

Titrateable Acidity

Titrateable Acidity (abbreviated as **TA**) is an approximation of the *Total Acidity* of a solution, and has long been used in the production of wine. It is usually expressed in units of grams per liter (g/L), although other formats are also used. Titrateable Acidity is often mistakenly confused with Total Acidity, but they are not the same thing. While Total Acidity is a more accurate measurement of the total acid content of a solution, Titrateable Acidity is used because it is easier to measure. Although titrateable acidity does not measure all acids, TA is generally considered a better way to measure perceivable acidity in sour beer and cider than pH.

TA vs pH

pH

In chemistry, pH is the negative log of the activity of the free (dissociated) hydrogen ion (H⁺) in an aqueous solution. Solutions with a pH less than 7 are said to be acidic and solutions with a pH greater than 7 are basic or alkaline. Pure water has a pH of 7.

The pH scale is traceable to a set of standard solutions whose pH is established by international agreement.

Primary pH standard values are determined using a concentration cell with transference, by measuring the potential difference between a hydrogen electrode and a standard electrode such as the silver chloride electrode.

Measurement of pH for aqueous solutions can be done with a glass electrode and a pH meter, or using indicators.

pH measurements are important in medicine, biology, chemistry, agriculture, forestry, food science, environmental science, oceanography, civil engineering, chemical engineering, nutrition, water treatment & water purification, and many other applications ^[5].

pH is best tested in sour beers using a [pH Meter](#) and is most useful for biological parameters.

Microbial growth, vitality, and death are evaluated based on pH rather than TA. Therefore pH should be used when testing sanitizer, [Wort Souring](#), starter cultures, etc.

TA

Titration is an attempt to quantify an unknown substance with a known one.

Titrateable acidity asks how much of a given base (in our case sodium hydroxide, NaOH) neutralizes the acid(s) (lactic, phosphoric, etc.) in a volume of liquid, thus estimating both free hydrogen ions and hydrogen ions that are bound to weak acids that can react with the strong base and be neutralized

The units of TA are g/L

Titrateable acidity does not target a specific acid in the liquid you are measuring.

Titrateable acidity can be expressed in terms of different acids:

In wine, TA is generally expressed in terms of tartaric acid (molecular weight of 150.09).

In sour beer, TA is expressed in terms of lactic acid (molecular weight 90.08, which is where the "0.9" number comes from in the equation below).

To express TA in terms of a specific acid, the molecular weight of the specified acid is used in the TA calculation.

TA expressed in units of lactic acid) is as follows:

$$\text{TA(as lactic acid)} = \frac{\text{mL } 0.1\text{M NaOH} \times 0.9}{\text{mL beer} \times \text{specific grav of beer}}$$

<h2 style="text-align: center;">pH vs Titratable Acidity</h2> <p style="text-align: center; font-size: small;">More Information Online WWW.DIFFERENCEBETWEEN.COM</p>		
	pH	Titratable Acidity
DEFINITION	A measurement of the concentration of free protons (H ⁺ ions) in a solution.	A measurement of the sum of free protons and un-dissociated acids in a solution. Gives the total acidity as an approximate value
UNIT OF MEASUREMENT	pH is unit-less.	The unit of measurement for titratable acidity is grams per litre (g/L).
PURPOSE	Measures the total dissociated protons in a solution.	Measures the total dissociated and non-dissociated acids in a solution.

pH versus Titratable Acidity

- pH is a measure of [H⁺] only
- pH in wine depends on both the concentration of acids present and their relative degrees of dissociation
- Titratable acidity measures free [H⁺] plus all undissociated acids that can be neutralised by a base
- pH and TA are not the same thing, nor do they have a linear relationship!

Determining Titratable Acidity with Sodium Hydroxide

1. Using Phenolphthalein Indicator Solution

Follow these steps to determine the Titratable Acidity (often just referred to as TA) in your must or wine.

1. Add a known amount of grape juice to a beaker (usually 10 or 15 milliliters).
2. Add additional water if the juice is rather dark. The amount of water you add is not critical, adding water does not change total amount of acid in your sample. Do not, however, add more water than 5 times the amount of juice.
3. Add about 5 drops of phenolphthalein. Phenolphthalein is an indicator that is clear when it is in a solution that is acidic, but will change to a purplish color when that solution becomes neutral to basic.
4. Add 0.1N NaOH (1/10 Normal Sodium Hydroxide) until the solution starts to turn pinkish and stay pinkish then note the amount of NaOH used for the titration. Make NaOH addition using a pipette graduated in milliliters. A 10 ml pipette works well.
5. Use the following formula to determine the TA of your wine or must. $TA = (\text{Number or milliliters of NaOH} / \text{Number of milliliters of juice}) \times 0.75$ The units for the TA in this calculation are: Number of grams of tartaric acid per 100 milliliters of juice.

2. Using a pH Meter

A pH meter substitutes for the color endpoint. When sodium hydroxide is added to wine, it increases the pH. Standard solution, usually at 0.1 N, is added until the pH meter reads 8.2. Follow these steps to determine the Titratable Acidity (often just referred to as TA) in your must or wine.

1. Calibrate the pH meter using a two point calibration. The most common buffer solutions used for calibration are pH 7 and pH 4 but pH 10 is also available. Our pH meter has two set screws with one marked pH 7 and the other pH 4 or 10. Fresh pH buffer solutions are important to assure accuracy in the calibration of the meter.
2. First, calibrate with pH 7 buffer because this is a weaker solution. If the meter does not read pH 7 with the pH 7 buffer, we turn set screw marked pH 7.0 to attain 7.0.
3. Then calibrate with the pH 4 buffer solution turning the set screw marked pH 4, or whatever method used for your meter.
4. Add a known amount of grape juice or wine into a beaker (usually 10 milliliters). Place the pH meter into the solution. At this point you can take a reading of the pH of the must or wine.
5. Add 0.1N NaOH (1/10 Normal Sodium Hydroxide) to the solution until the pH meter reads 8.2. In our set-up, we have a stand that supports a 10 ml burette with a stopcock on the bottom of the burette. The burette is calibrated in 0.1 increments. When the stopcock is opened, the solution is allowed to flow into the beaker. Closing the stopcock stops the flow of solution and allows a reading from the burette of how much solution has been dispensed. As the solution pH rises to around a pH of 6.0, changes occur faster so be careful as you pass pH 7.0 on your way to pH 8.2.
6. Use the following formula to determine the TA of your wine or must. $TA = (\text{Number or milliliters of NaOH} / \text{Number of milliliters of juice}) \times 0.75$ The units for the TA in this calculation are: Number of grams of tartaric acid per 100 milliliters of juice.

ASH CONTENT

Ash refers to the *inorganic residue remaining after either ignition or complete oxidation of organic matter in a foodstuff*. A basic knowledge of the characteristics of various ashing procedures and types of equipment is essential to ensure reliable results. Two major types of ashing are used: dry ashing, primarily for proximate composition and for some types of specific mineral analyses; wet ashing (oxidation), as a preparation for the analysis of certain minerals. Microwave systems now are available for both dry and wet ashing, to speed the processes. Most dry samples (i.e., whole grain, cereals, dried vegetables) need no preparation, while fresh vegetables need to be dried prior to ashing. High-fat products such as meats may need to be dried and fat extracted before ashing. The ash content of foods can be expressed on either a wet weight (as is) or on a dry weight basis.

Dry ashing refers to the use of a muffle furnace capable of maintaining temperatures of **500–600°C**. Water and volatiles are vaporized, and organic substances are burned in the presence of oxygen in air to CO₂ and oxides of N₂. Most minerals are converted to oxides, sulfates, phosphates, chlorides, and silicates. Elements such as Fe, Se, Pb, and Hg may partially volatilize with this procedure, so other methods must be used if ashing is a preliminary step for specific elemental analysis.

Wet ashing is a procedure for oxidizing organic substances by using acids and oxidizing agents or their combinations. Minerals are solubilized without volatilization. Wet ashing often is preferable to dry ashing as a preparation for specific elemental analysis. Wet ashing often uses a combination of acids and requires a special perchloric acid hood if that acid is used.

Importance of Ash in food

Ash content represents the total mineral content in foods. Determining the ash content may be important for several reasons. It is a part of proximate analysis for nutritional evaluation. Ashing is the first step in preparing a food sample for specific elemental analysis. Because certain foods are high in particular minerals, ash content becomes important. One can usually expect a constant elemental content from the ash of animal products, but that from plant sources is variable.

METHODS

Sample Preparation

It cannot be overemphasized that the small sample used for ash, or other determinations, needs to be very carefully chosen so that it represents the original materials. A **2–10gm** sample generally is used for ash determination. For that purpose, milling, grinding, and the like probably will not alter the ash content much; however, if this ash is a preparatory step for specific mineral analyses, contamination by microelements is of potential concern. Remember, most grinders and mincers are of steel construction. Repeated use of glassware can be a source of contaminants as well. The water source used in dilutions also may contain contaminants of some microelements. Distilled-deionized water always should be used.

Dry Ashing

Dry ashing is incineration at high temperature (**525°C or higher**). Incineration is accomplished with a muffle furnace. Several models of muffle furnaces are available, ranging from large-capacity units requiring either 208 or 240 V supplies to small benchtop units utilizing 110-V outlets.

Crucible selection becomes critical in ashing because the type depends upon the specific use. Quartz crucibles are resistant to acids and halogens, but not alkali, at high temperatures. Vycor^R brand crucibles are stable to 900°C, but Pyrex^R Gooch crucibles are limited to 500°C.

Ashing at a lower temperature of 500–525°C may result in slightly higher ash values because of less decomposition of carbonates and loss of volatile salts. Porcelain crucibles resemble quartz crucibles in their properties, but will crack with rapid temperature changes. Porcelain crucibles are relatively inexpensive and usually the crucible of choice. Steel crucibles are resistant to both acids and alkalies and are inexpensive, but they are composed of chromium and nickel, which are possible sources of contamination. Platinum crucibles are very inert and are probably the best crucibles, but they are currently far too expensive for routine use for large numbers of samples. Quartz fiber crucibles are disposable, unbreakable, and can withstand temperatures up to 1000°C. They are porous, allowing air to circulate around the sample and speed combustion. This reduces ashing times significantly and makes them ideal for solids and viscous liquids. Quartz fiber also cools in seconds,

virtually eliminating the risk of burns. All crucibles should be marked for identification. Marks on crucibles with a felt-tip marking pen will disappear during ashing in a muffle furnace. Laboratory inks scribed with a steel pin are available commercially. Crucibles also may be etched with a diamond point and marked with a 0.5 M solution of FeCl_3 , in 20% HCl. An iron nail dissolved in concentrated HCl forms brown goo that is a satisfactory marker. The crucibles should be fired and cleaned prior to use.

Procedure for Dry Ashing

AOAC International has several dry ashing procedures (e.g., AOAC Methods 900.02 A or B, 920.117, 923.03) for certain individual foodstuffs.

The general procedure includes the following steps:

1. Weigh a 5–10-g sample into a tared crucible. Predry if the sample is very moist.
2. Place crucibles in a cool muffle furnace. Use tongs, gloves, and protective eyewear if the muffle furnace is warm.
3. Ignite 12–18 h (or overnight) at about 550°C.
4. Turn off muffle furnace and wait to open it until the temperature has dropped to at least 250°C, preferably lower. Open door carefully to avoid losing ash that may be fluffy.
5. Using safety tongs, quickly transfer crucibles to a desiccator with a porcelain plate and desiccant. Cover crucibles, close desiccator, and allow crucibles to cool prior to weighing.

Calculation for Dry Ash content

$\% \text{ ash (dry basis) } = (\text{wt after ashing-tare wt of crucible}) / (\text{original sample wt} \times \text{dry matter coefficient}) \times 100$

Wet Drying

Wet ashing is sometimes called wet oxidation or wet digestion. Its primary use is preparation for specific mineral analysis and metallic poisons. Often, analytical testing laboratories use only wet ashing in preparing samples for certain mineral analyses (e.g., Fe, Cu, Zn, P), because losses would occur by volatilization during dry ashing. There are several advantages to using the wet ashing procedure. Minerals will usually stay in solution, and there is little or no loss from volatilization because of the lower temperature. The oxidation time is short and requires a hood, hot plate, and long tongs, plus safety equipment. The disadvantages of wet ashing are that it takes virtually constant operator attention, corrosive reagents are necessary, and only small numbers of samples can be handled at any one time. If the wet digestion utilizes perchloric acid, all work needs to be carried out in an expensive special fume hood called a perchloric acid hood.

Procedure for wet ashing

1. Accurately weigh a dried, ground 1-g sample in a 125-ml Erlenmeyer flask (previously acid washed and dried).
2. Prepare a blank of 3 ml of H₂SO₄ and 5 ml of HNO₃, to be treated like the samples. (Blank is to be run with every set of samples.)
3. Add 3 ml of H₂SO₄ followed by 5 ml of HNO₃ to the sample in the flask.
4. Heat the sample on a hot plate at ca. 200°C (boiling). Brown-yellow fumes will be observed.
5. Once the brown-yellow fumes cease and white fumes from decomposing H₂SO₄ are observed, the sample will become darker. Remove the flask from the hot plate. Do not allow the flask to cool to room temperature.
6. Slowly add 3–5 ml of HNO₃.
7. Put the flask back on the hot plate and allow the HNO₃ to boil off. Proceed to the next step when all the HNO₃ is removed and the color is clear to straw yellow. If the solution is still dark in color, add another 3–5 ml of HNO₃ and boil. Repeat the process until the solution is clear to straw yellow.
8. While on the hot plate, reduce the volume appropriately to allow for ease of final transfer. Allow the sample to cool to room

temperature, then quantitatively transfer the sample to an appropriately sized volumetric flask.

9. Dilute the sample to volume with ultrapure water, and mix well. Dilute further, as appropriate, for the specific type of mineral being analyzed.

Microwave Ashing

Both wet ashing and dry ashing can be done using microwave instrumentation, rather than the conventional dry ashing in a muffle furnace and wet ashing in a flask or beaker on a hot plate. The CEM Corporation (Matthews, NC) has developed a series of instruments for dry and wet ashing, as well as other laboratory systems for microwave-assisted chemistry. While the ashing procedures by conventional means can take many hours, the use of microwave instrumentation can reduce sample preparation time to minutes, allowing laboratories to increase their sample throughput significantly. This advantage has led to widespread use of microwave ashing, especially for wet ashing, both within analytical laboratories and quality control laboratories within food companies.

Other ash measurement

The following are several special ash measurements and their applications:

1. Soluble and insoluble ash (e.g., AOAC Method 900.02) – Applied to fruits.
2. Ash insoluble in acid – A measure of the surface contamination of fruits and vegetables and wheat and rice coatings; contaminants are generally silicates and remain insoluble in acid, except HBr.
3. Alkalinity of ash (e.g., AOAC Method 900.02, 940.26) – Ash of fruits and vegetable is alkaline; ash of meats and some cereals is acid.
4. Sulfated ash (AOAC Method 900.02, 950.77) – Applied to sugars, syrups, and color additives.

Note: The analysis of **ash content in foods** is simply the burning away of organic **content**, leaving inorganic minerals. This helps determine the amount and type

of minerals in **food**; important because the amount of minerals can determine physiochemical properties of **foods**, as well as retard the growth of microorganisms.