

Wine Analysis – Free SO₂ by Aeration/Oxidation Method

Scott Lacy, Purple Grin Winery

Perhaps the most common wine fault of the amateur winemaker (and too many commercial winemakers) is the improper management of sulfites to control oxidation. The typical manifestation may include odors of vinegar, solvents and browning.

Once fermentation has completed, it is very important to store and treat the wine under appropriate conditions. Malolactic fermentation (MLF), if desired for the variety of wine, should be completed as soon as possible at 70 degrees Fahrenheit prior to adding sulfites that will limit the health of the malolactic bacteria (MLB).

Once MLF has been completed, as confirmed by paper chromatography or other method, the wine should be dosed with potassium metabisulfite (K-meta) to a level appropriate for the pH of the wine. The aging vessel should then be topped up to the stopper or bung to minimize air contact, and then stored at cellaring temperatures (certainly less than 65 deg F). Lower temperatures are better, consistent temperatures are critical.

Now that you've got a stable condition for your wine, you'll want to keep it that way. Over time, the "free" sulfite molecules from the K-meta will attach themselves to bad players in the carboy, tank or barrel. The "total" amount of sulfites remains the same, but the "free" sulfites diminish. As we add additional K-meta, we add to both the "total" and "free" SO₂ tally. Good notes on your additions will help keep track of your total SO₂, which many like to limit for further quality issues.

The following method will allow the winemaker to measure free SO₂ so that correct additions of K-meta can be made. The method is called aeration/oxidation because we aerate the SO₂ out of a wine sample and then oxidize it into a colored solution. We then take that colored solution and analyze for the amount of SO₂ absorbed.

There are many variations of this test, but I wanted to offer one that was easy and inexpensive to obtain, so that more of the wine made will be enjoyed without holding one's nose. The method uses a few common components and a few specialty chemicals available from several sources on the internet. I have listed part numbers and current pricing from suppliers who have treated me right. I have no other affiliation with them.

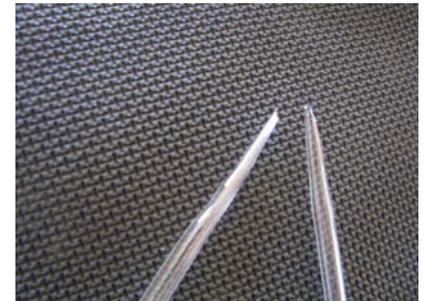
Item Description	Supplier	Part #	Price	Qty	Total
25mm x 200mm Glass Test Tube	Onlinesciencemail.com	TT109	\$1.95	2	\$3.90
#4 Rubber Stopper, 2-hole	Onlinesciencemail.com	DPSSTO3032-#4	\$0.57	1	\$0.57
24" length of 5mm Glass Tubing	Onlinesciencemail.com	DPSTUB107-5	\$1.75	1	\$1.75
Pipette, 1 ml	Onlinesciencemail.com	PIP307	\$3.50	1	\$3.50
Pipette Filler Bulb	Onlinesciencemail.com	PIP315	\$14.50	1	\$14.50
SO ₂ Color Indicator	piwine.com	SO2I-21	\$2.25	1	\$2.25
Phosphoric Acid, 25%	Hardware, Tile/Grout Care Aisle	Read MSD Sheet	\$8.99	1	\$8.99
Hydrogen Peroxide, 3%	Local Drugstore Shelf	Read Label	\$3.99	1	\$3.99
Aquarium Air Pump, 1-1.5 lpm	youraquariumcenter.com	AT2K2	\$7.99	1	\$7.99
Aquarium Tubing, 6 ft	youraquariumcenter.com	AK1	\$2.99	1	\$2.99
Tartaric Acid, granulated	Local Wine Supply Shop	On-hand			
Sodium Hydroxide, 0.2N	Local Wine Supply Shop	On-hand			
Syringe, plastic, graduated, 10 ml	Local Wine Supply Shop	On-hand			
Distilled Water, 1 gal	Grocery Store				
		Total Cost			\$50.43

I am hopeful that some of the above is already a part of your wine making tool collection. Please don't suck on a pipette to fill it. The filler bulb will be handy for years to come.

The one chemical that trips up many is the phosphoric acid. It is sold as a grout haze remover for use after tiling a floor. Since it is not for consumption, the concentration is not typically listed on the label. The actual concentration is not too important. We're just going to add enough to make that free SO₂ jump out of the wine. You can find a particular manufacturer's data with a web search for it's Material Safety Data Sheet (MSDS), if the hardware store doesn't have it available (trust me, if they stock it, they have the MSDS). I bought a quart manufactured by Aqua Mix Inc. Funny enough, they sell this at Amazon.com for \$9.10, but the shipping is \$12. Small quantities are also available at piwine.com.

OK. So you're back from the Depot, and the UPS man has delivered a nice box. Let's get started.

First, you'll need to fabricate a couple of impingers from that glass tubing. You basically want to start with a 12-inch section. You can cut this by scoring with a file (or glass cutting wheel) and snapping like a stick. Hold a flame under the tubing, about four inches from the end, rolling the tubing between your fingers, to evenly heat a one-inch section. As the glass softens, gently pull the two ends apart, creating a thin-necked section. Raise it off the heat. After it cools for a minute or so, break the two pieces apart at the small area. You now have a long and short impinger, sort of miniature glass turkey basters. The points are sharp and fragile, so handle with some care. You can put a little more heat on the tips to soften the edges, and maybe close up the tips a little for nice, small bubble points.



You'll also want a one-inch piece of glass tubing for connecting the inter-stage tubing to the 2-hole stopper.

Now, carefully assemble as shown. Wetting the tubing will help with insertion into the stopper. I set the two test tubes into a 250 ml beaker or clear drinking glass with a paper towel behind them so they won't spill while I'm playing mad chemist. Adjust the first impinger so that it reaches very close to the bottom of the first tube. The second impinger will simply rest on the bottom of the second tube.



Now we'll need to make a few chemical solutions. This will likely take about 15 minutes prior to your actual test. I prefer to make mine fresh each time I test my wine, since some of these solutions are not stable, and won't last long.

1. Put 15 ml of peroxide in a small beaker. Add 30 ml of distilled water. Add four drops of the SO₂ indicator, or until you achieve a light, but visible level of color when mixed.
2. In a separate beaker, add about 20 grains of tartaric to 100 ml of distilled water. While stirring the colored solution, add this diluted tartaric acid one drop at a time, until it turns from a light blue to a dark blue or purple.
3. Pipette exactly 1.0 ml of your 0.2N NaOH into another beaker. Add 19 ml of distilled water. You now have 20 ml of 0.01N NaOH. Plenty for our use here.
4. Rinse your pipette. With good lighting on your setup, add your 0.01N NaOH one drop at a time to your colored indicator beaker. Swirl between drops. When the purple starts changing back to light blue, stop, cleanup a little, and get your wine sample. You've got enough color solution for three or four tests.
5. Put 20 ml of your wine into the first test tube.
6. Put 10 ml of your color solution into the second test tube.
7. Put in your air pump. Insert the short impinger into the color solution. The tip should sit at the bottom. Make sure the other end of the tubing is securely connected to the short tubing in the rubber stopper.

8. Now add 20 ml of the phosphoric acid into the wine sample tube, and quickly and carefully put on the stopper. Don't get too excited and break the test tube. Just a firm seal is needed.
9. The air pump should be bubbling up through your wine/acid, and transferring the free SO₂ to the impinger that is bubbling up through your color indicator. You'll let this go on for fifteen minutes. But if you don't see it turn purple in a few minutes, you may not have any free SO₂ to measure.
10. After that 15 minutes, leave the air pump running. The bubbles make for a fine mixer.
11. Now carefully add your 0.01N NaOH using your 1.0 ml pipette, being careful that each drop gets into the sample, not collecting on the side of the test tube or impinger.
12. Stop exactly when your solution turns back to the light blue color. Now record the amount of 0.01N NaOH you used.



Free SO₂ (ppm) = 16 x ml of 0.01N NaOH used to return the blue color.

So 2.5 ml of NaOH would mean 40 ppm of free SO₂.

Since you have some additional solution, I would suggest verifying your measurement prior to additions to the wine.

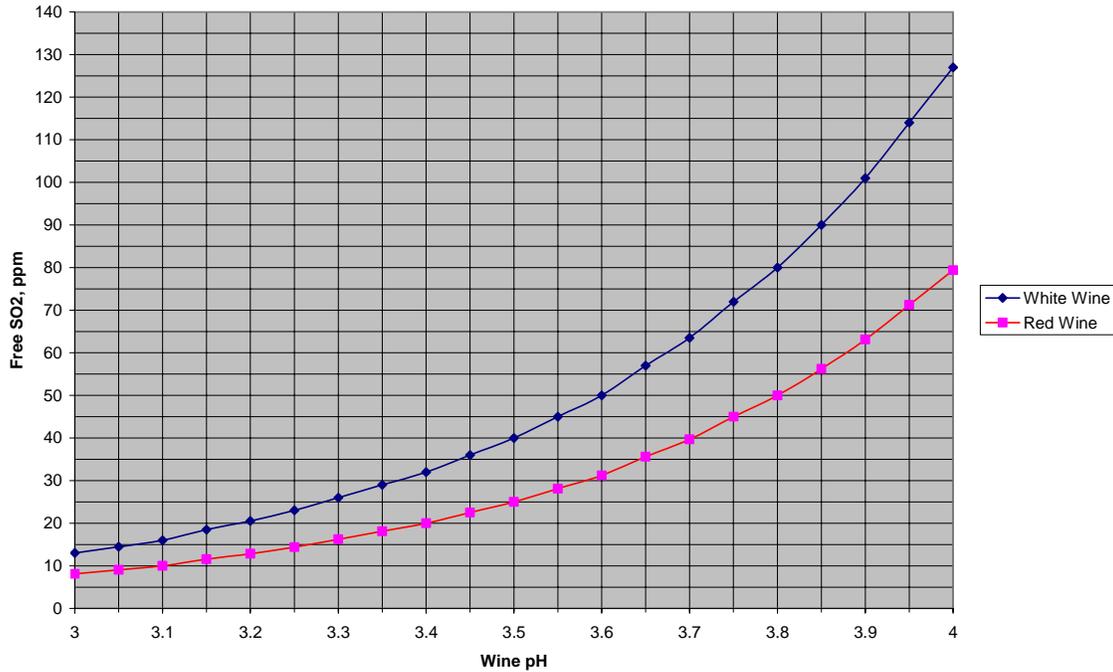
A special thanks to the forum gang at winepress.us, Jay Spence at Concetta Cellars, Jon Iverson for his book [Home Winemaking Step by Step](#), and Vinquiry for their posted method.

Free SO₂ Management Summary
 Scott Lacy, Purple Grin Winery

So let's quickly review sulfite management and additions. It is suggested that wine is best protected at molecular sulfite (SO₂) levels of 0.5 ppm for red wines and 0.8 ppm for white wines. This concentration depends on the pH of your wine. If you don't have a pH meter, a casual guess based on the varietal of wine will have to do. Finding someone to measure your sample for you would be better. A school, private or municipal laboratory may be able to get you a reading with only a minute of effort.

Use the chart below to determine the correct free sulfite level based on your type of wine and pH. Check your sulfite levels often, perhaps monthly if unsure, using an aeration/oxidation setup. A reduction of sulfite levels by ten or twenty percent is not dangerous. But keep good notes so that you don't surprise yourself. Experience will allow you to adjust your testing frequency, based on exposure, vessel type and events such as racking or aeration.

Free SO₂ Required



Also, bear in mind that potassium metabisulfite (a.k.a. "K-meta") provides only 570 mg of SO₂ per gram, or 57% by weight.

Here's an example: We measured a white wine with a pH of 3.65 at 40 ppm free SO₂. From the chart we read a target level of 57 ppm. So we want to add another 17 ppm.

Let's say we have one full carboy of this wine, 5 gallons or (5 x 3.785) = 19.0 liters = 19 kg.

The metric system is a gift here, since 1mg in 1kg is one part in a million, or a ppm.

(17 ppm needed addition) x (19 kg or liters of wine) / (0.57 SO₂ per k-meta) = 567 mg k-meta needed

Obviously, a scale would be nice to measure this small amount. Again, there is a fix for those without...

1 teaspoon of k-meta weighs about 5.9 grams. I am not comfortable measuring out roughly 1/10th of a teaspoon!

So, in this case, I would add one teaspoon of k-meta to 100 ml of distilled water, and then add (0.567/5.9*100ml) = 9.6 ml of this solution to the carboy. Stir completely.