

Vinmetrica SC-300 Pro Kit™ User Manual

Vinmetrica $SC-300^*$ Pro Kit is a simple and robust device that provides high accuracy in determination of sulfite (SO₂), pH and titratable acidity (TA) levels in wine. These are essential parameters to control in the effort to make high quality wines. The Pro kit includes lab accessories for the SC-300 Analyzer.

Table of Contents:

Materials Provided in the Kit	2
Things you will need	2
Why Test for SO ₂ , pH and TA?	3
Theory of Operation	4
Setup	5
Setting up the SC-300 for the first time	5
Assembling the Pro Kit Equipment	6
Burette Maintenance	8
Burette Reading	9
Instrument Operation ¹	10
Procedures1	12
Measuring Free Sulfite (SO ₂) by Titration 1	12
Measuring Total Sulfite (SO ₂) by Titration ¹	15
Calibration of pH1	15
Measuring pH1	16
Measuring Titratable Acidity (TA) by Titration1	17
Finishing up 1	19
Warranties, Liabilities & Hazards	20
Appendix A - Test Mode2	21
Appendix B - Sulfite & TA Adjustments	23
Appendix C1 - Troubleshooting: pH and TA Issues	24
Appendix C2 - Troubleshooting: SO ₂ Issues	26

*US Patent pending

Materials provided in the kit:

- 1. Vinmetrica SC-300 SO₂/pH instrument (Part number SC-300-1)
- SO₂ Electrode (Part number SC-100-3), blue polycarbonate housing
- 3. pH Electrode (Part number SC-200-3), red or blue polycarbonate housing
- 4. Two 5 mL syringes
- 5. Two 3 mL polyethylene transfer pipettes
- 6. One 25 mL serological sampling pipette
- 7. One 5mL serological sampling pipette
- 8. 100 mL polypropylene titration beaker
- SO₂ Reagent set (Part number SC-100-2):
 SO₂ Titrant solution (0.0156N) Acid reagent
- Reactant solution
- 10.pH/TA reagent set (Part number SC-200-8):pH 4.01 Reference solutionpH 7.00 Reference solutionTA Titrant (0.133N NaOH)
- Lab Support stand (the large blue metal base with its long metal rod) (Part number SC-300-3)
- 12. Vinmetrica Electrode Holder (Part number SC-300-8)
- 13. Double Burette Clamp (Part number SC-300-6)
- 14. 10 mL or 25 mL Glass burette with Teflon Stopcock (with ~2 grams of Burette detergent) (Part number SC-300-7)
- 15. Magnetic Stirrer (includes two AA batteries, stir bar and 25 mL cylindrical container) (Part number SC-300-4)

Things you will need:

- 1. Two standard AA batteries (alkaline type).
- 2. Distilled water, which usually can be found at your local grocery store. It's handy to have a wash bottle for rinsing. Rinse bottle available from Vinmetrica (Part number SC-100-17)
- (Optional) 1N Sodium Hydroxide solution (if you want to do <u>total</u> SO₂). Available from Vinmetrica (Part number SC-100-7)



Figure 1. The SC-300 instrument

Why Test for SO₂, pH and TA?

Testing for sulfite (SO_2) is crucially important for making sure your wine does not spoil by oxidation or from microbial growth. Having the proper SO_2 level in your wine gives you this protection. By monitoring your SO_2 levels, you can make adjustments when needed, especially before starting primary fermentation, after malolactic fermentation has completed, after racking or when ready to bottle. To correctly adjust sulfite, you need values for your current "Free SO_2 " level and your wine's pH, both of which can be measured with the Vinmetrica SC-300 analyzer.

The key parameter in protecting your wine is "Molecular SO_2 " which for most wines should be at 0.8 ppm (mg/L) following primary fermentation. This in turn depends on the "Free SO_2 " (it can also be referred to as "unbound" SO_2) and the pH. Overall, you can reach your target Molecular SO_2 by measuring and adjusting your free SO_2 levels and considering your wine's particular pH. See Table 1.

Table 1. Free SO₂ concentrations necessary to attain 0.8 mg/L Molecular SO₂ at a designated pH.

Free SO ₂ (ppm)	13	16	21	26	32	40	50	63	79	99	125
pH	3.0	3.1	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0

We recommend using a sulfite calculator for determining how much sulfite to add to your wines after taking a sulfite measurement with the SC-300 Pro Kit. Winemaker Magazine's Sulfite Calculator at http://winemakermag.com/guide/sulfite can walk you through the process. See Appendix B for more information on how to adjust your wine for sulfite.

Monitoring your wine's pH is especially important for the first few months of the wine making process. Proper pH and Titratable Acidity (TA) levels influence mouth feel and provide wine stability. During malolactic fermentation, the pH can increase somewhat and should be monitored. Typically, wine pH and TA are inversely related; when pH goes up, TA goes down and vice versa. Adjustments may be made to your wine to prevent wine instability. See Appendix B for more information on adjustments.

Theory of Operation:

1. <u>Sulfite (SO_2) :</u> The SC-300, with the SO₂ electrode and reagents provided, can be used to determine sulfite levels in wine, musts, and other samples. It relies on the Ripper titration based on the quantitative reaction of the SO₂ with iodine (generated during the titration) which oxidizes the SO₂ in the sample under acid conditions.

 $IO_3^+ + 5I^+ + 6H^+ \rightarrow 3I_2 + 3H_2O$ generation of iodine (I₂) from iodate (IO₃⁻)

 $SO_2 + I_2 + H_2O \rightarrow 2I + SO_3 + 2H^+$ reaction of SO_2 and iodine

When all the SO_2 is titrated at the endpoint, excess iodine appears in solution. This is detected as current with the SO_2 electrode and signaled by audible and visual indicators. The endpoint is much more sensitive than the starch color change commonly employed for Ripper titration, and it is sharp and clear, even when titrating red wines and musts. From the known concentration of the titrant and its volume required to reach the endpoint, the free SO_2 is simply calculated.

- 2. <u>pH and TA:</u> The SC-300 kit also provides a pH electrode and reagents for calibration and determination of pH and titratable acidity (TA) values in wines and other samples. The pH value is simply determined by placing the calibrated pH electrode into a sample and reading the value. TA is determined by titrating a 5 mL sample of wine to an endpoint pH of 8.2 with the TA titrant (0.133N NaOH) from the syringe in the kit. From the known concentration of the TA titrant and its volume required to reach the endpoint, the TA is simply calculated (results are in units of g/L tartaric acid).
- 3. Potential measurements: In versions 3.1.1 and higher of the firmware, the SC-300 can display the voltage reading on an electrode attached to the pH connector.

Setup:

Setting up the SC-300 for the first time:

- 1. The SC-300 (See Figure 1) runs on two standard AA batteries (alkaline cells recommended). To insert the batteries, open the battery housing on the bottom of the back of the unit by removing the two screws and gently prying off the lid. Install the batteries with the negative '-' end contacting the coiled springs and the positive '+' end contacting the raised metal inserts then close the housing. If desired, you can prop the unit up using its folding stand.
- 2. Low Battery Detection: When the battery level is getting low, the instrument will show a low battery icon on the upper left side of the display but continues to operate without impairment to any function. Replace the batteries as soon as practicable. When the battery level drops too far, the instrument does not operate. It rapidly flashes the low battery icon for 3.0 seconds, beeps and shuts itself off.
- 3. Auto Shut-off: The SC-300 shuts off after 30 minutes. If this happens unexpectedly, just press the POWER button to resume from where you were.
- 4. Electrodes: When directed to do so, attach the desired electrode (SO₂ or pH, Figures 2 & 3) via the proper connector protruding from the top (on earlier model SC-300s, there is just a single connector for both electrodes). Be sure to secure the electrode plug to the BNC connector to insure proper function.



Figure 2. Attach the SO2 electrode to the phono plug connector on the SC-300 by pushing it straight on. Avoid a twisting motion.



Figure 3. Be sure the pH Electrode attachment is screwed into place on the BNC connector.

5. <u>SO₂ electrode</u>: Remove any protective cover from the electrode tip (some electrodes are shipped without one). This cover is for shipping protection and need not be used routinely. Put the electrode on its side, or hang it from the electrode holder if you have one. The SO₂ electrode is sturdy with its plastic housing, but do take care not to let things touch or strike the

platinum wires; they are somewhat fragile and will break if bent and straightened repeatedly. *Electrode care*: When done, always rinse with DI water and let air dry. There is no need to store the SO_2 electrode in any kind of solution.

6. <u>pH electrode:</u> Attach the pH electrode via the BNC connector on the top of the SC-300 (see Figures 2 & 3). *A note about the pH electrode*: The pH electrode is fragile and should always be handled carefully. Remove the liquid storage bottle by unscrewing the cap first, then gently pulling the bottle, followed by the cap, off the electrode. Rinse the electrode in a little distilled water before each use. *Electrode care*: Do not touch the glass bulb, nor attempt to wipe it with anything. When necessary, you may gently blot excess liquid away from the electrode surface, but avoid directly touching it. The pH electrode should always be kept in the liquid storage bottle with its pH storage solution (available from Vinmetrica) when not in use. Rinse the electrode through the hole in the cap about an inch, then gently screw the bottle onto the cap so that the electrode is in contact with the solution in the bottle.

Assembling the Pro Kit Equipment:

- 1. Remove the items from their packaging: the Lab Support Stand, the Vinmetrica Electrode Holder, the Double Burette Clamp, the Glass Burette and the Magnetic Stirrer. Carefully pull out the glass burette from its cardboard cylindrical container and out of its container. Use caution not to exert too much force on the burette as it is fragile and can break.
- 3. Open the Magnetic Stirrer from its packaging, then insert the double AA batteries included. For pH measurements or SO₂ & TA Titrations, you can use the 25 mL cylindrical container included with the magnetic stirrer. Another option is to replace the cylindrical container with the titration beaker (Figure 4) included with the SC-300.
- 4. Assemble the Lab Support Stand by attaching the long metal rod and screwing it into the large metal base. We have found that adding a drop of super glue to the threads of the metal rod can prevent loosening of the assembly. Then place the magnetic stirrer below the lab support stand as shown below in Figure 4.
- 5. To attach the Vinmetrica Electrode Holder, loosen the thumb screw on the Holder and slide it onto the Lab Support Stand rod (Figure 5), then tighten the thumb screw to adjust it to an appropriate height (Figure 6). Make sure the Vinmetrica logo can be read upright.
- 6. Attach the Double Burette clamp to the Lab Support Stand using its thumb screw to adjust for height, as shown in Figure 7 below.



Figure 4. The Magnetic stirrer and beaker with the pH electrode inserted into the Electrode Holder to the wine sample.

Figure 5. The Vinmetrica Electrode Holder slides onto the Lab Support Stand.

Figure 6. The thumb screw makes adjusting The Vinmetrica Electrode Holder's height easy.

Figure 7. The Double Burette Clamp slides easily onto the Lab Support Stand.

7. Carefully open the Double Burette Clamp's spring arms with your thumb and forefinger and place the burette in between the four indentations in the double burette clamp's spring arm's plastic knobs (See Figure 8). Slowly release the clamp spring to secure the burette between these four spring arm plastic knobs (See Figure 9). You can adjust the burette at any time by raising and lowering it within the spring arm clamps of the Double Burette Clamp. When making these adjustments, hold onto the burette and do not apply too much force to it as the burette is delicate. Use the end of the Vinmetrica electrode holder as a guide for the tip of the burette. Do not try and force the burette into the cutout at the end of the electrode holder. This may cause your burette to snap and break. Finally, insert the electrode that you wish to use into one of the open side slots (as seen in Figure 11). Once the rubber neck of the electrode is further stabilized into the slot.

You are now ready to titrate!

Note: The magnetic stir bar that is placed within the 25 ml cylindrical container or titration beaker can potentially damage the SO_2 and pH electrodes. When adjusting the height of the electrodes make sure that the stir bar in the beaker is below the bottom of the electrode. You do not want the spinning magnetic stir bar to strike the SO_2 or pH electrode.

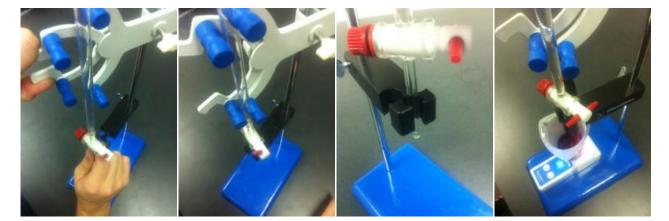


Figure 8. The glass burette should be placed between two sets of spring arm knobs on the Double Burette Clamp.

Figure 9. The burette sits firmly between the notches of the four spring arm knobs of the Double Burette Clamp.

Figure 10. The glass burette tip can be slid into the Electrode Holder's front open slot for accurate titrations.

Figure 11. The Pro Kit Assembly is complete. Go take some measurements!

Burette Maintenance

Keep the burette clean and wash with deionized (distilled) water when you are finished using the burette. This is especially important for TA titrations because the TA Titrant is caustic and can etch the burette. Every so often, you will want to clean your burette with a cleaning solution to maintain accuracy of the burette. The included ~2 grams of Burette Detergent is specially formulated to clean glassware. The dry detergent ships in an Eppendorf tube that has markings with approximate half gram increments. Upon first receiving the detergent, mix approximately half a gram (about an 1/8th of a teaspoon) with about 50 milliliters (mL) of distilled water into a small bottle. We recommend capping the bottle and shaking well for a minute or until the detergent is dissolved. Bubbles may form but the detergent will still be effective. The detergent solution should be made fresh every time you clean the burette, as it loses its potency quickly if stored as a liquid.

After you have run distilled water through your burette, close the stopcock, and fill with the Burette Detergent solution. You can open and close the stopcock over a waste bucket or sink to allow the solution to pass into the tip of the stopcock. We recommend letting this detergent solution sit in the burette for one hour. Dispense the detergent solution then rinse the burette with distilled water twice more. During this time we recommend opening and closing the stopcock to make sure the stopcock is turning easily. If it is not, remove the stopcock and clean it thoroughly. Once drained, allow to hang dry by hanging the burette upside down with the stopcock in the open position. Once the burette is dry, flip it back over and cover the top opening with a piece of tape. This will prevent dust from getting in that can cause aberrations when reading the burette. Remove the tape when you're ready to titrate.

Burette Reading

ALWAYS use eye protection and preferably gloves (latex or nitrile) when using glassware and chemical reagents. To get the most accurate results when titrating, there are a few things to keep in mind. We recommend reading from the bottom of the meniscus (See Figure 13 on the following page). First, use a thick sheet of white paper, note card or the back of a business card and draw a black band down the center of the paper about an inch and a half thick (See Figure 12). When taking a measurement, hold the paper about an inch behind the burette and the black band about a half an inch below the meniscus (See Figure 14). This provides a clear view of the bottom of the meniscus which helps make a precise, consistent measurement. Second, when filling the burette make sure the titrant (in most cases this will be either the SO₂ Titrant or the TA Titrant) has completely filled the bottom of the burette including within the tip. Sometimes bubbles can be trapped in the tip of the burette but can usually be dislodged by opening and closing the stopcock while the burette is hovering over a waste container. We also recommend washing a couple of milliliters of the titrant you are using through the burette to remove any excess water or contaminants that may remain from a previous titration. Finally, make sure there are not any large bubbles in the burette after filling. If there are, cover the top of the burette with some saran wrap (or parafilm if you have it) and make sure the stopcock is in the closed position. Then take the burette out of its clamp and hold the saran wrapped end tightly. Rotate and invert the burette to allow the bubbles to move out of the column of titrant. Once the bubbles have been displaced, you are ready to titrate.

Note: the gradations on your burette may be different than shown in the figures below. These photos are of the 10 mL burette, the 25 mL burette will have fewer gradations. Please take a look at your burette and determine the values for the gradations (lines).



Figure 12. On a thick white sheet of paper or a business card, use a black marker to draw a band approximately an inch and a half thick. This card will assist you in reading the burette accurately.

Figure 13. The Meniscus of the water column. This curve is formed because of interactions of water with the glass burette. We recommend measuring the titrant volume from the bottom of the meniscus. In the picture the value is about 3.27 mL.

Figure 14. Reading the Meniscus of the water column is easier and more precise if you use the paper with the black band held up an inch behind the burette and its black band 1/2 an inch below the meniscus. In this picture the value from Figure 17 is further resolved to 3.26 mL.

Before beginning the titration, record the starting titration volume, using the thick white paper with the black band, and then begin titrating, slowly. With enough practice, you will eventually be able to read the burette without black banded paper. Record the final titration volume using the same technique. You can also checkout these websites for more burette info: http://www.titrations.info/pipette-burette or http://www.csudh.edu/oliver/demos/buretuse/buretuse.htm

Instrument Operation:

- 1. <u>Disconnect any electrodes</u>. Turn on the instrument by pressing the POWER button <u>briefly</u> (Note: depressing the POWER button longer than two seconds at start-up will cause the instrument to enter *Test Mode;* see Appendix A). The instrument will go through a power-up sequence. After a few seconds the instrument will start in whatever mode was last selected. The mode is indicated by the yellow LED panel on the left. Select the desired mode by pressing the MODE button.
- 2. <u>SO₂ mode:</u> This is for titration in determining ppm of SO₂ (see below under 'Procedures'). The SO₂ electrode must be used, but <u>do not attach the electrode yet</u>. [*Warning: The pH electrode can be damaged by connecting it to the instrument in SO₂ mode*. As a safety precaution, you must press the ENTER button after selecting SO₂ mode to confirm that the SO₂ electrode is attached before the mode will be enabled. However, scrolling through the MODE selections (such that you pass by the SO₂ selection) with a pH electrode attached is safe, as long as you do not press ENTER to confirm the SO₂ mode.] After pressing ENTER, you should see the display show a value less than 50 (usually 0.0) and the green "PROCEED" LED should be on. <u>Now you can attach the SO2 electrode.</u>
- 3. **[Optional]** Precondition the SO₂ Electrode: *All SO₂ electrodes are pre-conditioned at Vinmetrica before shipment. Therefore normally you should skip this step. If you think your electrode is not functioning properly you may try pre-conditioning the electrode. See Appendix C2 for more information on preconditioning the SO2 electrode.
- 4. <u>pH mode:</u> In this mode, the meter measures the pH. The pH electrode must be attached. If the instrument has not yet been calibrated, the message "doCal" scrolls across the screen, and you need to do a calibration (see Calibration of pH below). We recommend re-calibrating the instrument for pH once each day of use. *Warning:* Be careful not to press ENTER when the pH electrode is attached while the instrument is in SO₂ mode, as this can potentially damage the pH electrode. If this does happen, and the pH electrode does not respond correctly, place it into the pH 4 Reference solution for an hour and it should return to normal function.
- 5. <u>Potential mode</u> (Firmware version 3.1.1 and higher) (pH LED flashing) In this mode the instrument displays the potential (in volts) coming from an electrode attached to the pH connector. This can be used with certain electrodes, for example, galvanic oxygen probes, potassium, or sodium electrodes, or to view the raw voltage reading of a pH electrode. In this

mode, when the red STOP LED is illuminated, the values on the screen are negative; otherwise the values are positive.

- 6. <u>TA mode</u>: This is for titration in determining titratable acidity (TA). As in the pH mode, the pH is displayed and the pH electrode must be attached. The green ("PROCEED") LED is lit if the pH is below 8.2, while the red "STOP" LED is lit if the pH is 8.2 or greater. (see below under Measuring TA by Titration)
- <u>CAL mode</u>: This is for calibrating the pH electrode, which must be attached. The display initially shows CAL for a few seconds as it prepares to read pH and lets readings settle. Thereafter, the display shows the measured pH level with two decimal places.
- 8. Calibration works with one of the following reference calibration sets:

pH 4.01 and 7.00 or "4/7" pH 7.00 and 10.00 or "7/10" pH 3.00 and 7.00 or "3/7"

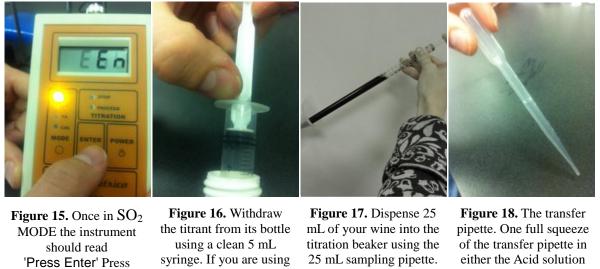
Vinmetrica recommends use of the pH 4/7 Reference Solution set provided with the kit.

9. The magnetic stirrer has two modes. Pressing the "light bulb" button on the magnetic stirrer activates a light underneath the sample and the stirrer. The power button activates just the stirrer. After pressing either button, the stirrer remains active for 60 seconds, a feature to conserve its batteries. If during the titration it turns off, simply press the button again for it to continue. We recommend using the light mode because it helps us indicate when the stirrer stops. Plus when doing TA titrations watching the wine turn from deep red to dark green is cool!

Note: When using the magnetic stirrer, be sure that the electrode does not touch the spinning stir bar as there is a slight chance that it can damage the glass bulb of the pH electrode or the platinum wires of the SO_2 electrode. If you are using the Vinmetrica Electrode Holder, adjust the electrode's height so that its probe end is above the level of the stir bar.

Procedures Measuring Free SO₂ by Titration:

1. With the electrode disconnected, turn on the instrument and select SO_2 mode with the MODE button. Press ENTER to confirm selection of SO_2 mode (Figure 15). The display should show a value less than 20, usually 0.0. Now attach the SO2 electrode.



- the glass burette, use the Make sure this is clean or Reactant should be ENTER to activate SO₂ syringe to fill it. before putting the pipette approximately 2 mL. MODE. into your wine container! 2. Fill the syringe by drawing up the SO_2 Titrant (the bottle with the blue label) (Figure 16). Expel bubbles and set the plunger on the syringe to a readable point, preferably the 5.0 mL point. Make sure the outside of the syringe is dry, to minimize any inaccuracies. [Note: the 5.0 mL setting allows determination of up to 100 ppm SO₂ in a standard 25 mL wine sample.] If using the burette, use the syringe to dispense the SO₂ titrant into the top of the burette. Make sure the burette stopcock is in the closed position (where the red handle is horizontal). When filling the burette make sure the SO₂ titrant has completely filled the bottom of the burette including the tip. Sometimes bubbles can be trapped in the tip of the burette but can usually be dislodged by opening and closing the stopcock while the burette is above a waste container. If you spill any titrant on the outside of the burette, be sure to clean it up with a paper towel or dry rag. If the spilled titrant is not cleaned from the outside of the burette you may introduce these spilled titrant droplets into the wine sample leading to an inaccurate reading. Be sure to record your starting burette or syringe volume; refer to 'Burette Reading' section under the Setup section for how to measure accurately.
 - 3. Place 25 mL of wine or must in the titration vessel. We recommend using the 25 mL sampling pipette provided in the kit: draw the sample up to the 0 mL mark, and then dispense the sample into your titration vessel by letting the tip of the pipette touch the side of the vessel while the

sample drains (See Figure 17). **NEVER pipette any reagents by mouth! Also make sure the pipette you are using is completely clean before submerging into your wine sample.**

- 4. Using the transfer pipettes (see Figure 18), add about 2 mL Acid Reagent and 2 mL Reactant solution to the titration beaker (the two transfer pipettes should come with red and yellow labels, with red for the Acid Reagent and yellow for the Reactant). It is not necessary to be extremely accurate in this step; with these pipettes, 2 mL is roughly the amount that fills the pipette up to the 2 mL mark after a single thorough squeeze of the bulb. To preserve the shelf life of these reagents take care not to cross contaminate the transfer pipettes. If they do get contaminated rinse them off with distilled water and let air dry. **Caution: the Acid reagent is corrosive and can cause damage to clothing, skin and eyes. The reagents should not be ingested. ALWAYS use safety glasses! We recommend the use of laboratory latex or nitrile gloves during this procedure. If any solutions contact skin or eyes, flush with plenty of water.**
- 5. If you are using a magnetic stirrer, turn it on to stir at a moderate rate. The Magnetic Stirrer provided with the SC-300 Pro Kit operates at a suitable preset speed. Make sure your electrode is not being struck by the spinning stir bar. To prevent this, we recommend using the Vinmetrica Electrode Holder to stabilize your electrode.
- 6. Rinse the electrode briefly with distilled water. Insert the electrode into the titration beaker so that the tip is completely submerged to just above the circulation gaps (cutouts at the tip of the electrode) but above the level of the stir bar (approximately half an inch from the bottom of the titration beaker). If you are using the Vinmetrica Electrode Holder adjust it to a similar level.
- 7. If you are stirring manually, begin now; use a constant moderate swirling motion. If the electrode is not held in a stand, hold it against the side of the vessel with one finger and grasp the vessel with the remaining fingers so that the two move together. (See Figure 19).
- 8. Verify that the current is less than 50 and the green ("PROCEED") LED is lit (See Figure 20). If the current is greater than this, and/or the red ("STOP") LED is lit and the buzzer sounds, your sample has less than 2 ppm SO₂ and there is no need to proceed. (Verify that your SO₂ has been preconditioned; see step 3 in the **Instrument Operation** section of the manual).
- 9. Titrate the sample by adding the SO_2 Titrant drop wise from the syringe (See Figure 19) or from the burette (See Figure 20), being sure to note the starting volume mark on the syringe or burette. Try to accomplish the titration as rapidly as possible (in 3 minutes or less), but be careful near the endpoint so as not to overrun it here, dispense one or two drops at a time. Be sure to maintain stirring or swirling throughout the entire procedure. If the magnetic stirrer turns off, turn it back on.



Figure 19. Manual stirring technique. Hold the electrode against the side of the titration beaker and swirl gently; add SO2 Titrant with other hand.

Figure 20. Automated stirring technique. Turn on the magnetic stirrer; add SO2 Titrant by slowly opening the burette stopcock valve.

Figure 21. Make sure that the "PROCEED" LED is lit. You should be reading close to 0.0 when you first start. You are now ready to titrate! Figure 22. Once the device beeps for 15 seconds or 20 sets of "beep-beep" you are done with the titration. The red "STOP" LED will also remain lit.

- 10. During the titration, the LCD display will show transient currents, the red "STOP" LED will briefly illuminate, and the beeper will sound ("beep-beep!"). These transient indicators will last longer and longer as you approach the endpoint (Figure 22). Take the endpoint as the first addition of Titrant that causes the display to exceed 50, <u>and</u> the red LED and beeper to stay on, for longer than 15 seconds (or a count of 20 sets of "beep-beep"). It is important to maintain stirring or swirling to detect the endpoint well. Do not add titrant while the red "STOP" LED is lit. Read the remaining titrant volume off of the syringe or burette.
- 11. Calculate the volume of titrant used "V" (using the Syringe: Starting volume minus final volume, e.g.; Burette: final volume minus starting volume), e.g., V = 5.0 mL 3.5 mL = 1.5 mL)
- 12. The free SO₂ content is calculated in units of parts per million (ppm) or mg/L as:

ppm (mg/L) Free SO₂ =
$$\frac{64 * V * N * 1000}{2 * S}$$

Where $V = mL SO_2$ Titrant needed to reach the endpoint; N = normality (concentration) of the Titrant; and S = mL of your wine sample.¹ If you use a 25 mL wine sample as directed and the SO₂ Titrant's normality is 0.0156 as supplied in the kit, then the calculation is simply:

**ppm (mg/L) Free SO₂ =
$$20 * V$$** (i.e. 20 times V)

^{1 64 [}mg SO2/mmol SO2]* V [mL] * N [meq/mL] * 1000 [mL/L]

^{2 [}meq/mmol SO2] * S [mL]

Measuring <u>Total</u> SO₂ by Titration (optional - requires 1N NaOH):

- 1. Place 25 mL wine or must in the titration vessel (See Figure 17).
- 2. Add 10ml 1N sodium hydroxide (Vinmetrica Part number SC-100-17) and mix well. Let stand approximately 10 minutes.
- 3. Using the transfer pipettes, add approximately 8 mL of the Acid Reagent and 2 mL of the Reactant solution to the vessel. Remember that if you are using the transfer pipettes in the kit, 2 mL is the amount that fills the bulb with a vigorous squeeze, so dispense four of these for the Acid Reagent.
- 4. Proceed from step 5 in the Free SO_2 procedure above. The result calculated will be total SO_2 , rather than free SO_2 in parts per million (ppm) or mg/L.

Calibration of pH:

- 1. Be sure the pH electrode is attached to the unit, via the BNC connector. Then select CAL mode by pressing the MODE button until the CAL LED illuminates.
- 2. Choose a calibration set that corresponds to the range you are working in. Usually for wine this will be at pH values below 4, so use the 4/7 set. If you have a source of a pH 3.00 reference solution, you can use this in place of pH 4.
- 3. Rinse the electrode with DI water, shake or blot off excess liquid gently, and place the electrode into a small vessel (like the pH 4 Reference Solution cap) containing one of the reference solutions (e.g. pH 4.01).
- 5. The instrument will determine which calibration solution is being used, and will display the apparent pH value. This may be different by as much as 0.40 from the value of the reference solution (e.g. the LCD may display 4.41 when the pH electrode is sitting in the pH 4.01 reference solution). When the pH level is sensed as stable, the nominal value is shown on the display, flashing, and the "CAL" LED flashes to convey that calibration for this value is ready. Press the ENTER button to accept the calibration.
- 6. The display stops flashing, scrolling the message **'Lood CAL'**, and four beeps are rapidly sounded to indicate success. [Note: if an error occurs during this process, the message **'bAd**

CRL' will scroll and a single beep will sound; the instrument will then continue to wait for a stable pH level. Repeat step 4.] Following the '**Lood CRL**' message, the display will now show the calibrated pH value.

- Now rinse the electrode again and place it in the second member of the calibration set (e.g., pH 7.00 reference solution). Repeat the process to get a second 'Lood LAL' message. Exit into pH or TA mode.
- 8. "Force Calibrate" feature [firmware v3.0.6 and later]: if your instrument displays an apparently stable value that is within 0.5 pH value of the target (i.e., 3.5 to 4.5 for pH 4.01, or 6.5 to 7.5 for pH 7.00), but the CAL LED does not flash within a reasonable amount of time (1-2 minutes at most, typically), you can force the instrument to accept the calibration by following these steps:
 - a. Use the MODE button to select TA mode.
 - b. Press and hold the MODE until the display goes from "**CAL**" to "**CAL**" i.e., the 'C' will change to lower case 'c'.
 - c. The display will then show **PH4**. Place your pH electrode in the pH 4.01 reference solution and press ENTER.
 - d. The apparent pH will be displayed. Allow it to come to a stable reading, then press ENTER. If the reading is within the Cal tolerance range (default 0.5 pH as above, but see Appendix A for Cal tolerance settings), the **'Lood CAL'** message will scroll and the four beeps will sound as usual. Otherwise the message **'bAd CAL'** will scroll and a single beep will sound. In that case you can return after the next step and try PH4 again.
 - e. The display will then show **PH7**. Place your pH electrode in the pH 7.00 reference solution and press ENTER, then repeat the procedure in step d.

Measuring pH:

- 1. Make sure the pH electrode is attached. Calibrate it as described above, if necessary. Select pH mode with the MODE button.
- 2. Rinse the pH electrode with DI water. Gently shake off or carefully blot away excess liquid.
- 3. Place the electrode in the solution to be tested. Be careful not to let the electrode strike any surfaces.

4. Allow the pH reading to stabilize. Typically this takes about 10-15 seconds. Read the pH value on the display. Note: Once you've completed the pH test you can start the TA test. Start with precisely a 5 mL wine sample before beginning the pH & TA combined measurement. You can use a clean 5 mL sampling pipette provided in the kit to pipette this amount.

Measuring Titratable Acidity (TA) by Titration:

- 1. If you are working with a sample of must, we recommend homogenizing your sample in a blender before proceeding; otherwise your TA values can be very inaccurate. Take 100 mL or more of your must and put it in a blender on high for 30 seconds. Allow solids to settle for 2 minutes before sampling or use a cheese cloth or mesh strainer to remove solids.
- 2. Fill the syringe by drawing up the TA Titrant (0.133N NaOH). Expel bubbles and set the plunger on the syringe to a readable point, preferably the 5.0 mL point. [Note: the 5.0 mL setting allows determination of up to 10 g/L TA in a standard 5 mL wine sample.] If you are using the burette, use the syringe to dispense the TA titrant into the top of the burette. Make sure the burette stopcock is in the closed (the red handle is horizontal) position. When filling the burette make sure the TA titrant has completely filled the bottom of the burette including the tip. Sometimes bubbles can be trapped in the tip of the burette but can usually be dislodged by opening and closing the stopcock while the burette is above a waste container. If you spill any TA titrant on the outside of the burette, be sure to clean it up with a paper towel or dry rag. If the spilled titrant is not cleaned from the outside of the burette you may introduce these spilled titrant droplets into the wine sample leading to an inaccurate reading. Be sure to record your starting burette or syringe volume. Caution: the TA Titrant is caustic and can cause damage to clothing, skin and eyes. We recommend use of laboratory safety glasses and latex or nitrile gloves during this procedure. If any solutions contact skin or eyes, flush with plenty of water.
- 3. Place 5.0 mL wine or must in the titration vessel (100 mL polypropylene beaker). We recommend using the 5 mL pipette provided in the kit: draw sample up to the 0 mL mark, then dispense the sample into your titration vessel by letting the tip of the pipette touch the side of the vessel while the sample drains. For best accuracy, do not blow out the liquid that remains in the tip. Add about 15 ml of deionized (DI) water (distilled water).
- 4. Turn on the instrument. Make sure the pH electrode is attached. If necessary, calibrate it as described above. Select TA mode with the MODE button.
- 5. If you are using a magnetic stirrer, turn it on to stir at a moderate rate. Be sure the stir bar will not strike the electrode in the following steps. (See Figure 24)

- 6. Rinse the electrode briefly with DI water. Insert the electrode into the titration vessel so that the tip is fully submerged to just above the circulation gaps (cutouts at the tip of the electrode).
- 7. If you are stirring manually, begin now; use a moderate swirling motion. If the electrode is not held in a stand, hold it against the side of the vessel with one finger and grasp the vessel with the remaining fingers so that the two move together while swirling (See Figure 23).



Figure 23. Manual stirring technique. Hold the electrode against the side of the titration beaker and swirl gently; add TA Titrant with other hand.

Figure 24. Automated stirring technique. Turn on the magnetic stirrer; add TA Titrant by slowly opening the burette stopcock valve.

Figure 25. Make sure that the green "PROCEED" LED is lit. You should be reading a pH close to what you expect your wine is at. You are ready to titrate!

Figure 26. Once the pH arrives at or passes 8.20 you are done with the titration. The red "STOP" LED will be lit and the instrument will be beeping

- 8. Verify that the pH is less than 7 and the green ("PROCEED") LED is lit (see Figure 29). If the pH is greater than this, and/or the red ("STOP") LED is lit and the beeper sounds, your sample has less than 0.1g/L TA and there is no need to proceed.
- 9. Titrate the sample by adding the TA Titrant drop wise from the syringe or burette, being sure to note the starting volume mark on the syringe or burette. During the titration, the pH will gradually rise from its starting value (below 4 usually). As you approach pH 7, go slowly in adding successive drops of titrant so as not to overrun the endpoint. Be sure to mix thoroughly after each successive drop of titrant. Take the endpoint as the first addition of TA Titrant that causes the pH to stay above 8.2 for longer than 15 seconds. The red "STOP" LED and the beeper will provide additional indication of the endpoint (See Figure 26). Read the endpoint volume off of the syringe or burette. To silence the beeper after the endpoint, select pH mode, or turn off the instrument.
- 10. Calculate the TA value as:

$$TA\left(g/LTartaric\right) = \frac{V*0.133*75}{S}$$

where V = mL Titrant needed to reach the endpoint; 0.133 = normality of the Titrant, <math>S = mL sample. If you use 5 mL of sample as directed, and the Titrant is 0.133 N as supplied, then the calculation is simply

TA = 2 * V (*i.e.* 2 times V)

Note: to express these values as % tartaric acid, divide by ten; e.g. if the TA is 7.1 g/L, that is equivalent to 0.71 % tartaric acid.

Finishing up:

- 1. Turn off the instrument.
- 2. Rinse the SO₂ electrode and syringe with distilled water. Let air dry.
- 3. Be sure to rinse the pH electrode with distilled water and then store it in its storage solution as directed under 'Setting up the SC-300 for the first time', item 6 (page 6 of this manual).
- 4. Store all reagents tightly capped and away from heat and sunlight.
- 5. Discard waste samples and solutions in accordance with local regulations. Acidic solutions can be neutralized by slow addition of baking soda (sodium bicarbonate) with stirring until effervescence ceases.

Technical assistance: info@vinmetrica.com tel. 760-494-0597

WARRANTIES AND LIABILITIES

- 1. The materials provided in the kit, as described on pages 1 and 2 above, ("Materials") are warranted as follows: The SC-300 instrument, SO₂ electrode and non-reagent accessories are warranted against defects in workmanship for 24 months from date of purchase. The reagents are warranted to perform as described herein up until any stated expiration date or 6 months after purchase, whichever is later. The pH electrode is warranted for 12 months. THE WARRANTIES IN THESE TERMS AND CONDITIONS ARE IN LIEU OF ALL OTHER WARRANTIES, EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, NONINFRINGEMENT, OR FITNESS FOR A PARTICULAR PURPOSE, SAID WARRANTIES BEING EXPRESSLY DISCLAIMED.
- 2. Buyer agrees that its sole and exclusive remedy against Vinmetrica shall be limited to the repair and replacement of Materials or parts of Materials, provided Vinmetrica is promptly notified in writing, prior to the expiration of the warranty period specified above, of any defect. Vinmetrica's liability for any damages due Buyer shall be limited to the purchase price of the Materials.
- 3. VINMETRICA'S MAXIMUM LIABILITY FOR ALL DIRECT DAMAGES, INCLUDING WITHOUT LIMITATION CONTRACT DAMAGES AND DAMAGES FOR INJURIES TO PERSONS OR PROPERTY, WHETHER ARISING FROM VINMETRICA'S BREACH OF THESE TERMS AND CONDITIONS, BREACH OF WARRANTY, NEGLIGENCE, STRICT LIABILITY, OR OTHER TORT WITH RESPECT TO THE MATERIALS, OR ANY SERVICES IN CONNECTION WITH THE MATERIALS, IS LIMITED TO AN AMOUNT NOT TO EXCEED THE PRICE OF THE MATERIALS. IN NO EVENT SHALL VINMETRICA BE LIABLE TO BUYER FOR ANY INCIDENTAL, CONSEQUENTIAL OR SPECIAL DAMAGES, INCLUDING WITHOUT LIMITATION LOST REVENUES AND PROFITS.

HAZARDS AND TOXICITY

All Materials offered by Vinmetrica are intended for use by individuals who are familiar with laboratory procedures and their potential hazards. The Materials contain chemicals which may be harmful if misused. Due care should be exercised with all Materials to prevent direct human contact. Glassware can break and chemicals can splash during experiments; *Always use safety glasses*. We strongly recommend using nitrile or latex gloves and wearing long pants, long sleeves and closed toed shoes. Keep out of reach of children.

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Appendix A - Test Mode

Test Mode provides various special functions that may be useful in testing the device, for example, if troubleshooting is necessary.

- Test Mode is entered when the POWER button is pressed longer than 2 seconds while turning the instrument on. Remove the pH electrode if it is attached.
- Test Mode is organized into sections. Press the POWER button briefly to move to the next section. After the last section, Test Mode restarts the first. WARNING! DO NOT MOVE THROUGH SECTIONS WITH THE pH PROBE ATTACHED! This can damage the electrode. Only connect the pH electrode if needed in section 2 or 4. Always remove the pH electrode before leaving these sections.
- Combinations of the yellow MODE LEDs are illuminated to indicate the section number as shown in the table below.
- The Stop LED (red) is illuminated when an error is detected by the instrument The Proceed LED (green) is illuminated to indicate no error detected. The green LED does not guarantee proper functioning; it only indicates that no problem could be automatically detected. The user should make careful observations to discern proper operation.
- To exit Test Mode, hold the POWER button down (5-10 seconds) until the instrument shuts off. If the device does not shut off after 10 seconds of holding down the button, move to the next section by releasing, then pressing again the POWER button briefly; then try to exit again.

Yellow LEDs	Section	Equipment Required	Description
0000 000	1. Version	None.	The version number of the instrument software is displayed.
0000	2. Burn-in	None	The instrument goes through a continuous "burn-in" cycle, exercising relay, sound, LEDs, and display.
0000	3. pH	pH probe or precision voltage source. Do not exceed +/- 0.5 V.	An uncalibrated pH level is shown in two alternating parts. First, the integer portion of pH level is shown (1 to 14). Next, three decimal places are shown. Readings above 14.00 are shown as "". Readings below 0.00 are shown as
0000	4. SO ₂	SO ₂ probe or SO ₂ probe simulator (e.g., 500 kOhm resistor)	The SO_2 current in nanoamperes is displayed. For values under 10, one decimal place is shown.
0000	5. pH Voltage	pH probe or precision voltage source. Do not exceed +/- 0.5 V.	The raw voltage output from the instrument's pH amplifier is displayed as X.XX volts. Readings can range from 0.00 to 4.10.

Yellow LEDs	Section	Equipment Required	Description
	6. SO ₂ Voltage	SO_2 probe or SO_2 probe simulator.	The raw voltage output from the instrument's current amplifier is displayed as X.XX volts. (.XXX if less than 1.00)
0000	7. DAC Test	None. Disconnect probe.	The Digital-Analog Converter (DAC) is cycled through its 32 levels. <i>Note</i> : Ignore red LED error indication.
0000	8. Battery Voltage	Install two AA batteries	The battery voltage is displayed as X.XX volts.
0000	9.Character Set	None.	Every ASCII character (space) to ~ is displayed. Due to the limitations of the 7-segment format, some characters are not used by the software.
000	10. Number Display	None.	The display cycles through showing every possible digit and every decimal point.
0000 0000	11. Sound Test	None.	The beeper is turned on continuously.
0000	12. pH CAL values [firmware 3.0.6 and later]	None	Displays current CAL values for pH 7 and 4, in mV. Pressing ENTER cycles between these. If a pH CAL reset has been done, displays CAL value for pH 3 rather than 4.
000	13. pH CAL reset [firmware 3.0.6 and later]	None.	Displays " PrESS EntEr "; Press ENTER to reset pH CAL parameters to default values. Message " Good CAL rSt " then scrolls.
0000	14. CAL tolerance [firmware 3.0.6 and later]	None.	Displays " Cal tol " then displays the current value of the tolerance required to allow calibration. Default is 0.5 pH units i.e., the displayed pH must be within 0.5 units of the target pH to allow calibration. Pressing ENTER cycles between settings of 0.25 - 0.50 - 0.75 - 1.00 - 1.25 - 1.50
00	15. SO ₂ Baseline	None. Disconnect SO ₂ electrode [only fw v3.1.0 and later]	Sets baseline value for SO2 mode. Let message scroll 5 sec., then press ENTER

Appendix B - Sulfite and TA Adjustments

Using the Winemaker Magazine Sulfite Calculator:

Winemaker Magazine's Sulfite Calculator at <u>http://winemakermag.com/guide/sulfite</u> is an excellent tool for calculating how much sulfite should be added to your wine. We will briefly go over the process here for some clarification.

1. Select a 'Preferred method of Sulfite addition:'; we recommend using a 10% solution of Potassium metabisulfite (KMBS). [You can prepare this solution by weighing out 10g of KMBS and dissolving it in a FINAL volume of 100 mL DI water.]

2. Next choose the wine type (red or white).

3. Keep the 'Desired Molecular SO_2 ' at 0.8 mg/L and enter your wine's pH value (hit your keyboard's 'Enter' button after entering the value).

4. After entering in your 'Desired molecular SO_2 ' and your wine's pH, the calculator will ask you to enter the 'Desired level of free SO_2 '. The calculator already 'knows' what this value is (based on the data you entered), and it <u>should</u> appear in the 'Notes:' section below the entry fields, but the one fault of this app is that, at least when we run it, it won't tell you until you go through the calculation once! So, for now, just enter "100" for the 'Desired level of free SO_2 ' and proceed; you will come back to this step in a moment to get the right answer. (see below).

5. Now input the "Current level of Free SO_2 " which you determined from measuring Free SO_2 with the Vinmetrica SC-300.

6. Finally, enter the "volume of wine to be corrected". Choose liters or gallons; we prefer "liters" because the answer is returned in mL. The 'Amount of sulfite to be added:', should be in mL or fluid ounces of 10% sulfite solution or in grams if you use sulfite powder as your sulfite additive.

7. Now press the blue 'Calculate' button. Ignore the answer below the "Amount of sulfite to be added:" and look below to the 'Notes:' section. You should now see the message "1. The recommended level of free SO_2 for this type of wine, molecular SO_2 & pH is: [your value] mg/L";

8. Go back to step 4. and enter this value for the 'Desired level of free SO_2 '.

9. Press 'Calculate' again to get the correct amount of sulfite to add.

We recommend double checking your calculations. Also, be sure you are using fresh KMBS! Once you have added the recommended amount of sulfite, stir your wine thoroughly and take another SO_2 measurement after waiting at least 30 minutes. If the measurement matches the 'Desired level of free SO_2 ' then you are done; otherwise, make incremental additions and repeated SO_2 measurements until you reach your desired level.

Adjusting TA in your wine:

We recommend adjusting your titratable acidity levels by adding tartaric acid (for non-grape wines, fruit acids can be added but use caution - malic acid, if dosed improperly, can make wine overly

sour or tart). If your wine's pH is too high, indicating low acidity, and TA levels too low, you can add tartaric acid to decrease the pH and increase TA. By measuring TA, you can figure out how much tartaric acid to add without making your wine overly tart or sharp. As a rough rule of thumb, adding 1 g of tartaric acid per liter of wine will increase the TA by 1 g/L (0.1%) and reduce the pH by 0.1 pH unit. If your TA is too high before bottling, you can try "cold stabilization". This results in precipitation of potassium acid tartrate (potassium bitartrate) to decrease the tartness. Another method to decrease your TA level is to add calcium carbonate or potassium carbonate (CaCO₃ or K₂CO₃). For the chemically inclined, we recommend Zoecklein's book "Wine Analysis and Production" which goes over theory and practice behind these adjustment techniques and many wine analytical techniques.

Appendix C1 - Troubleshooting: pH and TA Issues

I can't calibrate the pH on my SC-300

When calibrating your pH electrode, remember these points:

- 1. The displayed pH may not be correct until after you press ENTER.
- 2. If the instrument signals stable pH but displays "Bad Cal" after pressing ENTER, try laying it flat on the table; when the next stable signal is signaled, press the ENTER button quickly without handling the instrument. Sometimes the instrument may pick up noise from its environment, particularly if you handle it at the last second, while it's trying to achieve a stable reading. This sensitivity is usually only an issue during calibration.
- 3. If values appear to drift, leave the electrode in the pH 4.01 reference solution for 30 minutes.
- 4. If you intend to read pH values in samples that are at a different temperature than ambient, it's best to have your reference solutions at that temperature also before calibrating.
- 5. If the displayed pH value is within 0.5 pH units of the target, but the CAL light fails to flash in the expected "ready-to-calibrate" manner, you can try the "force calibrate" feature (available in firmware 3.0.6 and higher, described in the manual under "Calibration of pH")
- 6. If the displayed pH value is outside of the default tolerance of 0.5 pH (but not more than 1.5 pH units), you can temporarily broaden this tolerance to get calibration to go. See Test Mode, stage 14 in Appendix A. Call or Email us for help if you need it.
- 7. Finally, refer to the next FAQ question if these steps do not help.

What should I do if my pH electrode is acting sluggish, erratic and is difficult to calibrate?

Reconditioning and cleaning of pH electrodes:

Even in normal use and storage, performance of pH electrodes may show deterioration over time, which typically shows up as noisy, erratic or sluggish electrode readings, and/or difficulty calibrating. Assuming the meter itself is working (see "Meter test" below), then there are two main causes for this:

- 1. Clogging of the reference junction (most likely).
- 2. Fouling of the glass membrane (happens occasionally, or after prolonged service).

The following procedures will often provide renewed stability and pH sensitivity. If the electrode cannot be restored by one of these methods, it needs to be replaced.

Unblocking the reference junction:

The reference electrode junction is usually the problem when the electrode can't calibrate in its expected ranges. This junction is a fine-pored frit that allows electrical contact of a reference electrode with the solution being tested. It can become clogged over time.

- 1. Soak electrode in hot (NOT boiling!) water, about 60 °C, for 5 10 mins. Allow to cool to room temperature then place in pH 4 reference solution for 5 minutes. Try to recalibrate. If this does not work, try remedy 2.
- 2. Place the pH electrode into the pH storage solution (available from Vinmetrica part number SC-200-10 or a solution of 2.5M KCl with optionally added 0.01M KHP) at 60 °C and allow electrode and solution to cool to room temperature, then place in pH 4 reference solution for 5 minutes. Try to recalibrate. If this doesn't work, try remedy 3.
- 3. Soak in 0.1M HCl (note: this can be made by diluting 1 mL of the SO₂ Acid Solution with 20 mL DI water) or 0.1M nitric acid (HNO₃) for 1 hour. Rinse with DI water, then place in pH 4 reference solution for 5 minutes. Try to recalibrate. If this does not work, try remedy 4.
- 4. Soak in 1:10 dilution of bleach in a 0.1 0.05 % solution of liquid detergent in hot water with vigorous stirring for 15 mins. Rinse with DI water, then place in pH 4 reference solution for 5 minutes. Try to recalibrate.

Cleaning the pH electrode's glass membrane:

The glass bulb is a thin membrane of a special kind of glass that actually does the job of responding to the pH of the solution. It can sometimes become dirty and poorly responsive.

1. Immerse electrode tip in 0.1M HCl (see above for how to make) for about 15 secs., rinse with distilled water, then immerse in 0.1M NaOH (you can use a little of your TA Titrant for this) for another 15 sec. Cycle the electrode through these solutions several times (rinsing with DI water

in between), then rinse and check for performance in pH buffer 4.00 and 7.00.

2. Some other tricks: protein deposits can be removed by soaking in 1 % pepsin in 0.1M HCl for 15 mins. Inorganic deposits may be removed by soaking in 0.1M tetrasodium EDTA solution for 15 mins. Grease and oil deposits may be removed by rinsing the electrode in mild detergent in methanol solution.

Instrument test:

You want to be sure that the instrument is responding correctly. A quick test is to simply short out the electrode connector:

- 1. Put the instrument in pH mode.
- 2. Remove the electrode to expose the BNC connector at the back of the instrument. Short out the terminals on the connector, using a paper clip or similar metal piece to touch the center pin of the connector to its outer metal sheath.
- 3. With the input shorted out, the reading should be pH 7.00 +/- 0.50. If out of this range, the meter is probably bad. Contact us at info@vinmetrica.com or tel. 760-494-0597.
- 4. Bear in mind that this test is not 100% fool-proof (the instrument might still have trouble reading pH values different from 7.00), but generally if this test passes, it is much more likely to be an electrode problem.

Help! What do I do if my pH electrode was attached to my SC-300 when in SO₂ mode? Is my pH electrode broken?

No, don't worry, your pH meter should still work. To fix it, leave your pH electrode in the pH 4 reference solution for an hour and it should return to normal function. Enabling SO₂ mode with the pH electrode attached could cause the pH electrode to wear out more quickly. Because of its design, SO₂ mode cannot be accessed unless you press 'ENTER' after pressing the 'MODE' button, this is another safeguard we installed to prevent this from becoming a problem.

Appendix C2 - Troubleshooting: SO₂ Issues

How stable are the reagents?

The SO₂ reagents and the pH/TA reagents are all warranted to last for 6 months. We have found that, in fact, except for the TA Titrant, our reagents are stable for well over 12 months if stored tightly capped, out of the heat and direct sunlight. And of course, these reagents will last much longer if not

cross-contaminated with each other! The TA Titrant can pick up atmospheric CO_2 and lose potency over time, so we recommend that you replace it every six months.

How can I check the accuracy of my reagents?

It's rare that the SO_2 reagents go bad, but if you are concerned about it, run the 'Ascorbic Acid Test' method located in the FAQ section of the website to check your SO_2 reagents. If you are worried about your TA Titrant, you can run the 'KHP test', also located on the Vinmetrica website in the FAQ section under 'Manuals, Tests and How To Videos' at <u>vinmetrica.com/FAQ/</u>

I added the calculated amount of sulfite to my wine but the numbers are still low!

This is a common occurrence with several explanations, any or all of which may be happening.

1. Make sure you are using fresh sulfite powder. Potassium metabisulfite degrades over time and that stuff you bought 2 years ago is probably bad now!

2. Make sure that you stir your wine thoroughly when you add sulfite. If you pour a 10% solution of KMBS into your wine, it sinks like a battleship! A sample taken off the top will read low unless the wine is stirred.

3. A significant portion of the sulfite you added may have ended up 'bound', particularly if your free SO_2 was very low to begin with. This bound SO_2 does not show up when you measure free SO_2 , and it is not protecting your wine. You will need to add more sulfite until your free SO_2 comes up to the right level. Sometimes you must add 2 or even 3 times more sulfite than you first calculated.

I'm getting strange results in SO₂ mode; how do I know if my instrument is working correctly?

We recommend that you always start in SO_2 mode with the electrode disconnected. After pressing ENTER to confirm SO_2 mode, *then* connect the SO_2 electrode. This avoids incorrect background settings that may make the instrument <u>appear to be insensitive or unresponsive</u>.

For SO_2 measurements with the SC-300, there are several quick tests you can do to make sure the instrument is not faulty.

- 1. Be sure the battery is good per the manual's instructions.
- 2. Remove the electrode to expose the BNC connector at the back of the instrument. Turn on the instrument and select SO_2 mode. Short out the terminals on the connector, using a paper clip or similar metal piece to touch the center pin of the connector to its outer metal sheath. The device should indicate "STOP" with its red LED and buzzer or beeper. If this does not happen there may be a problem with the instrument; contact us for more information.
- 3. Connect the electrode and put it in about 20 mL of distilled water; add about 1 ml (half a bulb squeeze) of each of the acid solution and the reactant and swirl in the usual way keeping con-

stant motion. The instrument may or may not indicate STOP as above. If it does not, add a drop of the SO₂ Titrant solution. This should make the STOP condition occur. [If it doesn't you may have an electrode problem read in the next section below how to fix this.] Now add one drop of a concentrated sulfite solution (1-10% is fine) and verify that the STOP signal ends and the PROCEED light illuminates. If this happens, your electrode is probably OK as well.

4. Finally, you can check your SO₂ reagents with the ascorbic acid (vitamin C) test located on our website <u>vinmetrica.com/FAQ</u>.

<u>Preconditioning of the electrode</u> Note: as of October 2013, all SO2 electrodes are shipped pre-conditioned, so this procedure should not normally be needed. Sometimes when the SO₂ electrode is first shipped it can be shipped "hot". A hot electrode is one that is overly sensitive as indicated by high readings (and STOP conditions) when inserted into solutions that should be giving no signal, like pure water or water plus reactant and acid with a drop of 10% KMBS added. If your electrode shows this behavior it generally can be fixed by pre-conditioning as follows:

- 1. First, turn the power on and press the MODE button until the instrument is in SO₂ mode, then press ENTER. Attach the SO₂ electrode to the SC-300 analyzer. Put 20 mL of distilled water (deionized water) in a beaker and add half a bulb squeeze of the acid solution, half a bulb squeeze of the reactant solution and let the SO₂ electrode sit in this solution.
- 2. Then add one drop of 10% potassium metabisulfite solution (KMBS) to the beaker with the electrode in it. Swirl gently. The instrument LCD should now read 0.0 (in units of nanoamps) or close to it. If it is reading significantly higher than 20 on the screen, let the electrode sit in the solution for half an hour.
- 3. After half an hour rinse the electrode with distilled water. Put it in about 20 mL of distilled water. Again add half a bulb squeeze of each of the acid solution and the reactant and swirl in the usual way keeping constant motion. The instrument may or may not indicate STOP as above. If it does not, add a drop of the SO₂ Titrant solution. This should make the STOP condition occur. [If it doesn't you may have an electrode problem]
- 4. Now add one drop of KMBS solution (1-10% is fine), swirl and verify that the STOP signal ends and the PROCEED light illuminates. If this is the case, your electrode has been conditioned. If the electrode is still "hot" and the PROCEED light does not illuminate, let it sit in 20 mL of distilled water with a half bulb full of the acid solution for a few hours. Now repeat the test from step 3. If it works, great! If not, call us and we will try to troubleshoot or replace your electrode.

Technical assistance: info@vinmetrica.com tel. 760-494-0597