Determination of Titratable Acidity

Technically, titratable acidity is the sum of all of the free (dissociated) and bound (undissociated) protons in a solution. This measurement correlates to the total concentration of acid molecules in wine including all of the tartaric, malic, citric, lactic, acetic and succinic acids as well as which form they are in.[1,2] The TA of a juice or wine can give you a good indication of the sensory perception of acidity. Though there is a little bit of initial set-up, TA can also be measured easily in the winery lab with a well calibrated pH meter, a burette and a stir plate. All of the reagents listed are readily available wine suppliers as well as online retailers.

Degassing

Carbonic acid, produced by CO_2 dissolved in juice and wine, will be included as part of the titratable acidity if it is present, so it is important to degas the juice or wine if there is the possibility of dissolved CO_2 (i.e. fermentation has begun or recently ended)[3]. There are several ways you can degas wine depending on the equipment you have:

- Hook a vacuum pump to a one arm flask. Pour juice or wine into the flask, stopper the flask, and engage the vacuum. Sink/water based vacuum aspiration can also be used.
- Pour a small amount of juice or wine (30 mL or so) into a 250 mL Erlenmeyer flask and cover the top with a rubber stopper with a hole in it. Put your thumb/finger over the hole and shake vigorously. Release your thumb and allow gas to escape. Do this three times. Work over the sink to reduce the mess.
- Heat 30 mL of wine in an Erlenmeyer flask at 60 °C for 5 minutes, then let it cool for 5 minutes. Be careful that the juice/wine does not boil or scorch.

NaOH solution

The accuracy of the TA measurement relies on properly standardized NaOH. NaOH can be made in-house from reagent-grade powder purchased commercially. It is important to be aware that NaOH solutions will absorb CO_2 from the atmosphere to form Na_2CO_3, thus lowering the concentration of NaOH in the solution. For this reason, NaOH should be stored in closed plastic containers in the refrigerator when not in use. This solution should be stable for at least two months. Solutions in titration burettes should be refreshed before use each day. If possible, standardize the NaOH solution once per week. If you made your own from powder, you must standardize before use. The procedure for standardization can be found at the end of this protocol.

Procedure for Determination of Titratable Acidity[3,4]

1. Before you begin, make sure the pH meter has been calibrated and checked against a standard. Also, check that the sample does not contain CO_2.

2. Fill the titration burette with 0.1 N NaOH. Be careful to remove any bubbles in the tip of the burette (flush with NaOH until the bubble flows out).
3. Place a stir bar in a 250 mL glass beaker and add approximately **100mL distilled water**. The exact volume is not crucial.
4. Place the beaker on the stir plate underneath the burette of 0.1N NaOH and position the pH probe so the tip of the probe is under the surface of the water. Be careful to avoid hitting the probe with the stir bar. Make sure there is a clear path from the burette to the liquid in the beaker so all NaOH dispensed will be added to the solution.
5. Adjust the pH of the solution to 8.2 Distilled water has no buffer capacity, so it may be difficult to get to 8.2 exactly. One approach is to add a small amount of juice (one squirt from a plastic pipette) then titrate back to 8.2 with NaOH. Alternatively, some companies produce pH buffer at pH of 8.2, which can be used. After you have reached 8.2, refill the burette with 0.1N NaOH and **record the volume**.
6. Measure **10mL of juice/wine** to the beaker using a volumetric or graduated pipette. Make sure you have recorded the burette volume before you begin titration.
7. Titrate quickly with 0.1N NaOH until the pH meter reads close to 6.0, then slow the titration to drop-wise addition until you reach pH = 8.2. Allow the pH to stabilize before recording the final volume. Try to complete your titration within +/- 0.1 pH point from 8.2.
8. Record the final volume and determine the change in volume. **Multiply the change in volume by 0.75** to determine titratable acidity in g/L (tartaric equivalents). If you have standardized your NaOH solution, you can use the formula:

   TA (g/L tartaric) = 75 x molarity of NaOH x (volume of NaOH in ml/volume of sample in ml)

9. The same buffering solution can be used 3-4 times before being changed. If using multiple times, make sure to return to 8.2 (or near) to start the next trial.

Reagent Standardization

Ideally, reagents should be standardized once per week and every time they are made fresh or a new bottle is opened. The basic calculation is as follows:

N(reagent) = N (standard)*Vol (Standard)/Vol (reagent)

<u>To standardize 0.1 N NaOH for TA determination</u>:

Procedure: Pipette 10mL of 0.1 N H_2SO_4* into a 250 mL Erlenmeyer flask. Add a few drops of 1% phenolphthalein. Titrate to a light pink endpoint with 0.1N NaOH.

Calculation: (0.1N)(10mL)/(mL of NaOH used) = N of NaOH

Parameters: 0.095-0.105N

*Reagent grade H_2SO_4 is available from Fisher Scientific and is stable for several years when kept in a sealed container in the refrigerator.

References

(1) Boulton, R.; Singleton, V. L.; Bisson, L. F.; Kunkee, R. E. *Principles and Practices in Winemaking*; Chapman and Hall, Inc: New York, 1996.

(2) Jackson, R. S. *Wine Science: Principles and Applications*, 4 edition.; Academic Press: Amsterdam, 2014.

(3) Iland, P.; Bruer, N.; Edwards, G.; Weeks, S.; Wilkes, E. *Chemical Analysis of Grapes and Wine*; Patrick Iland Wine Promotions PTY LTD: Campbelltown, Australia, 2004.

(4) Zoecklein, B.; Fugelsang, K. C.; Gump, B. H.; Nury, F. S. *Wine Analysis and Production*; Springer: New York, 1995.