2710 TESTS ON SLUDGES*

2710 A. Introduction

This section presents a series of tests uniquely applicable to sludges or slurries. The test data are useful in designing facilities for solids separation and concentration and for assessing operational behavior, especially of the activated sludge process.


2710 B. Oxygen-Consumption Rate

1. General Discussion

This test is used to determine the oxygen consumption rate of a sample of a biological suspension such as activated sludge. It is useful in laboratory and pilot-plant studies as well as in the operation of full-scale treatment plants. When used as a routine plant operation test, it often will indicate changes in operating conditions at an early stage. However, because test conditions are not necessarily identical to conditions at the sampling site, the observed measurement may not be identical with actual oxygen consumption rate.

2. Apparatus

a. Oxygen-consumption rate device: Either:
   1) Probe with an oxygen-sensitive electrode (polarographic or galvanic), or
   2) Manometric or respirometric device with appropriate readout and sample capacity of at least 300 mL. The device should have an oxygen supply capacity greater than the oxygen consumption rate of the biological suspension, or at least 150 mg/L · h.
   b. Stopwatch or other suitable timing device.
   c. Thermometer to read to ± 0.5°C.

3. Procedure

a. Calibration of oxygen-consumption rate device: Either:
   1) Calibrate the oxygen probe and meter according to the method given in Section 4500-O.G, or
   2) Calibrate the manometric or respirometric device according to manufacturer’s instructions.

b. Volatile suspended solids determination: See Section 2540.

c. Preparation of sample: Adjust temperature of a suitable sample portion to that of the basin from which it was collected or to required evaluation temperature, and maintain constant during analysis. Record temperature. Increase DO concentration of sample by shaking it in a partially filled bottle or by bubbling air or oxygen through it.

d. Measurement of oxygen consumption rate:
   1) Fill sample container to overflowing with an appropriate volume of a representative sample of the biological suspension to be tested.
   2) If an oxygen-sensing probe is used, immediately insert it into a BOD bottle containing a magnetic stirring bar and the biological suspension. Displace enough suspension with probe to fill flared top of bottle and isolate its contents from the atmosphere. Activate probe stirring mechanism and magnetic stirrer. (Note: Adequate mixing is essential. For suspensions with high concentrations of suspended solids, i.e., >5000 mg/L, more vigorous mixing than that provided by the probe stirring mechanism and magnetic stirrer may be required.) If a manometric or respirometric device is used, follow manufacturer’s instructions for startup.

   3) After meter reading has stabilized, record initial DO and manometric or respirometric reading, and start timing device. Record appropriate DO, manometric, or respirometric data at time intervals of less than 1 min, depending on rate of consumption. Record data over a 15-min period or until DO becomes limiting, whichever occurs first. The oxygen probe may not be accurate below 1 mg DO/L. If a manometric or respirometric device is used, refer to manufacturer’s instructions for lower limiting DO value. Low DO (<2 mg/L at the start of the test) may limit oxygen uptake by the biological suspension and will be indicated by a decreasing rate of oxygen consumption as the test progresses. Reject such data as being unrepresentative of suspension oxygen consumption rate and repeat test beginning with higher initial DO levels.

   The results of this determination are quite sensitive to temperature variations and poor precision is obtained unless replicate determinations are made at the same temperature. When oxygen consumption is used as a plant control test, run periodic (at least monthly) replicate determinations to establish the precision of the technique. This determination also is sensitive to the time lag between sample collection and test initiation.

4. Calculations

If an oxygen probe is used, plot observed readings (DO, milligrams per liter) versus time (minutes) on arithmetic graph paper and determine the slope of the line of best fit. The slope is the oxygen consumption rate in milligrams per liter per minute.

If a manometric or respirometric device is used, refer to manufacturer’s instructions for calculating the oxygen consumption rate.
Calculate specific oxygen consumption rate in milligrams per gram per hour as follows:

\[
\text{Specific oxygen consumption rate, } (\text{mg/g})/h = \frac{\text{oxygen consumption rate, } (\text{mg/L})/\text{min}}{\text{volatile suspended solids, g/L}} \times \frac{60 \text{ min}}{\text{h}}
\]

5. Precision and Bias

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

6. Bibliography


2710 C. Settled Sludge Volume

1. General Discussion

The settled sludge volume of a biological suspension is useful in routine monitoring of biological processes. For activated sludge plant control, a 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\(^1\) (Section 2710D).

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 (2540F). Results from 2540F are not comparable with those obtained with the procedure herein.

2. 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 approximately, but no more than, 4 rpm (peripheral tip speed of approximately 1.3 cm/s). See Figure 2710:1.

b. **Stopwatch.**
c. **Thermometer.**

3. 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 stopwatch, 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.

Test results are applicable to a particular test site and are significantly affected by variables such as suspension temperature, sampling and agitation methods, dimensions of settling column and stirring rods, stirring speed, and time between sampling and start of the determination.

4. Precision and Bias

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

5. Reference

2710 D. Sludge Volume Index

1. General Discussion

The sludge volume index (SVI) is the volume in milliliters occupied by 1 g of a suspension after 30 min settling. SVI typically is used to monitor settling characteristics of activated sludge and other biological suspensions.\(^1\) Although SVI is not supported theoretically,\(^2\) experience has shown it to be useful in routine process control.

2. Procedure

Determine the suspended solids concentration of a well-mixed sample of the suspension (See Section 2540D).

Determine the 30 min settled sludge volume (See Section 2710C).

3. Calculations

\[
\text{SVI} = \frac{\text{settled sludge volume (mL/L)} \times 1000}{\text{suspended solids (mg/L)}}
\]

4. Precision and Bias

Precision is determined by the precision achieved in the suspended solids measurement, the settling characteristics of the suspension, and variables associated with the measurement of the settled sludge volume. Bias is not applicable.

5. References


6. Bibliography


2710 E. Zone Settling Rate

1. General Discussion

At high concentrations of suspended solids, suspensions settle in the zone-settling regime. This type of settling takes place under quiescent conditions and is characterized by a distinct interface between the supernatant liquor and the sludge zone. The height of this distinct sludge interface is measured with time. Zone settling data for suspensions that undergo zone settling, e.g., activated sludge and metal hydroxide suspensions, can be used in the design, operation, and evaluation of settling basins.\(^1\)\(^3\)

2. Apparatus

a. **Settling vessel:** Use a transparent cylinder at least 1 m high and 10 cm in diameter. To reduce the discrepancy between laboratory and full-scale thickener results, use larger diameters and taller cylinders.\(^1\)\(^3\) Attach a calibrated millimeter tape to outside of cylinder. Equip cylinder with a stirring mechanism, e.g., one or more thin rods positioned within two rod diameters of the internal wall of settling vessel. Stir suspension near vessel wall over the entire depth of suspension at a peripheral speed no greater than 1 cm/s. Greater speeds may interfere with the thickening process and yield inaccurate results.\(^4\) Provide the settling vessel with a port in the bottom plate for filling and draining. See Figure 2710:2.

b. **Stopwatch.**
c. **Thermometer.**

3. Procedure

Maintain suspension in a reservoir in a uniformly mixed condition. Adjust temperature of suspension to that of the basin from which it was collected or to required evaluation temperature. Record temperature. Remove a well-mixed sample from reservoir and measure suspended solids concentration (Section 2540D).

Activate stirring mechanism. Fill settling vessel to a fixed height by pumping suspension from reservoir or by gravity flow. Fill at a rate sufficient to maintain a uniform suspended solids concentration throughout settling vessel at end of filling. The suspension should agglomerate, i.e., form a coarse structure with visible fluid channels, within a few minutes. If suspension does not agglomerate, test is invalid and should be repeated.

Record height of solids-liquid interface at intervals of about 1 min. Collect data for sufficient time to assure that suspension is exhibiting a constant zone-settling velocity and that any initial reflocculation period, characterized by an accelerating interfacial settling velocity, has been passed.

Zone settling rate is a function of suspended solids concentration, suspension characteristics, vessel dimensions, and laboratory artifacts. With the filling method described above and a sufficiently large cylinder, these artifacts should be minimized. However, even with careful testing suspensions often may behave erratically. Unpredictable behavior increases for sludges with high solids concentrations and poor settling characteristics, and in small cylinders.

4. Calculations

Plot interface height in centimeters vs. time in minutes.\(^1\)\(^3\) Draw straight line through data points, ignoring initial shoulder or reflocculation period and compression shoulder. Calculate interfacial settling rate as slope of line in centimeters per minute.
5. Precision and Bias

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

6. References


7. Bibliography


2710 F. Specific Gravity

1. General Discussion

The specific gravity of a sludge is the ratio of the masses of equal volumes of a sludge and distilled water. It is determined by comparing the mass of a known volume of a homogeneous sludge sample at a specific temperature to the mass of the same volume of distilled water at 4°C.

2. Apparatus

Container: A marked flask or bottle to hold a known sludge volume during weighing.

3. Procedure

Follow either a or b.

a. Record sample temperature, T. Weigh empty container and record weight, W. Fill empty container to mark with sample, weigh, and record weight, S. Fill empty container to mark with water, weigh, and record weight, R. Measure all masses to the nearest 10 mg.

b. If sample does not flow readily, add as much of it to container as possible without exerting pressure, record volume, weigh, and record mass, P. Fill container to mark with distilled water, taking care that air bubbles are not trapped in the sludge or container. Weigh and record mass, Q. Measure all masses to nearest 10 mg.

4. Calculation

Use a or b, matching choice of procedure above.

\[
SG_{T,4^\circ C} = \frac{\text{weight of sample}}{\text{weight of equal volume of water at } 4^\circ C} - \frac{S - W}{R - W} \times F
\]

The values of the temperature correction factor F are given in Table 2710:I.

<table>
<thead>
<tr>
<th>Temperature $^\circ C$</th>
<th>Temperature Correction Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>0.9991</td>
</tr>
<tr>
<td>20</td>
<td>0.9982</td>
</tr>
<tr>
<td>25</td>
<td>0.9975</td>
</tr>
<tr>
<td>30</td>
<td>0.9957</td>
</tr>
<tr>
<td>35</td>
<td>0.9941</td>
</tr>
<tr>
<td>40</td>
<td>0.9922</td>
</tr>
<tr>
<td>45</td>
<td>0.9903</td>
</tr>
</tbody>
</table>

Figure 2710:2. Schematic diagram of settling vessel for zone settling rate test.

Table 2710:I. Temperature Correction Factor
b. Calculate specific gravity, $SG$, from the formula

\[
SG_{T/4\degree C} = \frac{\text{weight of sample}}{\text{weight of equal volume of water at } 4\degree C} = \frac{(P - W)}{(R - W) - (Q - P)} \times F
\]

For values of $F$, see Table 2710:I.

### 2710 G. Capillary Suction Time

#### 1. General Discussion

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.

#### 2. Apparatus

- **Test materials and apparatus** may be fabricated (see Figure 2710:3) or are commercially available. The unit includes a paper support block, stainless steel reservoir with 18-mm ID and 25-mm height, and a digital timer.

- **CST paper**,†

- **Thermometer** to read ± 0.5°C.

- **Pipet**, 10-mL, plastic with tip trimmed to allow free passage of sludge flocs.

#### 3. 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 varia-

---

* Venture Innovations, P.O. Box 53631, Lafayette, LA 70505; or Triton Electronics Ltd., Bigods Hall, Dunmow, Essex, England, CM63BE; or equivalent.
† Available from CST apparatus supplier or use Whatman No. 17 chromatography grade paper cut into 7- × 9-cm sections with grain parallel to long side.
tions, 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.

4. 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.

5. Bibliography


2710 H. Time-to-Filter

1. General Discussion

The time to filter (TTF) correlates with capillary suction time (CST) and is similar to the specific resistance to filtration if sludge solids content and filtrate viscosity do not vary among compared samples. The test requires approximately 200 mL sludge and can be used to assist in the daily operation of sludge dewatering processes or to evaluate sludge-conditioning polymers and dosages.

Testing with a smaller volume is possible in applications to evaluate water drainage rate subsequent to jar tests and settleable solids determination (see Section 2540F). In this case, drain collected sludge from one or more Imhoff cones after decanting as much supernatant as possible; use a small-volume TTF apparatus.

The test consists of placing a sludge sample in a Buchner funnel with a paper support filter, applying vacuum, and measuring the time required for 100 mL of sample to collect. While similar to the specific resistance to filtration test, the time-to-filter test is superior because of its ease of use and simplicity.

2. Apparatus

a. Time-to-filter large-volume or small-volume (Figure 2710:4) assembly.

b. Filter paper*

c. Stopwatch.

3. Procedure

Place paper filter in funnel and make a firm seal by pre-wetting with a small volume of water with vacuum on. If using large-volume apparatus, take a 200-mL sample of sludge. With vacuum pump providing a constant vacuum of 51 kPa, pour sample into funnel. Start stopwatch or timer and determine time required for 100 mL of sample to collect in graduated cylinder. This is the time to filter. Make a minimum of three replicate determinations.

For the small-volume test, use 7 to 10 mL sludge. Record time required for 50% of sample to collect in graduated cylinder. Compare this time to filter only to other results using the same sample volume.

Sludge suspended solids concentration has a significant effect on test results. In evaluating sludge-conditioning products, compare results for which initial suspended solids concentrations are comparable. Make a rough correction for

* Whatman No. 1 or 2 or equivalent.
different solids contents by dividing the time-to-filter value by its corresponding solids concentration. However, variations in solids concentration occur in full-scale applications, and the time-to-filter results may be interpreted as indicating the overall rate of water release from sludges, including the effect of differing solids concentrations.

4. Precision and Bias

Variations in vacuum pressure, support filter type, sludge temperature, and sample volume can affect test results. Triplicate analyses of 18 sample sets of conditioned and unconditioned alum sludge resulted in an average method precision of 19 s (approximately 4% of the average value) for the large-volume TTF test. Triplicate analyses of 9 sample sets of conditioned and unconditioned alum sludge resulted in a method precision of 9 s (approximately 6% of the average value) for the small-volume TTF test. Method bias, which refers to the agreement between the value determined by the test method and the real value, cannot be determined.

5. Bibliography


2710 I. Modified Settled Sludge Volume

1. General Discussion

See Section 2710C.
While 2710C determines settled sludge volume using a 1-L graduated cylinder equipped with a stirring mechanism, the settling column used with the procedure herein is a wide 2-L cylindrical vessel to allow suspension volume to be determined relative to the 1-L graduated test results. Use of a settling column having a diameter wider than the 1-L graduated cylinder has been suggested and the 2-L settling column is frequently used. Results from Method 2710C are not currently considered comparable with those obtained with the procedure herein.

2. Apparatus

a. Settling column: Use a 2-L graduated cylindrical vessel of approximately 13 cm diam, 19 cm height (outer dimensions) equipped with a stirring mechanism consisting of four thin rods extending the length of the column, with the outer rods positioned within two rod-diameters of the cylinder wall. Provide a stirrer able to rotate the stirring rods at approximately, but no more than, 2 rpm (peripheral tip speed of approximately 1.2 cm/s). See Figure 2710:5.

b. Stopwatch.

c. Thermometer.

3. Procedure

Start test as soon as possible after sample collection. Distribute solids in sample without breaking flocculated particles apart by gently inverting or swirling sample container three times. Gently pour 2.0 L sample in settling column and insert stirring rods. Activate stirring mechanism, start stopwatch, 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 of the suspension occupied at measured time intervals, e.g., 5, 10, 15, 20, 30, 45 and 60 min. Take readings after stirring rod rotates past graduated scale and disturbance at top of settling suspension is minimal.

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

Test results are applicable to a particular test site and are significantly affected by variables such as suspension temperature, sampling and agitation methods, dimensions of settling column and stirring rods, stirring speed, and time between sampling and start of the determination.

4. Precision and Bias

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

5. References