

Vinmetrica SC-300 Kit™ User Manual

The Vinmetrica SC-300 is a simple and robust device that provides high accuracy in determination of sulfite (SO₂), pH and titratable acidity (TA) levels in wines, ciders, and other liquids. These are essential parameters to control in the effort to make high quality wines.

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Materials provided in the kit:

- 1. Vinmetrica SC-300 SO₂/pH controller unit (Part number SC-300-1)
- 2. SO₂ Electrode (Part number SC-100-3), blue polycarbonate housing
- 3. pH Electrode (Part number SC-200-7), blue (pre-2021) or grey (since Jan 2021) polycarbonate housing, with storage bottle and storage solution
- SO₂ Reagent set (Part number SC-100-2): SO₂ Titrant Solution (0.0156N) SO₂ Acid Solution

SO₂ Reactant Solution

- pH/TA Reagent Set (Part number SC-200-8): pH 4.01 Reference Solution pH 7.00 Reference Solution TA Titrant (0.13 N NaOH)
- 6. Two 5 mL syringes
- 7. Two 3 mL polyethylene transfer pipettes
- 8. One 25 mL serological pipette
- 9. One 5mL serological pipette
- 10. 100 mL polypropylene beaker



Figure 1. The SC-300 instrument with the SO_2 and pH/TA electrodes.

Things you will need:

- 1. Two standard AA batteries (alkaline type).
- 2. Distilled water (aka purified water by deionization), which can be found at most grocery stores.
- 3. (Optional but highly recommended) It's handy to have a wash bottle for rinsing. Rinse bottle available from Vinmetrica (Part number SC-100-17, or included in the Deluxe Lab Accessory Kit).
- (Optional) Deluxe Lab Accessory Kit which includes: magnetic stirrer, burette (10mL or 25mL), lab support stand, electrode holder, and wash bottle. Available from Vinmetrica (Part Number SC-300-9).
- 5. (Optional) Sodium Hydroxide solution, 1N concentration (if you want to do <u>total</u> SO₂). Available from Vinmetrica (Part number SC-100-7)

Why Test for SO₂, pH and TA?

Testing for sulfite (SO₂) is crucially important for making sure your wine does not spoil by oxidation or from microbial growth. By monitoring your SO₂ 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 SO₂" 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 0.5 to 0.8 ppm (mg/L) following secondary 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 Analyzer Kit. Winemaker Magazine's Sulfite Calculator at <u>https://winemakermag.com/1301-sulfite-calculator</u> 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 also 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:

<u>Sulfite (SO₂)</u>: The SC-300, with the SO₂ electrode and reagents provided, can be used to determine sulfite (or SO₂) 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.

 $SO_2 + I_2 + H_2O \rightarrow 2I^- + SO_3 + 2H^+$ 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.

<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 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* (or 7.0 if preferred, see below) 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).

<u>Potential measurements:</u> In firmware versions 3.1.1 and higher, the SC-300 can display the voltage reading on 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 some countries, pH 7.0 is used as the endpoint; see Instrument Operation, Step 5 (page 7).

Setup:

Setting up the SC-300 for the first time:

- 1. The SC-300 (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, then close the housing. If desired, you can prop the unit up using its folding stand.
- 2. <u>Low Battery Detection:</u> When the battery level is getting low, the instrument shows a low battery icon on the upper left side of the display but continues to operate without impairment of 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. <u>Auto Shut-off:</u> The SC-300 shuts off after 30 minutes. If this happens unexpectedly, just press the POWER button to resume from where you were.
- 4. <u>Electrodes:</u> 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 ensure proper function.



Figure 2. Attach the SO2 electrode to the connector on the SC-300.



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 (most electrodes are shipped without one). This cover need not be used routinely. Put the electrode on its side, or hang it from an electrode stand 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. Attach the SO₂ electrode to the proper

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connector. In some cases this is a BNC-style plug as in figure 2; on older units this may be a phono style plug (not pictured). *Electrode care*: When done, always rinse with DI water and let air dry. **There is no need to store the SO₂ electrode in any kind of solution.**

5. <u>pH electrode</u>: Attach the pH electrode via the BNC connector on the top of the SC-300 (Figures 3). The pH electrode is fragile and should always be handled carefully. Its approved temperature range is +1 to +60 °C. Do not use it outside this range. Remove the liquid storage bottle from the electrode by unscrewing the cap first, then gently removing the bottle and pulling off the cap. Rinse the electrode with 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. When finished using the electrode, rinse the electrode with DI water and gently blot or shake off excess water. Push the electrode through the hole in the cap about an inch and gently screw the bottle onto the cap so that the electrode is in contact with the solution in the bottle. **The pH electrode should always be kept in the liquid storage bottle with its pH Electrode Storage Solution (Part Number SC-200-10) when not in use.** We recommended replacing the pH Electrode Storage Solution once a year or when the solution becomes cloudy or moldy.

<u>NOTE:</u> see Appendix D for information about the newer, grey polycarbonate pH electrode.

Instrument Operation:

- 1. 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 determining ppm of SO₂ by titration (see below under 'Procedures') and the SO₂ electrode must be used. Make sure to attach the SO₂ electrode. *On older models, as a safety precaution, you must press the ENTER button when prompted after selecting SO₂ mode to confirm that the pH electrode is NOT attached before the mode will be enabled. On single connector units (pre-2014), the pH electrode can be temporarily misadjusted by connecting it to the instrument in SO2 mode.* You should see the display show a value less than 50 (usually 0.0) and the green "PROCEED" LED should be on.
- 3. <u>pH mode</u>: In this mode, the meter measures the pH. The pH electrode must be attached. *If the instrument has never been calibrated, the message "do cRL" scrolls across the screen, and you need to do a calibration before using this mode (see Calibration of pH below). We recommend re-calibrating the instrument for pH once each day of use.*
- 4. <u>Potential mode</u> (Firmware version 3.1.1 and higher) (pH LED flashing): In this mode the instrument displays the potential (in volts, version 3.1.1, or millivolts, v 3.1.2 and higher) coming from an electrode attached to the pH connector. This can be used with certain electrodes, for example, the Vinmetrica Dissolved Oxygen probe, ORP, potassium, or sodium electrodes, or to view the raw voltage reading of a pH electrode. In this mode, on older instruments, when the red STOP LED is illuminated, the values on the screen are negative; otherwise the values are positive. On newer instruments (version 3.2.d and higher), the sign of the mV reading is displayed on the screen directly. Latest firmware versions 3.1.3 and 3.2.E read a range of about +430 to -240 mV; older versions read about +330 to -330 mV.
- 5. <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 the TA endpoint (normally 8.2, but can be set to 7.0, see Appendix A, Test Mode, section 16), while the red "STOP" LED is lit if the pH is above the endpoint (see below under Measuring TA by Titration).
- 6. <u>CAL mode</u>: This is for calibrating the pH electrode, which must be attached. The display initially shows "*cRL*" 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.

7. 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.

8. (Optional) The magnetic stirrer (Included in the SC-300 Pro Kit, or bought separately) 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. Turn on the instrument and select SO₂ mode using the MODE button. On older instruments, you need to press ENTER to confirm selection of SO₂ mode (Figure 4). The display should show a value less than 20, usually 0.0. Now attach the SO₂ electrode. [You do not need to repeat this start-up sequence each time you do a test]



Figure 4. Once in SO₂ Mode the instrument may request that you press ENTER to activate SO₂ Mode.



Figure 5. Withdraw the titrant from its bottle using a clean 5 mL syringe. If you are using the glass burette, use the syringe to fill it.



Figure 6. Dispense 25 mL of your wine into the titration beaker using the 25 mL sampling pipette. Make sure this is clean before putting the pipette into your wine container!



Figure 7. The transfer pipette. One full squeeze of the transfer pipette in either the Acid solution or Reactant should be approximately 2 mL.

2. Fill the syringe by drawing up the SO₂ Titrant Solution (the bottle with the blue label) (Figure 5). 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.] Be sure to record your starting syringe volume.

If using a burette, use the syringe to dispense the SO₂ Titrant Solution into the top of the burette. Make sure the burette stopcock is in the closed position (i.e., the 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 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 (Figure 6). **NEVER pipette any reagents by mouth! Make sure the pipette you are using is completely clean before submerging into your wine sample.** (We recommend using a 50% ethanol solution, <u>Do Not</u> use a sulfite solution to sanitize before sampling)
- 4. Using the transfer pipettes (Figure 7), add about 2 mL SO₂ Acid Reagent and 2 mL SO₂ Reactant Solution to the titration beaker (be sure to label the transfer pipettes to avoid cross contamination, we recommend "A" for Acid and "R" 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 out with distilled water and let air dry. **Caution: the SO₂ Acid Solution 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. (Optional) If you are using the magnetic stirrer, place the stir bar in the beaker, place the beaker on top of the magnetic stirrer and turn the magnetic stirrer on. The magnetic stirrer operates at a suitable preset speed. Make sure your electrode is not struck by the spinning stir bar. To prevent this, we recommend using the 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). 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 (Figure 8).
- 7. (Optional) If you are using the magnetic stirrer, ensure the tip of the electrode is completely submerged to just above the circulation gaps but above the level of the stir bar (approximately half an inch from the bottom of the titration beaker). If you are using the Electrode Holder adjust it to a similar level. If needed, you can add up to 25mL of DI water to raise the liquid level.

- 8. Verify that the current is less than 50 and the green ("PROCEED") LED is lit (Figure 10). 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.
- 9. Titrate the sample by adding the SO₂ Titrant drop wise from the syringe (Figure 8) or from the burette (Figure 9), 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. <u>Be sure to maintain stirring or swirling throughout the entire procedure.</u> If the magnetic stirrer turns off, turn it back on.



Figure 8. Manual stirring technique. Hold the electrode against the side of the titration beaker and swirl gently; add SO₂ Titrant with other hand.

Figure 9. Automated stirring technique. Turn on the magnetic stirrer; add SO2 Titrant by slowly opening the burette stopcock valve.

Figure 10. Make sure that the "PROCEED" LED is lit. You should be reading close to 0.0 when you first start.

Figure 11. 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. Take the endpoint as the first addition of Titrant that causes the display to exceed 50, and 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 SO_2 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
$$SO_2 = 20 * V$$
 (i.e. 20 times V)

Measuring Total SO₂ by Titration (optional - requires 1N NaOH):

- 1. Place 25 mL wine or must in the titration vessel (Figure 6).
- 2. Add 10ml 1N Sodium Hydroxide (NaOH) (Part number SC-100-7) and mix well. Let stand approximately 10 minutes.
- 3. Using the transfer pipettes, add approximately 8 mL of the SO₂ Acid Solution and 2 mL of the SO₂ Reactant Solution to the vessel.
- 4. Proceed from step 5 in the Free SO₂ procedure above. The result calculated will be total SO₂, rather than free SO₂ in parts per million (ppm) or mg/L.

Calibration of pH:

- 1. Be sure the pH electrode is attached to the unit, then select CAL mode by pressing the MODE button until the "CAL" LED illuminates.
- 2. Choose a calibration set of solutions 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 (you can use the Reference Solution cap) containing the pH 7.00 reference solution. Gently stir or agitate the solution continuously.

^{1 64 [}mg SO2/mmol SO2] * V [mL] * N [meq/mL] * 1000 [mL/L] 2 [meq/mmol SO2] * S [mL]

<u>IMPORTANT!</u> It's usually best to keep the electrode moving in the solution during calibration and measurement; letting it sit static may cause drift and inaccurate readings!

- 4. 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 7.40 when the pH electrode is sitting in the pH 7.00 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.
- 5. 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 cAL' 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 cAL' 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 4.01 reference solution). Repeat the process to get a second 'Lood cRL' message. Exit into pH or TA mode.

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 and stir or agitate gently in a constant manner. Be careful not to let the electrode strike any surfaces.
- 4. Allow the pH reading to stabilize, stirring or gently agitating continously. Typically this takes about 10-15 seconds. Read the pH value on the display.

Measuring Titratable Acidity (TA) by Titration:

1. <u>Sample pretreatments</u>: If you are working with a sample of must, we recommend homogenizing your sample in a blender before proceeding. 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.

If your sample has appreciable outgassing of CO_2 , as in a sparkling wine or newly-fermenting must, degas the sample by repeated shaking, then venting, in a closed small jar or sample bottle until no more gas evolves.

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, you can 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 and attach the pH electrode. If necessary, calibrate it as described above. Select TA mode with the MODE button.
- 5. (Optional) If you are using the magnetic stirrer, place the stir bar in the beaker, place the beaker on top of the magnetic stirrer, and turn on the magnetic stirrer. Be sure the stir bar will not strike the electrode in the following steps. (Figure 13)
- 6. Rinse the electrode briefly with DI water. Insert the electrode into the beaker so that the tip is fully submerged to just above the circulation gaps (cutouts at the tip of the electrode). You may add up to 15 mL more DI water to raise the liquid level if needed.
- 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 beaker with the remaining fingers so that the two move together while swirling (See Figure 12).



Figure 12. Manual stirring technique. Hold the electrode against the side of the titration beaker and swirl gently; add TA Titrant with other hand.

Figure 13. Automated stirring technique. Turn on the magnetic stirrer; add TA Titrant by slowly opening the burette stopcock valve.

Figure 14. Make sure that the green "PROCEED" LED is lit. You should be reading a pH close to what you expect your wine is at.

Figure 15. 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 (Figure 14). If the pH is greater than this, there is an error. Check your sample and setup.
- 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 the TA endpoint (8.2 or 7.0, depending on your setup; see Appendix A -Test Mode, section 16) for longer than 15 seconds. The red "STOP" LED and the beeper will provide additional indication of the endpoint (Figure 15). 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, 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. Electrodes can be left attached to instruments that have two connectors. On older models with a single connector, always remove the pH electrode when shutting down.
- 2. Rinse the SO₂ electrode and syringe with distilled water. Let air dry.
- 3. Rinse the pH Electrode and store it in the pH Electrode Storage Solution vial as directed under 'Setting up the SC-300 for the first time' item 6.
- 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.
- 6. For prolonged storage, remove the batteries from the unit.

Technical assistance: info@vinmetrica.com tel. 760-494-0597 x102

WARRANTIES AND LIABILITIES

- 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 pH electrode is warranted for 1 year. The reagents are warranted to perform as described herein up until any stated expiration date or 6 months after purchase, whichever is later. 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.

Vinmetrica

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Appendix A - Test Mode

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

- To enter Test Mode, first turn off the instrument. Now press and hold the POWER button about 2-3 seconds, then release. You should now see the firmware version number. The latest version 3.2.E, or 3.1.3 on older models.
- 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.
- 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.
- 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.

Yellov LEDs	Section	Equipment Required	Description
0000 0000	1. Version	None.	The version number of the instrument firmware is displayed, e.g. 3.1.2
0000	2. Burn-in	None	The instrument goes through a continuous "burn-in" cycle, exercising 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 14.000 are shown as "". Readings below 0.000 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.
0000	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)

Yellow LEDs	Jechon	Equipment Required	Description
	7.DAC Test	None. Disconnect probe.	The Digital-Analog Converter (DAC) is cycled through its 32 levels.
0000	8.Battery Voltage	None.	The battery voltage is displayed as X.XX volts.
0000	9.Character Set	None.	Every ASCII character (space) to \sim is displayed. Due to the limitations of the 7- segment format, some characters are not used by the software.
0000	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	None. [only firmware v3.0.6 and later]	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	None. [only firmware v3.0.6 and later]	Displays "PrE55 EntEr"; Press ENTER to reset pH CAL parameters and DAC to default values. Message "Good cRL r5t" then scrolls.
0000	14. pH DAC Set	None. [only firmware v3.1.2 and later]	Displays "Ph dRc 5EE" then displays the DAC index for pH bias voltage, followed by the voltage value itself. Default is 16, range 0 - 31. Press ENTER to increase the DAC value by 1; press MODE to decrease by 1. Can be used to change the baseline pH value. Note: values outside the range 12-20 are for diagnostic purposes only and will not be retained after exiting Test Mode.
000	15. SO ₂ Baseline	None. Disconnect SO ₂ electrode [only fw v3.1.0 and later]	Sets baseline value for SO ₂ mode. Let message scroll 5 sec., then press ENTER. Normal values are 0.02 to 0.10
000	16. TA endpoint	None [only firmware v3.1.2 and later]	Displays " 5EE ER PE " then displays TA endpoint value. Press ENTER to toggle between default of 8.2 (USA standard) or 7.0 (European standard).

Appendix B - Sulfite and TA Adjustments

Using the Winemaker Magazine Sulfite Calculator:

Winemaker Magazine's Sulfite Calculator at <u>https://winemakermag.com/1301-sulfite-calculator</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. Enter the "volume of wine to be corrected". Choose liters or gallons; we prefer "liters" because the answer is returned in mL.

4. Enter the wine's pH. If you know the % Alcohol by volume and temperature, enter these also (but you don't have to).

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

6. Leave the "**Desired** level of free SO₂"at 0 for now. If you want, enter a different value for "Desired molecular SO₂"; we usually use the default of 0.5 mg/L

7. Now look below to the 'Notes:' section. You should see the message "1. The recommended level of free SO₂ for this type of wine, molecular SO₂ & pH is: [your value] mg/L. Redo the calculation using this value for desired free SO₂ level, if required." Enter this value in the "Desired level of free SO₂" box.

8. Press 'Calculate' to get the correct "Amount of sulfite to be added:". The value will be in mL or fluid ounces of 10% sulfite solution, or in grams if you use sulfite powder as your sulfite additive.

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 SO₂ measurement after waiting at least 30 minutes (we recommend waiting overnight if practical). If the measurement matches the 'Desired level of free SO₂' then you are done, otherwise make incremental additions and repeated SO₂ measurements until you reach your desired level.

Adjusting TA in your wine:

If your wine's pH is too high, and TA level is too low, you may want to increase the acidity. There are various ways to do this. We recommend adding tartaric acid; for non-grape wines, fruit acids are sometimes used. Use caution, for if overdosed with added acid, the wine becomes too tart. Remember it's always easy to add more acid, while it's not so easy to reduce acidity. 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 about 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 decreasing 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.

Also, check out books and discussions about winemaking techniques on Daniel Pambianchi's web page: <u>http://techniquesinhomewinemaking.com/</u>

Appendix C1 - Troubleshooting: pH and TA Issues

Check out Troubleshooting Guide on line at https://vinmetrica.com/troubleshooting-guide/

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

When calibrating your pH electrode, remember these points:

FIRST, be sure the pH electrode has been stored at least 24 hours in a proper electrode storage solution (Vinmetrica's product is 3M potassium chloride in 10 mM potassium hydrogen phthalate; other similar products may be used). The entire bottom 1 inch of the electrode needs to have been submerged for at least 24 hours. If this has NOT happened, wait until it has!

SECOND, Remember: It's usually best to keep the electrode moving in the solution during calibration and measurement; letting it sit static may cause drift and inaccurate readings!

ALSO:

- 1. The displayed pH may not be correct until <u>after</u> you press ENTER.
- 2. If the instrument signals stable pH but displays "**bRd cRL**" 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 outside of the default tolerance of 0.5 pH (but not more than 1.5 pH units), you can change the baseline of the pH value. See Test Mode, stage 14 in Appendix A (available in firmware 3.1.2 and higher). Call or Email us for help if you need it.
- 6. 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/or is difficult to calibrate?

AGAIN, be sure the pH electrode has been stored at least 24 hours in a proper electrode storage solution (Vinmetrica's product is 3M potassium chloride in 10 mM potassium hydrogen phthalate; other similar products may be used). The entire bottom 1 inch of the electrode needs to have been

submerged for at least 24 hours. If this has NOT happened, wait until it has!

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 3M 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 30 secs., rinse with distilled water, then immerse in 0.1M NaOH (you can use a little of your TA Titrant for this) for another 30 sec. Cycle the electrode through these solutions a few times (rinsing with DI water in between), then rinse and check for performance in pH buffer 4.00 and 7.00.

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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 (i.e. 6.5 to 7.5). If out of this range, the meter may be 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.

pH test with cream of tartar:

A quick way to check your calibration and pH accuracy is to measure the pH of a saturated solution of cream of tartar which has a pH of 3.55 at 25 degrees Celsius:

- 1. Get pure cream of tartar (grocery store stuff is fine, provided it's pure), or reagent grade potassium hydrogen tartrate, also known as potassium acid tartrate or potassium bitartrate. Call it KHT for short.
- 2. Place about 1/8 teaspoon of KHT in 20 mL of distilled water. Mix well for about 30 seconds. You want to be sure the solution is saturated, i.e., everything that can dissolve, has dissolved. There should be some undissolved solid left.
- 3. Decant or filter the solution off the solids if possible.
- 4. This solution has a standard pH of 3.55 at 25 degrees C (78 degrees F). It should be within 0.02 pH of this value at temperatures from 20 to 30 degrees Celsius. We usually are OK with a value between 3.50 and 3.60. Discard after 24 hours.

Appendix C2 - Troubleshooting: SO₂ Issues

Check out Troubleshooting Guide on line at https://vinmetrica.com/troubleshooting-guide/

How stable are the reagents?

The SO₂ reagents and the pH/TA reagents are all warranted to last for 6 months and have a "use-by" date on the label that is 2 years from date of manufacture. Make sure they are 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! If the solutions become cloudy or show signs of microbial growth, they should be replaced.

How can I check the accuracy of my reagents?

1. It's rare that the SO₂ reagents go bad, but if you are concerned about it you can run the 'Ascorbic Acid Test' method located in the FAQ section of the website <u>https://vinmetrica.com/wp-content/uploads/2012/04/Ascorbate_stdization_procedure-500-mg.pdf</u> to check your SO₂ reagents.

2. The pH test with cream of Tartar mentioned above is a good way to check the accuracy of the calibration performed with your pH reference solutions.

3. If you are worried about your TA Titrant, you can run the 'KHP test', <u>https://vinmetrica.com/wp-content/uploads/2012/04/KHP_standardization.pdf</u> also located on the Vinmetrica website in the Support section 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 3 years ago may be bad now!

2. Make sure that you stir your wine well 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?

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. 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 SO₂ Acid Solution and the SO₂ Reactant Solution 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, with a current of 100-300 nA. [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 test passes, your system is detecting the titration endpoint correctly. If not, if you have a male RCA-terminated electrode (see below), try very slightly crimping the outer grounding ring (using a pair of pliers or similar tool) on the male terminated plug to ensure good contact with the female connector. Then repeat the above test.



more information.

3. The platinum wires of your SO₂ electrode could be dirty (crust, debris, etc.) even though you may not be able to see anything. First, soak the SO₂ electrode in your Acid Solution for about 10 minutes and rinse with DI water. Using the back edge of a pocket knife or something similar, very gently scrape the two platinum wires, being sure not to bend or break them. Thoroughly rinse with DI water and try your test again.

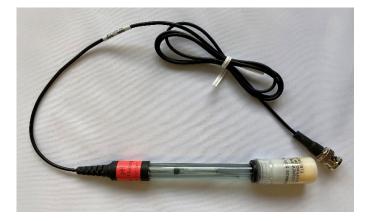
4. If the above tests don't work, remove the electrode from the connector at the back of the instrument. Turn on the instrument and select SO_2 mode. Short out the terminals on the connector, using a paper clip or similar metal piece to touch the center hole of the connector to its outer metal sheath. The device should indicate "STOP" with its red LED and buzzer or beeper, and the current should go to 1999. If this does not happen there may be a problem with the instrument; contact us for

5. Finally, you can check your SO₂ reagents with the ascorbic acid (vitamin C) test located on our website <u>https://vinmetrica.com/wp-content/uploads/2012/04/Ascorbate_stdization_procedure-500-mg.pdf</u>.

<u>Appendix D – 2021 pH electrode</u>

As of January 6, 2021, Vinmetrica is providing a new type of pH electrode. These are identical in operation and use to the older style electrodes, but have a few physical differences.

1. They are grey in color, though they are made of the same sturdy polycarbonate housing material.



2. They have a removable sensor protector. This can be unscrewed to better access the vicinity of the glass bulb (pH sensor) for cleaning. However, you do not want to clean the glass bulb itself by physical contact in any way – contact us if you have questions. Don't try to use the electrode without its protector in place – the glass bulb is very fragile.





3. They have a porous ceramic frit for a reference junction. The earlier models used a polymeric material. This does not require any additional attention on your part, but we found that this reference junction is less affected by lack of stirring, and the pH accuracy below pH 3.2 is slightly improved.

Technical assistance: info@vinmetrica.com tel. 760-494-0597 x102