

Volatile Acidity (VA) Estimation Using Steam Distillation

Volatile acids – such as acetic acid – can form in wines as a result of stressful fermentation and the unchecked proliferation of spoilage bacteria. Hence, estimates of the amount of VA accumulation provide a useful measure of the wine's overall health. Healthy, newly fermented dry red wines typically exhibit VA levels in the 0.2-0.4 g/L range. By bubbling steam through a diluted wine sample volatile acids are released from the wine and collected in the resulting distillate. Since nonvolatile acids (such as tartaric, malic and lactic) are left behind in the wine sample, the quantity of volatile acids in the original sample can be quantitatively estimated by titration of the distillate with standardized NaOH solution. VA is typically expressed in terms of grams/Liter of acetic acid.

Procedure

Setup glassware and apparatus as shown in Figure V1.

- a. Fill 500 mL boiling flask with distilled water (approx. 350 mL).
- b. Add 2-3 boiling stones to the boiling flask to prevent "bumping".
- c. Turn-on the heating mantle to the MAX temperature setting.

While waiting for the water to boil:

- d. Add approx. 75 mL of distilled water to the steam distillation flask
- e. Volumetrically transfer 20 mL of wine sample to the steam distillation flask

When boiling flask reaches 85 °C:

- f. Connect stainless steel transfer tube between boiling and steam distillation flasks. ^{Note 1}
 - g. Position Erlenmeyer flask under drip adapter to collect distillate.
 - h. Start flow of cooling water to Liebig condenser.
 - i. Light Bunsen burner, adjust to lowest flame and place under steam distillation flask. ^{Note 2}
- j. Allow steam to bubble through the contents of the steam distillation flask until 200 mL of clear distillate is collected in the Erlenmeyer flask (approximately 30-40 minutes).
 - k. Add 3 drops of phenolphthalein indicator to the distillate.
 - l. Titrate distillate using standardized 0.1 N NaOH solution.
 - m. Estimate VA as follows:

$$\text{VA (g/L)} = (\text{mL of NaOH titrant required})(0.1 \text{ N NaOH})(0.060)(1000) / (\text{mL of wine sample})$$

Note 1

The steam transfer tube used here consists of a length of stainless steel tubing which passes through drilled No. 4 rubber stoppers at each end. The stainless steel tubing must extend to the bottom of the steam distillation flask to allow the steam to bubble through the diluted wine sample. Care must be taken to insure that the rubber stoppers seal tightly within the glassware exit ports/joints.

Note 2

Heat from the Bunsen burner is needed to prevent the steam from condensing and rapidly filling the distillation flask. If water does begin to build-up in the flask, increase the heat. In the opposite case, if volume of the distillation flask drops by 50% then lower/remove heat until the volume increases to the original level.

Procedural Notes

Carbon dioxide in new wine as well as winemaking additions of sulfur dioxide and potassium sorbate can be volatilized by steam distillation and can contribute to erroneously elevated estimates of wine volatile acidity. New wines should be thoroughly degassed via vacuum aspiration. SO₂ additions can be neutralized by adding 3 drops of 3% (“drugstore”) hydrogen peroxide to the wine sample being tested. Validation studies at the *MoundTop MicroVinification* labs have demonstrated that this technique recovers about 80% of the acetic acid in samples of known concentrations. Hence, multiplying the empirical results by 1.25 provides a good final estimate.

Equipment List

Note: All connecting glassware uses 24/40 standard tapered joints.

500 mL 2-neck boiling flask (24/40)	Laboratory stand and miscellaneous clamps
Stainless steel steam transfer tube ^{Note 1}	Hoses for condenser cooling water
250 mL 2-neck steam distillation flask	25 mL buret, buret clamp and stand
Anti-foaming splash head	0.1 N NaOH titration solution
75-degree distillation elbow	20 mL volumetric pipette
300 mm Liebig condenser (24/40)	Electromantle 500-ml heating mantle
Distillation drip adapter	Bunsen burner (with LP gas source)
500 mL Erlenmeyer flask	

Optional equipment: ^{Note 3}

IMC 2400 precision digital thermometer
IMC 8011-2009 temperature probe
Drilled #4 rubber stopper (for temperature probe)

Note 3

The specified optional equipment is used in the *MoundTop MicroVinification* lab to perform ebulliometric determinations of percent alcohol in wine. Since this equipment is already available it has been incorporated into the equipment setup in order to provide additional and useful information during the distillation procedure.

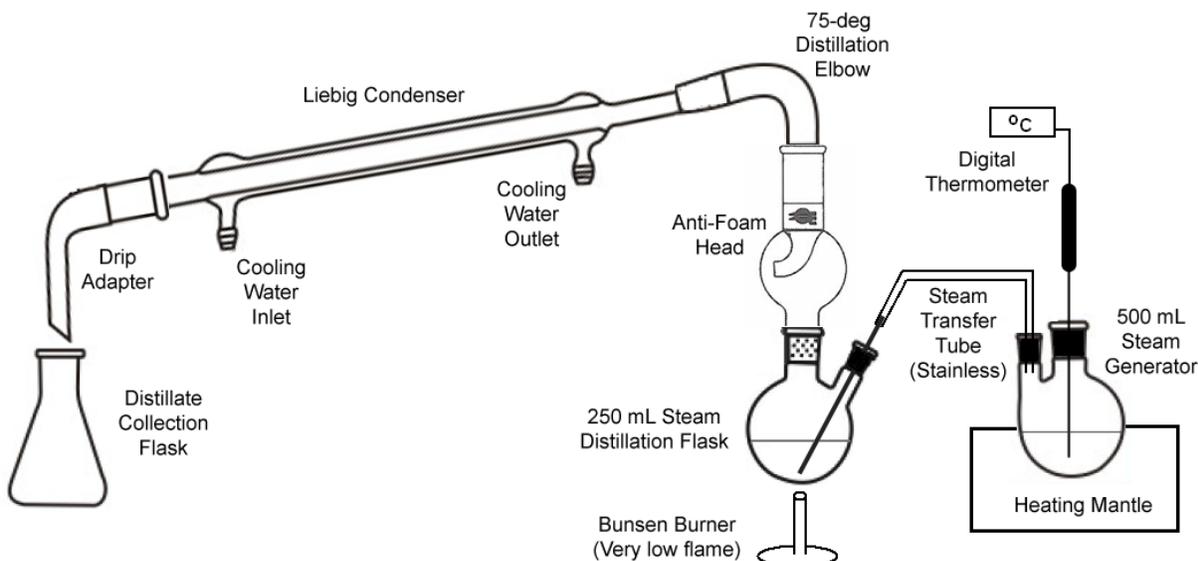


Figure V1. Volatile Acidity Steam Distillation Apparatus