--	--
10.0	10.2	10.1	9.8	9.8	10.0	9.9	10.0	10.0	10.2	--	--	--	--
Subsample of < 62 μm subsample, in percent, using equation 4													
8.42	8.55	8.85	8.88	8.94	8.83	8.89	8.85	8.85	8.81	--	--	--	--
8.68	8.83	8.77	8.82	8.57	8.59	8.63	8.30	8.69	8.49	--	--	--	--
8.67	8.81	8.75	8.53	8.49	8.66	8.57	8.66	8.65	8.81	--	--	--	--
Subsample of > 62 μm subsample, in percent, using equation 2													
10.5	10.8	9.7	10.0	8.8	9.8	9.7	10.1	10.3	10.3	--	--	--	--
9.9	8.5	9.6	9.2	11.1	10.0	10.1	12.5	9.2	9.9	--	--	--	--
10.3	9.5	9.0	9.7	10.5	9.8	10.5	10.1	10.3	10.3	--	--	--	--
Subsample of > 62 μm subsample, in percent, using equation 5													
1.28	1.31	1.17	.121	1.07	1.19	1.17	1.23	1.25	1.25	--	--	--	--
1.34	1.16	1.31	1.25	1.52	1.37	1.38	1.71	1.25	1.34	--	--	--	--
1.38	1.28	1.20	1.30	1.41	1.31	1.41	1.35	1.38	1.38	--	--	--	--

Table 4. Summary statistics of data from sample splitter

[Direction of pour toward tube number indicated; DW, distilled water; R, rinse. Original data from C.J. Durham and S.W. McKenzie, 1985.]

Description of sample	Test	Temperature (°C)	Volume (ml.)	Direction of pour	Subsample, volume in milliliters					Subsample, in percentage of total from equation 1				
					Maximum	Minimum	Median	Mean	Standard deviation	Maximum	Minimum	Median	Mean	Standard deviation
DW	1	24	2,500	1	258	237	248.0	248.6	7.11	10.4	9.5	10.0	10.0	0.30
DW	2	23	2,500	1	255	242	250.0	249.8	4.52	10.2	9.7	10.0	10.0	.19
DW	3	25	2,500	1	256	244	248.0	249.5	4.14	10.3	9.8	9.95	10.0	.16
DW	4	24	2,500	1	265	234	251.0	249.8	11.39	10.6	9.4	10.0	10.0	.46
DW	5	26	2,500	6	265	236	248.0	248.9	9.40	10.6	9.5	9.95	10.0	.37
DW	6	25	2,500	6	268	234	248.0	249.2	11.32	10.8	9.4	9.95	10.0	.46
DW	7	24	2,500	6	254	245	250.0	249.8	2.66	10.2	9.8	10.0	10.0	.12
DW	8	23	2,500	6	254	242	249.0	249.5	3.54	10.2	9.7	10.0	10.0	.14
DW	9	23	2,500	6	256	241	249.0	248.9	4.36	10.3	9.7	10.0	10.0	.18
DW	1	4	2,500	1	260	241	249.0	249.3	5.33	10.4	9.7	9.95	10.0	.20
DW	2	4	2,500	6	259	241	251.0	249.6	5.58	10.4	9.7	10.0	10.0	.23
DW	1	21	250	6	25.2	24.1	24.7	24.7	.35	10.2	9.7	10.0	10.0	.14
DW	2	21	250	1	25.2	24.0	24.5	24.7	.51	10.3	9.7	10.0	10.0	.21
DW	3	22	250	1	25.3	24.3	24.8	24.7	.31	10.2	9.8	10.0	10.0	.13
DW	4	22	250	1	25.4	24.5	25.0	24.9	.30	10.2	9.8	10.0	10.0	.12
DW	5	22	250	1	25.5	24.4	24.7	24.9	.38	10.2	9.8	9.9	10.0	.16
DW	6	22	250	1	25.7	24.4	24.8	25.0	.44	10.3	9.8	9.9	10.0	.17
DW	1	23	100	1	10.1	9.3	9.7	9.6	.25	10.5	9.7	10.1	10.0	.25
DW	2	23	100	1	9.9	9.2	9.6	9.6	.21	10.3	9.6	10.0	10.0	.21
DW	3	23	100	6	14.9	6.8	9.4	9.8	2.54	15.2	6.9	9.6	10.0	2.59
DW	4	23	100	6	15.0	7.9	8.4	9.8	2.44	15.3	8.0	8.6	10.0	2.49
DW	1	23	~150 R	6	25.7	7.9	12.1	13.9	5.76	18.5	5.7	8.7	10.0	4.14
DW	2	23	~100 R	6	16.7	5.8	10.2	10.1	3.65	16.6	5.8	10.0	10.0	3.62
DW	3	23	~125 R	1	14.4	9.9	11.3	12.0	1.79	12.1	8.3	9.4	10.0	1.50
DW	4	23	~100 R	1	18.9	5.6	9.2	10.4	4.39	18.1	5.4	8.8	10.0	4.20

Table 4. Summary statistics of data from sample splitter--*Continued*

Description of sample	Test	Temper-ature (°C)	Volume (ml.)	Direction of pour	Subsample					Subsample, in percentage of total				
					Maximum	Minimum	Median	Mean	Standard deviation	Maximum	Minimum	Median	Mean	Standard deviation
					Volume, in milliliters					From Equation 1				
Sand and DW	1	24	2,500	6	265	229	250.5	249.4	13.1	10.6	9.2	10.0	10.0	0.52
Sand and DW	2	24	2,500	6	263	237	249.0	249.4	8.82	10.5	9.5	10.0	10.0	.34
Sand and DW	3	24	2,500	1	261	239	251.0	249.7	7.48	10.5	9.6	10.0	10.0	.29
Sand and DW	4	24	2,500	1	264	233	250.5	249.2	11.2	10.6	9.6	10.1	10.0	.46
Sand and DW	5	24	2,500	1	261	237	249.0	249.2	8.35	10.5	9.5	10.0	10.0	.35
					Concentration, in milligrams per liter					From Equation 2				
Sand and DW	1	24	2,500	6	994	890	932.1	937.2	36.3	10.61	9.50	9.95	10.0	.39
Sand and DW	2	24	2,500	6	1,025	848	947.8	949.1	64.0	10.80	8.94	9.99	10.0	.67
Sand and DW	3	24	2,500	1	982	920	966.3	960.9	19.2	10.22	9.57	10.0	10.0	.20
Sand and DW	4	24	2,500	1	978	894	950.2	941.6	31.0	10.39	9.49	10.1	10.0	.33
Sand and DW	5	24	2,500	1	1,022	944	997.2	987.9	291.1	10.35	9.55	10.1	10.0	.29
					Volume, in milliliters					From Equation 1				
Urban-volume	1	23	1,954	6	200	188	195.0	194.3	3.65	10.3	9.7	10.0	10.0	0.18
Urban-volume	2	23	1,899	6	194	182	189.0	188.7	4.30	10.3	9.6	10.0	10.0	.23
Urban-volume	3	23	1,817	6	184	177	181.5	180.9	2.02	10.2	9.8	10.0	10.0	.12
					Concentration, in milligrams per liter					From Equation 2				
Urban < 62 µm	1	23	1,954	6	1530	1441	1513.8	1503.4	28.5	10.2	9.6	10.1	10.0	.20
Urban < 62 µm	2	23	1,899	6	1502	1411	1470.5	1468.0	27.6	10.2	9.6	10.0	10.0	.20
Urban < 62 µm	3	23	1,817	6	1523	1470	1499.2	1498.5	18.0	10.2	9.8	10.0	10.0	.13
					Concentration, in milligrams per liter					From Equation 2				
Urban > 62 µm	1	23	1,954	6	224	183	209.0	207.5	11.5	10.8	8.8	10.1	10.0	.56
Urban > 62 µm	2	23	1,899	6	290	196	228.4	231.5	26.4	12.5	8.5	9.90	10.0	1.12
Urban > 62 µm	3	23	1,817	6	244	208	236.4	232.1	11.5	10.5	9.0	10.2	10.0	.49

Table 4. Summary statistics of data from sample splitter--*Continued*

Description of sample	Test	Temperature (°C)	Volume (ml.)	Direction of pour	Subsample					Subsample, in percentage of total				
					Maximum	Minimum	Median	Mean	Standard deviation	Maximum	Minimum	Median	Mean	Standard deviation
					Concentration, in milligrams per liter					From Equation 2				
Urban-total	1	23	1,954	6	1727	1660	1718.0	1710.9	21.5	10.1	9.7	10.0	10.0	.13
Urban-total	2	23	1,899	6	1715	1672	1702.1	1699.5	13.0	10.1	9.8	10.0	10.0	.09
Urban-total	3	23	1,817	6	1762	1702	1729.6	1730.7	16.7	10.2	9.8	10.0	10.0	.12
					Concentration, in milligrams per liter					From Equation 4				
Urban < 62 µm	1	23	1,954	6	1530	1441	1513.8	1503.4	28.5	8.94	8.42	8.85	8.79	.17
Urban < 62 µm	2	23	1,899	6	1502	1411	1470.5	1468.0	27.6	8.83	8.30	8.66	8.64	.16
Urban < 62 µm	3	23	1,817	6	1523	1470	1499.2	1498.5	18.0	8.81	8.49	8.66	8.66	.11
					Concentration, in milligrams per liter					From Equation 5				
Urban > 62 µm	1	23	1,954	6	224	183	109.0	207.5	11.5	1.31	1.07	1.22	1.21	.07
Urban > 62 µm	2	23	1,899	6	290	196	228.4	231.5	26.4	1.71	1.16	1.34	1.36	.15
Urban > 62 µm	3	23	1,817	6	244	208	236.4	232.1	11.5	1.41	1.20	1.37	1.34	.07

Table 3.--Variation in water and sediment splits made with cone splitter.

	High-concentration Test			Low-concentration Test		
Discharge-port	Mixture Mass, Kg	Sediment Mass, mg	Concentration, ppm	Mixture Mass, Kg	Sediment Mass, mg	Concentration, ppm
1	0.19415	46370.	238830.	0.15941	22.	138.
2	0.19256	45970.	238730.	0.15577	20.	128.
3	0.20226	48270.	238653.	0.16506	21.	127.
4	0.21646	51620.	238473.	0.15811	19.	120.
5	0.20850	49810.	238896.	0.14212	18.	127.
6	0.21363	51060.	239011.	0.15586	19.	122.
7	0.21277	50740.	238474.	0.14783	19.	129.
8	0.21225	50710.	238916.	0.15015	20.	133.
9	0.20458	48870.	238879.	0.14764	19.	129.
10	0.20584	49120.	238631.	0.16471	20.	121.
Mean	0.20630	49250.	238749.	0.15467	20.	127.4
s.d.	0.00813	1940.	187.	0.00760	1.	5.52
s.d.x100/mean	3.94%	3.93%	0.078%	4.91%	5.%	4.33%

	Clear-water test	Clear-water test (repeat)
1	0.240	0.255
2	0.247	0.254
3	0.255	0.260
4	0.254	0.255
5	0.243	0.234
6	0.237	0.233
7	0.230	0.230
8	0.234	0.235
9	0.226	0.232
10	0.236	0.246
Mean	0.240	0.245
s.d.	0.010	0.012
s.d.x100/mean	4.17%	4.90%

From Skinner, J.V., and Szalona, J.J., 1980

Evaluation of plastic cone splitter for water and sediment.

Data generated 1990 for plastic cone splitter					
Test of water splitting ability					
Outlet	Test A	Test B	Test C	Test D	Mean
1	1.0097	1.0270	1.0248	1.0248	1.0208
2	1.0130	1.0171	1.0120	1.0125	1.0137
3	1.0020	1.0022	0.9981	0.9976	1.0000
4	1.0210	1.0147	1.0147	1.0142	1.0161
5	0.9690	0.9535	0.9534	0.9546	0.9576
6	0.9681	0.9595	0.9542	0.9567	0.9596
7	0.9925	0.9864	0.9808	0.9868	0.9866
8	1.0091	1.0192	1.0133	1.0160	1.0144
9	1.0103		1.0265	1.0235	1.0201
10	1.0055	1.0204	1.0222	1.0166	1.0162
Minimum	0.9681	0.9535	0.9534	0.9545	0.9576
Maximum	1.0210	1.0270	1.0235	1.0586	1.0208
Test of solids splitting ability					
Outlet	Sediment concentration (mg/L)				
1		259.6		Laboratory A	
2		264.4		Laboratory A	
3		272.2		Laboratory A	
4		265.1		Laboratory A	
5		257		Laboratory B	
6		257		Laboratory B	
7		261		Laboratory B	
8		280.7		Laboratory C	
9		263.7		Laboratory C	
10		278.7		Laboratory C	
Mean		265.9			
Median		263.7			
Minimum		257			
Maximum		280.7			
% RSD		3.2			

From Gray, J.R., and Ferguson, S.A., 1990

Evaluation of plastic cone splitter for water and sediment.

Data generated 1992 for plastic cone splitter

Test of water spliting ability

	Cone Splitter 1			Cone Splitter 2	
	Mass of water (g)				
Outlet	Test A	Test B	Test C	Test D	Test E
1	207.0	207.3	208.3	215.0	201.8
2	202.7	202.4	200.7	210.0	205.2
3	200.1	200.4	197.6	215.2	220.5
4	196.5	197.2	195.2	208.9	211.8
5	191.1	191.4	191.4	195.6	201.6
6	196.1	196.0	195.4	184.2	191.1
7	194.1	194.3	195.2	185.3	190.6
8	193.1	191.9	193.3	178.3	183.0
9	199.0	201.2	200.0	182.6	184.0
10	204.2	206.5	208.8	202.1	197.6
Mean	198.4	198.9	198.6	197.7	198.7
Minimum	191.1	191.9	191.4	178.3	183.0
Maximum	204.2	207.3	208.3	215.2	220.5
Stan Dev	5.1	5.6	6.0	14.3	12.0
% RSD	2.6	2.8	3.0	7.2	6.0

Test of solids splitting ability - all samples through Cone Splitter 1

	All solids < 63 um			20% sand/80% silt/clay	
Outlet	Sediment Concentration (mg/L)				
1	975	983		1003	1017
2	930	996		975	982
3	984	985		915	955
4	962	985		987	1040
5	976	965		1018	1008
6	989	982		980	1008
7	987	988		1018	1016
8	979	987		1005	1000
9	969	985		957	1005
10	981	853		992	1015
Mean	973.3	970.9		985.0	1004.5
Minimum	930.0	853.0		915.0	955.0
Maximum	989.0	996.0		1018.0	1040.0
Stan Dev	17.3	42.3		31.1	22.6
% RSD	1.8	4.4		3.2	2.2

From Shampine, W.J., Schroder, L.J., and Gilroy, E., 1992

Evaluation of Teflon cone for water and solids. Comparison of suspended sediment content of subsamples from all ten parts of Teflon cone splitter. River water with artificially high coarse particle content.

Subsample volume, mL	Total (SS) mg/L	% < 0.063 mm	Fines conc. (< 0.063 mm)	Coarse conc. (> 0.063 mm)
339	1370	12.63	173	1197
270	1430	11.8	169	1261
297	1260	11.63	147	1113
273	1310	12.76	167	1143
300	1270	13.21	168	1102
333	1170	14.87	174	996
300	1450	11.73	170	1280
321	1240	14.68	182	1058
306	1280	14.56	186	1094
347	1410	20.48	289	1121

From Martin, J.D., 1993

Evaluation of Teflon cone duplicates for water and sediment. Comparison of subsamples from two parts of the Teflon cone splitter.

		Subsample A		**Subsample B**	
River	date	(SS), mg/L	%<0.063mm	(SS), mg/L	%<0.063mm
Little Buck River	19920713	48	97.63	34	88.99
Little Buck River	19920805	11	87.5	8	72.22
White River	19910501	51.1		80.2	
White River	19910506	19.8		17.4	
White River	19910509	66.8		68.5	
White River	19910513	115.8		117.1	
White River	19910516	86.7		84.6	
White River	19910520	102		102.9	
White River	19910523	147.7		149	
White River	19910528	160.9		161.1	
White River	19910530	130.3		131.6	
White River	19910603	229		238.7	
White River	19910606	293.5		293.2	
White River	19910610	132.7		132.7	
White River	19910613	84		89.3	
White River	19910617	376.7		352.7	
White River	19910620	101.4		101.8	
White River	19910624	86.4		87.8	
White River	19910627	84		81.4	
White River	19910701	66.2		63.2	
White River	19910703	67.9		63.6	
White River	19910708	68.2		65.3	
White River	19910711	48.2		42.7	
White River	19910715	336		336.8	
White River	19910718	47.2		51	
White River	19910722	55.6		60	
White River	19910725	54.4		55.7	
White River	19910806	31	49.47	32	49.57
White River	19910808	36.2		34.1	
White River	19910812	45.6		48.6	
White River	19910815	46	54.94	45	61.14
White River	19910819	47.8		46.6	
White River	19910822	45.5		44.9	
White River	19910826	49	61.45	51	55.15
White River	19910829	37.5		38.6	
White River	19910909	60.1		58.9	
White River	19910912	31.3		29.2	
White River	19910919	34.6		30.2	
White River	19910926	29.9		34.8	
White River	19911003	32.9		38.4	
White River	19911008	29.1		27.2	
White River	19911017	77.1		76.3	
White River	19911024	73		71.4	

White River	19911028	98	97.25	99	71.83
White River	19911030	279	82.28	285	90.7
White River	19911104	66	91.56	66	87.62
White River	19911127	89	100	89	
White River	19920116	63	82.69	75	62.69
White River	19920227	99	55.99	90	49.64
White River	19920305	72	76.86	78	66.8
White River	19920420	368	93.72	353.9	
White River	19920424	155	92.34	154.7	
White River	19920429	176	88.83	168.4	
White River	19920623	527	89.45	428	89.32
White River	19920625	383	91.5	448	93.6
White River	19920723	274	87.08	272	88.2
Sugar Creek	19920618	761	96.57	894	97.8
Sugar Creek	19920713	128	78.59	113	80.4
Little Buck River	19930614	47	38.81	64	24.73
Little Buck River	19930728	23	4.08	33	7.94
Little Buck River	19930728	33	7.79	36	7.06
White River	19930623	100	34.74	102	42.78
White River	19930623	96	48.33	95	42.72
Kessinger River	19930908	37	87.5	48	62.15
White River	19930706	344	74.78	338	76.7
White River	19930719	269	65.31	272	60.2
White River	19930907	497	79.5	233	70.13
Big Walnut Creek	19930715	316	75.6	308	75.76
Big Walnut Creek	19930715	127	69.26	306	68.24
Sugar Creek	19930609	191	76.36	118	73.8
Sugar Creek	19930721	44	44.22	36	66.41

From Crawford, C., (Project Chief), White River NAWQA, 1993

Teflon cone versus traditional sediment method. Comparison of Teflon cone-processed and traditionally collected suspended sediment samples.

		Cone processed sample		Traditional sample - A		Traditional sample - B	
Station	Date	(SS), mg/L	%< 0.063mm	(SS), mg/L	%< 0.063mm	(SS), mg/L	%< 0.063mm
6713500	51493	27	91.76	24	92.45	25	91.88
6713500	60293	11	88.46	12	85.31	14	84.71
6713500	71593	14.5	89.44	14	91.43		
6713500	72893	5	80.56	3	91.3		
6713500	80593	1130	58.75	1260	60.82	1080	71.09
6713500	80693	13	91.09	13	94.44		
6713500	81093	6	77.27	4	77.27	3	79.41
6713500	81093	1130	76.66	995	60.7	943	63.04
6719505	50693	14	70	17	60.61		
6719505	60893	32	30.34	28	26.55	46	19.02
6719505	70193	32	67.76	82	28.45		
6719505	70793	32	31.45	33	24.09		
6719505	80393	7	63.89	47	16.02		
6714000	50793	20	92.11	17	96.83	19	89.5
6714000	51793	524	80.9	469	71.16	464	80.14
6714000	60293	807	44.99	418	62.32	420	55.35
6714000	70693	13	90.32	10	92.66	10	93.4
6714000	80293	26	81.6	23	88.3	23	90.79
6720500	60393	145	95.63	146	96.14	148	93.38
6720500	80693	56	91.84	54	93.01		
6731000	50393	25	92.31	25	77.89		
6731000	60193	238	86.49	263	74.16	255	71.48
6731000	61893	561	63.61	641	57.55	683	52.79
6731000	80493	98	88.64	98	86.6	97	85.61
6753400	50493	52	84.71	53	77.34		
6753400	50993	31	80.12	30	73.8	32	79.89
6753400	71393	38	48.6	15	90.86	18	80.19
6753400	81293	22	75.21	22	76.53	25	83.33
6753990	50593	212	95.7	207	90.89		
6753990	52993	1720	88.26	2350	77.46	2340	78.25
6753990	60493	265	94.94	259	91.95	259	92.08
6753990	71393	581	86.27	945	88.87	913	89.93
6753990	81293	302	85.4	274	82.9	297	80.17
6752000	50493	11	79.17	8	90.54		
6752000	61093	7	68.75	9	64.86	8	65.22
6752000	70893	10	51.47	7	60.23		
6754000	60993	117	80.95	122	77.27		
6754000	70993	127	98.08	126	95.9		
6754000	71593	522	90.16	514	87.25		
6754000	81393	108	92.35	110	89.25		
6759910	51293	48	54.82	72	35.59	46	56.58
6759910	60793	72	99.38	79	95.56	84	92.12
6759910	62193	657	19.73	836	16.78	739	17.99
6759910	80993	159	70.06	155	72.4	143	79.48
6765500	60393	61	85.03	70	87.68	124	65.07
6765500	80993	61	77.5	71	71.1	65	76.62
6765500	81193	83	72.93	87	77.61		

From Dennehy, K., (Project Chief), South Platte NAWQA, 1993

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