Inderstanding Laboratory Wastewater Tests: II. Solids (TS, TSS, TDS, TVS, TFS

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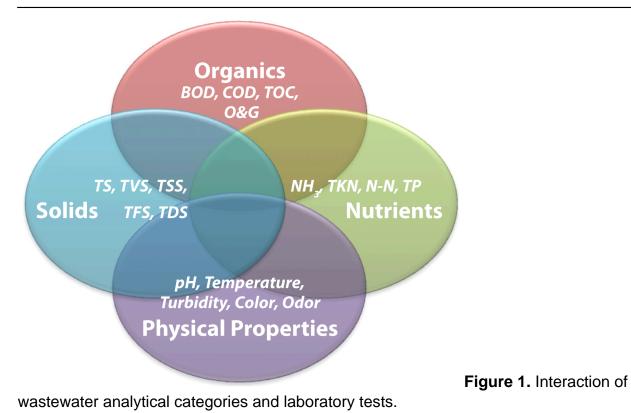
- Solids (TS, TSS, TDS, TVS, TFS)
- Total Suspended Solids (TSS)
- <u>Total Dissolved Solids (TDS)</u>
- Total Solids (TS), Total Volatile Solids (TVS), and Total Fixed Solids (TFS)

Since the implementation of the Clean Water Act and subsequent creation of the U.S. Environmental Protection Agency (EPA) in the early 1970s, industrial, institutional, and commercial entities have been required to continually improve the **quality** of their process wastewater effluent discharges.

At the same time, population and production increases have increased water use, creating a corresponding rise in wastewater **quantity**. This increased water use and process wastewater generation requires more efficient removal of by-products and pollutants that allows for effluent discharge within established environmental regulatory limits.

The determination of wastewater **quality** set forth in environmental permits has been established since the 1970s in a series of laboratory tests focused on four major categories:

- 1. **Organics -** A determination of the concentration of carbon-based (i.e., organic) compounds aimed at establishing the relative "strength" of wastewater [e.g., biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC), and oil and grease (O&G)].
- 2. **Solids -** A measurement of the concentration of particulate solids that can dissolve or suspend in wastewater [e.g., total solids (TS), total suspended solids (TSS), total dissolved solids (TDS), total volatile solids (TVS), and total fixed solids (TFS)].
- 3. **Nutrients -** A measurement of the concentration of targeted nutrients (e.g., nitrogen and phosphorus) that can contribute to the acceleration of *eutrophication* (i.e., the natural aging of water bodies).
- 4. **Physical Properties and Other Impact Parameters -** Analytical tests designed to measure a varied group of constituents directly impact wastewater treatability (e.g., temperature, color, pH, turbidity, odor).



Although wastewater analytical tests are often separated into categories, it is important to understand that these tests are not independent of each other (Figure 1). In other words, a potential contaminant identified by one test in one category can also be identified in another test in a separate category. For example, the organics in a wastewater sample represented by BOD will also be represented in the spectrum of solids, either as suspended (TSS) or dissolved (TDS) particulates. For most people a complete understanding of the standard methods required to accurately complete critical wastewater analytical tests is not necessary. However, a fundamental understanding of the theory behind, and working knowledge of, the basic procedures used for each test and the answers to commonly asked questions about each test can be a valuable tool for anyone involved in generating, monitoring, treating, or discharging process wastewater.

This publication is designed to provide a solid working knowledge of one of the major wastewater analytical test categories: **Solids.**

Solids (TS, TSS, TDS, TVS, TFS)

Analytical tests aimed at establishing the concentration of **solids** (i.e., **particulate matter**) suspended or dissolved in wastewater are used to control physical, chemical, and biological wastewater treatment processes and assess compliance with environmental regulatory permits. Today there are four common laboratory tests used to determine the concentration of the five forms of solids in wastewater that of are of interest:

- 1. TSS (biochemical oxygen demand)
- 2. TDS (total dissolved solids)
- 3. TS (total solids)
- 4. TVS (total volatile solids)

5. TFS (total fixed solids)

The World of the Micron

In today's world of solids removal in wastewater treatment, the typically unfamiliar unit of "micron" has emerged. As solids removal technology improves, it allows the removal of smaller and smaller particles—today, even in the "submicron" range. So, what is a micron? A micron (or micrometer, abbreviated μ m) is a unit of length that equals 1 millionth of a meter or 1 thousandth of a millimeter.

Interrelationships of Wastewater Solids (Part 1):

The interrelationships among the various forms of solids and the analytical tests used to measure their concentrations in wastewater can be confusing. Figure 2 graphically represents the interrelationships that the various solids analytical tests have to each other. What is important to keep in mind is that the total solids (TS) contained in a wastewater sample can be analyzed based using two basic viewpoints: **particulate size** or **organic content**.

Figure 2. Interrelationships of various forms of solids found in wastewater.



TS = total solids TSS = total suspended solids TDS = total dissolved solids VSS = volatile suspended solids VDS = total dissolved solids FDS = fixed dissolved solids TVS = total volatile solids TFS = total fixed solids

Particulate Size

Analysis of TS in a wastewater sample by the size of the particulates involves the use of the equation:

TS = TSS + TDS

where TS is total solids, TSS is total suspended solids, and TDS is total dissolved solids. The dividing line that determines whether a particulate is suspended (TSS) or dissolved (TDS) in a wastewater sample is the ? 1.5 μ m nominal pore size of the glass fiber filter used in the TSS or TDS test method.

Organic Content

Analysis of TS in a wastewater sample by organic content involves the use of the equation:

TS = TVS + TFS

where TS is total solids, TVS is total volatile (i.e., organic) solids, and TFS is total fixed (i.e., inorganic) solids. The dividing line that determines whether solids are organic (TVS) or inorganic (TFS) in a wastewater sample is whether they ignite and combust (TVS) in a muffle furnace or are left behind as an incombustible residue (TFS)

Interrelationships of Wastewater Solids (Part 2):

Confusion comes from the fact that a solid form from the **particulate size** equation can be subdivided into the forms from the **organic content** equation. As an example, the TSS recovered from a wastewater sample can be placed into a muffle furnace and the TVSS and TFSS determined based on the amount of TSS that is combusted. However, if one keeps in mind the two separate equations as presented (i.e., TS = TSS + TDS and TS = TVS + TFS), then confusion can be minimized.

Mathematical Note: Always remember, in any equation in the form of

$\mathsf{A}=\mathsf{B}+\mathsf{C}$

knowing the value of any two variables (i.e., B and C, A and B, or A and C) will yield the value of the third variable by a rearrangement of the equation.

Is My Wastewater "High-Strength"?

Wastewater generated by commercial, industrial, and institutional facilities is typically referred to as "high-strength" compared to typical household wastewater. Table 1 shows the typical concentrations (mg/L) of solids found in untreated domestic wastewater. This table can be used to understand how nonsanitary process wastewater compares to typical domestic wastewater.

Table 1. Typical Concentrations of Solids in Untreated Domestic Wastewater.		Typic Conc ntrati	ce
		n	
		(mg/l	∟)
	Constitu	Μ	
	ents	е	
		di	
		u	
		m	
	TS (total		
	solids)	0	2
			6
			0
	-TSS	21	4
	(total su	0	0
	spended		0
	solids)		
	–VSŚ	16	3
	(total	0	1
	volatile		5
	suspend		-
	ed		
	solids)		
	—FSS	50	8
	(total	00	5
	lulai		J

Constituents	Typical (Low	Concentration (mg/ Medium	L) High	
Constituente	2011	fixed su	-	
		spended		
		solids)	-	
		-TDS	50 8	
		(total dis		
		solved	0	
		solids)		
		—VDS	20 3	
		(total	0 4	
		volatile	0	
		dissolve)	
		d solids)	
		—FDS	30 5	
		(total dis	s 02	
		solved	0	
		solids)		
		Note. Adapted		
		from Metcalf &		
		Eddy, Inc., 2003.		

Total Suspended Solids (TSS)

TSS is typically determined through conducting an analytical test, while TDS can be determined through a subsequent calculation (if TS is known) or an additional analytical test. It is important to note that the term "total suspended solids" is a misnomer. All solids in a wastewater sample during the TSS test are either retained as TSS on a standard glass-fiber filter or pass through the filter and are identified as TDS. Thus, all solids larger than the nominal gap openings in the filter are identified as suspended solids whether they were suspended in the wastewater sample or not. TSS actually is a combination of floatable, settleable, and suspended solids retained by the filter.

The nominal gap openings of the standard glass-fiber filter used in the TSS test is 1.5 micron (μ m), which places it at the approximate delineation point between particle filtration (> 1.0 μ m) and microfiltration (0.1–1.0 μ m). In theory, all particulates larger than 1.5 μ m will be retained on the TSS glass-fiber filter.

Basic TSS Test Procedures



- A specific type and size of glass-fiber filter without organic binder (e.g., Whatman grade 934H, Gelman type A/E, Millipore type AP40) is placed in a weigh pan, dried in a 103–105 °C oven, and then allowed to cool in a desiccator. The initial weight of the filter is then determined on an analytical balance and recorded.
- 2. The glass-fiber filter is removed from the weigh pan and fitted into a clean filter funnel or filter crucible, and the funnel or crucible is attached to a stopper in the top of a sidearm flask connected to a vacuum system.
- 3. The vacuum system is turned on, creating suction, and the glass-fiber filter is rinsed with deionized (DI) water to ensure proper seating.
- 4. A measured volume of well-mixed wastewater (typically 25, 50, or 100 ml) is poured into the funnel or crucible and allowed to drain under suction until all free water moves through the filter. (Note: In theory, the larger the volume of wastewater added to the funnel/crucible, the more accurate the TSS result. However, care must be taken to not "blind" or block the gap openings in the filter to the point where wastewater remains in the funnel/crucible even under suction.)
- 5. The sides of the funnel are washed three times with 10 ml of DI water to ensure no solids remain outside of the filter. When no free water remains on the glass-fiber filter, the suction is stopped, and the filter is carefully removed from the filter or crucible apparatus and placed back in the original weigh pan.
- 6. The weigh pan and filter are again dried in a 103–105 °C oven and then allowed to cool in a desiccator. The final weight of the filter is determined on an analytical balance and recorded.
- 7. The difference between the initial and final pan/filter weights are used to calculate the concentration (mg/L) of TSS based on the volume of wastewater filtered in Step 4.

TSS Typical Mathematical Calculation:

You conduct a TSS test. The initial weight of the clean, dried, and cooled weigh pan and glassfiber filter was **1.1685 g**. The final weight of the dried and cooled weigh pan and glass-fiber filter was **1.1897 g**. The volume of wastewater filtered was **50 ml**. What is the concentration (mg/L) of TSS for the wastewater sample?

Information Provided:

- Wastewater volume filtered = 50 ml
- Initial pan/filter weight = 1.1685 g
- Final pan/filter weight = 1.1897 g

Formulas needed:

- Final weight (g) Initial weight (g) = Weight of solids (g)
- Weight of solids (g) x 1000 mg/g = Weight of solids (mg)
- TSS (mg/L) = Weight of solids (mg) x 1000 ml/L Sample volume (ml)

Calculation 1: Final weight (g) - Initial weight (g) = Weight of solids (g)

Final weight 1.1897 g Initial weight ? 1.1685 g Weight of solids 0.0212 g

Calculation 2: Weight of solids (g) x 1000 mg/g = Weight of solids (mg)

Weight of solids 0.0212 g Initial weight × 1000 mg/g _ Weight of solids 21.2 mg

Calculation 3: Weight of solids (mg) x 1000 ml/L Sample volume (ml) = TSS (mg/L)

21.2 mg x 1000 ml/L 50 ml = 21,200 mg/L 50 = 424 mg/L TSS

Total Volatile Suspended Solids (TVSS)

Some poultry processing wastewater treatment facilities take the TSS test a step further to determine what fraction of these suspended solids are organic in nature. To do this, after the TSS concentration has been determined, the glass-fiber (i.e., nonflammable) filter is placed in a 500 °C muffle furnace. This high temperature will *volatize*, or burn off, any organic matter present on the filter. After cooling, the glass-fiber filter is weighed again, with the weight loss representing the **total volatile suspended solids** (TVSS). The remaining solids are inorganic and often are called inert or fixed suspended solids.

Chemistry Note: In laboratory analytical solids tests, "volatile" refers to matter lost upon ignition of solids in a muffle furnace for 1 hr at 550 °C. The matter lost will include almost all (but not 100%) of the plant and animal-based organic material, and any other chemicals used in the poultry processing plant that are combustible (e.g., organic acids).

My lab reports list my results in "ppm"—what does that mean?

Most wastewater test results will be reported in either milligrams per liter (mg/L) or parts per million (ppm). The good news: these two units are equal and thus are interchangeable! However, make sure you always take note of the units reported. Some wastewater parameters (e.g., heavy metals) often are reported in *smaller* units, such as micrograms per liter (μ m/L) or parts per billion (ppb).

Total Dissolved Solids (TDS)

As previously described, TDS are the particulates that are small enough to pass through the

glass-fiber filter utilized in the TSS test (in theory, particulates less than 1.5 μ m in size), and are captured in the flask along with the remaining filtered wastewater. Again, if the concentration (mg/L or ppm) of TS is known, then the concentration of TDS can be calculated by rearranging the equation:

TS = TSS + TDS to solve for TDS: TDS = TS - TSS

However, if the concentration of TS is not known, then a measured volume of well-mixed wastewater from the sidearm capture flask can be subjected to the same procedure described for the TS test later in this publication. The resulting TDS concentration can then be added to the TSS concentration, and a TS value can be calculated.

Using the TSS and TDS Test:

Suspended solids measurement is utilized in a variety of ways in poultry wastewater treatment facilities. On raw postscreened wastewater, it gives the operator a relatively quick method of assessing incoming wastewater strength. As TSS increases, representing additional particulates of pollutants in the influent stream, so does biochemical oxygen demand (BOD) and chemical oxygen demand (COD).

In biological systems, both TSS and TVSS are used to quantify the amount of bacterial microbial solids present (i.e., it is used to determine the level of "bugs" present in the treatment system). Finally, virtually all systems, both pretreatment and full treatment, have TSS concentration and/or loading limits that must be achieved to maintain compliance.

All available water and wastewater laboratory analytical tests are established by the American Public Health Association (APHA) in the publication "Standard Methods for the Examination of Water and Wastewater," commonly referred to as "Standard Methods." In the most recent edition of "Standard Methods," the following two tests are established for TSS and TDS Measurement:

- 2540 C. Total dissolved solids dried at 103–105 °C
- 2540 D. Total suspended solids dried at 103–105 °C

Note: Always ensure the laboratory running your analytical tests is using the EPA-approved method listed in your regulatory discharge permit, and that the method used is listed on any results reports received from the lab.

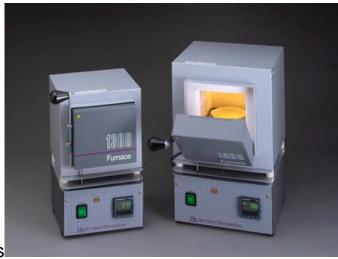
Total Solids (TS), Total Volatile Solids (TVS), and Total Fixed Solids (TFS)

As previously stated, the second form of solids of interest to the poultry processing wastewater operator is the **composition of the particulate matter** in a wastewater stream. In this case, the TS present are divided into TVS and TFS, represented by the equation:

The TS equation here divides all the particulates in a wastewater sample into two categories. In this case, instead of dividing the category based on particulate size, the division is based on whether the particulate matter contains the chemical clement **carbon** (making it by definition **organic**), or the particulate matter **does not** contain carbon (making it by definition **inorganic**)

TVS (total volatile solids) in the equation above are associated with the particulate matter that is defined as **organic** (i.e., carbon-containing) because these solids are combustible and will burn or volatilize under high temperatures. On the other hand, **TFS** (commonly referred to as "ash") contain no carbon and are noncombustible, and thus remain within a fireproof container after all liquid is evaporated and all combustible solids volatilize.

The **total solids** (TS) laboratory analytical test must be completed first, before the total volatile solids (TVS) test can be completed. The **total fixed solids (TFS) or ash** can be obtained through calculation if TS is known (by rearranging the equation **TFS = TS – TVS**), or by weighing the ash contents of the test container after the TVS test is completed.



Basic TS Test Procedures

- 1. A porcelain or metal crucible of appropriate size is thoroughly cleaned, dried in a 103–105 °C oven (or ignited in a 500 °C muffle furnace if TVS will subsequently be run), allowed to cool in a desiccator, and weighed on an analytical balance.
- 2. A measured volume of well-mixed wastewater (typically a minimum of 100 ml) is poured into the crucible. Note: In theory, the larger the volume of wastewater added to the crucible, the more accurate the TS result.
- 3. The crucible is then placed in a drying oven set at about 95 °C until all free liquid is evaporated. Note: The temperature of the drying oven is maintained below 100 °C while free water remains in the crucible. This ensures that the wastewater sample does not begin to boil and spill out of the crucible during the evaporation process.
- 4. Once all free liquid has evaporated, the temperature of the drying oven is increased to 103–105 $^{\circ}\text{C}$ for 1 hr.
- 5. The crucible is then removed from the drying oven, allowed to cool in a desiccator, and weighed on an analytical balance.
- 6. The difference in the initial and final weights is used to calculate the concentration (mg/L) of TSS based on the volume of wastewater evaporated in Step 2.

Basic TVS Test Procedures

- 1. The dried, cooled, and weighed crucible from the TS test is placed in a 500 °C muffle furnace for 1 hr.
- 2. The crucible is then removed from the muffle furnace, allowed to cool in a desiccator, and weighed on an analytical balance
- 3. The difference between the initial weight from Step 1 and final weight in Step 2 is used to calculate the concentration (mg/L) of TVS based on the volume of wastewater initially evaporated in the TS test.

Typical Mathematical Calculations for TS, TVS, and TDS

You conduct a TS test. The initial weight of the clean, dried, and cooled crucible was 56.2234 g. The final weight of the dried and cooled crucible was 56.2975 g. The volume of wastewater evaporated for the TS test was 100 ml. You then place the crucible in a muffle furnace to complete a TVS test. The final weight of the ignited and cooled crucible was 56.2356 g. What is the concentration (mg/L) of TS, TVS, and TFS for the wastewater sample?

Information Provided:

- Wastewater volume evaporated = 100 ml
- Initial crucible weight (TS test) = 56.2234 g
- Final crucible weight (TS test) = 56.2975 g
- Final crucible weight (TVS test) = 56.2356 g

Formulas needed:

- TS = TVS + TFS
- For TS: Final weight (g) Initial weight (g) = Weight of TS (g)
- For TVS: Initial weight (g) Final weight (g) = Weight of TVS (g)
- Weight of solids (g) x 1000 mg/g = Weight of solids (mg)
- TS or TVS (mg/L) = Weight of solids (mg) x 1000 ml/L Sample volume (ml)

Calculation 1: Final weight (g) - Initial weight (g) = Weight of solids (g)

Final weight 56.2975 g Initial weight ? 56.2234 g _ Weight of solids 0.0741 g

Calculation 2: Weight of solids (g) x 1000 mg/g = Weight of solids (mg)

Weight of TS 0.0741 g \times 1000 mg/g TS 74.1 mg

Calculation 3: Weight of TS (mg) x 1000 ml/L Sample volume (ml) = TS (mg/L)

74.1 mg x 1000 ml/L 100 ml = 741 (mg/L)

Calculation 4: Initial weight (g) – Final weight (g) = Weight of TVS (g)

Initial weight 56.2975 g Final weight ? 56.2356 g _ Weight of solids 0.0619 g

Calculation 5: Weight of TVS (g) x 1000 mg/g = Weight of TVS (mg)

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Weight of TVS 0.0619 g \times 1000 mg/g _ TVS 74.1 mg
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Calculation 6: Weight of TVS (mg) x 1000 ml/L Sample volume (ml) = TVS (mg/L)

61.9 mg x 1000 ml/L 100 ml = 619 mg/L TVS

Calculation 7: TS = TVS + TFS ? TFS = TS - TVS

TS 741 mg/L TVS ? 619 mg/L _ TFS 122 mg/L

Final answer: TS = 741 mg/L, TVS = 619 mg/L, TFS = 122 mg/L

All available water and wastewater laboratory analytical tests are established by the American Public Health Association (APHA) in the publication "Standard Methods for the Examination of Water and Wastewater," commonly referred to as "Standard Methods." In the most recent edition of "Standard Methods," the following two tests are established for measuring TS, TVS, and TFS.

- 2540 B. Total Solids Dried at 103–105 °C
- 2540 E. Fixed and Volatile Solids Ignited at 500 °C

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