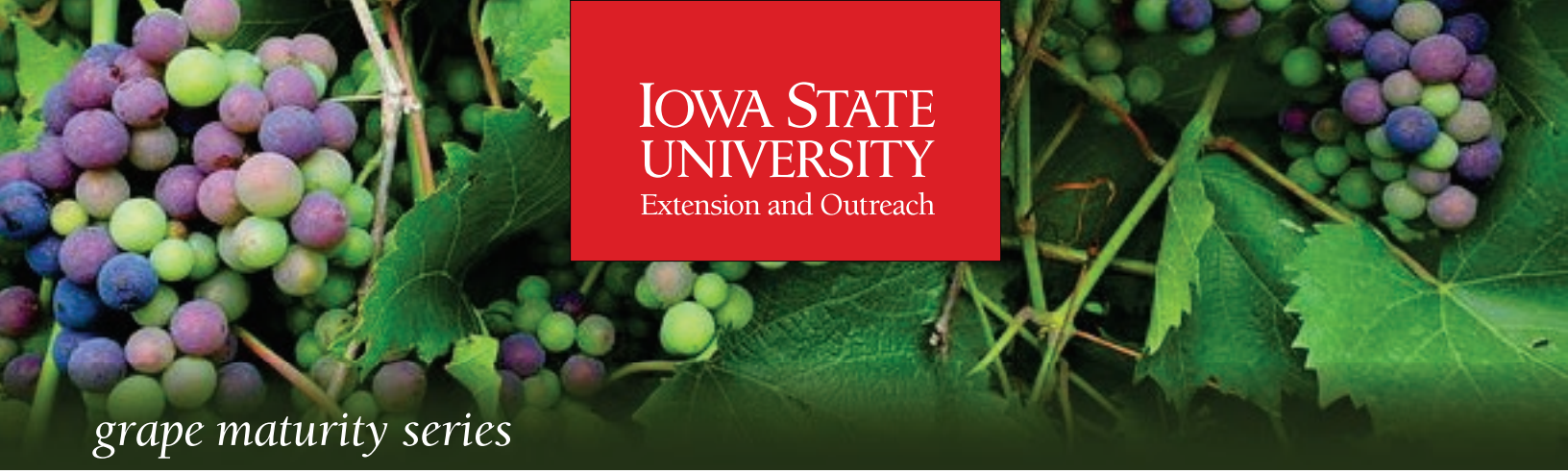


Estimating Grape Maturity by Titratable Acidity

What is titratable acidity

Titratable acidity (TA) is a measurement that approximates total acidity (fixed plus volatile acidity) in must and wine, and is indicative of the sensory perception of tartness. TA measures both the dissociated (free hydrogen ions) and un-dissociated acids neutralized by a base (usually sodium hydroxide). Additional information about the strength and concentration of acids in grapes and wine is provided by the measure of the free hydrogen ion concentration, known as pH. The pH also affects proteins, oxidation and color, and determines the suitability for bacterial growth, the solubility of potassium bitartrate and calcium tartrate, and the effectiveness of sulfur dioxide and potassium sorbate. See Iowa State University Extension and Outreach publication FS 49D: Estimating Grape Maturity with the Potential Power of Hydrogen (pH).

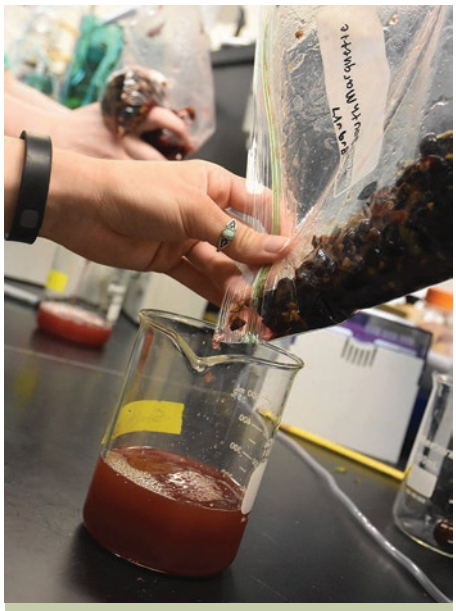
What happens during grape ripening

The major organic acids in must are tartaric, malic and, to a lesser extent, citric. Tartaric acid is characteristic to grapes, while malic acid can be found in many other fruits such as apples, cherries and peaches. Of these acids, tartaric and malic account for more than 90% of the total acid constituents of the juice. In mature *Vitis vinifera* grapes, tartaric is the predominant acid, ranging from 5–10 g/L. In many of the mature cold climate hybrid wine grapes, malic acid is the most abundant, ranging from 5–15 g/L.

These organic acids in wine are primarily derived from grapes. However, other acids are formed during the fermentation. The acids primarily produced during and after the alcoholic fermentation are acetic, lactic, and succinic.

A solid understanding of the organic acid composition of the must is very important for the winemaker to:

- Determine the harvest time and wine style.
- Decide on any must treatments prior to fermentation.
- Monitor the stability of a wine, e.g. if malolactic fermentation is intended, or not.
- Comply to minimum and maximum acid level regulations set by the TTB.



Preparing juice samples for analysis of titratable acidity.

How to measure titratable acidity

The TA content of the must is determined by titrating a sample with an alkaline solution (base) such as sodium hydroxide to a color endpoint using phenolphthalein as the color indicator or with the use of a pH meter to an endpoint of 8.2. Note that the color endpoint procedure can be difficult to execute with red must or wine due to the color of the sample. In most countries, TA is either expressed as grams of tartaric acid per 100 milliliters (g/100mL = %) or as g/L.

General notes for titratable acidity procedures

- Personal protective equipment should include safety glasses, nitrile gloves, closed toed shoes, and a lab coat.
- When using a pH meter, calibrate prior to any measurement. Be sure to follow the operating procedure recommended by the manufacturer.
- Samples containing carbon dioxide should be degassed prior to analysis. Degas by heating a small (25 mL) sample to incipient boil for 30 seconds and then let it cool.
- Always use boiled and cooled distilled water.
- The normality of sodium hydroxide should be periodically checked by titrating against standardized hydrochloric acid (see procedure below).

Standardizing sodium hydroxide solution for titrations

To properly calculate TA, sodium hydroxide solution must be standardized to know the normality. Standardized solutions can be purchased from suppliers and used within the 'use by' date. However, the normality should be checked periodically in the lab because the concentration will change (lose strength) over time. The procedure for determining normality is as follows:

1. Pipet 10 mL of 0.1 N hydrochloric acid into a 250 mL Erlenmeyer flask.
2. Add about 50 mL of water and 3 drops of Methyl red pH indicator.
3. Place sodium hydroxide solution in a 25 mL burette.
4. Titrate the hydrochloric acid with sodium hydroxide until the endpoint is reached, indicated by the development of a lemon yellow color.
5. Record the volume of sodium hydroxide used in the titration.

$$\text{Normality of sodium hydroxide} = \frac{(V)(N)}{(v)}$$

V = volume of hydrochloric acid, 10 mL
N = normality of hydrochloric acid, 0.1 N
v = volume of sodium hydroxide used

Note: The newly calculated normality of sodium hydroxide should be used in titratable acidity calculations.

Titratable acidity to a color endpoint with phenolphthalein

Materials

- 250 mL beaker
- 500 mL wide mouth Erlenmeyer flask
- 2550 mL burette and burette stand
- 1 mL pipet or disposable transfer pipet
- 5 mL or 10 mL pipet
- Stir plate and magnetic stirrer (optional)
- Pipet bulb or pump
- Distilled water
- 0.1 Normal (N) sodium hydroxide, standardized
- Phenolphthalein indicator

Procedure

1. Place 200 mL of boiled and cooled distilled water into a 500 mL Erlenmeyer flask.
2. Add 1 mL of phenolphthalein indicator. If using a disposable transfer pipet, approximately 20 drops is equal to 1 mL.
3. Fill a burette with standardized 0.1 N sodium hydroxide and secure it in the stand. Place the flask of water/indicator solution under the beaker. Titrant and sample need mixing during titration, a stir plate with stirrer is optional or use manual swirling of the flask.
4. Titrate the water with 0.1 N sodium hydroxide from the burette to a definite pink endpoint. The endpoint is defined when the water turns a pale/rose pink that holds for at least 15–30 seconds.
5. Using a pipet, add 5 mL of degassed, room temperature must or wine sample to the flask.
6. Write down the initial volume of sodium hydroxide in the burette.
7. Titrate the sample by adding 0.1 N sodium hydroxide from the burette to the beaker containing the sample. The titration is complete when the same distinct endpoint is reached (as in step 3).
8. Write down final volume of sodium hydroxide from the burette. Subtract the initial from the final volume of sodium hydroxide to determine the total volume used in the titration.

$$\text{T A as tartaric acid } \left(\frac{\text{g}}{\text{L}}\right) = \frac{(\text{V})(\text{N})(75)}{(\text{v})}$$

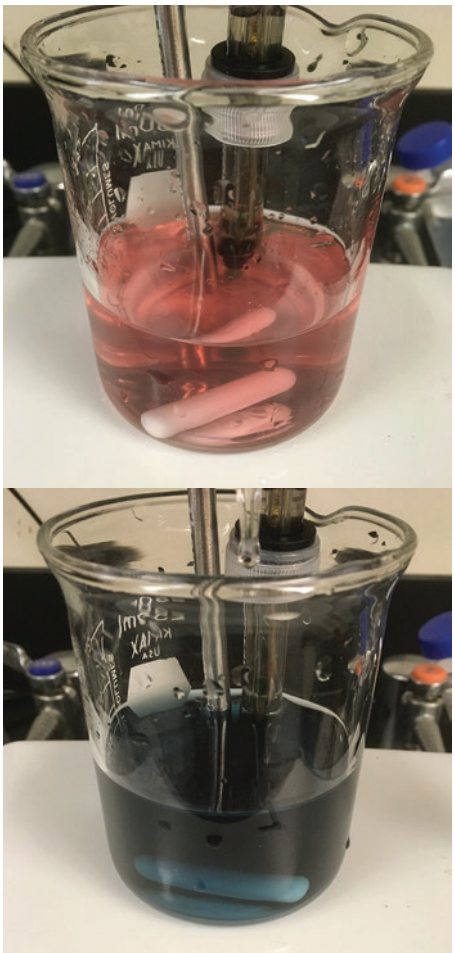
V = volume (mL) of sodium hydroxide solution used for titration
N = normality of sodium hydroxide solution
v = must/wine sample volume (mL)
75 = milliequivalent factor for tartaric acid

Note: Given the normality (N) of sodium hydroxide is 0.1 and the sample volume (v) is 5 mL, the equation can be simplified to: TA as tartaric acid (g/L) = (V) * 1.5

Titratable acidity using a pH meter

Materials

- Two, 250 mL beakers
- 25 mL burette and burette stand
- 5 mL pipet or 10 mL pipet
- 1 mL pipet
- Pipet bulb or pump
- Stir plate and magnetic stirrer
- pH meter with calibration buffers 4 and 7
- Distilled water
- 0.1 Normal (N) sodium hydroxide, standardized



Titration of titratable acidity of Marquette red wine using a pH meter (before titration, on the top; at the end of titration at pH 8.2, on the bottom).

Procedure

1. Calibrate the pH meter using manufacturer's instructions, with buffer solutions, first with pH 7 and then pH 4.
2. Place about 100 mL of boiled and cooled distilled water into a 250 mL beaker.
3. Fill a burette with standardized 0.1 N sodium hydroxide and secure it in the stand. Place the beaker on a stir plate with magnetic stirrer positioned under the burette. Place the pH meter in the beaker.
4. Add sodium hydroxide from the burette into the beaker to adjust the pH of the water mixture to pH 8.2, ensuring the reading holds for 15–30 seconds. Record the volume of sodium hydroxide in the beaker, as the initial volume.
5. Using a pipet, add 5 mL of degassed, room temperature must or wine sample to the beaker.
6. Titrate the sample with sodium hydroxide until an endpoint of pH 8.2, ensuring the reading holds for 15–30 seconds. Note: Titrant can be added quickly until the pH reaches 6.5–7 pH changes will be more rapid as endpoint is approached.
7. Record the volume of 0.1 N sodium hydroxide used in the titration. Subtract the initial from the final volume of sodium hydroxide to determine the total used in the titration. (Subsequent titrations can be completed in the same beaker by repeating steps 5-7.)

$$\text{T A as tartaric acid } \left(\frac{g}{L}\right) = \frac{(V)(N)(75)}{(v)}$$

V = volume (mL) of sodium hydroxide solution used for titration
N = normality of sodium hydroxide solution
v = must/wine sample volume (mL)
75 = milliequivalent factor for tartaric acid

AUTHORS

Prepared by Aude Watrelot, assistant professor of enology in the department of food science and human nutrition at Iowa State University; Jennie Savits, enology field specialist with the Midwest Grape and Wine Industry Institute at Iowa State University; Maureen Moroney, research associate with the Midwest Grape and Wine Industry Institute at Iowa State University.

REFERENCES

Amerine, M. A. and M. A. Joslyn. 1950. Table Wines: The Technology of their Production. University of California Press.

Amerine M. A. and C. S. Ough. 1980. Methods for Analysis of Musts and Wines. New York: John Wiley and Sons.

Dharmadhikari M.R. and K.L. Wilker. 2001. Wine and Must Analysis in Microvinification. Mountain Grove: Southwest Missouri State University.

Gallander, James et. al. 1987. Manual for Wine Analysis and Laboratory Techniques. Wooster: Ohio Agricultural Research and Development Center, The Ohio State University.

Iland, Patrick, Nick Bruer, Greg Edwards, Sue Weeks, and Eric Wilkes. 2004. Chemical Analysis of Grapes and Wine: Techniques and Concepts. Adelaide, AU: Patrick Iland Wine Promotions Pty Ltd.

Thermo Scientific. 2014. Titratable Acidity of Red Wine by Manual Titration (Potentiometric). Application Note 010, AN-TITRACID-E 1114 RevA. https://assets.fishersci.com/TFS-Assets/LSG/Application-Notes/AN-TITRACID-E-1114-RevA-WEB.pdf