Settling Column Test Procedures

(modified October 2002)

PREP - DAY 1:

1. Prepare settling column. Prepare the 7-ft settling column as follows:

   A) Clean and assemble the column and ports.

   B) Test the column for leaks by filling with tap water.

2. Determine salinity. Using a bucket of sediment that has separated into solid and liquid phases, determine the salinity or the total dissolved solids (TDS) concentration of the pore water or dredging site water using a salinity or TDS meter or gravimetrically:

If using a meter:

   A) If needed, clean the probe with a solution of nitric acid.

   B) Following the instructions for the meter, calibrate the meter using water with a known salinity. Be sure to check the batteries and determine the temperature of the liquid.

   C) Determine the salinity/TDS of the pore water or dredging site water in ppt.

If determining the salinity gravimetrically, follow the procedure below:

   A) Label, dry, and tare (weigh) three aluminum dishes to 1-mg accuracy.

   B) Fill each tared dish with about 25 mL of filtered pore water or filtered dredging site water (to a depth of about 3/8 in.) and weigh. Larger samples can be used for larger dishes.

   C) Place samples in a forced convection oven at 180ºC (180ºC is preferable to 105ºC for saline samples) for approximately 4 hr for small samples (2-in. diameter) and approximately 24 hr for larger samples (4-in. diameter). Samples should be dried until there is no measurable weight change.

   D) Weigh the dried samples.

   E) Subtract the tare (weight of dried empty aluminum dish) from the wet weight to obtain the initial weight of the filtered pore water or dredging site water sample.

   F) Subtract the tare from the dried weight to obtain the weight of salt in the sample.

Use the following equation to determine salinity:

$$S = W_s \left(\frac{1000}{W_{pw}} \right)$$

where S = salinity of pore water, ppt
W_s = weight of salt in sample, g
W_{pw} = weight of pore or site water sample (water plus salt), g

3. Homogenize sediment samples. Combine and thoroughly mix all of the sediment samples to create a single uniform sample. Water of the correct salinity may need to be added to facilitate mixing.

4. Determine particulates concentration. Using the total solids analysis procedures given in Step 22, determine particulates concentration of the saturated sediment in triplicate.

5. Compute average moisture content. Average the three particulates concentrations to obtain the average particulates concentration of the sediment. Compute the average moisture content of the sediment sample based on the average particulates concentration using the following equation:

$$w = \left(\frac{1000 - \left(C_p / G_s \right)}{C_p} \right) 100\%$$

where w = moisture content, %
C_p = particulates concentration of sediment, g/L
G_s = specific gravity of particulates

PREP - DAY 2:

6. Assemble sampling equipment. Gather, clean, and label supernatant sample bottles (125 to 250 mL), sampling syringes, and catheters (up to 40 bottles may be needed in the first day or two). Gather, clean, and label six sample bottles (50 to 100 mL) for total solids (residue) analysis. Label, dry, and weigh six aluminum dishes for total solids analysis as described in Step 22. Wash, dry, and tare (weigh) filter papers for suspended solids analysis (up to 40 filter papers may be needed in the first two days). (See Step 23A for instructions on preparation of filter papers.) Clean vials for turbidity measurements, and calibrate turbidimeter. Remove cap nuts and bushings from the sampling valve assemblies on the settling column until they are needed for sampling the supernatant.

7. Estimate slurry concentration for settling test. Determine the concentration at which the dredge is pumping based on historical dredging data, grain-size distribution, and/or settling characteristics of the sample. Hydraulic dredges normally pump at 100 to 200 g/L for predominantly fine-grained slurries. Slurry concentrations for hydraulic dredges pumping a mixture of coarse- and fine-grained materials can be approximated by the following equation:

$$C_{sl} = \%\ Fines + (3 \times \%\ Coarse)$$

where C_{sl} = slurry concentration, g/L

8. Compute unit weights of sediment. Calculate the weights of pore water, salt, and particulates in 1 kg of sediment as follows:

A) Calculate the weight of the pore water in 1 kg of sediment:

$$W_{wkg} = \frac{1000\,w}{100 + w}$$

where W_{wkg} = weight of pore water in 1 kg of sediment, g/kg

B) Calculate the weight of salt in 1 kg of sediment:

$$W_{skg} = \frac{W_{wkg}\ S}{1000 - S}$$

where W_{skg} = weight of salt in 1 kg of sediment, g/kg

C) Calculate the weight of the particulates in 1 kg of sediment:

$$W_{pkg} = 1000 - W_{wkg} - W_{skg}$$

where W_{pkg} = weight of particulates in 1 kg of sediment, g/kg

9. Compute unit requirements for dilution of sediment. Determine the amounts needed to dilute 1 kg of sediment to the desired slurry concentration as follows:

A) Calculate the total volume after dilution of 1 kg of sediment:

$$V_{kg} = \frac{W_{pkg}}{C_{sl}}$$

where V_{kg} = total volume of slurry created from 1 kg of sediment, L/kg

B) Calculate the volume of pore water in 1 kg of sediment:

$$V_{pwkg} = \frac{W_{wkg}}{1000}$$

where V_{pwkg} = volume of pore water in 1 kg of sediment, L/kg

C) Calculate the volume of particulates in 1 kg of sediment:

$$V_{pkg} = \frac{W_{pkg}}{1000\, G_s}$$

where V_{pkg} = volume of particulates in 1 kg of sediment, L/kg

D) Calculate the volume of dilution water per 1 kg of sediment:

$$V_{dwkg} = V_{kg} - V_{pwkg} - V_{pkg}$$

where V_{dwkg} = volume of tap water needed to dilute 1 kg of sediment to desired slurry concentration, L/kg

E) Calculate the weight of salt required to slurry 1 kg of sediment:

$$W_{dskg} = \frac{1000\, S\, V_{dwkg}}{(1000 - S)}$$

where W_{dskg} = weight of salt needed to bring dilution water for 1 kg of sediment to the measured pore water salinity, g/kg

10. Compute slurry requirements. Determine the amounts of sediment, tap water, and salt to mix the required volume of slurry to fill the settling column to the desired height.

A) Calculate the required weight of sediment:

$$W_t = V_t / V_{kg}$$

where W_t = required total weight of sediment sample for settling test, kg
V_t = volume of slurry required for settling test, L (typically, 90 to 120 L)

B) Calculate the volume of tap water needed to create slurry for settling test:

$$V_{wt} = W_t \, V_{dwkg}$$

where V_{wt} = volume of tap water, L

C) Calculate the weight of salt needed to create slurry for settling test:

$$W_{st} = W_t \, W_{dskg}$$

where W_{st} = weight of salt to bring column slurry to pore water salinity, g

11. Prepare slurry for testing. Using a 4-ft Lightning or barrel mixer, mix the required quantity of sediment, tap water, and salt in a large container (40- to 55-gal drum) for approximately 15 min to create the slurry for settling tests. Mix longer if the sediment is not well-mixed or thoroughly hydrated.

12. Sample slurry. Allow approximately 5 min for the coarse-grained material to fall out of the slurry and obtain grab sample from the upper half of the slurry in a 250-mL sample bottle. Determine the particulates concentration of the slurry (See Step 22). Typically, the particulates concentration should range from 100 to 130 g/L. If the concentration is greater than 160 g/L, consider diluting the upper slurry to a particulates concentration of 150 g/L. If the concentration is less than 50 g/L, consider decanting clear supernatant from the settled slurry in the barrel to achieve a particulates concentration of 60 g/L.

TEST INITIALIZATION - DAY 1:

13. Mix slurry. Collect a 100-mL sample of the clarified supernatant and use it to verify the salinity of the slurry as given in Step 2. Then, thoroughly mix slurry in drum/barrel for at least 15 min. Next, slow the mixing and allow the coarse, medium, and some fine sand to settle out of the slurry for about 5 min.

14. Fill settling column. Turn off the mixer and rapidly fill the settling column with the slurry of fine-grained sediment. The column can be filled by transferring the slurry by bucket or by pump. Transferring 2 or 3 gal of slurry at a time in a 5-gal bucket works well; a 1-gal pitcher can be used to fill the bucket. If transferring by pumping, a centrifugal, rotary screw, or positive displacement pump with a ¾- or 1-in. I.D. hose works well. Continue this process until column is filled to the required height (usually at or above 6.4 ft). Place a cover on the top of the column to prevent supernatant evaporation.

15. Sample initial slurry concentration. Collect samples from each 1-ft column port beginning at the 6-ft port and place them in the six 125-mL sample bottles. The test has

begun; take special care not to disturb the column until the test is complete. Determine the particulates concentration of all six samples (see Step 22) and calculate average particulates concentration for the start of the test.

16. Record initial conditions. Record the average particulates concentration as the initial concentration, the initial time, and the initial height of the slurry in the column on the Settling Column Data Sheet.

ZONE SETTLING TEST - DAY 1:

17. Test for zone settling. If an interface develops at the top of the column between supernatant and settled solids within the first 2 hr (typically within 15 to 30 min), zone settling is occurring in the column. If the slurry is saline (greater than 1 to 3 ppt) and zone settling does not occur, restart the test at a lower initial slurry concentration.

18. Record interface height as a function of time. If zone settling occurs, record the solids interface height and the time of the reading every 10 to 30 min over the first 6 to 8 hr of the test on the Settling Column Data Sheet. The frequency of the readings should be sufficient to capture about every 0.2 ft of change in the height of the interface. Readings should also be taken occasionally throughout the first 24 hr of the test. (If a compression settling test is also being run, interface height readings are continued after the first 24 hr for the compression settling test as described below in Step 21.)

FLOCCULENT SETTLING TEST - DAYS 1 - 15:

19. Prepare sampling port. Shortly before sampling from a port for the first time, flush the port of the initial slurry by opening the valve and draining to waste about 50 mL of material through the port. Then, if sampling a supernatant sample for total suspended solids, insert the sampling bushing into the sampling port and screw on the cap nut. Tighten the cap nut until the bushing forms a seal with the sampling catheter.

20. Sample collection and measurement. If zone settling occurs or if the slurry is saline (greater than 1 to 3 ppt), sample only the supernatant for total suspended solids (from all ports, up to 6, above the solids interface); otherwise, sample the slurry for total solids at port heights of 6, 5, 4, and 3 ft (if the port at 6 ft is not available, sample the slurry at port heights of 5.5, 4.5, 3.5, and 2.5 ft).

A) Take 50- to 60-mL samples for total solids and 100- to 120-mL samples for total suspended solids. Samples for total suspended solids should be taken from the center of the column using a catheter. The catheter may be marked to show when it has been inserted sufficiently to reach the center of the column and when it has cleared the valve during removal.

B) The column should be sampled during the first day at approximately 1 (if any ports are available for sampling), 2, 4, 7, 12, and 24 hr after the start of the test. After the first day, the column should be sampled at approximately 2, 3, 4, 7, 11, and 15 days after the start of the test. If significant changes in concentrations are observed in the 4-day samples, sample on days 6, 9, 12, and 15.

C) Record the sampling time and the height of slurry or supernatant water as well as the interface height on the Settling Column Data Sheet prior to each sampling period.

D) Record any other observations or information that would be helpful in interpreting the test results such as ports sampled, presence and thickness of scum, oil, grease, precipitates (such as bands of iron oxide), height of coarse material buildup at the bottom of the column, color bands in the settled solids, surface debris falling through the supernatant water while sampling for TS or TSS, etc.

E) Measure the turbidity of each sample as well as the particulates concentration by total solids analysis (see Step 22) or total suspended solids analysis (see Step 23) immediately after sampling. The turbidity and suspended solids concentration may change within 6 hr due to flocculation, coagulation, and precipitation.

F) Take digital pictures of the testing, as desired to document the test.

COMPRESSION SETTLING TEST - DAYS 1 - 15:

21. Record interface height as a function of time. Record the solids interface height and the time of the reading on the Settling Column Data Sheet about twice a day throughout the first 5 days of the compression settling test, about once a day during days 6 to 10, and every other day during days 11 to 15. The frequency of the readings should be sufficient to capture about every 0.05 ft of change in the height of the interface. If zone settling does not occur, the interface may be difficult to discern until the second or third day of the test.

LABORATORY ANALYSIS:

22. Total solids analysis. Total solids analysis should be performed on samples having particulates concentrations greater than about 3 to 5 g/L. Using the Total Solids Report Sheet to record the weights, total solids analysis should be conducted as follows:

A) Label, dry, and tare (weigh) the required number of dishes to 1 mg accuracy. Record the weight on the Total Solids Report Sheet.

B) Pour the samples into the appropriate tared dishes and weigh. Record the weight on the Total Solids Report Sheet.

C) Place the dishes in a forced convection oven at 105ºC to 180ºC (180ºC is preferred for saline samples) and dry for approximately 5 hr or until there is no measurable weight change.

D) Weigh the dried samples. Record the weight on the Total Solids Report Sheet.

E) Calculate the weight of the salt in the sample:

$$W_s = \frac{W_w \ S}{1000 - S}$$

where W_s = weight of salt in the sample, g
W_w = weight of water in the sample, g
S = salinity of pore water, ppt

F) Compute the weight of particulates in each sample using the following equation:

$$W_p = W_d \ - \ W_s$$

where W_p = weight of dry particulates, g
W_d = weight of dried sample (particulates and salt), g

G) Determine the particulates concentration of each sample using the following equation:

$$C_p = \frac{1000\, W_p}{(W_p / G_s) + W_w}$$

where C_p = particulates concentration of sediment, g/L
G_s = specific gravity of particulates

23. Suspended solids analysis. Total suspended solids (TSS) analysis should be performed on samples having concentrations less than 3 g/L. Membrane filters designed for gravimetric analysis of water samples perform best. The filters should have a pore diameter of 0.45 microns and filter diameter of 47 mm. The filters should be constructed of mixed cellulose esters, cellulose nitrate, or cellulose acetate, such as Millipore (# HAWP 047 00), Gelman Sciences (GN-6), and Whatman (WCN 7184-004), and Advantec MFS. Use of glass fiber filters is discouraged because their pore sizes are too large, but use of 0.7-micron pore size may produce acceptable results. Using the Suspended Solids Report Sheet to record the weights and volumes, total suspended solids analysis by filtration should be conducted as follows:

A) Prepare filters as follows:

a) Soak the filters in deionized/distilled (DD) water for 5 min.

b) Using DD water, wet the base of the filter holder.

c) Following the instructions on the vacuum pump or system, build up a vacuum pressure of approximately 25 psi.

d) Using the vacuum pressure, draw the water through the filter holder.

e) Place the filter (shiny side down) on the holder using forceps.

f) Place the funnel (top of holder) on the base of the filter holder and secure.

g) Pour approximately 10 mL of DD water into the filtration apparatus and vacuum the water through the filter and into the filter flask. Repeat the flushing two more times.

h) Place the filter in a labeled dish using forceps.

i) Making sure that the filter does not stick to the dish, air dry the filter for approximately 5 min.

j) Dry in a forced convection oven at 105ºC for approximately 15 min or until there is no measurable weight change.

k) Cool in desiccator for 5 min.

l) Immediately weigh the filter on an analytical balance and record the weight on the Suspended Solids Report Sheet. Use forceps to transfer the filter to the balance.

m) Place the filter back in the labeled dish using forceps and place in the desiccator for future use.

n) Prepare the required number (or at least eight at a time if limited space is available in desiccator) of filters for the test all at one time.

B) Perform total suspended solids test as follows:

a) Place a filter on the base of the filter holder using forceps and wet with DD water. Vacuum the water through the filter.

b) Shake the sample in the sample bottle.

c) Filter a sample volume that contains approximately 5 to 10 mg of TSS.

d) Using a 10-, 25-, 50-, or 100-mL to-deliver (TD) graduated cylinder,

measure and record the volume of sample to be filtered on the Suspended Solids Report Sheet.

e) Pour the sample onto the center of the filter paper and remove the water from the sample under vacuum pressure.

f) Rinse filter funnel and wash salt from the filtered solids on the filter paper with 10 mL DD water and then remove the water under vacuum pressure. Repeat the rinse two more times.

g) Release the vacuum pressure from the filter flask. Remove the filter from the filter holder using forceps and return the filter to the labeled dish.

h) Allow the filter to air dry, ensuring that the filter does not stick to the dish.

i) Dry the filter in the forced convection oven at 105ºC for approximately 30 min or until there is no measurable weight change.

j) Cool in the desiccator for 5 min.

k) Immediately weigh the filter on an analytical balance and record the weight on the Suspended Solids Report Sheet. Use forceps to transfer the filter to the balance.

l) Determine TSS (particulates concentration, C_p):

$$TSS = 10^6 \left(\frac{W_p}{V_t} \right)$$

where TSS = total suspended solids of sample, mg/L
W_p = weight of dried particulates, g
V_t = volume of sample, mL

COMPLETION:

24. After 15 days, drain supernatant water and stored settled material until the results are analyzed for completeness and reliability.

27. Thoroughly clean column and gaskets and allow them to dry.

28. Apply Dow Corning High Vacuum Grease on the column gaskets (O-rings) to provide a watertight seal and reassemble the column. Close all the sampling ports and cover the top of the column to keep clean between uses.

Settling Column Tests Equipment and Supplies:

Durable Equipment:

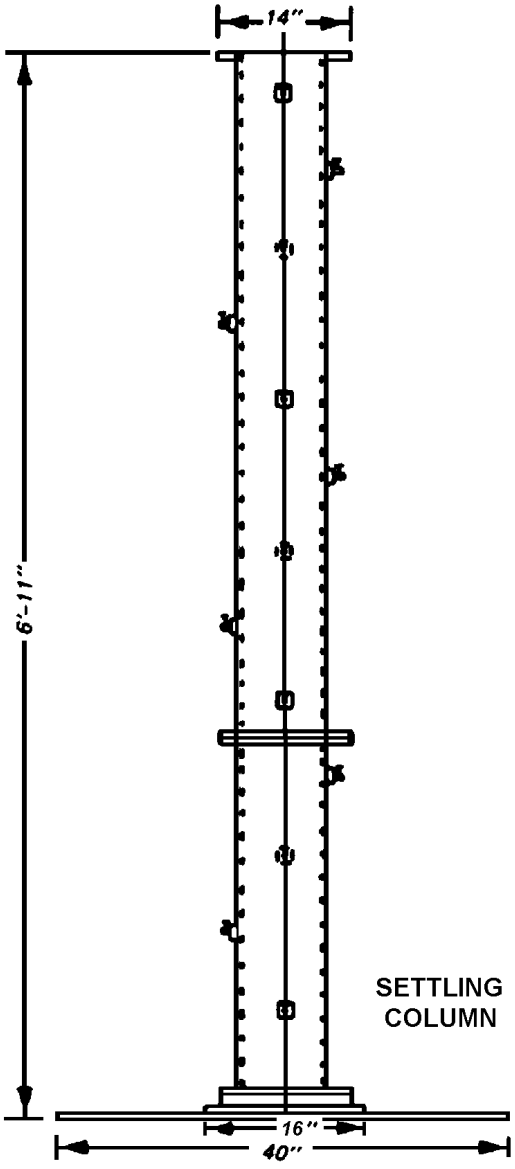
Settling column (design specifications in Appendix A)
S-C-T meter
Small hand-held mixer for mixing in 5-gal buckets
3-ft mixer for mixing slurry in 55-gal drum (should have clamp for mounting)
Analytical balance(s) with range from 0 to 200 g with readability to 0.1 mg
Scale to weigh sediment in buckets (100-kg capacity)
Forced convection oven
Deionized/distilled (DD) water source
32- to 55-gal plastic drum or barrel (44-gal can may be preferable)
Cover for top of column
Digital camera
Membrane filtration apparatus with vacuum setup
Desiccator
Graduated cylinders (TD) (10-, 25-, 50-, 100-, 1000-, 2000-, and 4000-mL sizes)

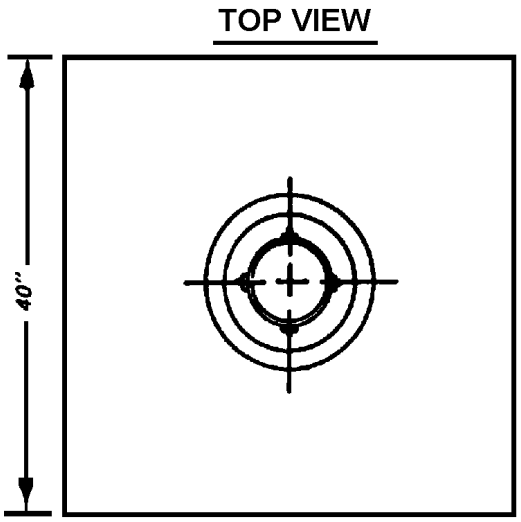
Expendable Supplies:

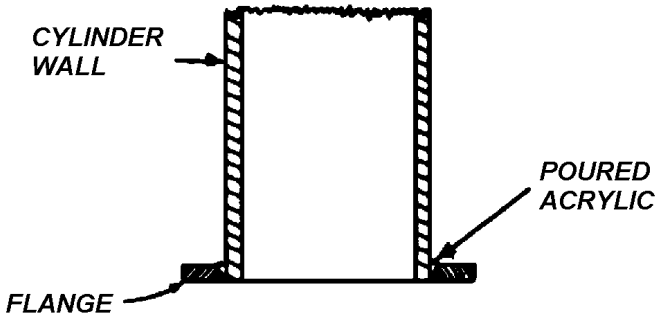
Nitric acid
Salt
5-gal buckets
Disposable aluminum weighing dishes
Sample bottles (125-, 250-, 500-, and 1000-mL sizes)
Labels for bottles
Waterproof markers
60-ml disposable syringes with catheter tip
Polypropylene catheters (HRI 8890-703518, size 10FR, 22-in length)
Filters (Millipore # HAWP 047 00, Gelman Sciences GN-6 # 63069, Whatman WCN 7184-004, and Advantec MFS)
Wash bottles
Forceps
Dow Corning High Vacuum Grease

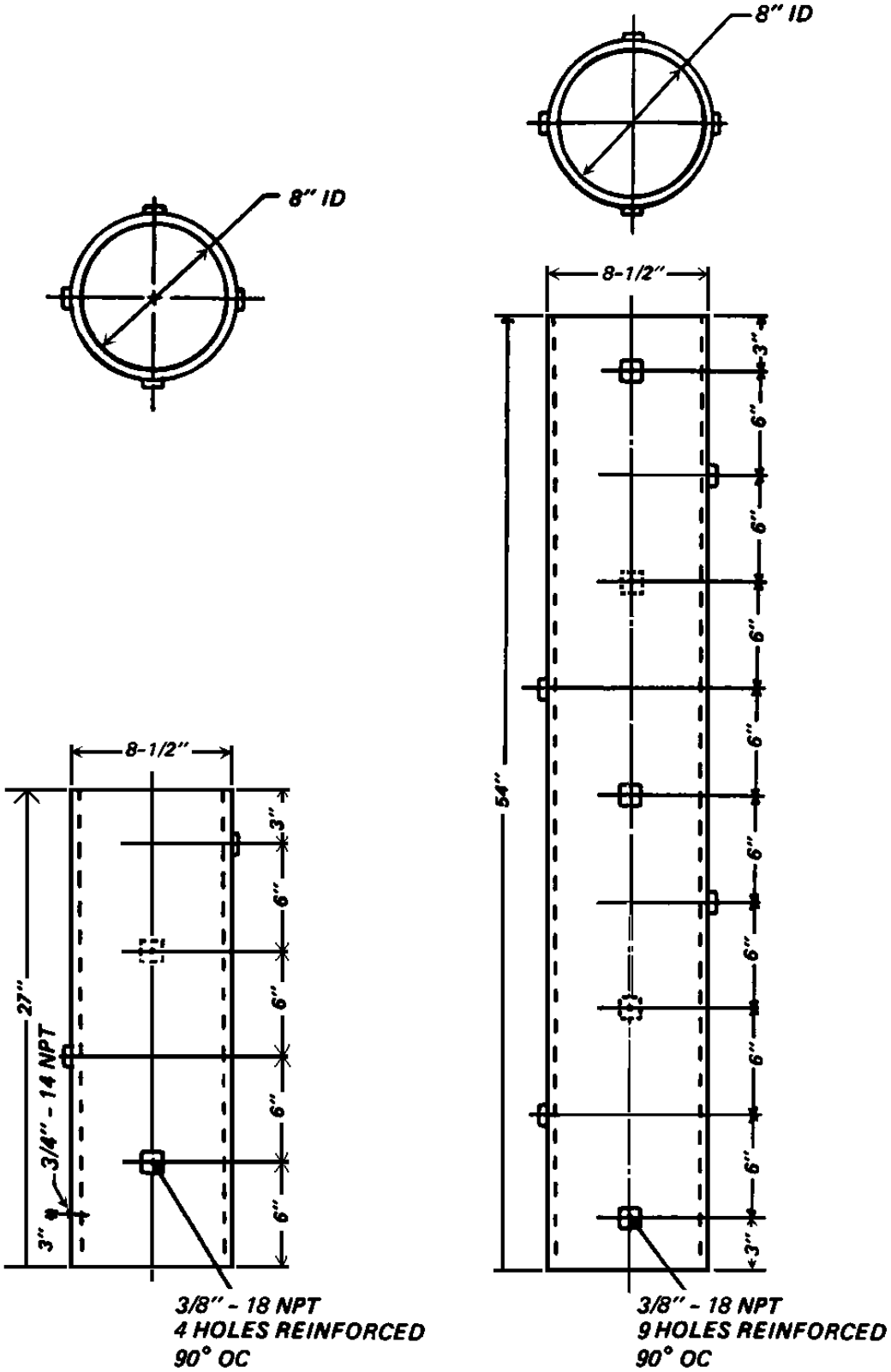
Appendix A

Schematic of Settling Column with Bill of Material, Possible Suppliers, and Pictures of Equipment



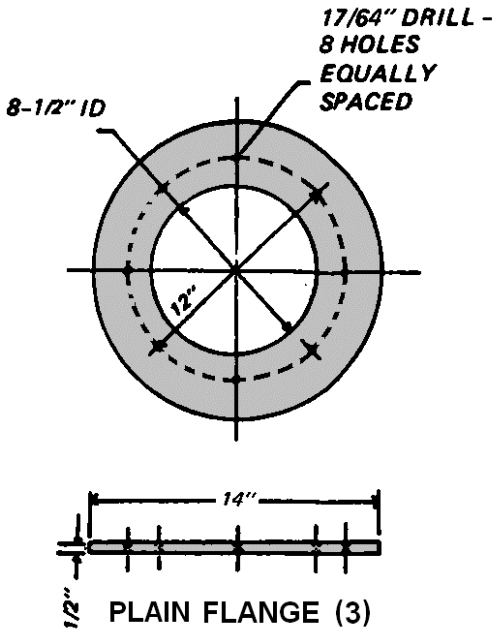




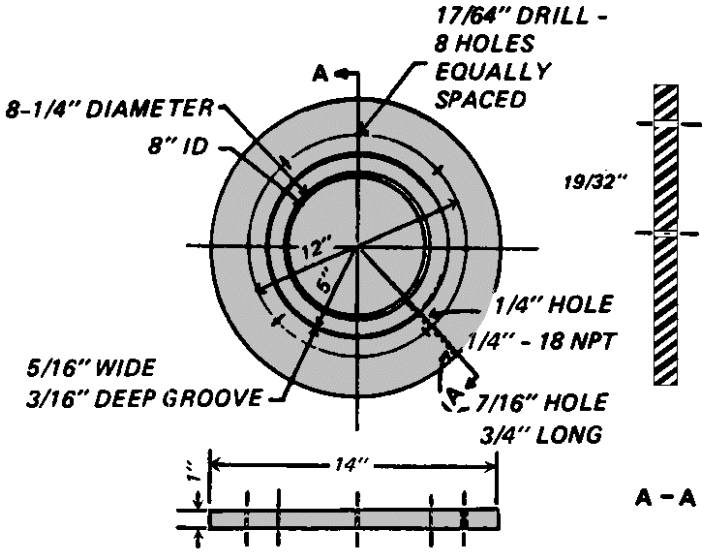


BOTTOM SECTION
OF COLUMN

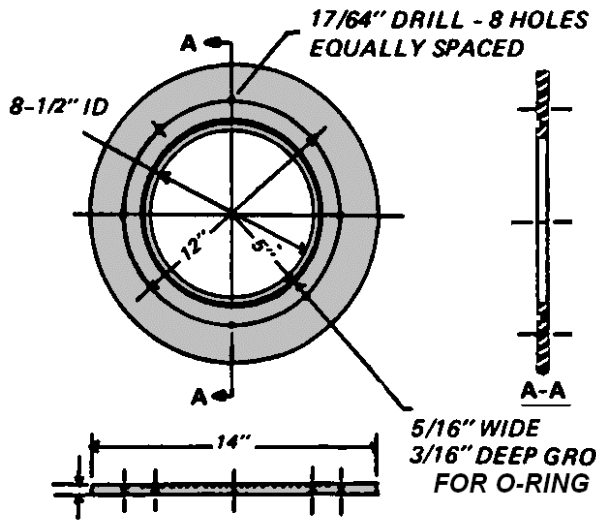
TOP SECTION
OF COLUMN



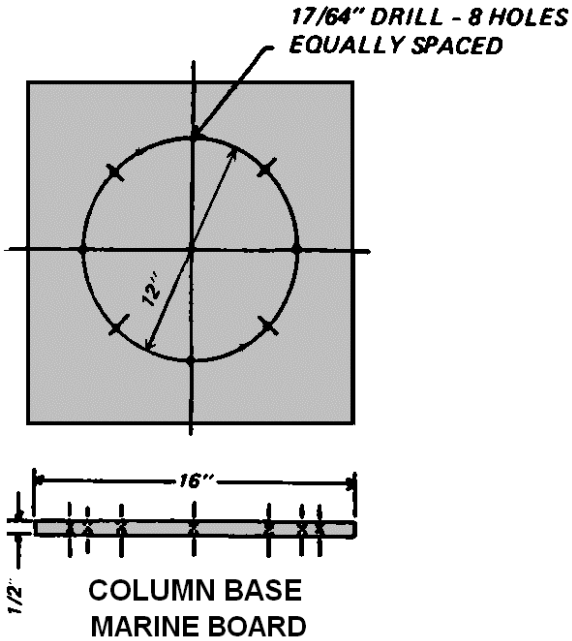
PLAIN FLANGE (3)



COLUMN BOTTOM



FLANGE FOR TOP OF
BOTTOM COLUMN (1)



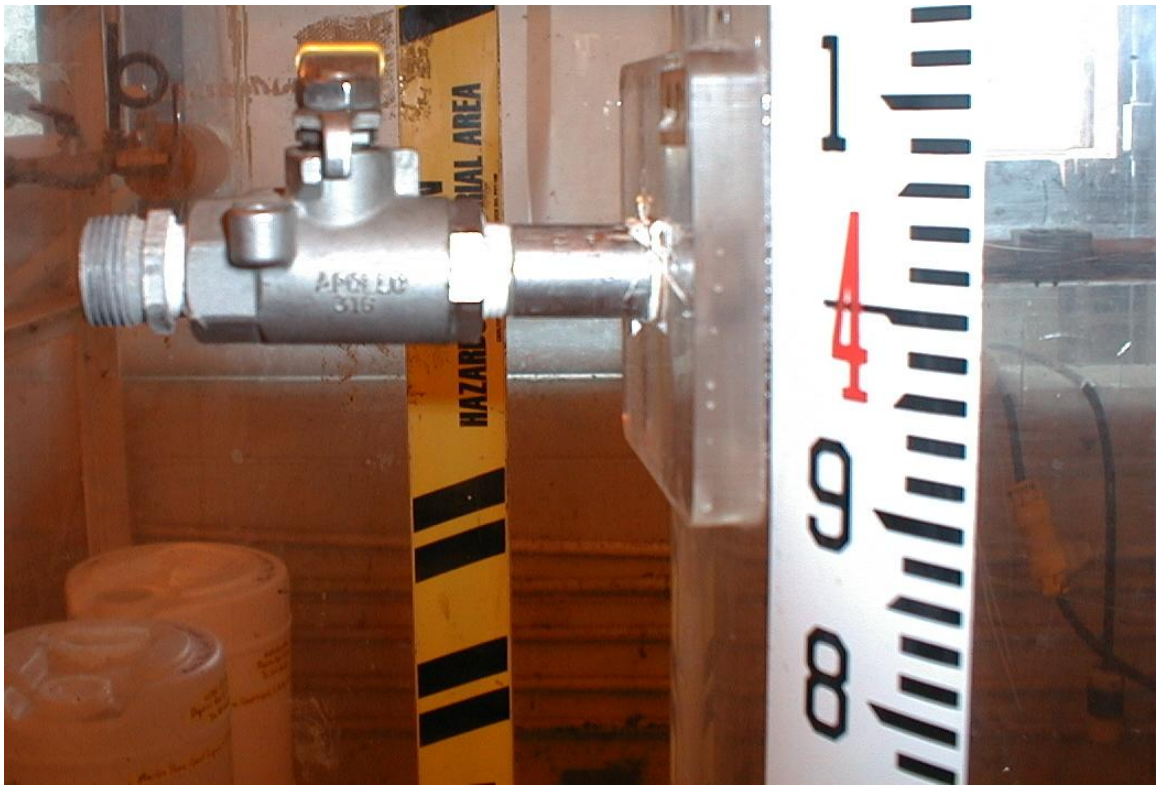
COLUMN BASE
MARINE BOARD

BILL OF MATERIAL

QTY	MATERIAL	SIZE
1	Ball Value (Stainless Steel)	3/4” I.D.
13	Sampling Valve Assemblies (Stainless Steel)	3/8” I.D.
1	Material for at Least 2 10 in.-Diameter O-Rings	1/4” Dia, 12’ Long
1	Waterproof Plywood	4’ x 4’ x 1/2”
1	Marine Board for Column Base	16” x 16” x 1/2”
8	Hex Head Bolt w/Washer and Nut	1/4”-20 UNC x 1-1/2”
8	Carriage Head Bolt w/Washer and Nut	1/4” x 3”
14	Plexiglas® Reinforcement for Column Holes	3” x 3” x 1/4”
1	Plexiglas® Flange for Top of Lower Section of Column	1/2” Thick, 14” O.D. 8-1/2”I.D.
3	Plexiglas® Flange - 2 for Top and Bottom of Upper Section and 1 for Bottom of Lower Section of Column	1/2” Thick, 14” O.D. 8-1/2”I.D.
1	Plexiglas® Plate for Column Bottom	1” Thick, 14” Dia
1	Plexiglas® Cylinder	27” Long, 1/4”Thick 8” I.D., 8-1/2” O.D.
1	Plexiglas® Cylinder	54” Long, 1/4”Thick 8” I.D., 8-1/2” O.D.

POSSIBLE SUPPLIERS

MATERIAL	VENDER
O-Ring	**Mississippi Rubber and Specialty Company** **715 East McDowell Rd** **Jackson, MS 39204** **(601) 948-2575**
Plexiglas®	**GE Polymer Shapes (Cadillac Plastic)** **600 South Royal Lane** **Coppell, TX 75019** **(972) 304-2525** **Magnolia Metal & Plastics Products, Inc.** **101 County Lane** **PO Box 820289** **Vicksburg, Ms 39182** **Phone (601) 638-6912** **Fax (601) 636-2552**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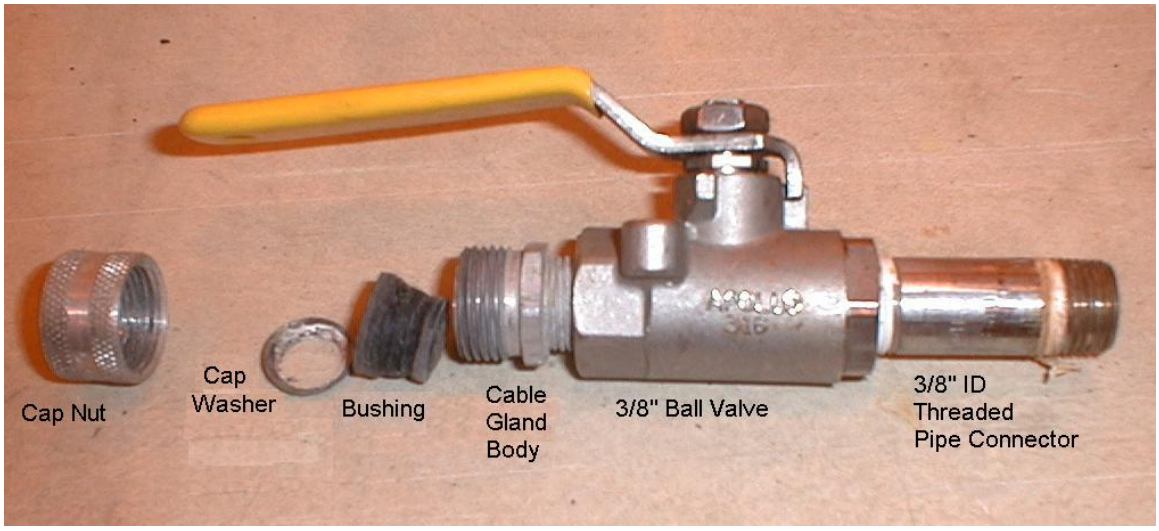
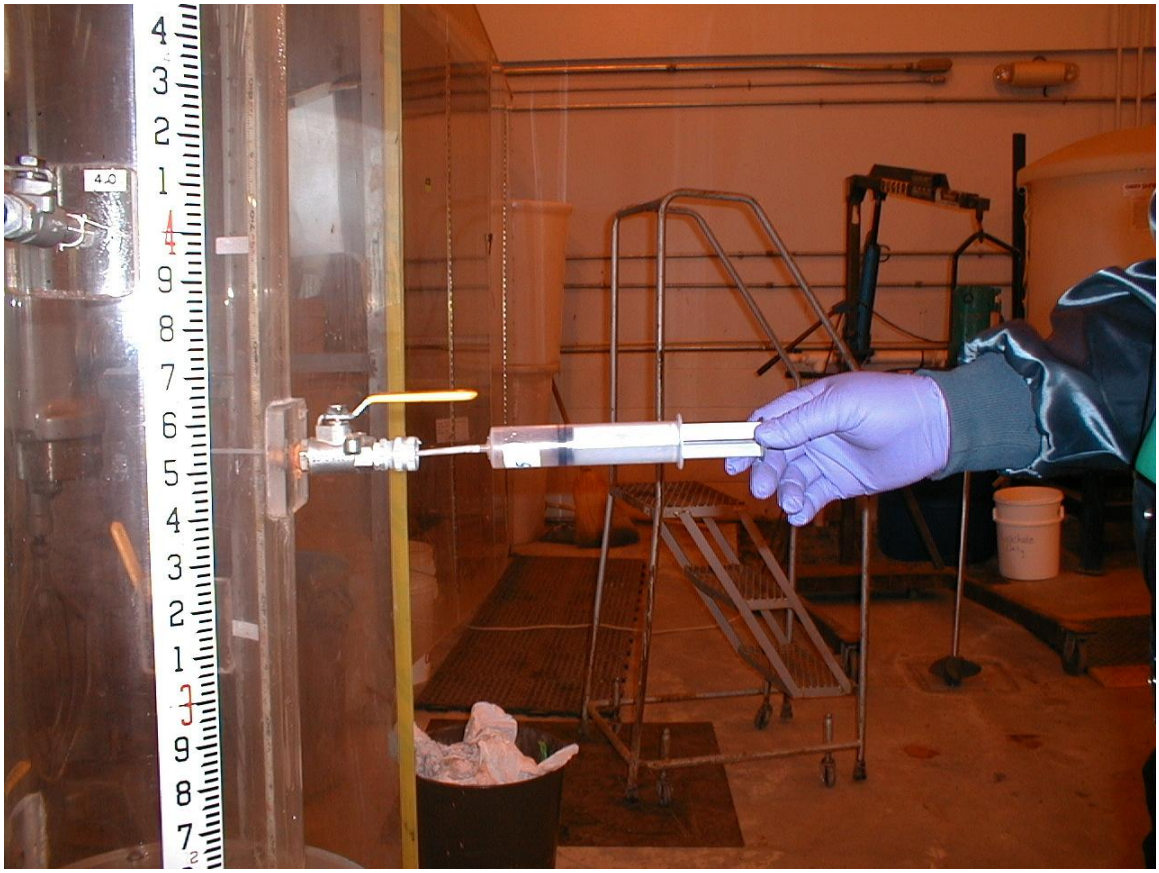
AREA
316
8
9


Cap Nut
Cap Washer
Bushing
Cable Gland Body
3/8" Ball Valve
3/8" ID Threaded Pipe Connector



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HOBART