

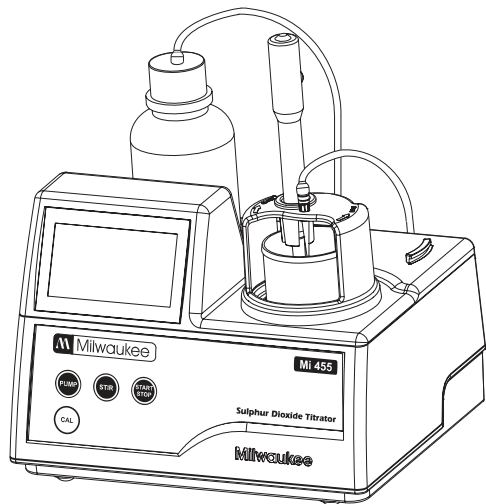
INSTRUCTION MANUAL

Milwaukee Wine Lab Minititrator



■ Mi455

Free & Total Sulphur Dioxide



 Milwaukee

www.milwaukeeinst.com

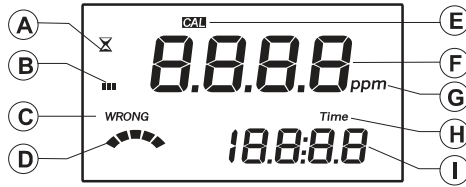
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FUNCTIONAL DESCRIPTION

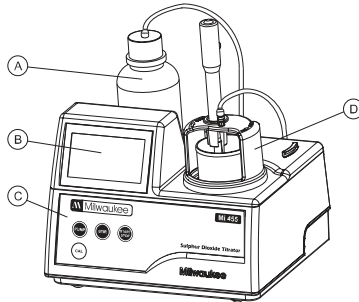
DISPLAY

- A. HOURGLASS ICON
- B. BATTERY STATUS ICON
- C. ERROR MESSAGE
- D. MEASURE STATUS
- E. CALIBRATION MODE INDICATOR
- F. MAIN DISPLAY
- G. MEASUREMENT UNIT
- H. TIMER MODE INDICATOR
- I. SECONDARY DISPLAY



FRONT PANEL

- A. TITRANT BOTTLE
- B. LIQUID CRYSTAL DISPLAY (LCD)
- C. KEYPAD
- D. ELECTRODE HOLDER

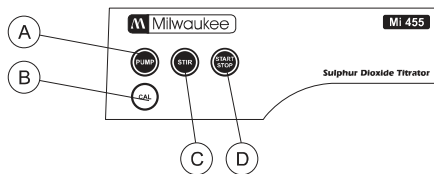


REAR PANEL

- E. ORP ELECTRODE
- F. PERISTALTIC PUMP TUBE
- G. BEAKER
- H. PERISTALTIC PUMP
- I. BNC ELECTRODE CONNECTOR
- J. FUSE
- K. POWER CABLE CONNECTOR
- L. POWER SWITCH

KEYPAD

- A. PUMP - to start/stop the pump
- B. CAL - to enter pump calibration mode
- C. STIR - to start/stop the stirrer while in measurement or purging mode
- D. START STOP - to start/stop titration or pump calibration



GENERAL DESCRIPTION

The **Mi455** is a low-cost, easy to use, microprocessor-based automatic titrator. It has a simple and reliable peristaltic pump that ensure high dosing repeatability. By performing pump calibration with the provided Milwaukee standards, the instrument accuracy is assured. The instrument comes with a pre-programmed analysis method designed for Free and Total Sulphur Dioxide measurements on wine samples. The instrument has a powerful and effective built-in algorithm to analyze the shape of the electrode response and to determine the reaction completion. This algorithm automatizes the analysis, makes all the necessary calculations and assures a simple and effective interface for the user.

By simply pressing the START STOP button, the instrument will automatically make the titration up to the equivalence point. The result is immediately displayed in convenient units, then the instrument is ready for another titration.

SIGNIFICANCE OF USE

An important reason for adding SO_2 is to avoid oxidation. When there is oxygen around, SO_2 itself becomes oxidized before phenol compounds in the wine, and so acts as an oxygen scavenger. Also SO_2 suppresses the activity of enzymes that cause browning and other problems.

What is really protecting your wine is molecular SO_2 . When you add SO_2 , depending of circumstances, some of it immediately becomes bound. The relationship between the amount of added SO_2 and the amount of SO_2 remaining free is complex. It is clear, however, that it is largely governed by the total SO_2 content of the wine. The rate of binding decreases as the free SO_2 concentration increases. The exact relationship between free and bound (total - free) SO_2 will vary from wine to wine.

Below 30-60 ppm, 33% to 50% of SO_2 addition becomes bounded. What remains is called "free" and it is divided in two parts. The larger, and relatively ineffective free part is

“bisulphite” (HSO_3^-). The smaller part of the free is the active molecular SO_2 . The amount of molecular SO_2 in your wine depends both on the level of free SO_2 present as well as pH. For instance, at pH 3.2, the amount of free SO_2 for 0.8 ppm molecular SO_2 is 22 ppm. At pH 3.5, you will need 43 ppm free - essentially double.

Free SO_2 concentration (ppm) for 0.8 ppm molecular SO_2 :

pH	30	31	32	33	34	35	36	37	38	39
Free SO_2	14	18	22	28	35	44	55	69	87	109

In most situations, 0.8 ppm molecular SO_2 during bulk storage and at bottling will provide you with adequate protection from oxidation and bacterial action. This includes prevention of malolactic bacteria as well.

It is important to remember that the amount of free SO_2 in the wine depends on three things: how much is added, how much was present before the addition and how much of your addition promptly becomes bound.

The level at which molecular SO_2 can be detected by the human senses is about 2.0 ppm. This is also the level which is needed for maximum protection of your wine. This is particularly true in the case of sweet, and most notably, botrytised wines.

The Milwaukee **Mi455** offers the possibility to test free or total SO_2 in all the wines including the red ones, that are difficult to test with manual methods because the color changes are hardly seen.

This meter is supplied with:

- Reagents set for 20 titrations
- Two 50 mL beakers
- Two 20 mL beakers
- Tubes set with cap
- ORP probe
- Stir bar
- Power cable
- One 230 mL bottle of Refill Solution
- One 1 mL syringe
- Instruction manual
- Warranty code

Note: Save all packing material until you are sure that the instrument works correctly.

Any defective item must be returned in its original packing.

PRINCIPLE OF OPERATION

Determination of sulphur dioxide in wine samples is made by titration of the sulphur dioxide present in wine with iodate. In this procedure an excess of iodine is added to the wine sample and it is titrated with iodate.

For precise results it is very important to know the exact sample volume, titrant volume and concentration.

The peristaltic pump has a good repeatability but the dosing volume depends on many factors as the diameter of the tube or the tube stretching. To compensate for all this errors, the pump need to be calibrated. The calibration of the pump is also needed in order to have high precision of the titrations.

The calibration procedure is in fact the analysis of a known solution. By doing this, the instrument makes a differential analysis between the standard and the wine sample. The pump volumetric debit and the real concentration of the titrant is compensated. Only the sample volume has to be precisely known.

SPECIFICATIONS

Range	0 to 400 ppm SO ₂
Resolution	1 ppm
Accuracy	5% of reading
Method	Ripper titrimetric method
Principle	Equivalence point redox titration
Sample volume	50 mL
ORP Electrode	MA924B/1 (included)
Pump debit	0.5 mL/min
Stirring speed	1500 rpm
Environment	0 to 50 °C (32 to 122 °F) ; max 95% RH non-condensing
Power supply	220V/50Hz; 10VA
Dimensions	208 x 214 x 163 mm (8.2 x 8.4 x 6.4") (with beaker)
Weight	2200 g (77,6 oz.).

Required Reagents

<u>Code</u>	<u>Description</u>	<u>Quantity/test</u>
Mi555-001	Standard (for Free & Total SO ₂)	50 mL
Mi555-002	Titrant (for Free & Total SO ₂)	
Mi555-003	Alkaline Reagent (for Total SO ₂)	5 mL
Mi555-004	Acid Reagent (for Total SO ₂)	5 mL
Mi555-005	Acid Reagent (for Free SO ₂)	5 mL
Mi555-006	Stabilizer (for Free & Total SO ₂)	1 packet

This instrument is in compliance with CE Directives.

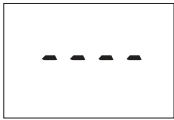
STARTUP

- Place the instrument on a flat table. Do not place the instrument on direct sun light.
- Connect the titrator to mains socket with ground connection and the correct voltage and frequency. See the label on the instrument rear for this.
- Place the peristaltic pump tube on the pump. See the Pump Tube Replacement section for the procedure.
- Remove the reagent bottle cap and place the bottle cap of the tubes set. Place the reagent bottle in the appropriate place on the titrator top.
- Connect the tubes with the peristaltic pump (inlet tube is connected with the reagent bottle, outlet tube is connected with the dosing tip).
- Turn the instrument ON using the power switch from the rear panel of the instrument and wait until it displays dashes.

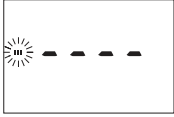
GUIDE TO DISPLAY CODES



This screen appears for a few seconds each time the instrument is turned ON.



Main screen display.

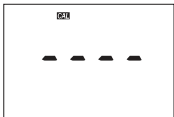


Main screen display with stirrer active.

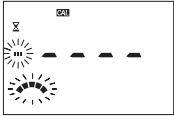


Purging mode message.

PUMP CALIBRATION MESSAGES



This screen appears each time the meter enters pump calibration mode. The meter is ready to start pump calibration by pressing the START STOP button.



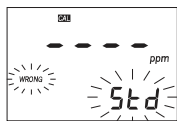
This screen appears while pump calibration is in progress. Pressing CAL or START STOP button, the minititrator returns to the main screen.



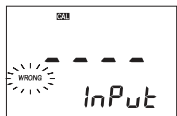
This screen appears for a few seconds before returning to the main screen, when pump calibration is done.



This error message appears when the sample concentration exceeds 400 ppm.

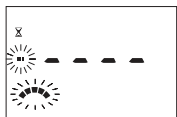


The used standard solution is wrong.



This error message appears when the input readings (mV) exceed the input limits (0-1000mV).

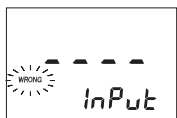
TITRATION MESSAGES



This screen appears each time a titration is running. When the START STOP button is pressed the minititrator returns to the main screen.



The titration result, expressed as concentration of sulphur dioxide in ppm (mg/L), is displayed at the end of the titration process. Press the START STOP button to return to the main screen.



This error message appears when the input reading exceeds the input limits (0-1000mV).



This screen appears when the sample concentration exceeds 400 ppm.

GENERAL TIPS FOR AN ACCURATE MEASUREMENT

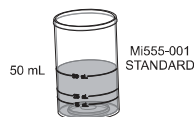
The instructions listed below should be carefully followed during testing to ensure best accuracy.

- Purge the peristaltic pump to have fresh titrant when starting a new analysis or calibration.
- Calibrate the peristaltic pump before performing an analysis.
- Analyze the wine immediately after the sample is obtained.

PUMP CALIBRATION PROCEDURE

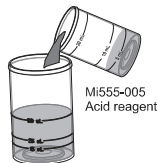
Warning: The calibration of the pump must be performed each time the pump tube, the reagent bottle or the pH electrode is changed. It is recommended to perform the pump calibration before each set of measurements.

- Using a clean pipette, fill the 50 mL beaker up to the 50 mL mark with **Mi555-001** Standard.



Note: Failure to use clean pipettes will result in erroneous readings.

- Place the stir bar in the beaker and put the beaker in the minititrator top.
- Fill the 20 mL beaker up to the 5 mL mark with the **Mi555-005** Acid Reagent and add the content to the 50 mL beaker.
- Place the beaker into the beaker holder.



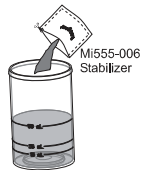
Place the probe holder on the top of the beaker and secure by turning clockwise.

- Open refill cap on the ORP electrode and immerse the electrode approximately 2 cm (0.8") into the sample to be tested. Do not touch stir bar with the tip of the electrode.

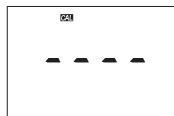
- Ensure there is titrant in the dosing tip. If not, hold the dosing tip over an empty container and press **PUMP** for a few seconds until the titrant is in the dosing tip.



- Add the contents of one powder packet of **Mi555-006** Stabilizer into the beaker.



- Press **CAL** button. The meter enters in pump calibration mode.



- Press **START STOP** in order to start the system calibration.



- At the end of the calibration, **DONE** appears for a few seconds and the meter returns to the measurement mode.



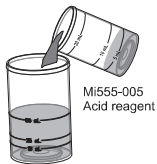
FREE SO₂ MEASUREMENT PROCEDURE

- Using a clean pipette, fill the 50 mL beaker up to 50 mL mark with the wine sample.

Note: It is important to be accurate in your measurements of Wine sample. Pipettes are recommended.



- Place the stir bar in the beaker and put the beaker in the minititrator top.
- Fill the 20 mL beaker up to the 5 mL mark with the **Mi555-005** Acid Reagent and add the content to the 50 mL beaker.



- Place the probe holder on the top of the beaker and secure by turning clockwise.
- Open refill cap on the ORP electrode and immerse the electrode approximately 2 cm (0.8") into the sample to be tested. Do not touch stir bar with the tip of the electrode.

- Insert dosing tip into holder. Ensure there is titrant in the dosing tip. If not, hold the dosing tip over an empty container and press **PUMP** for a few seconds until the titrant is in the dosing tip. The tip should be below the pump to ensure gravity flow.

Note: Be aware of any air bubbles in the tube it could effect the results.



- Add the contents of one powder packet of **Mi555-006** Stabilizer into the beaker.



- Press **START STOP** in order to start titration. The display will show "----" during titration, along with stirrer and pump tags blinking on the LCD.



- At the end of the titration, the Sulphur Dioxide concentration is displayed in mg/L (ppm).



TOTAL SO₂ MEASUREMENT PROCEDURE

- Fill the 50 mL beaker up to 50 mL mark with the wine sample.

Note: It is important to be accurate in your measurement of wine sample. Pipettes are recommended.

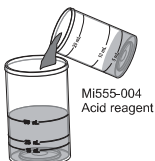


- Place the stir bar in the beaker and put the beaker in the minitrator top.

- Fill the 20 mL beaker up to the 5 mL mark with the **Mi555-003** Alkaline Reagent and add the content to the 50 mL beaker.



- Swirl the beaker and wait for 15 minutes.
- Using a clean pipette, fill the 20 mL beaker up to the 5 mL mark with the **Mi555-004** Acid Reagent and add the content to the 50 mL beaker.



- Open refill cap on the ORP electrode and immerse the electrode approximately 2 cm (0.8") into the sample to be tested. Do not touch stir bar with the tip of the electrode.

- Insert dosing tip into holder. Ensure there is titrant in the dosing tip. If not, hold the dosing tip over an empty container and press **PUMP** for a few seconds until the titrant is in the dosing tip. The tip should be below the pump to ensure gravity flow and minimize the chance of air bubbles in the tube.



- Add the content of one powder packet of **Mi555-006** Stabilizer into the beaker.



- Press **START STOP** button to start titration. The display will show “----” during titration, along with stirrer and pump tags blinking on the LCD.



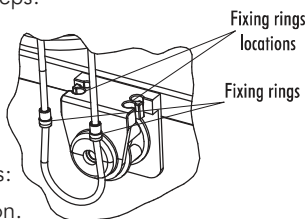
- At the end of the titration, the Sulphur Dioxide concentration is displayed in mg/L (ppm).



PUMP TUBE REPLACEMENT

To remove the tube of the peristaltic pump follow next steps:

- Detach the old tube system from the reagent bottle.
- Grasp one fixing ring of the peristaltic pump tube.
- Pull the tube until it's taken out from its location.
- Remove the other side of the tube.



To mount the new peristaltic pump tube follow next steps:

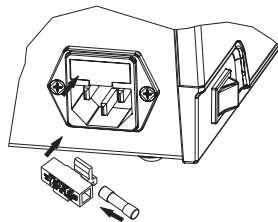
- Position one peristaltic pump fixing ring on its location.
- Stretch the tube over the peristaltic pump cylinders.
- Fix the second pump fixing ring on its location.
- Attach the tube to the reagent bottle.

Note: Purge the peristaltic pump until drops of reagent appears on the dosing tip by pressing the **PUMP** button.

FUSE REPLACEMENT

To change the fuse follow next steps:

- Disconnect the power cord from the rear panel of the instrument.
- Pull out the fuse holder located near the power cord connector.
- Replace the fuse with a similar one.
- Push the fuse holder with the fuse in the appropriate place.



ELECTRODE CONDITIONING & MAINTENANCE

PREPARATION PROCEDURE

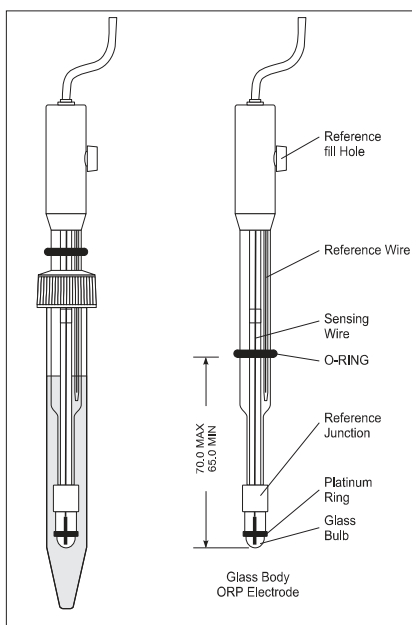
Remove the protective cap of the ORP electrode (MA924B/1).

DO NOT BE ALARMED IF SALT DEPOSITS ARE PRESENT. This is normal with electrodes. They will disappear when rinsed with water.

During transport, tiny bubbles of air may form inside the glass bulb affecting proper functioning of the electrode. These bubbles can be removed by “shaking down” the electrode as you would do with a glass thermometer.

If the bulb and/or junction is dry, soak the electrode in MA9015 Storage Solution for at least one hour.

If the filling solution (electrolyte) is more



than 2½ cm (1") below the fill hole, add **MA9011** 3.5M KCl Electrolyte Solution. For faster response, unscrew the fill hole screw during measurements.

STORAGE PROCEDURE

To minimize clogging and assure a quick response time, the glass bulb and the junction of the electrode should be kept moist and not allowed to dry out.

Replace the solution in the protective cap with a few drops of **MA9015** Storage Solution or, in its absence, Filling Solution (**MA9011**). Follow the Preparation Procedure before taking measurements.

Note: NEVER STORE THE ELECTRODE IN DISTILLED OR DEIONIZED WATER.

PERIODIC MAINTENANCE

Inspect the electrode and the cable. The cable used for connection to the instrument must be intact and there must be no points of broken insulation on the cable or cracks on the electrode stem or bulb. Connectors must be perfectly clean and dry. If any scratches or cracks are present, replace the electrode. Rinse off any salt deposits with water.

Probe Maintenance

Refill the reference chamber with fresh electrolyte (**MA9011**). Allow the electrode to stand upright for 1 hour. Follow the Storage Procedure above.

CLEANING PROCEDURE

- *Wine deposits* Soak in Milwaukee **MA9016** cleaning solution for 1 hour
- *Wine stains* Soak in Milwaukee **MA9016** cleaning solution for 1 hour

IMPORTANT: After performing any of the cleaning procedures, rinse the electrode thoroughly with distilled water, refill the reference chamber with fresh electrolyte and soak the electrode in **MA9015** Storage Solution for at least 1 hour before taking measurements.

ACCESSORIES

REAGENT SETS

MA9011	Electrode filling solution (230 mL)
MA9015	Electrode storage solution (220 mL)
MA9016	Cleaning solution (220 mL)
Mi555-001	Calibration standard (500 mL)
Mi555-002	Titratant solution (100 mL)
Mi555-003	Alkaline reagent (100 mL)
Mi555-004	Acid reagent for Total SO ₂ determination (100 mL)
Mi555-005	Acid reagent for Free SO ₂ determination (100 mL)
Mi555-006	Stabilizer reagent (40 pcs.)

OTHER ACCESSORIES

MA924B/1	ORP probe with 1 m cable
Mi0009	Stir bar (5 pcs.)
Mi0010	Titratant tube (2pcs.)
Mi0020	Beaker 50 mL (4 pcs.)
Mi0021	Beaker 20 mL (4 pcs.)

Recommendations for Users

Before using this product, make sure that it is entirely suitable for your specific application and for the environment in which it is used.

Operation of this instrument may cause unacceptable interferences to other electronic equipments, this requiring the operator to take all necessary steps to correct interferences.

Any variation introduced by the user to the supplied equipment may degrade the instrument EMC performance.

To avoid damages or burns, do not put the instrument in microwave ovens. For yours and the instrument safety do not use or store the instrument in hazardous environments.

For your Safety don't use or store the instrument in hazardous environments. To avoid damages or burns, do not perform any measurement in microwave ovens.

WARRANTY

This instrument is warranted against defects in materials and manufacturing for a period of 2 years from the date of purchase. Electrodes are warranted for 6 months.

If during this period the repair or replacement of parts is required, where the damage is not due to negligence or erroneous operation by the user, please return the instrument, electrode and probe to either distributor or our office and the repair will be effected free of charge.

Damage due to accidents, misuse, tampering or lack of prescribed maintenance is not covered by the warranty.

Milwaukee/Martini instruments reserves the right to make improvements in design, construction and appearance of its products without advance notice.

THANK YOU FOR CHOOSING



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