Preparing Standard Sodium Hydroxide Solution*

By Dr. Murli Dharmadhikari and Tavis Harris

Note: This article has been written at the request of the industry. It is written for wine lab workers with no background in chemistry.

In a wine laboratory, analyzing wine for TA, VA and $S0_2$ involves the use of a sodium hydroxide (NaOH) reagent. Winemakers usually buy sodium hydroxide solution of a known concentration (usually 0.1 Normal). This reagent is relatively unstable and its concentration changes over time. To ensure the accuracy of analytical results it is important to periodically check the concentration (Normality) of sodium hydroxide. If the concentration has changed then it must be readjusted to the original concentration or the new concentration (Normality) value needs to be used in calculations.

Sometimes a winemaker may wish to make his/her own NaOH solution instead of buying it. Whether making a new solution or checking the normality of an old solution it is important to know the procedure for making a standard (known concentration) solution of NaOH reagent. In the present article the standardization procedure along with the basic concept behind the titration procedure are explained.

Expressing concentration in solution

A solution consists of a solute and the solvent. Solute is the dissolved substance and solvent is the substance in which the solute is dissolved. A solute can be a solid or a liquid. In NaOH solution, sodium hydroxide (solid) is the solute and water (liquid) is the solvent. Note that the solute being a solid is measured in terms of weight (in grams) and the solvent water is measured in terms of volume. This is an example of expressing solution in weight per volume (w/v) basis.

In a solution consisting of two liquids the concentration is expressed in a volumes per volumes basis. For example the concentration of alcohol in wine is expressed as volume per volume. A 12% alcohol wine means it contains 12 ml of alcohol per 100 ml of wine.

Generally, in many solutions, the weight is given in grams and volume is given in milliliters or liters. At this point, it is important to establish the relation between the units of weight and volume. One kilogram (weight) of water at a temperature of maximum density and under normal atmospheric pressure has the volume of one liter. This means that one kilogram (weight) of water equals one liter of volume, and one gram of water by weight equals one milliliter of water by volume. Thus the units of weight (gram) and volume (ml) are similar and interchangeable.

The chemist expresses the concentration of a solution in various ways. The common expressions include Percent, Parts per million (ppm), Molar and Normal. It is important to have a clear understanding of these terms.

Percent

One of the simplest forms of concentration is the percent. This simply means units per 100 units, or parts per 100 parts. The percent concentration can be used in three ways. It can be weight per weight, volume per volume or weight per volume basis.

When winemakers use °Brix hydrometer to measure sugars in grape juice they are essentially measuring grams of sugar per 100 grams of juice. A juice sample of 18 °Brix means 18 grams of sugar per 100 grams of juice or commonly referred as 18%. In describing the alcohol content of a wine, percent alcohol content is expressed in terms of a volume per volume basis. In many cases, including in a laboratory, a solution is made by dissolving a solid in a liquid, usually water. In such a case the concentration is expressed in a weight per volume basis.

Parts per million

When dealing with a very small amount of a substance in solution, the concentration is often expressed in terms of parts per million. A 20 ppm concentration means 20 parts of solute dissolved for every 1,000,000 parts of solution. The unit of measurement can be weight or volume. Generally the ppm concentration is used to indicate milligrams of solute per liter of solution.

Molar solution

A molar solution implies concentration in terms of moles/liter. One molar (I M) solution means one mole of a substance (solute) per liter of solution. A mole means gram molecular weight or molecular weight of a substance in grams. So the molecular weight of a chemical is also its molar weight. To calculate the molecular weight one needs to add the atomic weights of all the atoms in the molecular formula unit. For example the molecule of NaOH consists of one atom each of sodium (Na), oxygen (0), and hydrogen (H). Their respective atomic weights are: Na - 23,0 - 16 and H - 1, so the molecular weight, is 23 + 16 + I = 40. Thus 40 grams of NaOH equals one mole of NaOH, and a 1 molar solution of NaOH will contain 40 grams of NaOH chemical.

Normal solution/Normality

The other form of concentration used relatively frequently is normality, or N. Normality is expressed in terms of equivalents per liter, which means the number of equivalent weights of a solute per liter of a solution. The term normality is often used in acid-base chemistry. The equivalent weight of an acid is defined as the molecular weight divided by the number of reacting hydrogens of one molecule of acid in the reaction.

Understanding equivalents requires knowing something about how a reaction works, so let's start there. Below is a basic equation for an acid and a base.

HCI + NaOH ------> NaCl + H20
or
Acid + base -------> salt + water

In our simple equation above you can see we have the acid and base reacting to form a salt and water, and that they react equally. The acid gives 1 H+ for every -OH given by the base. So for every mole of H+ one needs a mole of
-OH. This reaction is one-to-one reaction on a molar basis. One mole of acid has one reacting unit and one mole of base also has one reacting unit thus both acid and base has, in the above example, equal 1:1 reacting units. As stated above, for acids we define an equivalent weight as the molecular weight divided by the number of H+ donated per molecule. Above, the HCI gave up 1 H+ (proton) to the reaction.

Molecular weight of H_2SO_4 = 98.08 g = 49.04 grams per equivalent
\# of protons given   2 protons

Normality is the molecular weight divided by the grams per equivalent (all this results in the number of equivalents) in a given volume. For an 1 N solution we need 1 equivalent/liter. For hydrochloric acid (HCl) the equivalent weight is 36.46 grams. Therefore, for making an 1 Normal solution, 36.46 g/liter of HC1 is needed. Note that a 1 M solution is also 36.46 g/L. For molecules that can give off or accept only one proton per molecule, the Normality is equal to the Molarity.

Table 1. Molecular and Equivalent weights of some common compounds.

Chemical name	**Formula**	**Molecular weight g/mol**	**Equivalent weight g/equiv**
hydrochloric acid	HCI	36.46	36.46
nitric acid	HNO_3	63.01	63.01
sulfuric acid	H_2SO_4	98.08	49.04
phosphoric acid	H_3PO_4	98.00	32.67
tartaric acid	$C_4H_6O_6$	150.09	75.05
malic acid	$C_4H_6O_5$	134.09	67.05
citric acid	$C_6H_6O_7$	192.12	64.04
lactic acid	$C_3H_6O_3$	90.08	90.08
acetic acid	$C_2H_4O_2$	60.05	60.05
water	H_2O	18.02	18.02
sodium hydroxide	NaOH	40.00	40.00

potassium hydroxide	KOH	56.11	56.11

In the case where a molecule can give off or accept more than one proton, you need to adjust your calculation. For example, sulfuric acid with a formula of H_2SO_4 donates 2 separate protons. Using the molar mass of sulfuric acid, and knowing that one molecule can donate 2 protons we can find the equivalent weight.

With a molar mass of 98.08 grams, a solution containing 98.08 g in 1 liter would have a Molarity of 1 M and a Normality of 2 N. This is because every I mole of sulfuric acid (H_2SO_4) has 2 moles of H+ atoms.

Table 1 lists the molecular weights and equivalent weights of important acids and bases used in a wine laboratory.

Making 1 N solution of NaOH
From the discussion above, it should be clear that to make 1 Normal solution we need to know the, equivalent of NaOH, which is calculated by dividing Molecular weight by 1, that is 40 divided by 1= 40. So the equivalent weight of NaOH is 40. To make 1 N solution, dissolve 40.00 g of sodium hydroxide in water to make volume 1 liter. For a 0.1 N solution (used for wine analysis) 4.00 g of NaOH per liter is needed.

Standardization
Before we begin titrating that wine sample we have one more important step, standardization of NaOH solution. Standardization simply is a way of checking our work, and determining the exact concentration of our NaOH (or other) reagent. Maybe our dilution was inaccurate, or maybe the balance was not calibrated and as a result the normality of our sodium hydroxide solution is not exactly 1 N as we intended. So we need to check it. This is achieved by titrating the NaOH solution with an acid of known strength (Normality). Generally 0.1 N HCI is used to titrate the base. The reagent, 0.1 N HCI solution is purchased from a chemical supplier that is certified in concentration. That means it was standardized to a base of known concentration. "But isn't that going in circles?" you ask. No, because acids are standardized to a powdered base called KHP, or potassium hydrogen phthalate. This can be very accurately weighed out because it is a fine powder, and then is titrated with the acid.

To standardize NaOH, start by pipetting 10.0 ml of 0.1 N hydrochloric acid (HC1) into a flask. Add approximately 50 ml of water (remember, not tap water) and three drops of methyl red indicator. Fill a 25 ml buret with the 0.1 N sodium hydroxide solution and record the initial volume. Titrate the hydrochloric acid to the point at which a lemon yellow color appears and stays constant. Record the final volume.

Subtract the initial volume from the final to yield the volume of NaOH used, and plug that into the equation below.

$$\text{Normality of NaOH} = \frac{\text{Volume of HCI x Normality of HCI}}{\text{Volume of NaOH used}}$$

Titration Techniques
Before conquering volumetric analysis totally, we need to discuss some titration techniques. First of all, handle the buret with care. Avoid damaging the tip and petcock assembly because damage and leaks in these areas can and will alter performance. Also, be sure to always record your final and initial volume readings accurately by reading the bottom of the meniscus of the solution. Don't try to squeeze in that last sample and drain the buret past its lowest mark; take the time to refill it properly. For help in reading a buret, take a white index card and color a black square on it as shown. Hold this behind the buret scale when taking readings to aid in seeing the meniscus. Some burets actually come with a stripe painted on them for this reason.

Next, remember to stir your sample as you titrate. Whether using a stir plate (recommended) or stirring by swirling the flask manually, it is imperative that the solution be mixed. Be sure not to slosh the sample outside of the beaker/flask and don't allow the buret's contents to fall outside of the beaker. Also, lower your buret enough so that splatter from the sample does not exit the flask as you titrate. This is not only bad lab practice but can also be dangerous.

Safety is an important consideration when working with burets, acids and bases. Realize that you are handling corrosive chemicals and delicate glassware, treat it like an irreplaceable wine in the daintiest glass.

That means deliberately and with respect. Wear safety glasses and a labcoat at least, and gloves are also recommended. When filling a buret, take it out of the stand and hold it at an angle with the tip above the sink. That way any spills will drain into the sink and you can stand safely on the floor, not a stool. Leaning over the buret while it is on the benchtop is dangerous.
Be sure to have access to an eyewash station or something that can supply a stream of water to your body and/or eyes for 15 minutes, the OSHA recommended treatment for chemical spills to the eyes and body. Remember you will have sodium hydroxide in the buret at and above eye level so make sure your equipment is attached to a steady base.

Good laboratory practices can help you monitor the quality of your wines more accurately and efficiently. Volumetric analysis by titration is one of the most common techniques the winemaker employs to analyze his product. Improving your skills in this area is important in the quest for excellent wines on a consistent basis.

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