**Determination of total solids (VS) and volatile solids (VS)**

*Adapted from Standard Methods for the Examination of Water and Wastewater, 2540 Solids*

Total solids (TS) are all solids in a sample; everything except water or other solvents with low boiling points of less than 150°C. Most anaerobic digesters have an optimal volatile solids concentration for their slurry or feedstock. Dry AD requires at least 25% TS, plug-flow AD likes 10-15% TS, complete mix AD from 6-11% TS and up-flow and fixed film AD require even lower TS content. TS content is assayed by drying samples in a low-temperature oven until the sample mass stops dropping and only solids remain.

Volatile solids (VS) are solids that can be combusted and represent a fraction, hopefully a large fraction of the TS content of a feedstock material or slurry. VS are organic or carbon-based and represent that fraction of TS that can be converted into biogas. The higher the VS content the higher the biogas production. The best AD feedstock is at least 90% VS. VS content is assayed by combusting dried TS samples, first over a Bunsen burner and then in a high-temperature muffle furnace. Most, but not all, VS can be converted to biogas by microbes. But a small fraction of VS is refractory: difficult to digest and capable of producing biogas only after prolonged anaerobic digestion.

Ash is the material that remains after combustion of VS. Ash represents that fraction of TS that cannot be combusted and will not produce biogas. Ash is often not organic and can include sand, soil, minerals and other non-biodegradables.

**Equipment:**

* Chemical fume hood;
* Balance;
* Evaporating dishes;
* Ring stand & wire gauze;
* Bunsen burner & sparker;
* Oven and muffle furnace in a fume hood;
* Desiccator;
* Scoopulas; and
* Zip-lock bags.

**Sample collection:**

* Collect about one quart of good, representative samples from prep pit, hydrolyzer, digester, effluent tanks, and solids bay in plastic bottles with well-sealed caps.
* Either begin testing immediately or refrigerate the samples for no more than 24 hours.
* If using previously frozen samples thaw before testing.

**Protocol:**

1. Label evaporating dishes with graphite pencil on an unglazed surface and dry them in a 150°C oven for about one hour to remove any moisture. It is wise to place the oven in a fume hood for olfactory containment. If you believe the dishes are contaminated or need a deeper cleaning, dry them in the muffle furnace for one hour at 550°C.
2. Meanwhile, invert sample bottles to suspend any settled solids and collect 100 or 200 mL of samples using beakers that have been calibrated with graduated cylinders. The volume used will depend on the size of available evaporating dishes.
	* To ‘calibrate’ a beaker, use a graduated cylinder to add 100 and / or 200 mL of water into the beaker and mark the volume carefully with a sharpie. Beakers work better than cylinders for measuring ‘chunky’ slurries.
3. Remove the dried evaporating dishes from oven, cool until touchable, weigh and record their empty masses exactly.
4. Transfer the samples to the evaporating dishes, weigh the full wet dish and record the mass exactly.
5. Place the full dishes into the oven for 24 hours or more
6. Remove samples from the oven and place in a desiccator until just warm to touch. Weigh the evaporating dishes with dried samples and record masses exactly.
7. Place the samples back in the oven for 1 to 2 hours and weigh again. If the sample does not then drying is complete. If additional drying decreases sample mass, then continue to dry until sample mass is constant. Record the final dry mass exactly.
8. Once drying is complete, heat a muffle furnace to 550°C. If at all possible, place the furnace in a fume hood to vent the odors of combustion.
9. Set up a Bunsen burner in a fume hood, below a ringstand and wire gauze.
10. Place the dried samples over the flame and heat them until organic matter stops producing smoke.
11. Place the evaporated and ‘burned’ samples into the muffle furnace and bake for one hour at 550°C.
12. Remove samples from the muffle furnace to a desiccator or warm oven to cool. Be careful not to disturb and dislodge the ash.
13. When cooled enough to handle with bare hands, weigh the evaporating dishes containing residual sample ash and record the masses.
14. Scrape ash into labeled zip-lock bags to store for possible analysis.

**Calculations:**

***Total solids:***

$$\frac{mg total solids}{L}=\frac{\left(dish+dry mg\right)-dish mg}{sample volume mL}\*1000$$

***Volatile solids:***

$$\frac{mg volatile solids}{L}=\frac{\left(dish+dry mg\right)-(dish+ash mg)}{sample volume mL}\*1000$$

**Note:** TS and VS may also be calculated as a **percentage** of the initial sample mass.

