**VOLATILE ACIDS**

**1. OBJECTIVE AND IMPORTANCE OF EXPERIMENT**

The volatile-acids determination is widely used in the control of anaerobic waste treatment processes. In the biochemical decomposition of organic matter that occurs, facultative and anaerobic bacteria of wide variety hydrolyze and convert the complex materials to low-molecular-weight compounds. Among the low-molecular-weight compounds formed, the short-chain of fatty acids, such as acetic, propionic, butyric acid. These low-molecular-weight fatty acids are termed *volatile acids* because they can be distilled at atmospheric pressure.

An accumulation of volatile acids can have a disastrous effect upon anaerobic treatment if the buffering capacity (alkalinity) of the system is exceeded and the pH falls to unfavorable levels. At pH values below 6.5, methane bacteria seriously inhibited.

Volatile acids determinations are important in detecting the presence of unbalance conditions in anaerobic treatment units. The onset of unfavorable conditions can be detected almost immediately, and usually several days in advance of other methods, such as through pH measurements.

**1.1 Selection of Method**

Two methods of determining volatile acids are currently described in “Standard Methods”. One uses column-partition chromatography, and the other involves distillation. Several laboratories today also use gas chromatography or ion chromatography for the measurement of the concentration of individual volatile acids, although these procedures are not yet considered as standard.

**1.1.2 Principle of Distillation Method**

The direct distillation method is commonly used for the routine determination of volatile acids.

Anaerobic treatment reactors normally have a pH maintained in the range of 6.5 to 7.5, and under such conditions organic acids exist largely in the ionic form and cannot be distilled. By the addition of strong nonvolatile acid, such as sulfuric, the organic acids are converted to the un-ionized form which can be distilled.

Hydrogen sulfide (H2S) and CO2 are liberated during distillation and will be titrated to give a positive error. Eliminate this error by discarding the first 15 mL of distillate and account for this in the recovery factor.

Calculations and reporting are on the basis of acetic acid.

**2. EXPERIMENTAL PROCEDURE**

**2.1 Materials and Equipment:**

* Centrifuge
* Distillation flask (500 mL capacity)
* Condenser (about 76 cm long)
* pH meter or recording titrator
* Distillation assembly
* Measuring cylinder
* Erlenmeyer flask
* Graduated cylinder
* Glass beads
* Sulfuric acid, H2SO4 (1 + 1)
* Standard sodium hydroxide titrant, 0.1 N
* Phenolphthalein indicator solution
* Acetic acid stock solution, 2000 mg/L (Dilute 1.9 mL concentrate CH3COOH to 1000 mL with deionized water)

**2.2 Steps of the Experiment**

* Determine recovery factory (f):
	+ To determine the recovery factor, *f*, for a given apparatus, dilute an appropriate volume of acetic acid stock solution to 250 mL in a volumetric flask to approximate the expected sample concentration and distill as for a sample. Calculate the recovery factor:

*f =* a / b

 a = volatile acid concentration recovered in distillate, mg/L

 b = volatile acid concentration in standard solution used, mg/L

* Centrifuge 200 mL sample for 5 min.
* Place 100 mL supernatant liquor, or smaller portion diluted to 100 mL, in a 500 mL distillation flask.
* Add 100 mL distilled water, 4-5 glass beads to prevent bumping and 5 mL 1 + 1 H2SO4.
* Mix so that acid does not remain on bottom of flask.
* Connect flask to a condenser and adapter tube and distill at the rate of about 5 mL/min.
* Discard first 15 mL.
* Collect exactly 150 mL distillate in a 250 mL graduated erlenmeyer flask.
* Titrate with 0.1 N NaOH by using phenolphthalein indicator until the color turns pink.

**2.3 Calculations**

mg volatile acids as acetic acid / L = $\frac{mL NaOH x N x 60000}{ml sample x f}$

N = normality of NaOH

*f* = recovery factor