

برنامج المسار الوظيفي للعاملين بقطاع مياه الشرب والصرف الصحي

دليل المتدرب

البرنامج التدريبي كيميائي مياه الصرف الصحي - الدرجة الثالثة

Sludge analysis



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Settleable Solids (SS)

General discussion

Introduction

Settleable solids in surface and saline waters as well as domestic and industrial wastes may be determined and reported on either a volume (ml/L) or a weight (mg/L) basis.

The QC practices considered to be an integral part of each method are summarized in Table 2020: II.

Sampling and storage

- Collect samples in clean plastic or glass bottles.
- Refrigerate at 4 °C up to the time of analysis to minimize microbiological decomposition of solids.
- Analyze within 24 h. bring to room temperature before analysis.

Apparatus:

For volumetric determination:

- Imhoff cones.

For gravimetric determination:

- All equipment for suspended solids and a glass vessel with a minimum diameter of 9 cm.



Drying oven



Desiccator



Elect. Balance



Filtration System



Imhoff cones

Procedure:**a. Volumetric:**

Fill an Imhoff cone to the 1 L mark with a well-mixed sample. Settle for 45 min, gently agitate sample near the sides of the cone with a rod or by spinning, settle 15 min longer, and record volume of settleable solids in the cone as milliliters per liter.

If the settled matter contains pockets of liquid between large settled particles, estimate volume of these and subtract from volume of settled solids.

The practical lower limit of measurement depends on sample composition and generally is in the range of 0.1 to 1.0 ml / L. Where a separation of settleable and floating materials occurs, do not estimate the floating material as settleable matter. Replicates usually are not required.

Where biological or chemical floc is present, the gravimetric method (3b) is preferred.

b. Gravimetric:

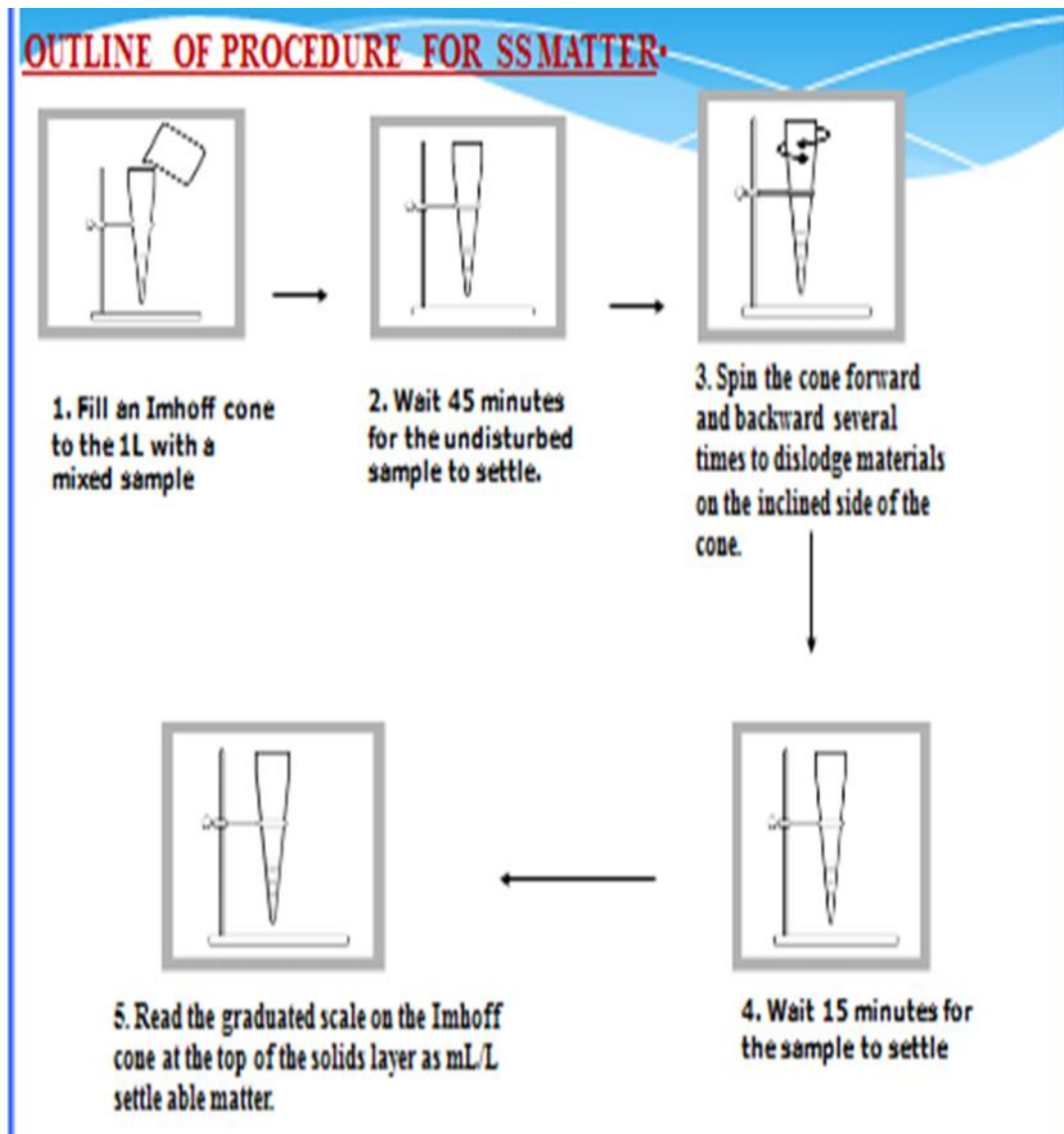
1) Determine total suspended solids.

2) Pour a well mixed sample into a glass vessel of not less than 9 cm diam using not less than 1 L and sufficient sample to give a depth of 20 cm. Alternatively use a glass vessel of greater diameter and a larger volume of sample. Let stand quiescent for 1 h and, without disturbing the settled or floating material, siphon 250 ml from center of container at a point halfway between the surface of the settled material and the liquid surface.

Determine total suspended solids (milligrams per liter) of this supernatant liquor. These are the nonsettleable solids.

Summary of method:

The amount of settleable matter in sewage treatment plant influent and effluent gives an empirical estimate of the type and extent of treatment required and the general quality of the water being discharged.



Calculation

$$\text{Settleable solids (mg/L)} = \frac{\text{total suspended solids (mg/L)}}{\text{nonsettleable solids (mg/L)}}$$

$$\text{Settleable solids (mg/L)} = A - B$$

Where: A = Total suspended solids (mg/L)
B = Nonsettleable solids (mg/L)

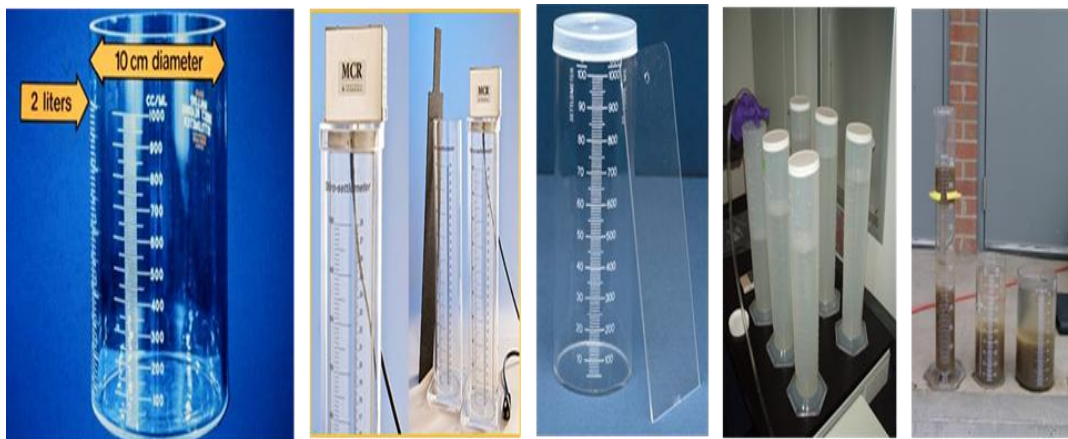
Settled Sludge Volume (SV30)

General Discussion

The settled sludge volume of a biological suspension is useful in routine monitoring of biological processes. For activated sludge plant control, 30 min settled sludge volume or the ratio of the 15-min to the 30 min settled sludge volume has been used to determine the returned-sludge flow rate and when to waste sludge.

The 30 min settled sludge volume also is used to determine sludge volume index 1

This method is inappropriate for dilute sludges because of the small volume of settled material. In such cases, use the volumetric test for settleable solids using an Imhoff cone.



Settling Column



Timer

Thermometer Types

Beaker

Apparatus

a. Settling column:

Use 1 L graduated cylinder equipped with a stirring mechanism consisting of one or more thin rods extending the length of the column and positioned within two rod diameters of the cylinder wall. Provide a stirrer able to rotate the stirring rods at no greater than 4 rpm (peripheral tip speed of approximately 1.3 cm/s).

b. Stopwatch.

c. Thermometer.

Procedure

Place 1.0 L sample in settling column and distribute solids by covering the top and inverting cylinder three times. Insert stirring rods, activate stirring mechanism, start the stop watch, and let suspension settle. Continue stirring throughout test. Maintain suspension temperature during test at that in the basin from which the sample was taken.

Determine volume occupied by suspension at measured time intervals, e.g., 5, 10, 15, 20, 30, 45, and 60 min.

Report settled sludge volume of the suspension in milliliters for an indicated time interval.

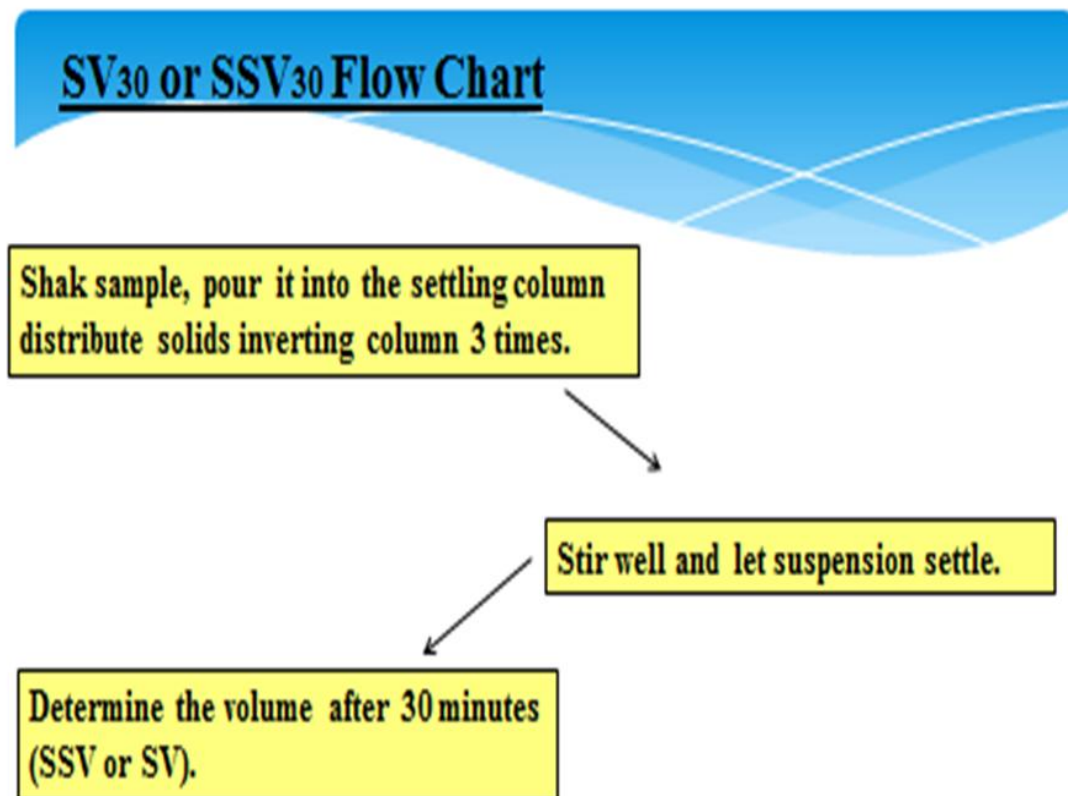
Variations in suspension temperature, sampling and agitation methods, dimensions of settling column, and time between sampling and start of the determination significantly affect results.

Precision and Bias

Bias is not applicable. The precision for this test has not been determined.

Reference

1. DICK, R.I. & P.A. VESILIND. 1969. The SVI—What is It? J. Water Pollut. Control Fed. 41:1285.



Sludge volume index (SVI) or stirred sludge volume index (SSVI)

Introduction

This method is taken from Standard Methods for the Examination of water and wastewater edition 19:1995.

Clarifier performance & effluent quality depend directly on sludge settling and thickening characteristics.

A measure of the sludge quality is the sludge volume index or SVI test.

SVI is the volume in ml occupied by one gram of MLSS after 30 minutes settling.

A more representative measure is the stirred sludge volume index or SSVI. Where, a sample of sludge is settled whilst being gently stirred for 30 minutes.

A normal SSVI result will range from 50-100 for a good settling sludge and 125 or higher for poor settling sludge.

Scope

This procedure describes how to measure Settled Sewage Volume Index.

Personal Responsible

Title	Technician
Responsibilities	Responsible to measure Settled Sewage Volume Index in wastewater samples.

Reagents

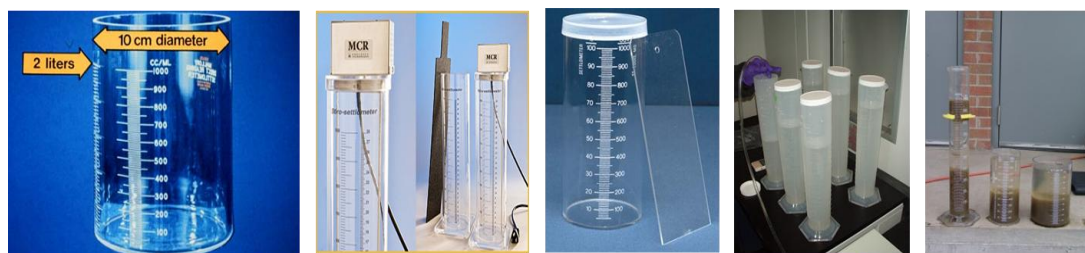
Hazards

Laboratory coats must be worn.

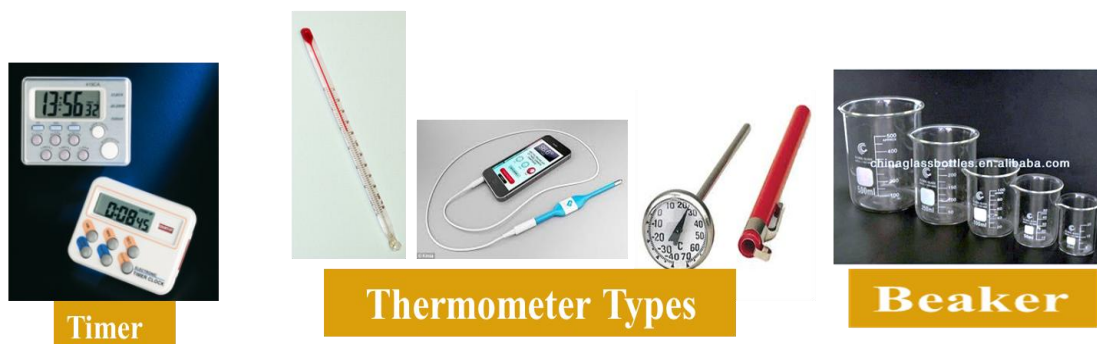
Safety goggles, gloves and eye protection must be worn.

Water Laboratory:

Distilled water.



Settling Column



Apparatus

1 Settling Column.

0.5 m deep and about 0.1 m in diameter with settlement impeded by a wire stirrer rotating at 1 r.p.m. The column is graduated to 50 units, each unit equals 60 ml.

2 Stop watch capable of measuring from 0 – 60 minutes.

3 Thermometer capable of measuring from 0 – 50 °C.

Procedure

1 Pour the well shaken sample into the settling column up to 50 ml mark and distribute solids inverting column 3 times.

2 Insert stirring rods, activate stirring mechanism and let suspension settle.

3 Continue stirring throughout test, maintain suspension temperature during test at that in the sludge basin from which the sample was taken.

4 Determine the volume occupied by the suspension after 30 minutes and report as SSV.

5 Determine the suspended solids concentration for the suspension and report as MLSS in g/L.

6 The individual SSV (ml/1000 ml) is calculated by dividing the volume of sludge after 30 min. by the volume taken for analysis then multiply by 1000.

$$\text{Individual SSV} = \frac{\text{Volume of sludge after 30 min (units/50 units)}}{\text{Volume of sample taken for analysis (units)}} \times 1000$$

7 The individual SSVI (ml/g) is calculated by dividing SSV by MLSS.

$$\text{Individual SSVI (ml/g)} = \frac{\text{SSV (ml/ 1000 ml)}}{\text{MLSS (g/1000 ml)}}$$

8 Determination of SSVI at 3500 mg/L

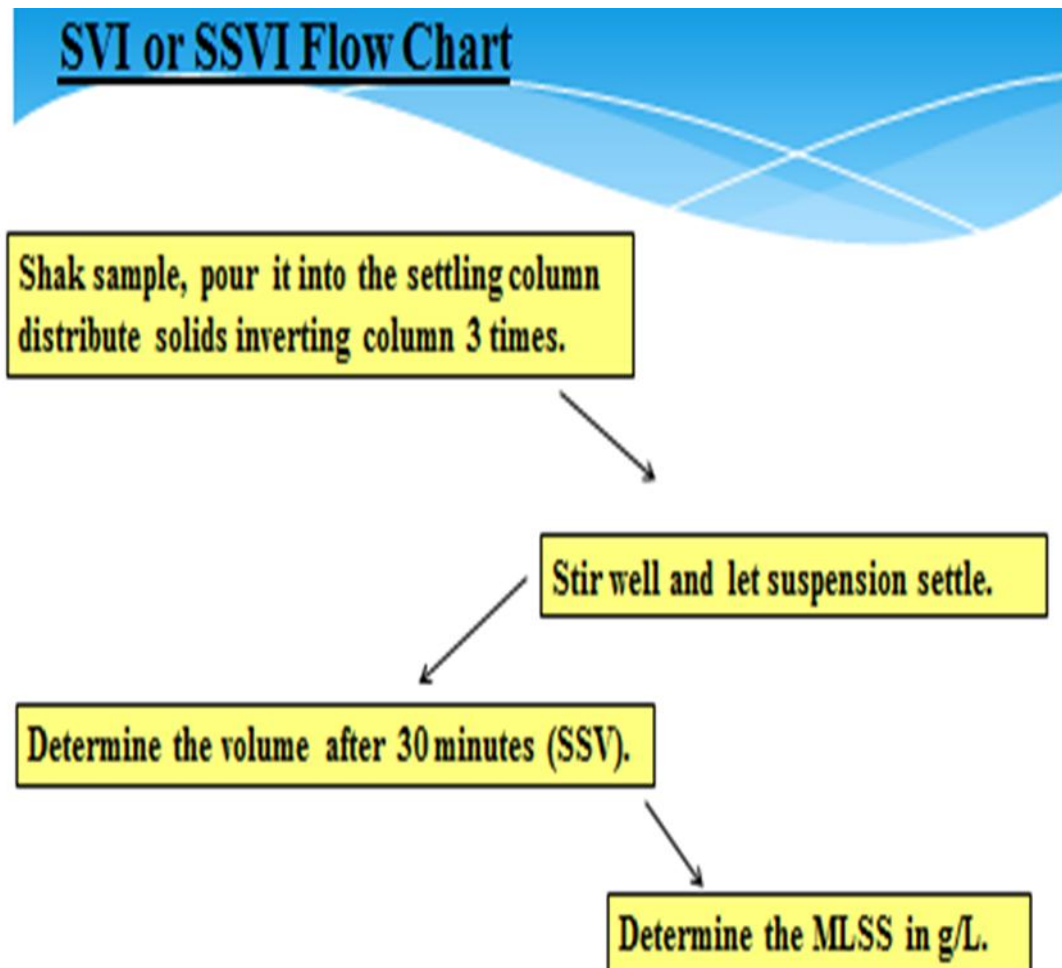
Plot the total suspended solids of the Mixed Liquor (MLSS) and Return Activated Sludge (RAS) against the SSVI for the same sample.

Determine the slope and intercept on the y axis.

$$\text{SSVI at 3500 mg/L} = \frac{3500 - \text{Intercept on y axis}}{\text{Slope}}$$

Reporting results

- 1 Enter all data onto the SSVI spread sheet on the computer, save the spread sheet and print out the SSVI work sheet.
- 2 Pass to the Senior Scientist or Laboratory Manager to sign off.
- 3 When the SSVI sheet has been signed off the daily report can be prepared.
- 4 The results must be reported to the nearest whole number.



Percentage of Dry & Volatile Solids (DS % & VS %)

Introduction

This method is based on the "Standard Methods for the Examination of Waters and Waste waters" 19th edition 1995, 2550 G.

A well-mixed sample is evaporated in a weighed basin and dried to constant weight in an oven at 103-105 °C. The increase in weight over that empty dish represents the total solids.

Total solids include total suspended solids and total dissolved solids.

The loss on ignition of dry residue volatile solids is also determined gravimetrically. The residue in the dish from the dried sludge solids determination is ignited in a furnace at 550 °C.

Interferences

1 The presence of substances which are volatile at temperatures below 105 °C or which decompose at temperatures below 105 °C to form volatile compounds will give falsely low results for the total solids determination.

2 For many purposes the loss on ignition determination is used as a measure of the organic solids content. In these circumstances the loss of volatile inorganic substances may occur giving high results, also there may be incomplete combustion of all organic substances giving low results.

Personnel responsible

The laboratory manager and his staff are responsible for measurement of total and volatile solids.

Health & Safety

- 1 Wear a lab coat when working in a laboratory.
- 2 Wear gloves when handling sewage and sewage sludge.
- 3 Use tongs when removing hot evaporating dishes from the furnace or oven.

Quality Control Standard (QC)

A synthetic sludge is made by mixing 5 g of macerated GF/C paper and 15 g of macerated china clay based photocopy paper (both previously dried in an oven at 105 °C overnight) and making up to 1 litre in a volumetric flask. This mixture gives a nominal %DM of 2% and a %VM of 60%. A QC sample should be analyzed with every batch of twenty samples.

This mixture is stable for 2 months. Mark with expiry date.



Cylinders



Desiccators



Steam bath



Wash bottle



Elec. Balance



Vol. Flask



Drying Oven



Muffle furnace



Beaker

Equipment & Apparatus Required

- 1 Analytical balance capable of weighing to 1 mg
- 2 Steam bath
- 3 Drying oven (set at 103 to 105 °C) ± 5 °C
- 4 Muffle furnace (set at 550 C) ± 20 °C
- 5 Tongs
- 6 Desiccator
- 7 Wash bottle for deionized water
- 8 Measuring cylinders
- 9 Evaporating dish "Porcelain or Nickel" 150-175 ml capacity suitable for ignition at 550 °C

Procedures

The following Quality Control checks for total solids and volatile solids must be used.

Quality Control Standard and control chart.

- Replicate drying to constant weight.
- Distilled water blank.

Quality Control Standard

- 1 A quality control standard (QC) must be run with each bath of samples.
- 2 The concentration used is 2% dried solids and 60% volatile matter.
- 3 An analyst independent of the analyst who carries out the analysis must prepare the quality control standard solution.
- 4 2% Dried solids and 60%. For volatile matter Quality Control Standard See section 2.2 for preparation procedure. This standard should be run as a sample

Total and Volatile Solids Quality Control Chart

“Shewert” Control charts have been set up using the standard material 2% dried solids, 60% volatile matter at as target concentrations.

For a more detailed explanation of Quality Control, charts and how they should be used refer to document LP 02 “Quality Control Charts”.

Plot the result from the quality control standard onto the control chart as soon as the result is available.

Replicate Drying to Constant Weight (Total solids only)

- 1 After drying on a water bath the evaporating basin is dried in an oven for 1 hour, cooled in a desiccator and weighed for a second time. The evaporating dish is then placed back in the oven for a further 30 minutes cooled in a desiccator and weighed for a third time.
- 2 The % change in weight between weight 1 and 2 and weight 1 and 3 must not be greater than 4%. If the difference is greater than this the evaporating basin and sample must be returned to the oven and dried for a further 30 minutes.

Distilled water blank

Distilled water blank must be included with each batch of samples.

Approximately 100 ml of water is treated as a sample.

After drying and weighing as 4.4.1 above the % change in weight between 1 and 2 and weight 1 and 3 must not be greater than 4%. If the difference is greater than this, an investigation must take place, do not report the results.

Preparation of the evaporating basin

- 1 A sufficient number of suitable clean evaporating basins should be fired in the furnace in advance of the work starting so that they are ready for use without delay. After firing, the evaporating dishes should be transferred to an oven and stored until needed. Then transfer to a desiccator prior to weighing.
- 2 The time that evaporating basins are out of the desiccator until they are weighed should be kept to a minimum.
- 3 After use, the evaporating basins should be cleaned: washed with tap water, rinsed with deionized water, and allowed to drain and dry.
- 4 Each evaporating basin should be clearly marked with an individual number or letter that will not be erased or lost on heating and firing.

Steam Bath

- 1 Ensure that the steam bath is filled to the correct level with water before switching on the power.
- 2 30 minutes before the evaporating basins are transferred to the steam bath switch on the heater. Ensure that the water is boiling before you start analysis.

Oven

A daily record of the oven temperature should be kept in the sludge solids log book. The temperature must be maintained at 105 ± 5 °C.

Muffle Furnace

A daily record of the furnace temperature should be kept. The temperature must be maintained at 550 °C.

Balance

- 1 The analytical balance should be ready for use, level, properly calibrated, and clean at all times.
- 2 Before each batch of weighing, the analyst must make a brief check to ensure that:
 - a. The balance is level,
 - b. The pan is clean,
 - c. The zero is properly set.
- 3 For accurate weighing, the doors to the balance chamber must be properly closed. A stable reading must be achieved. An "O" symbol is displayed if the weight reading is unstable. The weight reading must be recorded after the "O" symbol disappears. The analyst must ensure that the balance is clean after use.
- 4 Initial weighing and preparation of samples
- 5 Weigh a clean pre-prepared evaporating basin (see section 4.5) to the nearest 1 mg and record the weight on the sludge solids work sheet.

- 6 Pour a suitable volume of sludge sample into the evaporating basin (approximately 100-125 ml).
- 7 Weigh the evaporating basin and sludge sample to the nearest 1 mg and record the weight on the sludge solids work sheet. Repeat for all samples, QC standard and blank distilled water.
- 8 Transfer the evaporating basins to a pre-heated steam bath. Allow the basin to remain on the water bath until all visible water has evaporated.

First Drying & Weighing

- 1 Record the oven temperature on the sludge work sheet.
- 2 Immediately transfer the evaporating basin to the oven, from the steam bath.
- 3 Record the time when the drying began on the sludge work sheet.
- 4 Allow the evaporating basins to remain in the oven for 1 hour $\pm$ 5 minutes.
- 5 After 1 hour using suitable tongs, remove the evaporating dishes from the oven and transfer to the desiccator and allow cooling to room temperature (about 15 minutes).
- 6 Record the time when the drying was ceased on the sludge work sheet.
- 7 After cooling, transfer individually the cooled evaporating basins from the desiccator, to the balance pan.
- 8 Weigh the evaporating dishes and record the weights on the sludge work sheet.
- 9 Repeat steps for the remaining samples of the batch including the QC sample, and the distilled water blank.

Repeat Drying & Weighing

- 1 Record the oven temperature on the sludge work sheet.
- 2 Immediately transfer the evaporating basin to the oven for the second drying period.
- 3 Record the time when the drying began on the sludge work sheet.
- 4 Allow the evaporating basin to remain in the oven for 30 minutes $\pm$ 5 minutes.
- 5 Using suitable tongs remove the evaporating dishes from the oven and transfer to the desicator and allow cooling to room temperature (about 15 minutes).
- 6 Record the time when the drying was ceased on the sludge work sheet.
- 7 After cooling, transfer individually, the cooled evaporating dishes from the desicator, to the balance pan.
- 8 Weigh the evaporating dishes and record the weights on the sludge work sheet.

Quality Control Checks for Total Solids

- 1 Enter the first second and third weighing into the computer spread sheet for all samples, the quality control sample and distilled water blank.
- 2 The computer will calculate the dried solids of all the samples including the blank and quality control standard. If the quality control criteria are not met then the computer will indicate a QC failure and not calculate the results. The exception is the QC standard which must be plotted on the QC chart and determined manually.
- 3 You should calculate the dried solids QC before ashing the samples for the volatile matter determination.

Quality Control Checks after analysis

1 Plot the quality control standard result on the quality control chart and check that the standard meets the quality requirements (detailed in LP02 Quality Control Charts laboratory procedure). If the quality requirements are not met, report the failure to the Laboratory Scientist and Laboratory Manager. The results must not be reported.

Weighing to constant weight

If the difference between the initial weight of sludge and second weight and the initial weight and third weight is greater than 4% then the computer spread sheet will not calculate the result.

You must not report the result. Inform the Senior Chemist and Laboratory Manager.

Distilled water blank

See substitute distilled water.

If the weighings do not meet the QC criteria then transfer the basin to the oven for a further 30 minute period and weigh again.

If the weights still do not meet the QC criteria then report to the Senior Chemist and Laboratory Manager. **[Do not report the results]**

Determination of volatile matter

- 1- After the successful determination of the dried solids value the evaporating basin with the dried sludge samples, QC standard and blank should be placed in the muffle furnace using tongs at $550 \pm 20^\circ \text{C}$ for 1 hour. **[Record the furnace temperature]**.
- 2- After ashing remove the hot basin and ashed samples using tongs to an oven at $105 \pm 5^\circ \text{C}$ to allow them to cool (approximately 30 minutes).
- 3- Transfer the ashed sample and basin to the desiccator and allow cooling.
- 4- Weigh each sample the QC and the blank on the analytical balance.

- 5- The weighings into the computer spreadsheet. The computer will then calculate the volatile matter in the sample.

Quality Control Checks for Volatile Matter

- 1 Plot the QC standard result on the quality control chart and check that the standard meets the quality requirements (detailed in LP02 "Quality Control Charts" Laboratory procedure)
- 2 If the quality requirements are not met report the failure to the Laboratory Scientist and Laboratory Manager. You must not report the result.

Calculation of total and volatile solids

- 1 If all quality checks (in 4.13, 4.1.4 and 4.20 meet the requirements) calculate the % dried and % volatile solids, using the computer. Should the calculation need to be done manually then the following equations must be used:

Total solids = $(W_3 - W_1 / W_2 - W_1) \times 100\% \text{ w/w}$ (as a % of the wet sludge)

Volatile solids = $(W_3 - W_4 / W_3 - W_1) \times 100\% \text{ w/w}$ (as a % of the dried sludge)

Where:

W_1 = Empty Dish Weight.

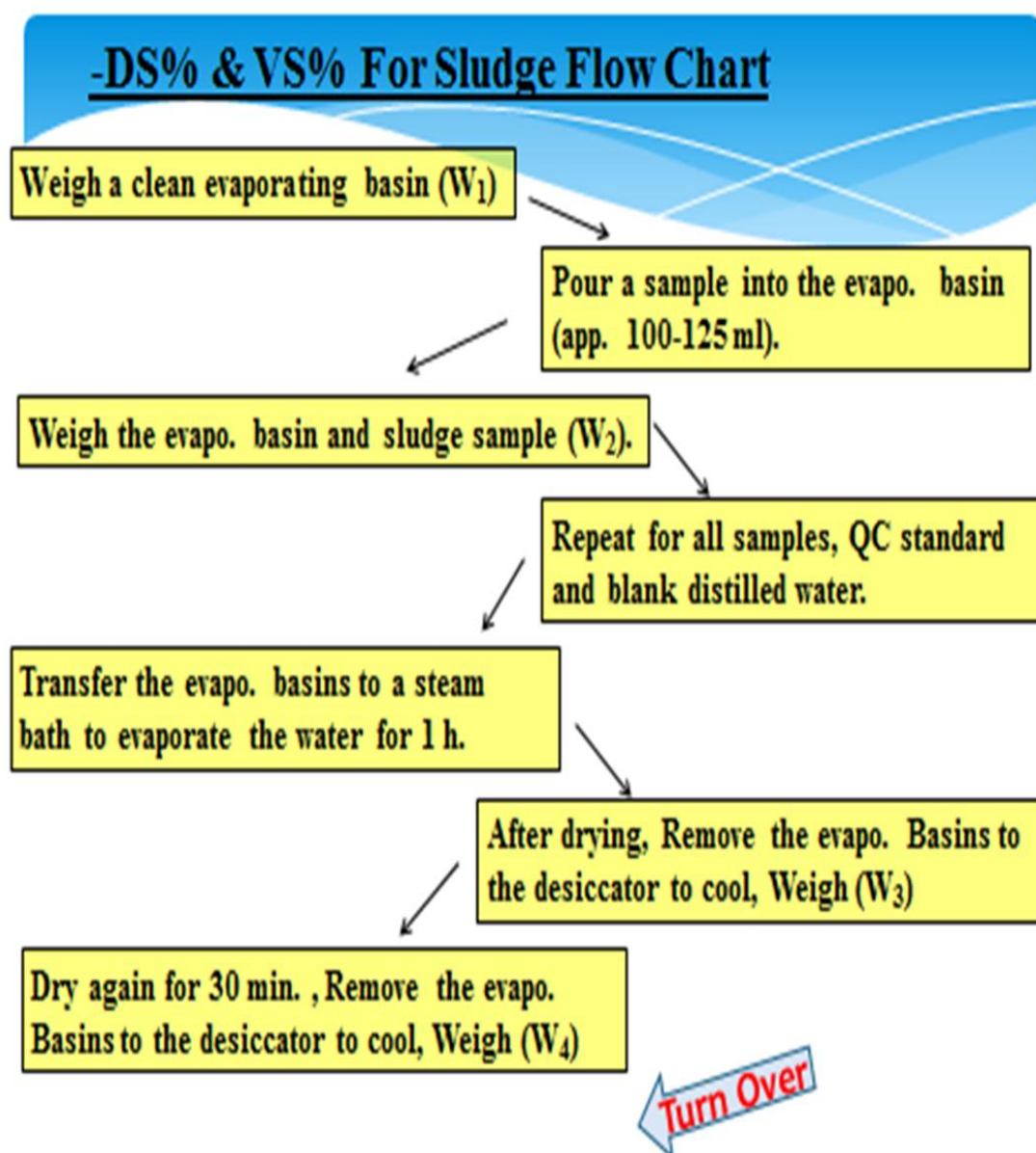
W_2 = Weight of dish + wet sludge.

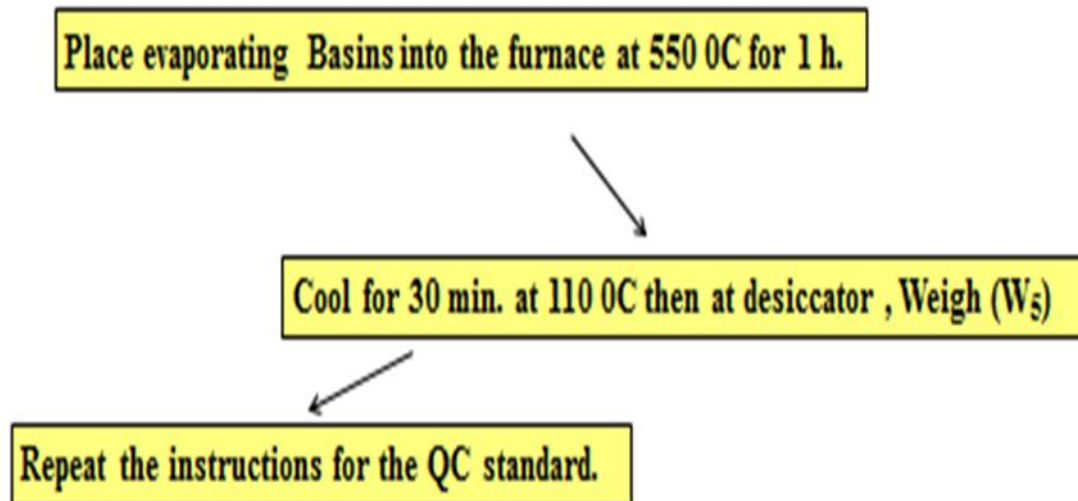
W_3 = Weight of dish + dry Sludge

W_4 = Weight of dish + ashed sludge.

Saving and reporting results

- 1 All % dried and volatile solids must be reported to 1 decimal place.
- 2 When all data has been entered and the quality control checks have passed, save the computer file by pressing (control + S)
- 3 Then print the sludge report sheet (control + P), and pass to the Senior Scientist or Laboratory Manager to sign off.
- 4 When the sludge report sheet has been signed off the daily report sheet can then be reported.
- 5 Sludge dried solids and volatile matter is automatically copied from the sludge report sheet to the daily report sheet.
- 6 File paper copies of all reports in the appropriate report file.





Capillary Suction Time (CST)

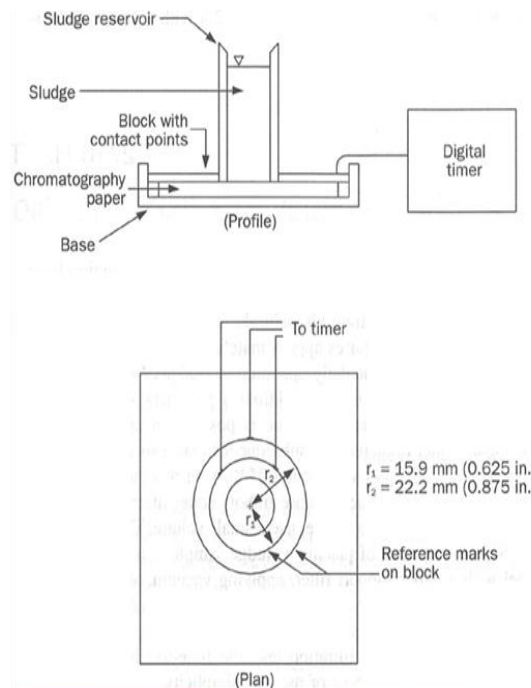
Introduction

The capillary suction time (CST) test determines rate of water release from sludge.

It provides a quantitative measure, reported in seconds, of how readily a sludge releases its water.

The results can be used to assist in sludge dewatering processes; to evaluate sludge conditioning aids and dosages; or, when used with a jar test and the settleable solids procedure, to evaluate coagulation effects on the rate of water release from sludges.

The test consists of placing a sludge sample in a small cylinder on a sheet of chromatography paper. The paper extracts liquid from the sludge by capillary action. The time required for the liquid to travel a specified distance is recorded automatically by monitoring the conductivity change occurring at two contact points appropriately spaced and in contact with the chromatography paper. The elapsed time is indicative of the water drainage rate. The CST test has been used as a relative indicator to characterize the performance of most sludge dewatering processes.



Wash bottle



Beaker



Capillary Suction Time (CST)

Apparatus

a. Test materials and apparatus may be fabricated or commercially available.

The unit includes a paper support block, stainless steel reservoir with 18-mm ID and 25 mm height, and a digital timer.

b. CST paper.

c. Thermometer to read ± 0.5 °C.

d. Pipet, 10 ml, plastic with tip trimmed to allow free passage of sludge flocs.

Procedure

Turn on and reset CST meter. Dry CST test block and reservoir. Place a new CST paper on lower test block with rough side up and grain parallel to the 9-cm side. Add upper test block, insert sludge reservoir into test block, and seat it using light pressure and a quarter turn to prevent surface leaks. Measure and record temperature of sludge. Pipet 6.4 mL sludge into test cell reservoir; if pipetting is difficult because of sludge consistency, pour a representative sludge sample into the test cell until it is full. The CST device will begin time measurement as liquid being drawn into the paper reaches the inner pair of electrical contacts. Timing ends when the outer contact is reached. Record CST shown on digital display. Empty remaining sludge from reservoir and remove and discard used CST paper. Rinse and dry test block and reservoir. Repeat for a minimum of five determinations per sample to account for measurement variation and to allow identification of any faulty readings due to leaks or spills.

Variations in sludge temperature and sample volume can affect CST results. Ensure that all analyses are run under similar conditions. Sludge suspended solids concentration has a significant effect on test results. In evaluating sludge conditioners or monitoring operation of a dewatering process, avoid this effect by ensuring homogeneity among sludge samples. Comparison of CST data from different sludge samples from the same source (especially if taken on different days) cannot be made with confidence unless suspended solids concentrations are comparable. Make a rough correction for different solids contents by dividing the sludge's CST value by its corresponding solids concentration.

Characteristics of CST paper may vary between lots. If comparison of CST values for distilled water indicates such variations, subtract times for distilled water blanks from sample times to improve comparisons.

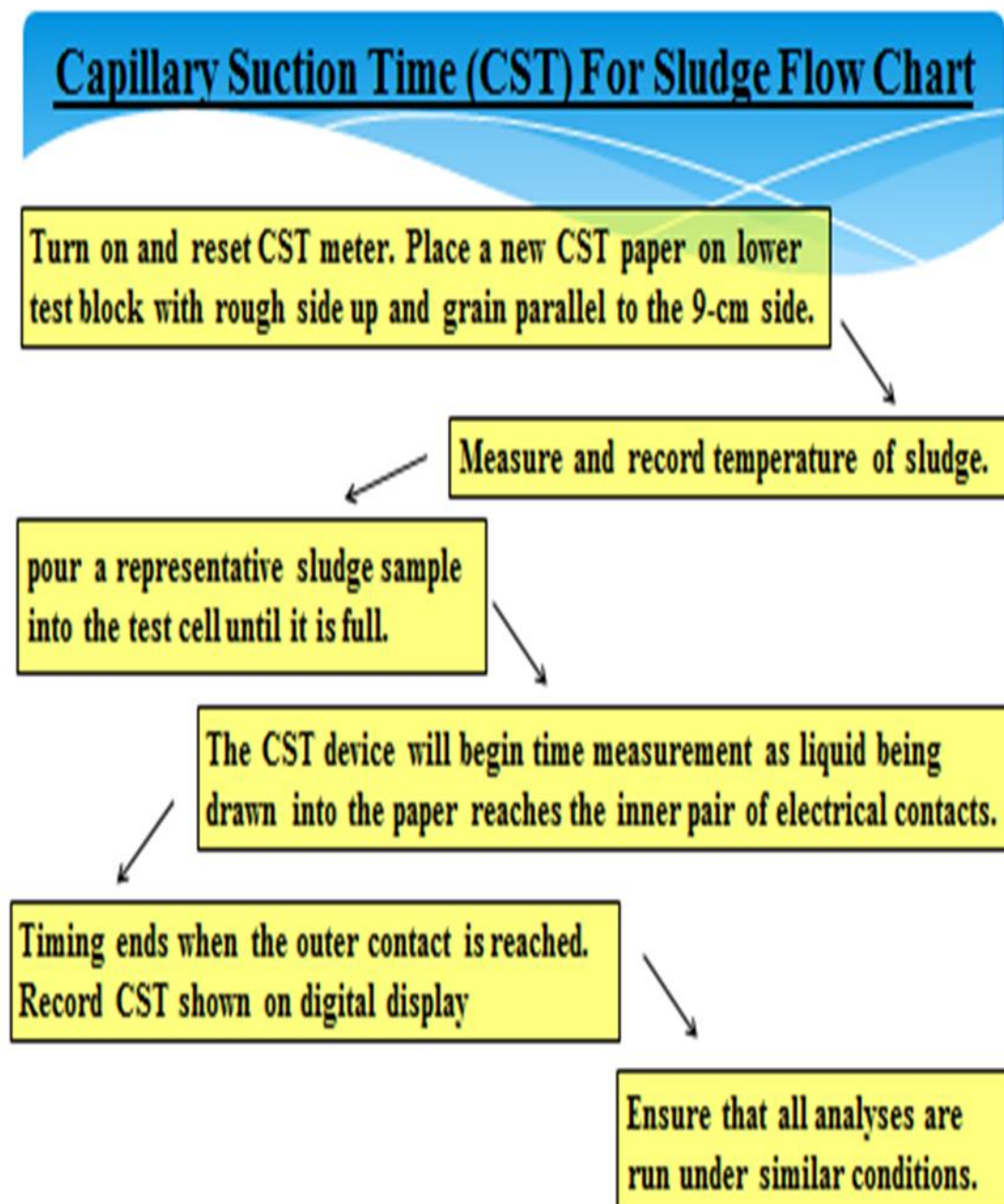
Record CST model used, paper type, sludge type, sludge temperature, and capillary suction time.

Measure solids concentration and CST of distilled water using the same paper to provide useful information.

Precision and Bias

Ten tests conducted on an anaerobically digested pulp mill sludge resulted in a mean CST of 363.2 s with a standard deviation of 36.2 s. Twenty tests using an anaerobically digested municipal wastewater sludge gave a mean of 85.2 s with a standard deviation of 14.12 s.

Triplicate analyses of 30 sample sets of conditioned and unconditioned alum sludge resulted in an average standard deviation of 1.0 s with means between 5 and 80 s. Method bias cannot be determined.



المراجع

- تم الإعداد بمشاركة المشروع الألماني GIZ
- و مشاركة السادة :-

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