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# Orion product and application guide to winemaker's chemistry analysis

Recommended electrochemistry instrumentation and detailed application notes for winemaking

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# Chemistry analysis throughout the winemaking process

From grape to glass, great winemaking starts with a commitment to quality. Wine's unique attributes, like color, taste and smell, are all highly dependent on a series of chemical reactions and equilibriums throughout the various stages of winemaking. Monitoring and controlling these chemical reactions is part of the science and artistry of producing an exceptional, balanced glass of wine.

| Winemaking Stage | Winemaking Process | Relevant Chemistry Analysis |
|---|---|---|
| Pre-fermentation processes | Monitoring grapes for harvest | Titratable acidity<br>pH monitoring |
| | Crush | Titratable acidity<br>Ammonia Nitrogen<br>pH monitoring |
| | Must adjustment | Titratable acidity<br>pH monitoring<br>Sulfer dioxide<br>Ammonia Nitrogen |
| Fermentation processes | Maceration | Temperature<br>pH monitoring<br>Ammonia Nitrogen<br>Sulfer dioxide |
| Post-fermentation processes | Clarification | Turbidity/Clarity measurement<br>Sulfur Dioxide |
| | Aging<br>Racking | Turbidity/Clarity measurement<br>Sulfur dioxide<br>Dissolved oxygen monitoring in wine tanks/barrels<br>pH monitoring<br>Titratable acidity<br>Temperature |
| | Filtration and bottling | Dissolved oxygen monitoring in wine bottles<br>Turbidity/Clarity measurement<br>Sulfur dioxide<br>Titratable acidity<br>pH |

# Detailed application notes for winemaking

# Our experience makes the difference

Ensuring your winery is equipped with reliable, easy-to-use instrumentation helps protect the quality of your product. This guide includes detailed application notes for the various stages in the winemaking process, as well as the recommended electrochemistry instrumentation, accessories and solutions.

We've been delivering liquid sensing solutions that make customers complex measurements routine to the pharmaceutical, food and beverage, and chemical industries for over 50 years. See what our durable, easy to use Thermo Scientific™ Orion™ meters and accurate, reliable sensors can do for your winemaking operation.

Depending on your sample throughput needs and budget, we have included application notes for our automated titrators, as well as, electrochemistry meters for manual titrations.

Our automated titrators can help improve your titrations by simplifying your process and helping to deliver consistently reliable results. Just program it once and our titrators take care of the rest - including addition of titrant, endpoint determination, results calculation, and data logging.

1. Start with an automated titrator or meter for pH, titratable acidity and ammonia nitrogen analysis.
2. Add an ORP electrode for sulfur dioxide analysis.
3. For dissolved oxygen analysis, select a convenient portable meter kit.
4. To measure the wine's visual clarity, add a turbidity measurement meter kit.

# Great wines start with great chemistry

## Analysis tools that make the difference you can taste

Ensure product quality and exceptional taste throughout the winemaking process with Thermo Scientific™ Orion™ automated titrators, pH and ion selective electrodes, meters and solutions. Our instruments and solutions have been delivering accurate measurements of critical parameters for over 50 years. Thermo Scientific Orion analysis instruments combine excellent meter features with proven electrode technology to provide a complete package to meet your demanding quality standards. Choose from an assortment of standard measurement kits below, or pick-and-choose from our selection of individual products based on the type of analysis you are performing.

### Dissolved Oxygen Analysis

Oxygen exposure can cause adverse effects during the winemaking process, including browning, aroma and flavor modification, and structural softening. For these reasons, dissolved oxygen (DO) measurement is extremely important for wineries. Optical DO sensor technology improves measurement efficiency with no required warm up time, maintenance solutions or stirring.

**Recommended product: Thermo Scientific™ Orion Star™ A323 Dissolved Oxygen (DO) Meter Kit - STARA3235**

This dissolved oxygen meter kit includes everything you need to start testing the oxygen exposure of wine samples, including a dissolved oxygen meter, optical DO sensor and portable carrying case.

### pH and Ammonia Nitrogen Analysis

pH and ammonia nitrogen are critical measurements that affect the fermentation process and flavor of wine. pH strongly relates to a wine's stability and quality, while the level of nitrogen during fermentation affects the taste. Titratable acidity is an indicator of how acidic the final product will taste. Ammonia nitrogen is a major component of assimilable nitrogen, which is important to yeast growth and metabolism.

**Recommended Product: Thermo Scientific™ Orion Star™ A214 pH/ISE Meter pH and Ammonia Kit - STARA2146**

This pH and ammonia meter kit includes everything you need to start testing the ammonia nitrogen of wine samples, including a meter, pH electrode, ammonia electrode, temperature probe, stirrer probe, ammonia standards and solutions.

### Visual Clarity (Turbidity) Analysis

Accurate visual clarity measurements (turbidity) of wines at various stages of the winemaking process are critical for determining suitability for bottling. An infrared light source allows readings which are not affected by the deep color of red wines or the blush color of rosé wines.

**Recommended product: Thermo Scientific™ Orion™ AQUAfast™ Turbidity Meter Kit - AQ3010**

This turbidity meter kit includes everything you need to start testing the turbidity of wine samples, including a turbidity meter, primary standards to verify reporting accuracy, vials and portable carrying case.

### pH and Titratable Acidity

pH and titratable acidity (TA) are critical measurements that affect the fermentation process and flavor of wine. pH strongly relates to a wine's stability and quality, while the titratable acidity is an indicator of how acidic the final product will taste.

**Recommended Product: Thermo Scientific™ Orion Star™ T910 pH Titrator Kit - START9102**

This Orion Star T910 pH titrator kit is designed for dedicated acid-base titrations including titratable acidity of juices and wines. Titration techniques include equivalence point titrations and preset pH endpoint titrations for versatile sample analysis as well as a direct pH measure function. The kit includes automated titrator, ROSS Sure-Flow pH electrode, temperature probe and stirrer probe.

# Trust Thermo Scientific products for your wine analysis

In addition to the recommended standard measurement kits listed on the previous page, we offer additional products that address chemical analyses critical to winemaking, depending on your needs and budget. Refer to the chart below, or call your Thermo Scientific Orion distributor to find the best solution for your requirements.

Depending on your sample throughput needs and budget, we have listed our automated titrator and electrochemistry meters for manual titrations. Our automated titrators can help improve your titrations by simplifying your process and helping to deliver consistently reliable results. Just program it once and the titrator takes care of the rest - including addition of titrant, endpoint determination, results calculation, and data logging.

| Type of Analysis | Alternative Products | Relevant Features |
|---|---|---|
| pH and Total Acidity | Orion Star T940 all-in-one titrator kit - START9402 | Automated solution for total acidity titrations and direct pH analysis plus the ability to perform redox and ion titrations |
| pH, Total Acidity, Ammonia Nitrogen | Orion Star A211 pH benchtop meter kit – STARA2115 | Budget-friendly meter alternative for pH and mV analysis |
| | Orion Star A321 pH portable meter kit – STARA3215 | Portable meter for on-the-spot testing of pH and mV readings |
| | Orion ROSS Sure-Flow 8172BNWP electrode and Orion stainless steel ATC probe 927007MD for temperature measurement | Sure-Flow pH electrode has very easy-to-clean junction that works best with samples containing viscous or solid materials |
| | 9512BNWP Standard Ammonia Electrode, 951006 ammonia standard, 951211 ionic strength adjuster | Add on to existing pH/mV meter or ion titrator for ammonia nitrogen analysis |
| | Orion Star A216 pH/DO benchtop meter kit – STARA2165 | Space-saving alternative for combined pH and DO benchtop measurement capabilities |
| Dissolved Oxygen | Orion Star A326 pH/DO portable meter kit – STARA3265 | Compact alternative for combined pH and DO portable measurement capabilities |
| | Orion 087020MD optical dissolved oxygen probe | Long 6 meter cable for deep wine vats |
| Sulfur Dioxide using ORP | Orion 9180BNMD ORP/ATC electrode | Built-in ATC temperature sensor for convenience |
| | Orion 9678BNWP Sure-Flow ORP electrode, Orion 927007MD ATC temperature probe and Orion 967901 ORP calibration standard | Add on to existing pH/mV meter or redox titrator for sulfur dioxide analysis |
| Visual Clarity (Turbidity) | Orion AQUAfast AQ4500 turbidity meter | Added capability to measure using white light or IR LED |
| Spectrophotometry | Talk to us about Thermo Scientific spectrophotometry products | Match your application requirements and budget |

APPLICATION NOTE



# Measuring pH in wine and juice

**Key Words:** pH, red wine, wine-making, juice, proteins, sulfides, tannins, polyphenols.

## Goal

The following application note includes the recommended equipment, procedures and maintenance for accurate pH readings.

## Introduction

Since pH plays a critical role in wine making, measurements are taken throughout the winemaking process, from juice to finished wine. Typical pH levels in wine range from 2.9 to 3.9. Various components of juice and wine can challenge the performance of the pH electrode, including proteins, sulfides, tannins, and polyphenols. This note includes recommended equipment, procedures, and maintenance to assure accurate pH readings.

## Recommended Equipment

- Thermo Scientific™ Orion™ pH meter (or equivalent)
- Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ pH Electrode (Cat. No. 8172BNWP), Orion Green pH Combination Electrode (Cat. No. GD9156BNWP), or equivalent
- Automatic Temperature Compensation (ATC) probe (Cat. No. 927007MD)
- Stirrer (Cat. No. 096019)
- Swing arm and electrode holder (Cat. No. 090043)
- pH electrode storage bottle (Cat. No. 910003)

## Recommended Solutions

- pH 4.01 and 7.00 buffers (Cat. No. 910104, 910107)
- Electrode filling solution (Cat. No. 810007 for 8172BNWP, or Cat. No. 910008-WA for GD9156BNWP)
- Orion ROSS storage solution (Cat. No. 810001) or pH electrode storage solution (Cat. No. 910001)
- Deionized water (DI)
- pH electrode cleaning solution A (Cat. No. 900021-WA)
- pH electrode cleaning solution D (Cat. No. 900024)
- ~75% alcohol solution (methanol or ethanol in water)

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## Meter Setup

Connect the pH electrode, ATC probe, and stirrer to the meter. Set measurement mode to pH. In Setup, set the stirrer speed to 3, pH resolution to 0.01, buffer set to USA and read type to auto or continuous.

Note: When the ATC is connected properly, the true temperature (not the default 25.0) will be displayed on the screen. The ATC will measure buffer and sample temperatures and will ensure precise automatic temperature-compensated readings.

## Electrode Calibration

Before sample testing, perform a two-point pH calibration using pH 4.01 and 7.00 buffers. (See Analysis instructions below for details on test protocol). The electrode slope should be between 92 and 102%.

## Sample Preparation

Place about 50 mL of sample in a small, clean beaker (about 100 mL size).

## Analysis

Place the pH electrode, ATC, and stirrer into the electrode stand. Rinse each with DI water. Place probes and stirrer into the sample, immersing about 1-2 inches into the solution. Stir the sample continuously. When the meter indicates the reading is stable, record the pH to two decimal places (e.g., 3.39) and the temperature to one decimal place. (If using Autoread mode, the meter will lock on the final reading and automatically log the readings, when the log function is turned on in Setup). Between readings, rinse the probes and stirrer with DI water to remove any remaining sample.

## Electrode Storage

After testing is complete, rinse pH electrode thoroughly with the ~75% alcohol solution or immerse for 5 minutes, then rinse thoroughly with DI water. Cover the fill hole and store pH electrode in a bottle of electrode storage solution. Change the storage solution biweekly or monthly. ATC should be stored dry.

## Electrode Maintenance

Fill the electrode to the level of the fill hole each day, prior to testing. Weekly or biweekly, empty the fill solution and replace with fresh fill solution.

## Electrode Cleaning

If the electrode begins to exhibit drift and/or is slow to respond, clean it as follows: clean the electrode with Orion Cleaning Solution A according to the instructions, to remove proteins and restore the pH membrane. If further cleaning is desired, use Orion Cleaning solution D to remove organic compounds and restore the pH membrane.

## Quality Control (QC)

Recommended QC procedures may include: calibration, calibration verification, sample duplicates, and/or QC samples.

**Results of Measuring the pH of Red Wine**

| Precision | 8172BNWP | GD9156BNWP |
|---|---|---|
| pH of Red Wine, avg. (n = 10) | 3.41 | 3.42 |
| Standard Deviation | 0.02 | 0.01 |
| Avg. Temp. (°C) | 21.9 | 21.9 |
| Avg. Response Time | < 30 sec | < 30 sec |
| **Accuracy** | | |
| pH 4 Buffer, avg. (n = 6) | 4.01 | 4.01 |
| Difference From Expected | 0.00 | 0.00 |
| Standard Deviation | 0.01 | 0.01 |

## Precision

Both Orion pH electrodes demonstrated excellent precision between test results for multiple replicates of wine and pH buffer as follows:

- Red wine - showing a standard deviation of <0.02 pH units
- pH 4 buffer - showing a standard deviation of 0.01 pH units.

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## Accuracy

Both Orion pH electrodes demonstrated excellent accuracy for multiple replicates of pH 4 buffer, showing a difference from expected value of 0.00 pH units (reads exactly as expected).

## Speed

Both Orion pH electrodes demonstrated excellent response time. The time to a stable reading averaged less than 30 seconds for wine samples and pH 4 buffer.

To purchase Orion meters, electrodes and solutions, please contact your local equipment distributor and reference the part numbers listed below:

### Ordering Information

| Product | Cat. No. |
|---|---|
| **Meters** | |
| Thermo Scientific™ Orion™ Versa Star Pro™ pH Benchtop Meter Kit with Stand, ROSS™ Sure-Flow™ pH Electrode, ATC Probe, Stirrer Probe, pH 4/7/10 Buffers and ROSS Storage Solution | VSTAR13 |
| Thermo Scientific™ Orion Star™ A211 pH Benchtop Meter Kit with Stand, ROSS™ Sure-Flow™ pH Electrode, ATC Probe, pH 4/7/10 Buffers and ROSS™ Storage Solution | STARA2114 |
| **Electrodes** | |
| Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ pH Electrode | 8172BNWP |
| Thermo Scientific™ Orion™ Green pH Electrode | GD9156BNWP |
| **Solutions** | |
| Thermo Scientific™ Orion™ pH 4.01 and 7.00 Buffers | 910104 and 910107 |
| Thermo Scientific™ Orion™ Filling Solution for 8172BNWP or GD9156BNWP pH Electrodes | 810007 or 910008-WA |
| Thermo Scientific™ Orion™ ROSS™ Storage Solution or pH Electrode Storage Solution | 810001 or 910001 |
| Thermo Scientific™ Orion™ pH Electrode Cleaning Solution A or D | 900021-WA or 900024 |

### References


1. Zoecklein et al. *Wine Analysis and Production.* Chapman and Hall. 1995.
2. AOAC International. *AOAC Official Method 960.19, pH of Wines.* Official Methods of Analysis (OMA), 16th edition. 1999. www.aoac.org


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APPLICATION NOTE



# Titratable acidity in wine by automatic titration

**Key words**
TA, wine, must, juice,titrametric, potentiometric, AOAC 942.15, Orion 8172BNWP, Orion 8102BNUMD, Orion Star T910, Orion Star T940.

## Introduction

Titratable acidity (TA), is a measure of the organic acid content in wine, juice, or must. These organic acids come from the grapes, the fermentation, and the bacterial activity. The acidity can affect the flavor, color, and stability of the wine. TA in wine, juice, or must is determined using the preprogrammed method T1A TA Wine. This method is a direct titration to a preset endpoint at pH 8.2 using 0.1M (0.1N) sodium hydroxide titrant. The method may be edited to perform titratable acidity in other samples as well.

## Recommended equipment

- Thermo Scientific™ Orion Star™ T910 pH Titrator or T940 All-In-One Titrator or equivalent
- Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ pH Electrode (Cat. No. 8172BNWP) or equivalent
- Orion™ Automatic Temperature Compensation (ATC) probe
- Analytical balance (for sample measurement by weight) or graduated 10 mL pipet (for sample measurement by volume)

## Required reagents and solutions

- Purchased or prepared sodium hydroxide (NaOH) standard titrant solution, 0.1 M (0.1N)
- Reagent grade water (RGW)
- pH buffers: pH 4, 7, and 10

**Optional:**

- Potassium hydrogen phthalate (KHP) acidimetric standard

Use suitable personal protective equipment (PPE) as recommended by the Safety Data Sheets (SDS) for the chemicals utilized during this procedure.

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Connect the Orion pH electrode, ATC, and the stirrer probe to the titrator. If not previously done, import the T1A T1A Wine preprogrammed method into the titrator from the Methods screen1. Rinse and fill the burette with 0.1M (0.1N) sodium hydroxide titrant. See the titrator user manual for details. If bubbles are visible in the tubing, dispense titrant (from the Burette screen) until the bubbles have been expelled. Tap tubing to dislodge bubbles that stick. Consider standardizing the titrant before titrating samples. See the following Titrant section.

### T1A TA Wine Method: Preprogrammed parameters

| Electrode | Parameter |
|---|---|
| Electrode Type | pH |
| Electrode Name | Edit as desired |
| Resolution | 0.01 |
| Buffer Group | USA |

| Titrant | Parameter |
|---|---|
| Titrant Name | NaOH |
| Titrant ID | Edit as desired |
| Conc. Input Mode | Standardization |
| Nominal Concentration | 0.1M |
| Standardize Tech | Equivalence Pt. |
| Number of Endpoints | 1 |
| Results Units | M |
| Standardize Reaction Ratio | 1 |
| Standard Name | KHP |
| Standard Amount | Variable weight |
| Standard Molecular Wt | 204.2 |
| Standard Purity | 100% |
| Pre-dose Titrant Volume | 0 ml |
| Max. Total Titrant Volume | 5 ml |
| Stand. Process Control | Routine |
| Pre-stir Duration | 5 sec |
| Stir Speed | Medium |

| Titration | Parameter |
|---|---|
| Titration Technique | Preset End Pt. |
| Number of Endpoints | 1 |
| Endpoint Values | 8.2 |
| Titration Type | Direct |
| Blank Required | No |
| Result Units | g/L |
| Reaction Ratio | 0.5 |
| Sample Mol. Wt. | 150.09 |
| Sample Amount | Fixed vol, 5.0 mL |
| Pre-dose Titrant Volume | 0 ml |
| Max total titrant volume | 10 ml |
| Titration Process Control | Routine |
| Pre-stir Duration | 5 sec |
| Stir Speed | Fast |
| Sample ID | Manual |


### Electrode preparation

Remove electrode from storage solution. Top up the fill solution to the bottom of the fill hole and leave the fill hole open during testing. Rinse thoroughly with RGW before and between titrations.

### Sample preparation

Accurately measure 5.0 mL of wine, juice, or must into a clean 100 mL beaker. Add RGW to the 60 mL mark on the beaker. The sample is ready to titrate.

### Sample titration

1. From the Home screen, select option to use a saved method, then select the T1A TA Wine reprogrammed method.
2. At the pre-titration screen, select the Calibrate option and calibrate the electrode with pH 4, 7, and pH 10 buffers.
3. After calibration, place the electrode, ATC, stirrer, and dispenser into the sample in the beaker. Ensure that the dispenser tip is inserted below the surface of the sample and start the titration.
4. When prompted, enter the exact weight of the sample.

### Results

| Sample | Results | RSD (n = 3) | % Recovery | Duration (min:sec) |
|---|---|---|---|---|
| Tartaric Acid Standard | 7.563 g/L | 0.25% | 100.8% | 2:33 |
| Red Burgudy Wine | 5.835 g/L | 1.24% | NA | 3:21 |
| Red | 13.33 g/L | 0.19% | 99.9% | 4:55 |

### Range

This preprogrammed titration method covers a range of TA that may be expected in wine, juice, or must.

### Method modifications

- **For other result units:** Edit the Titration section of the method and choose the desired unit.
- **For shorter titrations:** For routine titrations with well-established endpoint volumes, use a pre-dose to shorten the analysis time. Edit the pre-dose in the Titration section of the method. In general, set the pre-dose at a volume that is 0.5 mL less than the expected endpoint volume.

### Titrant

Over time, standard titrant solutions age and can change concentration. For higher accuracy, determine the exact concentration by standardizing the titrant. It is common to standardize on a weekly basis, but other standardization frequencies may be suitable.

1. Standardizing the Titrant
   a. Weigh about 0.05 g KHP into a clean 100 or 150 mL beaker. Record the exact weight to the nearest 0.0001g. Repeat twice more for a total of three beakers of KHP. Add RGW to the 60 mL mark on each beaker and stir for about 2 minutes or so until the KHP is completely dissolved.
   b. If the KHP purity is not 100%, edit the Titrant section of the method to enter the actual purity
   c. Select the Titratable Acidity preprogrammed method on the titrator.
   d. At the pre-titration screen, select the Standardize option and follow the prompts to standardize the titrant.
   e. The new standardized titrant concentration will automatically be saved and used for subsequent T1A TA method titrations.
2. Certified Standardized Titrant Solutions
   a. Some customers may prefer not to standardize their titrant, instead choosing to purchase and use certified standardized titration solutions. In this case, edit the Titrant section of the method and enter the certified concentration and titrant ID (i.e., lot number, if desired).

### Titrator and electrode care

Refer to the titrator and electrode user manuals for details on cleaning, storage, and maintenance recommendations to keep the titrator and electrode performing well. Main points for care are summarized as follows.

| Daily Care | Weekly or Biweekly Care | As Needed |
|---|---|---|
| • If bubbles are visible in the titrator tubing, dispense titrant until bubbles have been expelled<br>• Top up the electrode fill solution and leave the fill hole open during measurement<br>• Rinse electrode well with RGW between titration cycles<br>• Cover the fill hole and store electrode in storage solution overnight | • Drain and replace the fill solution of the electrode.<br>• Change the storage solution in the electrode storage bottle<br>• Consider standardizing the titrant on a weekly basis | • For slow or drifty electrode response, soak 15 minutes in 1% laboratory detergent while stirring. Rinse well with RGW afterwards<br>• If still slow or drifty, use Orion pH cleaning solution D per instructions<br>• See the user manuals for maintenance details |

### Notes

[1]Refer to the user manual for detailed instructions, if desired.

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To purchase Thermo Scientific laboratory products, please contact your local equipment distributor and reference the part numbers listed below:

| Product | Description | Cat. No. |
|---|---|---|
| Titrator kits | Thermo Scientific™ Orion Star™ T910 Titrator Standard Kit with 8102BNUWP Thermo Scientific™ Orion™ ROSS Ultra™ pH Electrode and ATC Probe | START9101 |
| | Thermo Scientific™ Orion Star™ T910 pH Titrator Sure-Flow™ Kit with 8172BNWP ROSS™ Sure-Flow™ pH Electrode and ATC Probe | START9102 |
| | Thermo Scientific™ Orion Star™ T940 All-In-One Titrator Standard Kit with 8102BNUWP ROSS™ Ultra pH Electrode and ATC Probe | START9401 |
| | Thermo Scientific™ Orion Star™ T940 All-In-One Titrator Sure-Flow™ Kit with 8172BNWP ROSS™ Sure-Flow™ pH Electrode and ATC Probe | START9402 |
| Titrators | Thermo Scientific™ Orion Star™ T910 pH Titrator without electrode | START9100 |
| | Thermo Scientific™ Orion Star™ T940 All-In-One titrator without electrode | START9400 |
| Electrodes | Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ pH Electrode | 8172BNWP |
| | Thermo Scientific™ Orion™ ROSS Ultra™ pH Electrode | 8102BNUWP |
| | Automatic Temperature Compensation (ATC) Probe | 927007MD |
| pH Buffers | Orion pH 4.00 Buffer, NIST traceable, 475 ml | 910104 |
| | Orion pH 7.00 Buffer, NIST traceable, 475 ml | 910107 |
| | Orion pH 10.00 Buffer, NIST traceable, 475 ml | 910110 |
| Reagent Grade Water | Thermo Scientific™ Barnstead™ Smart2Pure™ 12 UV Water Purification System | 50129890* |
| Reagents | 0.1M (0.1N) Sodium Hydroxide Titrant | |
| | Potassium Hydrogen Phthalate, primary or acidimetric standard grade | |
| Accessories | 100 or 150 mL beakers | |

*Please contact your local Thermo Scientific representative for support on ordering water quality products. For more information, visit thermofisher.com/waterquality.

**References**


- Acidity (Titratable) of Fruit Products, Method 942.15. Official Methods of Analysis (OMA). AOAC International, 2275 Research Blvd, Ste 300, Rockville, MD 20850-3250. USA.
- Patrick Iland et al. (2004). Titratable Acidity. In: Chemical analysis of grapes and wine: techniques and concepts, 2nd ed. Patrick Iland Wine Promotions PTY LTD, pp. 39 - 43.


Find out more at **thermofisher.com/titrator**

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# Titratable acidity of red wine by manual titration (potentiometric)

Key words: Titratable acidity, red wine, manual titration, potentiometric, organic acids

## Goal

The following application note demonstrates a simple titratable acidity titration method using a Thermo Scientific™ Orion™ pH Electrode and Meter to signal the endpoint.

## Introduction

Titratable acidity (TA) is a measure of acid content in wine, juice, or must. TA is usually reported in units of tartaric acid, malic acid, or citric acid. Since grapes contain significant amounts of organic acids, TA analysis is one of the most basic analyses in a winery lab. The acid content impacts the taste, color, and microbial stability of the juice. This application note demonstrates a simple TA titration method using an Orion pH Electrode and Meter to signal the endpoint.

## Recommended equipment

- Thermo Scientific™ Orion™ Versa Star Pro™ pH/mV Meter, Thermo Scientific™ Orion Star™ A211 pH/mV Meter, or equivalent Orion pH/mV Meter
- Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ pH Electrode 8172BNWP or Thermo Scientific™ Orion™ GD9156BNWP green pH Electrode, or equivalent
- Orion automatic stirrer probe and paddle (Cat. No. 096019)
- Orion swing arm and electrode holder (Cat. No. 090043)

- 10 mL burette, burette clamp, ring stand

## Recommended equipment

- Sodium hydroxide (NaOH) standard solution, 0.10 N or 0.067 N
- Orion pH 7.00 buffer (Cat. No. 910107)
- Orion pH 4.01 buffer (Cat. No. 910104)
- Orion 810007 ROSS filling solution or Orion 910008-WA Double Junction pH fill solution (for GD9156BNWP)
- Orion ROSS pH electrode storage solution (Cat. No. 810001) or Orion pH electrode storage solution (Cat. No. 910001)
- Deionized water (DI) of conductivity < 2 uS/cm
- Optional: potassium hydrogen phthalate (KHP); tartaric

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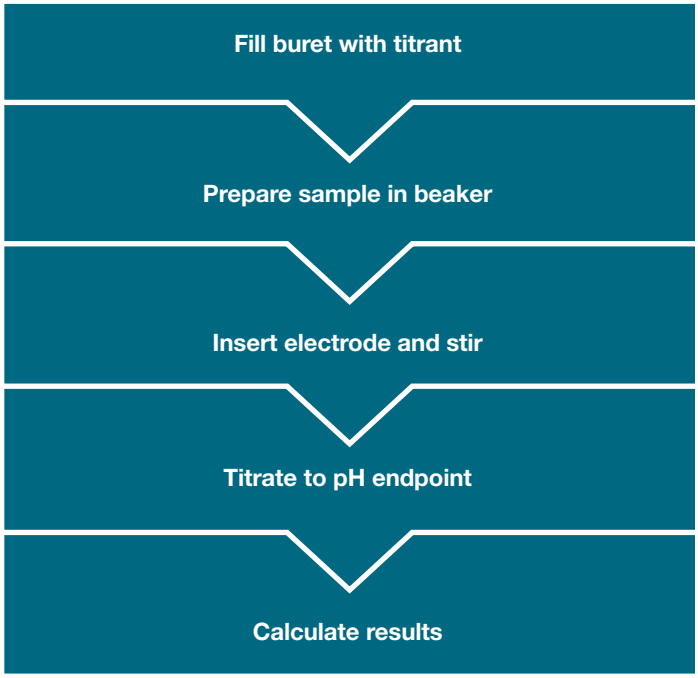
## Basic titration workflow




## Meter setup

Connect the Orion pH electrode and the stirrer probe to the meter. In Setup, select the pH mode, set read type to continuous, and set the stirrer speed to 3. Refer to the Orion meter user guide for more details.

## Electrode calibration

Perform a two-point pH calibration using pH 4.01 and 7.00 buffers. Stir the buffer during calibration. The electrode slope should be between 92 and 102%.

## Titration setup

Secure the burette on the clamp. Fill the burette with NaOH titrant solution and adjust the level to the zero mark. Eliminate bubbles.

## Sample preparation

Make sure the wine sample is at room temperature. To minimize interference from $CO_2$, degas an amount of the wine sample, e.g. 25 mL. Degas in a flask under vacuum for several minutes with agitation. Alternately, place a sample in an ultrasonic bath for a few minutes, bubble compressed air through to release excess $CO_2$, or heat a sample to almost boiling, agitate, and let cool.

## Sample titration

Pipette 5.0 mL of degassed wine sample into a 150 mL beaker containing 50 mL DI. Immerse the electrode and stirrer in the solution. Turn on the stirrer.

Titrate with the NaOH titrant at a faster rate in the beginning and a slower rate when the pH reaches 6.5. It is not necessary to wait for a stabilized reading before pH 6.5. Then, start to add titrant slowly until the pH just exceeds 8.2. This is the endpoint of the titration. Record the volume of titrant used (Vs). Calculate the TA in g/L tartaric acid in the wine sample using the equation on page 2. Repeat the titration as desired or required by user's protocol. After each titration, rinse the pH electrode and stirrer with DI and tap to remove excess water droplets.

## Quality Control (QC)

Recommended QC procedures may include: titrant standardization, blank titration, analysis of tartaric acid standard or QC sample, and/or duplicate sample. For details, refer to the Appendix.

## Results

Results obtained using standard solutions prepared in lab and an Orion 8172BNWP electrode.

### Titratable acids in red wine samples

| Red wine sample | TA (g/L tartaric acid) |
|---|---|
| Sample 1 | 5.82 |
| Sample 2 | 5.90 |
| Sample 3 | 5.87 |
| Results statistics | |
| Mean | 5.86 |
| RSD | 99.8% |

| QC sample* | TA (g/L tartaric acid) |
|---|---|
| Tartaric acid standard (5.04 g/L) | 5.05 |
| Results statistics | |
| Recovery | 99.8% |

* For details on QC sample, see Appendix

## Calculation

TA in sample:

$$\text{TA (g tartaric acid/L)} = \frac{(\text{N NaOH}) \times (\text{mLs NaOH}) \times 75)}{\text{mLs of sample}}$$

## Notes

1. If sodium hydroxide is 0.067N and 5 mL of sample are used, then the equation simplifies to TA = mLs of NaOH titrant used.
2. To report results as g malic acid/L (e.g. for apple wines), multiply tartaric acid results by 0.893.
3. To report results as g citric acid/L (e.g. for fruit wines), multiply tartaric acid results by 0.853.
4. For greater accuracy, titrate a blank (50 mL of DI water). If more than 0.10 mL of sodium hydroxide titrant is required to bring pH to 8.2 or higher for the blank, then subtract that volume from the sample titration. Calculate TA:

TA = (N NaOH) x (mLs NaOH $_{\text{(sample titration)}}$ – mLs NaOH $_{\text{(blank titration)}}$) x 75/ mLs of sample.

## Appendix

### Optional User Prepared NaOH Titrant Solution and KHP Solution for Standardization of Titrant

0.067 N NaOH titrant solution: Weigh 1.34 g NaOH pellets and quickly transfer to a 500 mL volumetric flask. Add DI to dissolve the pellets and fill to the mark. (Required reagents: NaOH, ACS grade)

0.033 N KHP solution: Dry KHP at 110 degrees C for 2 hours and cool in a dessicator. Weigh ~0.34 g of cool, dried KHP into a 50 mL volumetric flask and record the exact weight (Ws). Fill the flask to the mark with DI and mix well to dissolve the solid.

(Required reagents: KHP, ACS grade)

### Standardization of Titrant – recommended for User Prepared Titrant and for quality control of any titrant solution

Pipette 10 mL KHP solution to 50 mL DI in a 100 mL beaker. Rinse the electrode and stirrer with DI. Immerse the electrode and stirrer in the solution. Turn on the stirrer. Tap to release air bubbles trapped on the surface of the electrode.

Adding NaOH titrant from the burette, titrate at a moderate rate. It is not necessary to wait for a stabilized reading when the pH is still far from 8.2. Start to add titrant slowly when the pH approaches 8. When the pH reading exceeds 8.2, the titration has reached the endpoint. Record the volume of titrant used (Vt) at the endpoint. Repeat the standardization procedure if required by user's protocol. Calculate the NaOH titrant concentration (T, mol/L) as follows:

$$\text{Normality of NaOH} = \frac{\text{(N KHP) x (mLs KHP)}}{\text{mLs NaOH}}$$

Results of an example titrant standardization are summarized in the following table.

| Titrant standardization | Endpoint volume (mL) | NaOH (M) |
|---|---|---|
| Trial 1 | 5.60 | 0.0596 |
| Trial 2 | 5.57 | 0.0599 |
| Trail 3 | 5.61 | 0.0594 |
| Statistics | Mean | 0.0596 |
| | RSD | 0.4% |

### Optional analysis of tartaric acid standard – QC sample

Weigh ~1.00 g tartaric acid into a 200 mL volumetric flask and record the exact weight (Wd). Dissolve and fill to the mark with DI water. The concentration of the tartaric acid standard is 5 × Wd g/L. (The actual Wd was 1.0081 g in the Results section above. The concentration of the standard was calculated to be 5.0405 g/L). Pipette 5 mL of tartaric acid standard into a 100 mL beaker containing 50 mL DI. Titrate with the NaOH titrant using the titration techniques described in Standardization of Titrant section. Calculate the concentration of tartaric acid using the equation for TA in sample (noted in Results section).

Depending on your sample throughput needs and budget, we have developed application notes for both our electrochemistry meters for manual titrations and potentiometric titrators for automated titrations. Our automated titrators can help improve your titrations by simplifying your process and helping to deliver consistently reliable results. Just program it once and the titrator takes care of the rest - including addition of titrant, endpoint determination, results calculation, and data logging. Please visit thermofisher.com/titrator for more information.

| Product | Description | Cat. No. |
|---|---|---|
| Electrodes | Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ pH Electrode | 8172BNWP |
| | Orion Green pH Electrode | GD9156BNWP |
| Meters | Thermo Scientific™ Orion Star™ A211 pH Benchtop Meter Kit | STARA2115 |
| | Thermo Scientific™ Orion™ Versa Star Pro™ pH Meter Kit | VSTAR12 |
| | Thermo Scientific Orion Versa Star Pro™ pH/ISE/Conductivity/RDO/DO Meter Kit | VSTAR92 |
| Solutions | Thermo Scientific™ Orion™ ROSS™ All-in-One pH Buffer Kit | 810199 |
| | Thermo Scientific Orion ROSS pH Electrode Filling Solution | 810007 |
| | Thermo Scientific Orion Double Junction pH Electrode Fill Solution (For GD9156BNWP) | 910008-WA |
| Accessories | Thermo Scientific Orion Automatic Stirrer Probe and Paddle | 096019 |
| | Thermo Scientific Orion Swing Arm and Electrode Holder | 090043 |

**References**

1. Zoecklein et al. Wine Analysis and Production. Chapman and Hall. 1995.
2. AOAC International. AOAC Official Method 962.12, Acidity (Titratable) of Wines. Official Methods of Analysis (OMA), 16th edition. 1999. www.aoac.org

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# Nitrogen measurement in wine

**Key Words:** Nitrogen testing, ammonia nitrogen, winemaking analysis, nitrogen compounds, Yeast Assimilable Nitrogen (YAN), free alpha-amino nitrogen (FAN), primary amino nitrogen (PAN), fermentation testing

## Goal

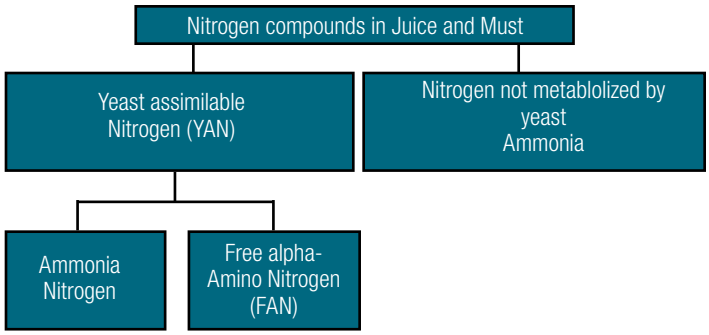
The following application note explains the importance of nitrogen testing in the winemaking process.

## Introduction

Nitrogen is a key nutrient for yeast growth, and is necessary for the successful fermentation of grape juice and must into wine. Nitrogen compounds in juice, must, and wine affect not only the fermentation, but the clarification, aroma, and final chemical composition of the wine. For these reasons, the analysis of nitrogen in the winemaking process is recommended for ensuring a quality wine.

The total nitrogen (N) content of grape juice/must is widely variable. It may be as high as >1000 mg N/L or as low as <50 mg N/L. However, not all nitrogen compounds in juice and must are available for yeast metabolism. The nitrogen that can be used by yeast is known as Yeast Assimilable Nitrogen or YAN. YAN is comprised mostly of ammonia (present as ammonium salts) and certain amino acids, often designated as free alpha-amino nitrogen (FAN[1]) or primary amino nitrogen (PAN). Therefore, YAN = ammonia nitrogen + amino nitrogen. The relationship of nitrogen compounds is expressed by Figure 1.

**Figure 1.**

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## Testing for Nitrogen

Testing for nitrogen before and during the fermentation is desired. YAN that is too low or too high can have negative impacts on the winemaking process and the wine. Some possible impacts are listed at Figure 2. Decisions on how much and what types of nitrogen to add will be informed by the results of the nitrogen testing.

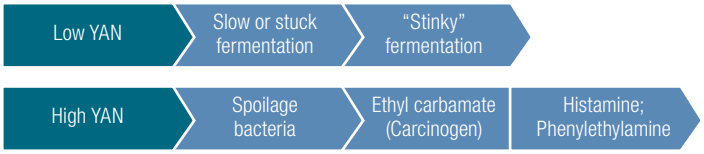


**Figure 2.**

## Methods for Nitrogen Testing

There are several options for nitrogen testing that are available for a wine analysis laboratory. Some tests are for ammonia, or amino nitrogen, and others are for both. Some methods for testing can be done in any laboratory, while others require the advanced instrumentation; typically found in a commercial laboratory. Here is a summary of some of the simpler tests that can be done in most any lab.

## Equipment Recommendations for Testing

We can match your testing requirements and budget to our analytical equipment. Call your authorized distributor or visit thermofisher.com.

**Methods for Testing Nitrogen**

| Product | Method | Equipment | Notes |
|---|---|---|---|
| Ammonia nitrogen | Ammonia Ion Selective Electrode (ISE) | pH/ISE meter | No filtration or color removal/blank required. Economical ammonia test option. A pH/ISE meter can also be used for pH, TA[3], $SO_2$ by Ripper/ORP, and potassium, with corresponding electrodes. |
| | Enzyme | Spectrophotometer or wine analyzer | Can also test for amino acids and other parameters, using different reagents. Initial equipment costs and prepared reagent costs tend to be higher. |
| Amino nitrogen | NOPA[2] | Spectrophotometer | Can also test for ammonia and other parameters, using different reagents. Initial equipment costs and prepared reagent costs tend to be higher. |
| | Enzymatic | Spectrophotometer or wine analyzer | Can also test for ammonia and other parameters, using different reagents. Initial equipment costs and prepared reagent costs tend to be higher. |
| YAN | Micro formol titration | pH meter and burette | One test gives both nitrogen forms. Uses small amounts of hazardous formaldehyde. Most economical option. |
| YAN and TA[3] | Micro TA and formol titration[4] | pH meter and burette | One test gives both nitrogen forms. Uses small amounts of hazardous formaldehyde. Most economical option. |

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**Equipment Recommendations for Testing**

| Ammonia Nitrogen | Amino Nitrogen + | YAN (and TA) |
|---|---|---|
| Thermo Scientific™ Orion Star™ A214 benchtop[5] or Star A324 portable pH/ISE meter | Talk to us about Thermo Scientific™ spectrophotometry products | Orion Star™ A211 Benchtop[5] or Orion Star A221 portable pH meter |
| Thermo Scientific™ Orion™ 9512HPBNWP High-Performance Ammonia Electrode | + Can test for ammonia and other parameters as well | Thermo Scientific™ Orion™ ROSS™ Sure-Flow™ 8172BNWP pH Electrode or Thermo Scientific™ Orion™ GD9156BNWP Green pH Combination Electrode |

## References


1. In some winegrowing areas, the term FAN is used to indicate free available nitrogen, rather than the term YAN. To avoid confusion, we will use the terms “amino nitrogen” and “ammonia nitrogen” here.
2. Nitrogen by o-phthaldialdehyde assay.
3. Titratable Acidity.
4. Combined Titrametric Analysis of TA and YAN, Barry H. Gump, Vitis Research. (bgump@fiu.edu; www.vitisresearch.com).
5. Comes with stirrer capability.


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APPLICATION NOTE



# Free and Total Sulfur Dioxide ($SO_2$) in wine (Ripper method) by automatic titration

Water Analysis Instruments,
Thermo Fisher Scientific

**Key words**
Sulfite, sulfurous acid, $SO_2$, juice, must, iodine, redox, Orion 9770BNWP, Orion Star T920, Orion Star T940, titrator.

**Preprogrammed methods**
T5 $SO_2$ Wine

**Introduction**
Free and Total sulfur dioxide in wine are determined using the preprogrammed method, T5 $SO_2$ Wine. To determine free sulfur dioxide, the sample is treated with appropriate reagents and titrated to the equivalence point using iodine/iodide titrant. To determine total sulfur dioxide, the sample is reacted with alkaline solution for a set period of time, then treated with appropriate reagents and titrated to the equivalence point using iodine/iodide titrant.

**Recommended equipment**
- Thermo Scientific™ Orion Star™ T920 Redox Titrator or T940 All-In-One Titrator or equivalent with a 20 mL burette
- Thermo Scientific™ Orion™ 9770BNWP platinum/iodide (chlorine) electrode or equivalent
- Glass pipette or graduated cylinder, 10 mL graduated
- 25 or 50 mL graduated cylinder
- 100 and 150 mL beakers

**Required reagents and solutions**
- Titrant: Purchased or prepared iodine/iodide titrant, 0.01M (0.02N)
- Standardizing Solution (optional): Purchased or prepared 0.01M (0.01N) standard sodium thiosulfate solution
- Reagent Solutions: Purchased or prepared 1N Sodium Hydroxide, purchased or prepared 25% Sulfuric Acid (1 + 3 $H_2SO_4$), prepared conditioning solution
- Solid Reagent: Sodium Bicarbonate
- Reagent grade water (RGW)

Use suitable Personal Protective Equipment (PPE) and ventilation as recommended by the Safety Data Sheets (SDS) for the chemicals utilized during this procedure.

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## Titrator setup

Connect the electrode and the stirrer probe to the titrator. If not previously done, import the T5 $SO_2$ Wine preprogrammed method into the titrator from the Methods screen[1]. Rinse and fill the burette with titrant. See the titrator user manual for details on setting up the titrator.

If bubbles are visible in the tubing, dispense titrant (from the Burette screen) until the bubbles have been expelled. Tap the tubing to dislodge bubbles. Consider standardizing the titrant before titrating samples. See Titrant section below.

**T5 $SO_2$ Wine method: Preprogrammed parameters**

| Electrode | Parameter |
|---|---|
| Electrode Type | Redox |
| Electrode Name | Edit as desired |

| Titrant | Parameter |
|---|---|
| Titrant Name | I2 |
| Titrant ID | Edit as desired |
| Conc Input Mode | Standardization |
| Nominal Concentration | 0.01 M[2] |
| Standardize Tech | Equivalence Pt. |
| Number of Endpoints | 1 |
| Results Units | M |
| Standardize Reaction Ratio | 2 |
| Standard Name | $Na_2S_2O_3$ |
| Standard Amount | Fixed volume, 10 mL |
| Standard Concentration | 0.010 M[3] |
| Pre-dose Titrant Volume | 4 mL |
| Max total titrant volume | 10 mL |
| Stand. Process Control | User defined |
| ΔE | 10 mV |
| ΔVmin | 0.02 mL |
| ΔVmax | 0.100 mL |
| dE/dt | 10 |
| t min | 2 seconds |
| t max | 8 seconds |
| Threshold | 250 |
| Pre-stir Duration | 5 sec |
| Stir Speed | Medium |

| Titration | Parameter |
|---|---|
| Titration Technique | Equivalence Pt. |
| Number of Endpoints | 1 |
| Titration Type | Direct |
| Blank Required | No |
| Result Units | mg/L |
| Reaction Ratio | 1 |
| Sample Amount | Fixed vol., 25 mL |
| Sample MW | 64.07 |
| Pre-dose Titrant Volume | 0.1 mL |
| Max total titrant volume | 10 mL |

| Titration (Contd.) | Parameter (Contd.) |
|---|---|
| Titration Process Control | User defined |
| ΔE | 5 mV |
| ΔVmin | 0.020 mL |
| ΔVmax | 0.100 mL |
| dE/dt | 10 mV/min |
| t min | 2 seconds |
| t max | 4 seconds |
| Threshold | 250 |
| Pre-stir Duration | 10 sec |
| Stir Speed | Medium |
| Sample ID | Manual |

## Electrode preparation

Before the first titration of the day, place electrode into conditioning solution for 10 or 15 minutes. Rinse thoroughly with RGW before and between titrations. At the end of the day, clean any foreign materials from the platinum sensor by wiping with a moistened lint-free wiper. Thoroughly rinse the electrode with RGW and store the electrode dry.

## Reagent solution preparation

**Prepared 25% Sulfuric Acid:** Place 750 mL of RGW in a large beaker or graduated cylinder. Add 250 mL of concentrated (98%) $H_2SO_4$. Mix and allow to cool. Store in a sealed container at room temperature.

**Conditioning solution:** Dispense 0.5 mL of titrant into a 150 mL beaker and add RGW to the 100 mL mark.

## Sample preparation

**Free $SO_2$:** Accurately add 25.0 mL of wine to a beaker. Add RGW to the 60 mL mark on the side of the beaker. Proceed to Sample Titration.

**Total $SO_2$:** Accurately add 25.0 mL of wine. Add 25 mL of 1 N NaOH to a beaker. Mix and allow 10 minutes reaction time. After 10 minutes, add RGW to the 60 mL mark on the side of the beaker. Proceed to Sample Titration.

## Sample titration

1. Free $SO_2$
   a. From the Methods screen, select option to run the saved method T5 $SO_2$ Wine.
   b. Rinse the electrode, stirrer, and dispenser with RGW. Place the electrode, stirrer, and dispenser into the prepared sample in the beaker. Ensure that the dispenser tip is inserted below the surface of the sample. Ensure that the reference pellet on the side of the electrode is immersed.

a. bicarbonate to the beaker. The sample will bubble and foam. Addition of the bicarbonate may be omitted if results are satisfactory without it.

b. Results are reported as mg/L $SO_2$.

1. Total $SO_2$

a. From the Methods screen, select option to run the saved method T5 $SO_2$ Wine.

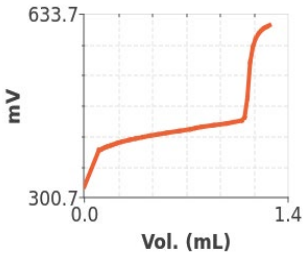
b. Rinse the electrode, stirrer, and dispenser with RGW. Place the electrode, stirrer, and dispenser into the prepared sample in the beaker. Ensure that the dispenser tip is inserted below the surface of the sample. Ensure that the reference pellet on the side of the electrode is immersed.

c. Add 10 mL of 25% sulfuric acid to the beaker. Start the titration immediately. When the stirrer starts to spin, immediately add about 1 g (¼ teaspoon) of solid bicarbonate to the beaker. The sample will bubble and foam. Addition of the bicarbonate may be omitted if results are satisfactory without it.

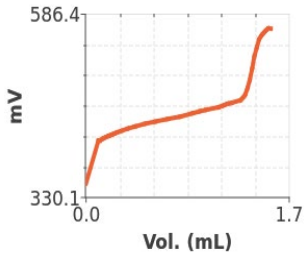
d. Results are reported as mg/L $SO_2$.

## Results

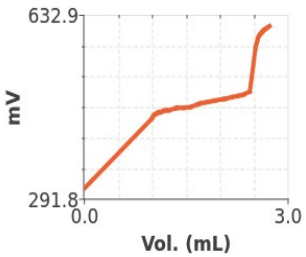
| Parameter | Sample | Average (n = 3) | SD (mg/L) | Analysis Time |
|---|---|---|---|---|
| Free $SO_2$ | White wine | 26.3 mg/L $FSO_2$ | 0.6 | 02:39 minutes |
| Free $SO_2$ | Red wine | 29.9 mg/L $FSO_2$ | 0.7 | 03:03 minutes |
| Total $SO_2$ | White wine | 65.9 mg/L $TSO_2$ | 0.8 | 03:49 minutes[4] |
| Total $SO_2$ | Red wine | 51.8 mg/L $TSO_2$ | 0.9 | 02:51 minutes[5] |



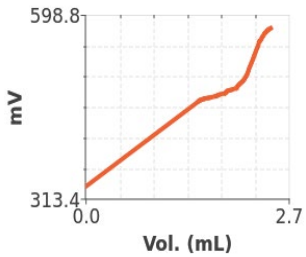

Free $SO_2$ in white wine




Free $SO_2$ in red wine




Total $SO_2$ in white wine




Total $SO_2$ in red wine

## Range

This preprogrammed titration method covers a range of about 25 to 250 mg/L of $SO_2$, when using 0.01 M (0.02 N) Iodine/Iodide titrant and the prescribed volume of sample.

## Method modifications

- For samples of a concentration less than 25 mg/L:
  - Double the sample volume tested, e.g., 50 mL.
  - Edit the method ($SO_2$ Wine), edit Titration, change Sample Amount to Variable Volume, and Save. When prompted by the titrator, enter 50 mL as the sample volume.
- For samples of a concentration of greater than 250 mg/L, choose one option:
  - Use a smaller sample volume, e.g., 15 mL. Edit the method ($SO_2$ Wine), edit Titration, change Sample Amount to Variable Volume, and Save. When prompted by the titrator, enter 15 mL as the sample volume.
- For shorter titrations:
  - Use a pre-dose to shorten the analysis time.
  - Edit the method ($SO_2$ Wine), edit Titration, change the Pre-dose Titrant Volume, and Save.
  - If desired, copy the method to create two methods: one method for free $SO_2$ and another method for total $SO_2$. Adjust the predose as desired for each method.
  - In general, a pre-dose that can be expected to work well will be a volume of about 1 mL less than the expected final volume of the titration.
  - For example, a sample with a concentration near

## Titrant

Over time, standard titrant solutions age and can change concentration. For higher accuracy, determine the exact concentration by standardizing the titrant. It is common to standardize on a weekly basis, but other standardization frequencies may be suitable.

1. Standardizing titrant
    a. Pipet 10.0 mL standardizing solution, 0.01M (0.01N) standard sodium thiosulfate, into a clean 100 mL beaker. Add about 60 mL of RGW to the beaker.
    b. Select the $SO_2$ Wine method. At the pre-titration screen, select the standardize option.
    c. Add 2 drops of 25% sulfuric acid to the beaker (to lower the pH to < 5).
    d. Start the titration.
    e. The new standardized titrant concentration will automatically be saved and used for subsequent $SO_2$ Wine method titrations.
2. Certified standardized titrant solutions
    a. Some customers may prefer not to standardize their titrant, instead choosing to purchase and use certified standardized titration solutions. In this case, edit the Titrant section of the method and enter the certified concentration and titrant ID (i.e., lot number, if desired).

## Titrator and electrode care

Refer to the titrator and electrode user manuals for details on cleaning, storage, and maintenance recommendations to keep the titrator and electrode performing well. Main points for care are summarized below.

| Daily Care | Weekly or Biweekly Care | As Needed |
|---|---|---|
| • If bubbles are visible in the titrator tubing, dispense titrant until bubbles have been expelled. Tap tubing to dislodge bubbles that stick.<br>• At the start of the testing, immerse the electrode in diluted titrant conditioning solution for 10 or 15 minutes.<br>• Rinse electrode well with RGW before and between titrations.<br>• Clean any foreign materials from the platinum sensor by wiping gently with a moistened lint-free wiper.<br>• Storage: Thoroughly rinse the electrode with RGW and store the electrode dry. | • Consider standardizing the titrant on a weekly basis, or more frequently, as desired.<br>• Prepare a small batch of conditioning solution for the week. | • For slow or drifty electrode response, soak 15 minutes in warm 1% laboratory detergent while stirring. Gently brush the platinum sensor and reference pellet with a *soft* toothbrush. Rinse well with RGW afterward.<br>• If still slow or drifty, use Orion pH cleaning solution C, immersing only the platinum sensor. Do not immerse the reference pellet. Rinse well, then soak in conditioning solution solution 10 or 15 minutes before use.<br>• See the electrode user manuals for maintenance details. |

## Notes

[1]Refer to the user manual for detailed instructions.

[2]If the concentration of the iodine titrant is listed in normality (N), divide by 2 to obtain the molarity (M). For example, 0.02N iodine ÷ 2 = 0.01M iodine.

[3]If the standardizing solution concentration is not 0.0100 M (0.0100 N), enter the true value.

[4]Using a predose of 1.0 mL. For a shorter run, try a predose of 1.5 or 2.0 mL.

[5]Using a predose of 1.5 mL.

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To purchase Thermo Scientific laboratory products, please contact your local equipment distributor and reference the part numbers listed below:

| Product | Description | Cat. No. |
|---|---|---|
| Titrators | Thermo Scientific Orion Star T920 pH Titrator without electrode | START9200 |
| | Thermo Scientific Orion Star T940 All-In-One Titrator without electrode | START9400 |
| Electrodes | Thermo Scientific Orion Platinum/Iodide (chlorine) Electrode | 9770BNWP |
| Accessories | 100 and 150 mL beakers | |
| | 25 or 50 mL graduated cylinder | |
| | 10 mL graduated pipet | |
| Reagent Grade Water | Thermo Scientific™ Barnstead™ Smart2Pure™ 12 UV Water Purification System | 50129890* |
| Reagents | 0.01M (0.02N) iodine/iodide titrant | |
| | 0.01M (0.01N) standard sodium thiosulfate solution | |
| | 1M Sodium Hydroxide | |
| | Sulfuric Acid, 98% or 25% | |
| | Sodium Bicarbonate, solid | |

*Please contact your local Thermo Scientific representative for support on ordering the best water purification system for your application. And visit our website at www.thermofisher.com/T900titratorseries.

## References


Zoecklein et al. *Wine Analysis and Production*, Chapman and Hall. 1995.

Iland et al. *Chemical Analysis of Grapes and wine: techniques and concepts*. 2nd Edition, 2013.


Find out more at **thermofisher.com/T900titratorseries**


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APPLICATION NOTE



# Sulfur dioxide in wine by manual ripper titration with a platinum ORP electrode

**Key Words**
Wine analysis, Ripper titration, sulfur dioxide, platinum ORP electrode, iodine titrant standardization, $SO_2$.

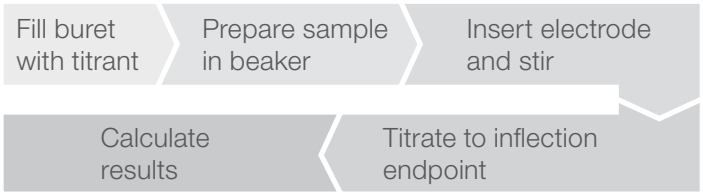
## Goal

The following application note describes a simple way to analyze the sulfur dioxide in wine using a Thermo Scientific™ Orion™ 9778BNWP or 9678BNWP ORP Electrode and the Ripper titration method.

## Introduction

$SO_2$ is widely used in wine production as a chemical antioxidant and inhibitor of microbial activity. $SO_2$ in wine is traditionally analyzed by Ripper titration using a color indicator. In this note, the same Ripper titration is performed by using an Orion platinum ORP electrode to signal the endpoint. The color and clarity of red wine does not interfere, thereby improving the results. This method provides a simple solution to $SO_2$ analysis in wine.

## Basic Titration Workflow




## Recommended Equipment

- Thermo Scientific™ Orion™ Versa Star Pro™ pH/mV meter, Thermo Scientific™ Orion Star™ A211 pH/mV meter, or equivalent Orion pH/mV meter
- Thermo Scientific Orion 9778BNWP Glass Combination Platinum ORP Electrode or Thermo Scientific™ Orion™ Sure-Flow™ 9678BNWP Platinum ORP Electrode
- Stirrer probe (Cat. No. 096019)
- Swing arm stand (Cat. No. 090043)
- Electrode polishing strip (Cat. No. 948201)
- 10 mL burette, burette clamp, ring stand

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## Required Reagents and Solutions

- Purchased or prepared Iodine ($I_2$) standard titrant solution, 0.01 M (0.02 N)
- Sodium hydroxide (NaOH), 1 N
- 25% sulfuric acid (1+3 $H_2SO_4$)
- Electrode filling solution for ORP electrode (Cat. No. 900011)
- Laboratory Reagent Water (LRW)

Optional:

- Standard sodium thiosulfate solution, 0.01 M (0.01 N)
- Sodium sulfite ($Na_2SO_3$)
- Sodium bicarbonate ($NaHCO_3$)

## Meter Setup

Connect Orion electrode and the stirrer probe to the meter. In Setup, select the mV mode, set read type to continuous, and set the stirrer speed to 3. Refer to the Orion meter and electrode user guides for more details.

## Titration Setup

Secure the burette on the clamp. Fill the burette with iodine titrant solution and adjust the level to the zero mark. Consider standardizing the titrant before titrating samples. See *Hints and Tips* on the following page.

## Sample Preparation

Make sure the wine sample is at room temperature.

**Total $SO_2$:** Add 25.0 mL of wine and 25 mL of 1 N NaOH to a 100 mL beaker. Mix and allow 10 min for hydrolysis then proceed immediately to the Sample Titration.

**Free $SO_2$:** Add 25.0 mL of wine to a 100 mL beaker. Proceed immediately to the Sample Titration.

## Sample Titration

The Ripper titration should be done relatively quickly to avoid loss of $SO_2$ to the air. Rinse the electrode and stirrer with LRW. Immerse the electrode and stirrer at least one inch below the liquid level in the beaker, with the stirrer just below the electrode. Turn on the stirrer. Stir gently so that a vortex is not created. Tap to release air bubbles trapped on the surface of the electrode.

**Total $SO_2$:** Add 10 mL of 25% sulfuric acid to the beaker.

**Free $SO_2$:** Add 5 mL of 25% sulfuric acid to the beaker.

Watching the mV reading, titrate at moderate speed with the iodine titrant. The mV values will not rise quickly, until near the endpoint (EP). The EP is considered the point where the largest mV change is observed per volume addition of titrant. See the example graph on the next page for a description of the EP. Depending on the ORP electrode, generally look for the EP to occur between 275 to 375 mV.

Record the volume of titrant used ($V_t$) at the EP. Repeat the titration as desired or required by your protocol. After each titration, rinse the electrode and stirrer with LRW and tap electrode to remove excess water droplets.

## Quality Control (QC)

Recommended QC procedures may include: standardization of the iodine titrant*, analysis of $SO_2$ standard* or QC sample, and/or replicates.

*See Application Note #016: Standardization of Iodine Titrant for Ripper Titration of Wines.

## Calculation of Free or Total $SO_2$

$SO_2$ (mg/L) = $V_I \times N_I \times 1280$

$V_I$ = Volume of iodine titrant used at the endpoint of the titration (mL)
$N_I$ = Normality of the iodine titrant (certified or standardized value)
1280 = (32g $SO_2$/equivalent × 1000 mg/g)/25 mL wine

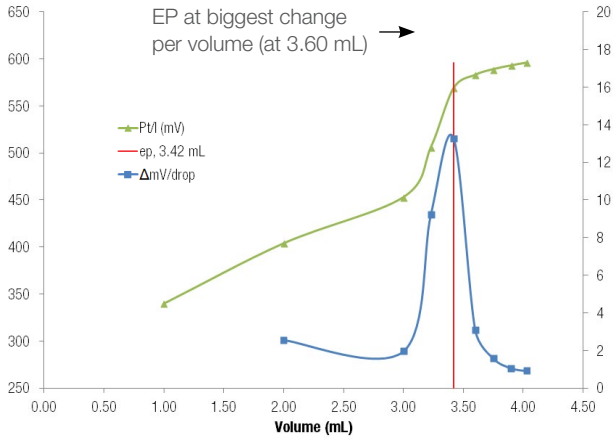
## Results - Total $SO_2$ by Manual Ripper Titration with Orion Platinum ORP Electrode

| Red Wine Sample | Endpoint Vol (mL) | Total $SO_2$ (mg/L) |
|---|---|---|
| 1 | 3.80 | 80 |
| 2 | 3.60 | 76 |
| 3 | 3.65 | 77 |
| Statistics | avg | 78 |
| | Stdev* | 2.2 |
| | RSD | 2.8% |

| $SO_2$ QC Sample | Total $SO_2$ Result (mg/L) | Within ±7 mg/L?* |
|---|---|---|

*per Zoecklein et. el, anticipated error is ±7 mg/L

**Graph of Endpoint - Total $SO_2$ in red wine**




## Endpoint Location – Platinum ORP Electrode

Note that at the EP, one drop of titrant will generally cause a mV change of >10 mV, while a mV change of >5 mV per drop generally means the EP is near, either quickly approaching or has just been passed. If another drop of titrant causes a smaller change than the last drop, the EP has passed, but if a larger change is observed, the EP is still approaching. Record the volume of titrant used ($V_t$) at the EP.

## Hints and Tips for $SO_2$ Titration with Platinum ORP Electrode

- Per Zoecklein, consider using sodium bicarbonate to minimize loss of $SO_2$ during titration as follows: After the 10 minute hydrolysis (for total $SO_2$) and just before adding the 25% sulfuric acid, quickly add a pinch of sodium bicarbonate (0.5g or less) to the sample. The solution will fizzle forming a $CO_2$ atmosphere to minimize loss of $SO_2$.
- Iodine Titrant Standardization: Iodine titrant solution ages and changes concentration over time. For higher accuracy, standardize the titrant daily or weekly before titrating samples. See Application Note #016: Standardization of Iodine Titrant for Ripper Titration of Wines.
- Refer to the electrode user guide for details on cleaning, storage, and maintenance recommendations to keep the electrode performing well. Main points for electrode care are summarized as follows.

| Daily Care | Weekly Care | As Needed |
|---|---|---|
| • Top up fill solution<br>• Store electrode in 4M KCl or water | • Drain and refill the fill solution<br>• Clean the electrode with 75% methanol or ethanol | • Clean the electrode with HCl or Orion 900023<br>• Measure mV of titrant to check electrode operation |

- Filling solution - each day top up the filling solution in the electrode. On a weekly basis, drain and refill the electrode with filling solution, for best performance.
- Electrode Storage – store the electrode in 4M potassium chloride solution or store in water.
- Periodically clean the ORP electrode by stirring 1 minute in 75% methanol or alcohol. Wipe the platinum sensor gently with a lint free wiper afterwards.
- If periodic cleaning and refilling described above does not maintain or restore performance, clean with 0.1 to 1M HCl or Orion 900023 pH cleaning solution C. Follow directions (user guide or instruction sheet).
- To check the operation of the electrode, immerse the electrode in a portion of the 0.02N iodine titrant. Expect the mV reading to be near 410 mV (±30 mV) within 1-2 minutes. If not, polish the platinum sensor gently with a lint free wiper, then drain and refill the electrode to restore proper readings.

**References**


1. Zoecklein et al. *Wine Analysis and Production*, Chapman and Hall. 1995.
2. Napa Valley College. *Laboratory Analysis of Musts and Wines*, Viticulture and Enology Department. 2007. http://www.napavalley.edu/

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To purchase an Orion platinum ORP electrode, or other related products, please contact your local equipment distributor and reference the part numbers listed below.

Depending on your sample throughput needs and budget, we have developed application notes for both our electrochemistry meters for manual titrations and potentiometric titrators for automated titrations. Our automated titrators can help improve your titrations by simplifying your process and helping to deliver consistently reliable results. Just program it once and the titrator takes care of the rest - including addition of titrant, endpoint determination, results calculation, and data logging. Please visit **thermofisher.com/titrator** for more information.

| Product | Description | Cat. No. |
|---|---|---|
| Meters | Thermo Scientific Orion Versa Star Pro pH Benchtop Meter | VSTAR10 |
| | Thermo Scientific Orion Star A211 pH Benchtop Meter | STARA2110 |
| | Thermo Scientific Orion Star A214 pH/ISE Benchtop Meter | STARA2140 |
| Electrodes | Thermo Scientific™ Orion™ Combination Platinum ORP Electrode, Glass Body, BNC Waterproof Connector | 9778BNWP |
| | Thermo Scientific™ Orion™ Sure-Flow™ Platinum ORP Electrode, Epoxy Body, BNC Waterproof Connector | 9678BNWP |
| Solutions | Thermo Scientific Orion Electrode Filling Solution for ORP Electrode | 900011 |
| | Thermo Scientific Orion pH Electrode Storage Solution | 910001 |
| Accessories | Stirrer Probe | 096019 |
| | Electrode Storage Sleeve and Base | 810017 |
| | Swing Arm Stand | 090043 |
| | Thermo Scientific™ Orion™ ATC Temperature Probe with Steel Body | 927007MD |
| Laboratory Reagent Water | Thermo Scientific™ Barnstead™ Water Purification Systems | Multiple |

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APPLICATION NOTE



# Sulfur dioxide in wine by enhanced manual ripper titration with platinum and iodide electrode

**Key Words**
Wine analysis, sulfur dioxide, Ripper titration, platinum and iodide electrode, mV endpoint titration, $SO_2$.

## Goal

The following application note explains how to analyze sulfur dioxide in wine using a Ripper titration with a Thermo Scientific™ Orion™ 9770BNWP Platinum and Iodide Electrode.

## Introduction

Sulfur dioxide ($SO_2$) is widely used in wine production as a chemical antioxidant and inhibitor of microbial activity. $SO_2$ in wine is traditionally analyzed by Ripper titration using a color indicator. In this note, the same Ripper titration is performed using an Orion 9770BNWP Platinum and Iodide Electrode to signal the endpoint at a known mV value. The color and clarity of red wine does not interfere, thereby improving the results. The Orion 9770BNWP Electrode is fast, low maintenance, requires no fill solution, and is long-lasting. The mV endpoint titration is comparable to a Titratable Acidity (TA) determination to pH 8.2. This method provides a simple solution to $SO_2$ analysis in wine.

## Basic Titration Workflow


## Recommended Equipment

- Thermo Scientific™ Orion™ Versa Star Pro™ pH/mV Meter, Thermo Scientific™ Orion Star™ A211 pH/mV Meter, or equivalent Orion pH/mV meter
- Orion 9770BNWP Platinum and Iodide Electrode (residual chlorine ISE)
- Stirrer Probe (Cat. No. 096019)
- Swing Arm Stand (Cat. No. 090043)
- Electrode Polishing Strip (Cat. No. 948201)
- 10 mL burette, burette clamp, ring stand

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## Required Reagents and Solutions

- Purchased or prepared Iodine ($I_2$) standard titrant solution, 0.01 M (0.02 N)
- Sodium hydroxide (NaOH), 1 N
- 25% sulfuric acid (1+3 $H_2SO_4$)
- Laboratory Reagent Water (LRW)

Optional:

- Standard sodium thiosulfate solution, 0.01 M (0.01 N)
- Sodium sulfite ($Na_2SO_3$) or potassium metabisulfite (KMBS)
- Sodium bicarbonate ($NaHCO_3$)

## Meter Setup

Connect the Orion electrode and the stirrer probe to the meter. In Setup, select the mV mode, set read type to continuous, and set the stirrer speed to 3. Refer to the Orion meter and electrode user guides for more details.

## Titration Setup

Secure the burette on the clamp. Fill the burette with iodine titrant solution and adjust the level to the zero mark. Consider standardizing the titrant before titrating samples. See *Hints and Tips* section on the following page.

## Sample Preparation

Make sure the wine sample is at room temperature.

**Total $SO_2$:** Add 25.0 mL of wine and 25 mL of 1 N NaOH to a 100 mL beaker. Mix and allow 10 min for hydrolysis. After 10 minute, proceed immediately to the *Sample Titration*.

**Free $SO_2$:** Add 25.0 mL of wine to a 100 mL beaker. Proceed immediately to the *Sample Titration*.

### Sample Titration

The Ripper titration should be done relatively quickly to avoid loss of $SO_2$ to the air. Rinse the electrode and stirrer with LRW. Immerse the electrode and stirrer at least one inch below the liquid level in the beaker, with the stirrer just below the electrode. Turn on the stirrer. Stir gently so that a vortex is not created. Tap the electrode to release air bubbles trapped on the surface of the electrode.

**Total $SO_2$:** Add 10 mL of 25% sulfuric acid to the beaker.

**Free $SO_2$:** Add 5 mL of 25% sulfuric acid to the beaker.

Watching the mV reading, titrate at moderate speed with the iodine titrant. Expect the endpoint (EP) to occur near 555 mV (±35 mV) when titrating red wine. The mV values will not rise quickly, until near the EP. The EP is considered the point where the largest mV change is observed per volume addition of titrant. See the example graph on the next page for a description of the EP.

Record the volume of titrant used ($V_t$) at the EP and the mV reading at the EP. For subsequent titrations of this wine today, titrate to this mV value. Repeat the titration as desired or required by user's protocol. After each titration, rinse the electrode and stirrer with LRW and tap electrode to remove excess water droplets.

## Quality Control (QC)

Recommended QC procedures may include: standardization of the iodine titrant*, analysis of $SO_2$ standard* or QC sample, and/or replicates.

*Application Note #016: Standardization of Iodine Titrant for Ripper Titration of Wines.

## Calculation of Free or Total $SO_2$

$SO_2$ (mg/L) = $V_I \times N_I \times 1280$

$V_I$ = Volume of iodine titrant used at the endpoint of the titration (mL)
$N_I$ = Normality of the iodine titrant (certified or standardized value)

1280 = (32g $SO_2$/equivalent × 1000 mg/g)/25 mL wine

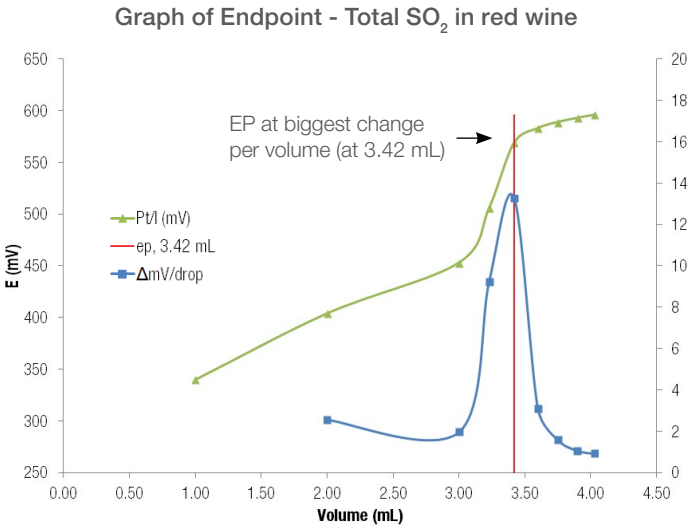
## Results - Total $SO_2$ by Manual Ripper Titration with Orion Platinum and Iodide Electrode (9770BNWP)

| Red Wine Sample | Endpoint Vol (mL) | Total $SO_2$ (mg/L) |
|---|---|---|
| 1 | 3.53 | 75 |
| 2 | 3.42 | 73 |
| 3 | 3.65 | 78 |
| Statistics | avg | 75 |
| | Stdev* | 2.4 |

| $SO_2$ QC Sample | Total $SO_2$ Result (mg/L) | Within ±7 mg/L?* |
|---|---|---|
| 68 mg/L | 63 | Yes |

## Endpoint Location – Platinum and Iodide Electrode

Note that at the EP, one drop of titrant will generally cause




a mV change of >10 mV, while a mV change of >5 mV per drop generally means the EP is near, either quickly approaching or has just been passed. If another drop of titrant causes a smaller change than the last drop, the EP has passed, but if a larger change is observed, the EP is still approaching. Record the volume of titrant used ($V_t$) at the EP and the mV reading at the EP. For subsequent titrations of this wine today, titrate to this mV value.

## Hints and Tips for $SO_2$ Titration with Platinum and Iodide Electrode

- Per Zoecklein, consider using sodium bicarbonate to minimize loss of $SO_2$ during titration as follows: After the 10 minute hydrolysis (for total $SO_2$) and just before adding the 25% sulfuric acid, quickly add a pinch of sodium bicarbonate (0.5 g or less) to the sample. The solution will fizzle forming a $CO_2$ atmosphere to minimize loss of $SO_2$.

- Iodine Titrant Standardization: Iodine titrant solution ages and changes concentration over time. For higher accuracy, standardize the titrant daily or weekly before titrating samples. See Application Note #016: Standardization of Iodine Titrant for Ripper Titration of Wines.

- Refer to the electrode user guide for details on cleaning, storage, and maintenance recommendations to keep the electrode performing well. Main points for electrode care are summarized as follows.

| Daily Care | Weekly Care | As Needed |
|---|---|---|
| • Store electrode dry | • Clean the electrode with 75% methanol or ethanol | • Gently polish sensor<br>• Measure mV of titrant to check electrode operation |

- Electrode storage - Thoroughly rinse the electrode with LRW water and store the electrode dry.

- Periodically clean the electrode by stirring 1 minute in 75% methanol or alcohol. Wipe the platinum sensor gently with a lint free wiper afterwards.

- If periodic cleaning and refilling described above does not maintain or restore performance, clean by gently polishing the platinum sensor with an Orion 948201 polishing strip. See electrode user guide for details.

- To check the operation of the platinum and iodide (9770BNWP) electrode, immerse the electrode in a portion of the 0.02N iodine titrant. Expect the mV reading to be near 670 mV (±20 mV) within 1-2 minutes. If not, polish the platinum sensor gently with a lint free wiper, then drain and refill the electrode to restore proper readings.

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Depending on your sample throughput needs and budget, we have developed application notes for both our electrochemistry meters for manual titrations and potentiometric titrators for automated titrations. Our automated titrators can help improve your titrations by simplifying your process and helping to deliver consistently reliable results. Just program it once and the titrator takes care of the rest - including addition of titrant, endpoint determination, results calculation, and data logging. Please visit thermofisher.com/titrator for more information.


**References**

1. Zoecklein et al. *Wine Analysis and Production*, Chapman and Hall. 1995.
2. Napa Valley College. *Laboratory Analysis of Musts and Wines*, Viticulture and Enology Department. 2007. http://www.napavalley.edu/


To purchase an Orion Platinum and Iodide Electrode, or other related products, please contact your local equipment distributor and reference the part numbers listed below.

| Product | Description | Cat. No. |
|---|---|---|
| Meters | Thermo Scientific Orion Versa Star Pro pH Benchtop Meter | VSTAR10 |
| | Thermo Scientific™ Orion Star™ A211 pH Benchtop Meter | STARA2110 |
| | Thermo Scientific™ Orion Star™ A214 pH/ISE Benchtop Meter | STARA2140 |
| Electrodes | Thermo Scientific™ Orion™ Platinum and Iodide Electrode (Residual Chlorine ISE) | 9770BNWP |
| Accessories | Stirrer Probe | 096019 |
| | Electrode Polishing Strip | 948201 |
| | Swing Arm Stand | 090043 |
| Laboratory Reagent Water | Thermo Scientific™ Barnstead™ Water Purification Systems | Multiple |

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APPLICATION NOTE



# Standardization of iodine titrant for ripper titration of wines

**Key Words:** Wine analysis, iodine titrant, Ripper titration, standardization workflow, sulfur dioxide titration, $SO_2$ titration

## Goal

The following application note explains how to determine the true concentration of the iodine titrant used for Ripper titration of wine by standardizing with a Thermo Scientific™ Orion™ 9770BNWP Platinum and Iodide Electrode or a Thermo Scientific Orion ORP electrode (e.g. 9778BNWP or 9678BNWP) and a standard solution of sodium thiosulfate.

## Introduction

Since the concentration of the iodine titrant changes over time, for best accuracy, determine the true concentration of the iodine titrant by standardizing with a standard solution of sodium thiosulfate. Typically it is best to standardize daily or weekly.

## Basic Standardization Workflow

1. Fill buret with titrant
2. Prepare standard in beaker
3. Insert electrode and stir
4. Titrate to endpoint
5. Calculate true titrant normality

## Required Reagents and Solutions

Purchased or prepared Iodine ($I_2$) standard titrant solution, 0.01 M (0.02 N); deionized or distilled water (DI); standard sodium thiosulfate ($Na_2S_2O_3$) solution, 0.01 M (0.01 N). Optional: sodium sulfite ($Na_2SO_3$) or potassium metabisulfite (KMBS).

## Meter and Titration Setup for Standardization

Prepare according to the $SO_2$ in Wine by Enhanced Manual Ripper Titration (Application Note 017) or Manual Ripper Titration (Application Note 018) method.

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## Standard Titration

Add 5.0 mL of 0.01 N sodium thiosulfate solution to 50 mL DI in a 100 mL beaker. Rinse the electrode and stirrer with DI. Immerse the electrode and stirrer at least one inch below the liquid level in the beaker. Turn on the stirrer. Tap to release air bubbles trapped on the surface of the electrode.

Watching the mV reading, titrate at moderate speed with the iodine titrant. The mV values will not rise quickly until near the endpoint (EP). The EP is considered the point where the largest mV change is observed per volume addition of titrant. Depending on the ORP electrode, generally look for the EP to occur between 325 to 425 mV when standardizing with sodium thiosulfate and the Orion platinum ORP electrode (e.g., Cat. No. 9778BNWP or Cat. No. 9678BNWP). Expect the EP to occur near 595 mV (± 20 mV) when standardizing with sodium thiosulfate and an Orion platinum and iodide electrode (Cat. No. 9770BNWP).

At the EP, one drop of titrant will generally cause a mV change of >10 mV, while a mV change of >5 mV per drop generally means the EP is quickly approaching or has just been passed. If another drop of titrant causes a smaller change than the last drop, the EP has passed, but if a larger change is observed, the EP is still approaching. Record the volume of titrant used (Vt) at the EP. When using the Orion platinum and iodide electrode, record also the mV value at the EP.

Repeat the standardization procedure if required by your protocol. When using the Orion platinum and iodide electrode, simply titrate to the EP mV value found for the first titration. When using the Orion platinum ORP electrode, titrate to the largest mV change. Calculate the normal concentration of iodine titrant (NI) for each portion of standard titrated, as follows:

$N_I = N_S \times 5 \div V_I$

$N_I$ = Normality of the iodine titrant (certified or standardized value)

$N_S$ = Normality of the standard thiosulfate solution

$V_I$ = Volume of iodine titrant used at the endpoint of the titration (mL)

Use the average $N_I$ result determined here when titrating wine samples.

## Optional QC Sample – $SO_2$ Standard

### Option 1

Weigh 0.2956 g sodium sulfite ($Na_2SO_3$) and ~1.0 g citric acid into a 1 L volumetric flask. Dissolve and fill to the mark with DI water. This is equivalent to 150 mg/L total $SO_2$ stock solution. Pipette 10 mL of the $SO_2$ stock solution into a beaker and add 50 mL of DI water. This is equivalent to 60 mg/L $SO_2$ in a 25 mL wine sample.

### Option 2

Dissolve 3 KMBS (Campden) tablets in 2L of DI water for 426 mg/L $SO_2$ solution. Pipette 4 mL of the $SO_2$ solution into a beaker and add 50 mL of DI water. This is equivalent to 68 mg/L $SO_2$ in a 25 mL wine sample.

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To purchase Orion meter, electrodes and solutions, please contact your local equipment distributor and reference the part numbers listed below:

Depending on your sample throughput needs and budget, we have developed application notes for both our electrochemistry meters for manual titrations and potentiometric titrators for automated titrations. Our automated titrators can help improve your titrations by simplifying your process and helping to deliver consistently reliable results. Just program it once and the titrator takes care of the rest - including addition of titrant, endpoint determination, results calculation, and data logging. Please visit **thermofisher.com/titrator** for more information.

**Ordering Information**

| Product | Cat. No. |
|---|---|
| **Meters** | |
| Thermo Scientific™ Orion™ Versa Star Pro™ pH Benchtop Meter | VSTAR10 |
| Thermo Scientific™ Orion Star™ A211 pH Benchtop Meter | STARA2110 |
| Thermo Scientific™ Orion Star™ A214 pH/ISE Benchtop Meter | STARA2140 |
| **Electrodes** | |
| Thermo Scientific™ Orion™ Redox/ORP Electrode, Glass Body | 9778BNWP |
| Thermo Scientific™ Orion™ Platinum and Iodide Electrode (Residual Chlorine ISE) | 9770BNWP |
| Thermo Scientific™ Orion™ Sure-Flow™ Redox/ORP Electrode, Epoxy Body | 9678BNWP |
| **Accessories** | |
| Electrode Storage Sleeve and Bottle | 810017 |
| Stirrer Probe | 096019 |
| Swing Arm Stand | 090043 |

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APPLICATION NOTE



# Measuring clarity in wine

**Key Words**
wine clarity, turbidity, beverage quality, fermentation, barrel testing, filtration, wine tank testing.

## Goal

The following application note explains how to measure the turbidity of red, white and rosé wine samples using a Thermo Scientific™ Orion™ AQUAfast™ turbidity meter. The analysis of wine turbidity may be used to evaluate chill haze, protein stability, and wine clarity. In this note, the evaluation of wine clarity is described.

## Introduction

Orion™ AQUAfast™ AQ3010 and AQ4500 Turbidity Meters allow quick and simple determinations of the clarity of white, rosé, and red wine samples. Understand how to measure the clarity or "turbidity" of various wine samples using the AQ310 model, or the infrared mode in the AQ500 model. As the light source is infrared, the turbidity measurement is independent of color.

## Recommended Equipment

- Orion™ AQ3010 Turbidity Meter and Orion™ AC3V25 Turbidity Vials

OR

- Orion™ AQ4500 Turbidity Meter and Orion™ AC2T24 Turbidity Vials

## Required Reagents and Solutions

- Orion AC301S Turbidity Standards (if using AQ3010)
- Orion AC45ST Turbidity Standards (if using AQ4500)
- Turbidity-free water (TFW), e.g., by filtration through 0.1 um filter, or equivalent water

## Solutions Preparation

None.

## Meter Setup

None.

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### Meter Performance Check/Calibration Verification

Orion AC301S and AC45ST styrene divinylbenzene (SDVB) polymer turbidity standards never need mixing. Do not shake the standards as this will introduce bubbles and cause them to read inaccurately until the bubbles dissipate.

### AQ3010

Check meter accuracy by reading one or more turbidity standards (included with the meter) at the level of interest. For example, read the zero (0.02) and the 20 NTU standard. The zero should read <0.1 NTU and the 20 NTU standard should read within ±10%, e.g., 18-22 NTU.

### AQ4500

Review certificate of analysis of the turbidity standards and record the expected turbidity values for the IR Ratio mode.

Set the meter to the IR Ratio mode. Check meter accuracy by reading one or more turbidity standards at the level of interest. For example, read the zero (0.02) and the 1 NTU standard. The zero should read <0.1 NTU and the 1 NTU standard should read within ±10% from the expected value according to the Certificate of Analysis.

If the AQ3010 or AQ4500 meter performance check fails, take corrective actions as follows:

1. Wipe the vial carefully with a lint-free wipe to remove all fingerprints and liquid drips from the exterior, handle the vial by the cap only, and remeasure.
2. Tap the vial gently three times and let the vial sit for 60 seconds to allow for bubbles to release, then remeasure.
3. Using a clean vial (which reads <0.1 NTU when filled with TFW), pour a fresh portion of turbidity standard into the clean vial, wipe carefully, and measure.

### Sample Vial (Cuvette) Storage, Soaking, and Rinsing

Store vials filled with TFW. Immediately after use, clean sample vials with laboratory detergent and rinse multiple times with TFW. Note: Standards may be stored in supplied glass sample vials until the standard reading is no longer in specification. See Meter Performance Check section for corrective actions when a standard reads out of specification.

### Sample Storage and Preparation

In general, allow the samples to warm to room temperature before measurement. Mix the sample well, but do not introduce bubbles by shaking the sample. Use a little of the sample to rinse a clean sample vial twice. Mix the sample again and fill the rinsed vial.

### Calibration - AQ3010

The meter is shipped precalibrated. The meter performance is very stable and does not require frequent calibration. If a standard reading is not within criteria, take all necessary corrective actions (as described in the Meter Performance Check section) to improve meter readings. If corrective actions fail and recalibration is necessary, perform the recalibration only on the points that failed and do so with fresh portions of standard poured into clean vials. Ensure that all fingerprints and liquid drips have been removed from the exterior of the vial with a lintfree wipe before using. Handle vials by the cap only.

### Calibration - AQ4500

The meter is shipped precalibrated. The meter performance is very stable and does not require frequent calibration. If a standard reading is not within criteria, take all necessary corrective actions (as described in the Meter Performance Check section) to improve meter readings. If corrective actions fail and recalibration is necessary, perform the recalibration in IR Ratio mode (see the Initial Calibration section of the Meter User Guide and an example on page 3).

### Analysis

Gently invert the filled sample vial a few times to mix the sample well without introducing bubbles. Wipe the sample vial to remove all traces of liquids and fingerprints, place into meter, and press the measure key. Record the reading. Press the measure key to take duplicate measurement(s). Continue until readings stabilize and results agree, for example, within 5% or ±0.02 NTU, whichever is higher.

### Quality Control (QC)

Recommended QC procedures include: calibration verification, turbidity-free water analysis (optional), and sample duplicates.

### Notes for Improved Accuracy of Low-level Samples

If improved accuracy is desired, pay close attention to:

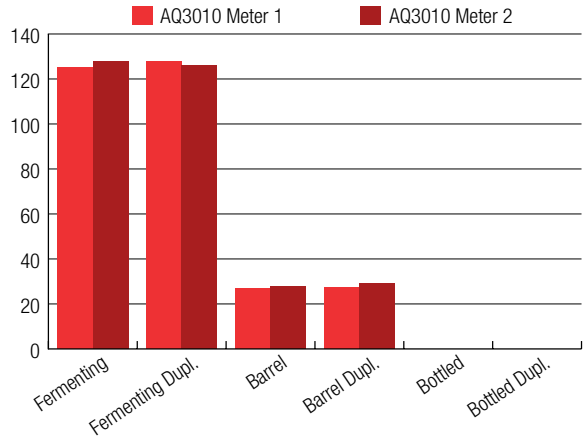
- The cleanliness of the sample vials.
- The quality of the TFW.
- The handling of the standards and samples.
- Use of matching vials.
- Storing clean vials filled with TFW.
- Use vials free of scratches or other imperfections.

For improved low-level accuracy, ensure that a clean vial filled with TFW reads < 0.1 NTU before using that vial to test highly filtered wine. If a clean vial does not read <0.1 NTU, discard it or set it aside for further cleaning. If no clean vials read <0.1 NTU, the TFW may need degassing or a cleaner source of TFW may be required. See ASTM D6855 Test Method for Test Method for Determination of Turbidity Below 5 NTU in Static Mode for more information about low level turbidity readings.

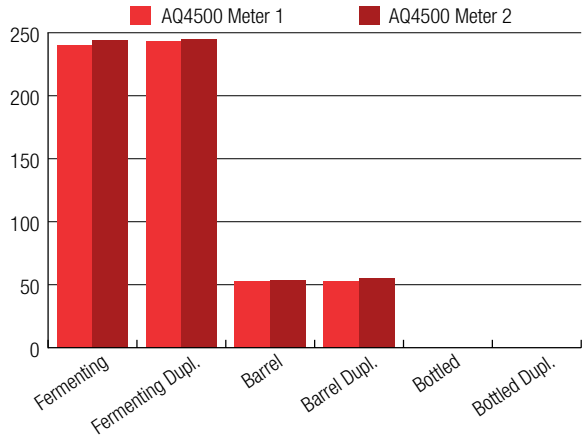
## Results

Various wine samples, taken at different stages of the winemaking process, were tested for turbidity on the AQ3010 and AQ4500.

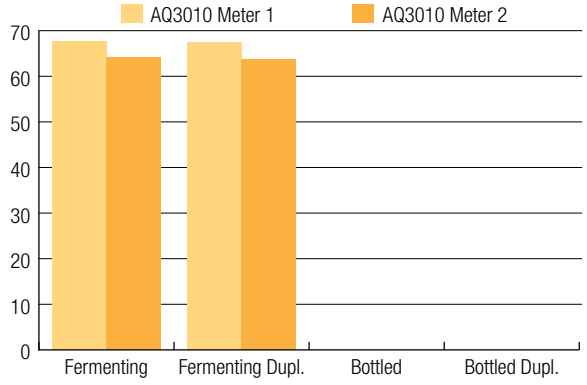
**Turbidity of Red Wine (AQ3010)**



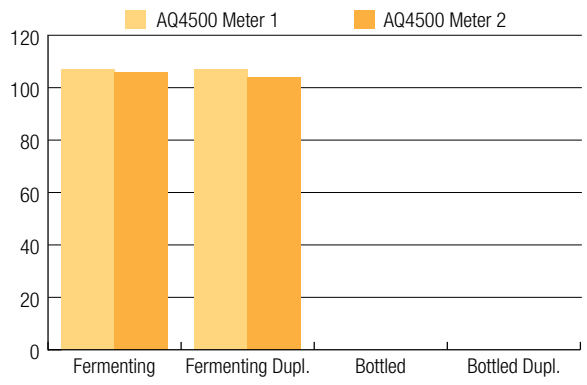

**Turbidity of Red Wine (AQ4500)**



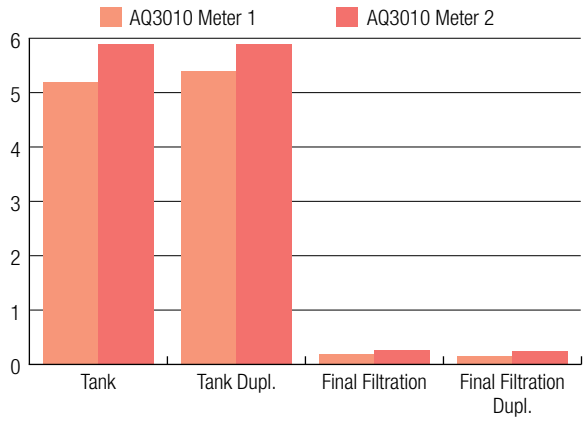

**Turbidity of White Wine (AQ3010)**



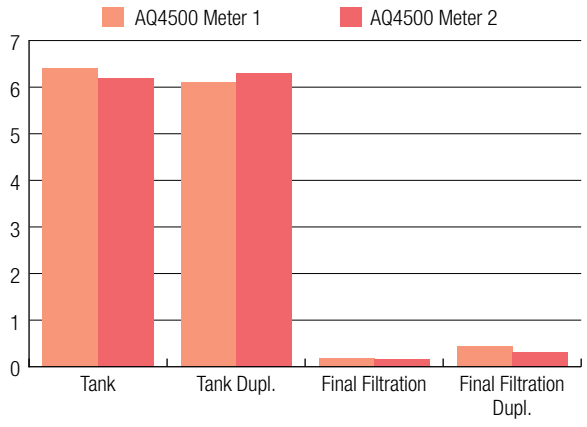

**Turbidity of White Wine (AQ4500)**




**Turbidity of Rosé Wine (AQ3010)**




**Turbidity of Rosé Wine (AQ4500)**



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## Summary

The Orion AQUAfast AQ3010 Turbidity Meter allows accurate measurement of red, white, and rosé wines at various stages of the wine-making process. The infrared light source allows readings which are not affected by the deep color of red wines or the blush color of rosé wines.

The Orion AQUAfast AQ4500 Turbidity Meter allows accurate measurement of red, white, and rosé wines at various stages of the wine-making process. When measurements are performed in the infrared ratio mode, readings are not affected by the deep color of red wines or the blush color of rosé wines.

To purchase an Orion turbidity meter, or other related products, please contact your local equipment distributor and reference the part numbers listed below.

### Results of Testing Turbidity Standards using an AQ3010 Meter

| Expected Value | AQ3010 Meter 1 | % Recovery | AQ3010 Meter 2 | % Recovery |
|---|---|---|---|---|
| 0.02NTU (<0.1) | 0.00 | NA | 0.00 | NA |
| 20NTU | 18.9 | 94.4% | 20.1 | 100.5% |
| 100NTU | 96.3 | 96.3% | 101 | 101.0% |
| 800NTU | 772 | 96.5% | 798 | 99.8% |

### Results of Testing Turbidity Standards using an AQ4500 Meter

| Expected Value | AQ4500 Meter 1 | % Recovery | AQ4500 Meter 2 | % Recovery |
|---|---|---|---|---|
| <0.1 | 0.00 | NA | 0.03 | NA |
| 0.93 | 0.95 | 102.2% | 0.93 | 100.0% |
| 9.54 | 9.30 | 97.5% | 9.65 | 101.2% |
| 99.4 | 99.6 | 100.2% | 99.8 | 100.4% |
| 708 | 742 | 104.8% | 722 | 102.0% |

### Ordering Information

| Product | Description | Cat. No. |
|---|---|---|
| Turbidity Meters | Thermo Scientific Orion AQUAfast AQ3010 Turbidity Meter | AQ3010 |
| | Thermo Scientific Orion AQUAfast AQ4500 Turbidity Meter | AQ4500 |
| Accessories | Thermo Scientific Orion Turbidity Vials, for use with the AQ3010 | AC3V25 |
| | Thermo Scientific Orion Turbidity Vials, for use with the AQ4500 | AC2T24 |
| Solutions | Thermo Scientific Orion Turbidity Standards (0, 1, 10, 100, 1000 NTU), for use with the AQ4500 | AC45ST |
| | Thermo Scientific Orion Turbidity Standards, for use with the AQ3010 | AC301S |

Find out more at **thermofisher.com/water**

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APPLICATION NOTE



# Measuring the dissolved oxygen of wine in tanks

**Key Words:** Wine quality, dissolved oxygen, DO, optical sensor, portable meter, beverage testing.

## Goal

The following application note describes how to reliably measure the oxygen content of wine directly in the tank, using a Thermo Scientific™ Orion™ Optical Dissolved Oxygen Sensor with automatic temperature compensation and a Thermo Scientific™ Orion Star™ A223 Dissolved Oxygen (DO) Portable Meter.

## Introduction

Wineries have become increasingly concerned with the oxygen incorporation in the wine during the bottling process. This is an extremely important issue that influences wine quality, stability, and longevity. Although oxygen is a part of the wine's natural aging process, adverse levels can cause discoloration to white wines and flavor degradation to both white and red varietals. The concentration of molecular oxygen should be measured in the wine before bottling begins, and throughout the entire wine making process.

By using a Thermo Scientific Orion Optical Dissolved Oxygen Sensor and a Thermo Scientific Orion Star A223 Dissolved Oxygen (DO) Portable Meter, reliable oxygen measurements can be made directly in the tanks which hold the wine.

## Equipment

- Orion Star A223 Dissolved Oxygen (DO) Portable Meter Kit – includes optical DO sensor, portable meter armor, field case and USB computer cable (Cat. No. STARA2235) or
- Orion Star A223 DO Portable Meter (Cat. No. STARA2230) or equivalent Orion portable DO meter
- Optical DO Sensor – includes calibration sleeve and stainless steel sensor guard (Cat. No. 087010MD)

## Solutions

- Deionized water (DI)

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## Luminescence-Based Dissolved Oxygen Method

The oxygen content of wine must be monitored throughout the wine-making process. Using the optical DO sensor with built-in automatic temperature compensation and a portable meter, reliable measurements can be directly in the tanks which hold the wine.

## Optical DO Sensor Setup

Refer to the Optical Dissolved Oxygen Sensor User Guide for detailed assembly and preparation instructions for the sensor. Place the optical DO sensor into the calibration sleeve and moisten the sponge in the calibration sleeve with deionized water. Connect the optical DO sensor to the 9-pin MiniDIN input on the meter. Once assembled, the optical DO sensor can be used immediately.

## Meter Setup

Turn the Star A223 Dissolved Oxygen (DO) Portable Meter on. The meter should automatically detect the type of DO sensor and update the measure type to optical DO. In the measurement mode, set the measurement units to mg/L. Access the setup menu and update the channel settings to the following, as needed:

- Measure Mode: Auto
- Measure Unit: mg/L
- Resolution: 0.01
- Read Type: Auto Read
- Baro Pressure: Auto
- Salinity Correct: Manual (0.0)

Update the instrument settings to the following, as needed:

- Export Data: On
- Data Log: On
- Date / Time: Set current date & time

## Sensor Performance Checks

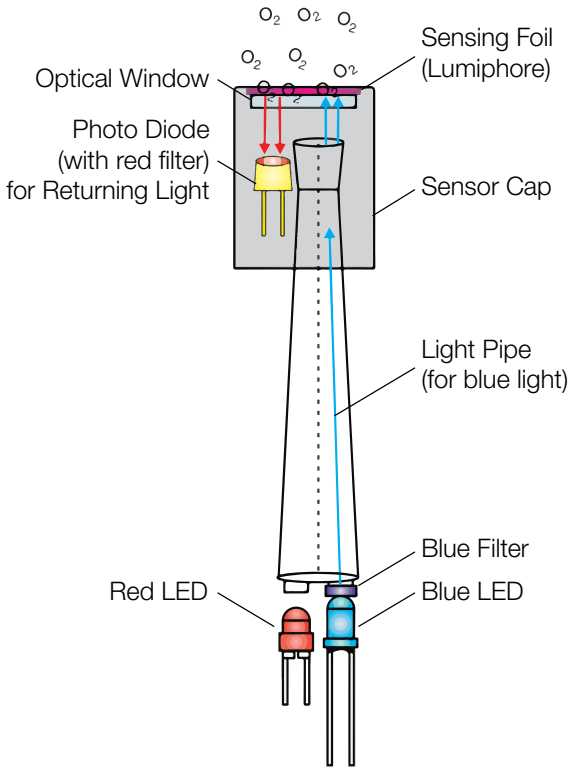
The optical DO sensor should read between 98 and 102 % saturation in the calibration sleeve after calibration. The optical DO sensor should stabilize during calibration within 2 minutes when working properly. Make sure to thoroughly rinse and blot dry the optical DO sensor after measuring samples and before placing into the calibration sleeve (see Comments section). Refer to the optical DO sensor user manual if the sensor does not pass the performance checks.

## Luminescence-Based Dissolved Oxygen Sensing

The Orion Optical Dissolved Oxygen Sensor measures dissolved oxygen in liquid with a luminescence-based optical sensor. Using an optical sensor, the luminescence-based method monitors the time it takes to quench an excited lumiphore, which is inversely proportional to the concentration of oxygen.

Luminescence-based Dissolved Oxygen Sensing is one of the three affirmed methods for dissolved oxygen measurement by the American Society for Testing and Materials (ASTM). The other two methods are the Winkler titration method and the electrochemical membrane method – both of which can also be performed using the Star A223 Dissolved Oxygen (DO) Portable Meter and Orion polarographic DO probe.

**Optical Sensor/Luminescence-Based Dissolved Oxygen Sensor**




## Sensor Rinsing, Soaking and Storage

After each sample measurement, rinse the optical DO sensor thoroughly with deionized water and blot the sensor dry with a lint-free cloth. For short term storage, overnight or between measurements, keep the optical DO sensor in the calibration sleeve or a biochemical oxygen demand (BOD) bottle with water-saturated air. For long-

### Sample Preparation and Preservation

Dissolved oxygen can be measured directly in the tank. Samples cannot be preserved. Measure samples on location for the best results, or immediately following sample collection. If samples must be collected and moved, use an air-tight container, fill completely with no air space, and keep the container sealed until immediately before measuring the sample.

### Calibration

Prepare the calibration sleeve by moistening the sponge, squeezing out excess water, and inserting the optical DO probe. Perform a water-saturated air (Air) calibration with the optical DO probe in the prepared calibration sleeve. The water from the sponge will saturate the air in the calibration sleeve and act as the calibration standard. A stable reading of 100.0 % saturation should be displayed within about two minutes during the calibration.

### Analysis

Rinse the optical DO sensor with deionized water and blot excess rinse water off with a lint-free cloth. If measuring below the tank surface, attach the stainless steel sensor guard to the sensor to weigh and sink the sensor to the desired tank depth.

Place the optical DO sensor in the tank, making sure that the temperature sensor is also submerged in the sample. Initiate a reading using the Auto Read measurement mode by pressing the measure key on the meter keypad. For best results, take a second reading to ensure the dissolved oxygen measurement is fully stabilized, as it may take the optical DO sensor one to two minutes to fully stabilize in the wine sample. Use the second stable value for the oxygen content of the wine. Both readings will be saved in the meter data log.

### Comments

It is important to thoroughly clean the optical DO sensor after sample measurement. Rinse with deionized water and thoroughly blot all excess water with a lint free cloth several times before putting the sensor in the calibration sleeve. Rinsing following the completion of all sample measurements should take 5 to 10 minutes.

The Star A223 meter data log collects up to 1000 measurement sets with time and date stamp and the non-volatile meter memory preserves data, even with loss of power. Download Orion Star Com software to facilitate the transfer of the data log from the meter to a computer at thermofisher.com/orionsoftware. Use the Orion Star Com software to export data to a Microsoft™ Excel™ spreadsheet or as a comma separated value file (.csv) or print data to a network or local printer.

### Quality Control (QC)

Recommended QC procedures may include: calibration, check of the thermistor (temperature sensor) response against a calibrated NIST-traceable thermometer, and measurement of a zero DO solution, such as 5% sodium sulfite.

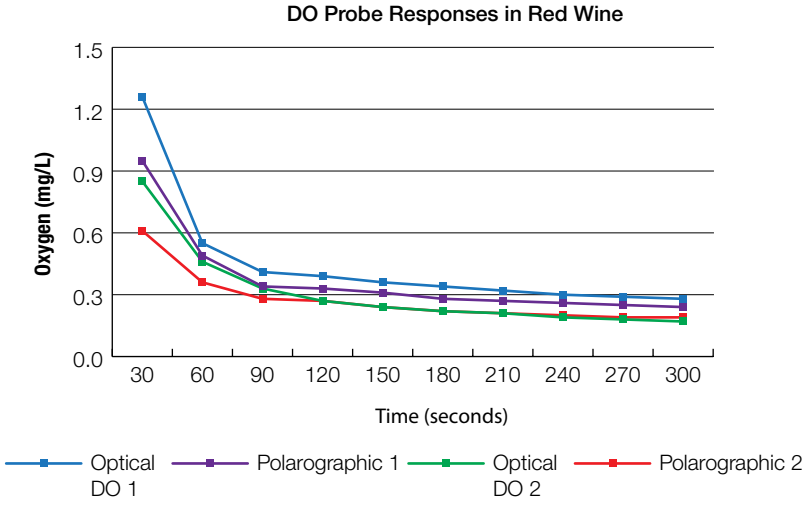
### Notes

- Keeping the calibration sleeve clean and free from water or sample droplets is essential to getting good calibration and read back values in water-saturated air. Rinse the optical DO sensor thoroughly with deionized water and wipe excess water with a lint-free cloth prior to putting the sensor in the calibration sleeve.
- Optical DO sensors do not require stirring or a sample stream for accurate measurements. The speed, accuracy, and precision of the optical DO sensor are equivalent or superior to the traditional polarographic sensor measurement.
- If readings are slow or inconsistent, ensure the temperature sensor is completely submerged in the sample.
- If the temperature sensor is not in the sample, the DO readings will be incorrect.
- The optical DO cap must be replaced every 365 days. The remaining cap life can be viewed in the channel setup menu. The meter will display an error message

## Results

### Dissolved Oxygen Readings in Wine Samples

| | Oxygen (mg/L) | | | |
|---|---|---|---|---|
| | Sample 1 | | Sample 2 | |
| Minute | Optical DO | Polargraphic | Optical DO | Polargraphic |
| 1 | 0.55 | 0.49 | 0.46 | 0.36 |
| 2 | 0.39 | 0.33 | 0.27 | 0.27 |
| 3 | 0.34 | 0.28 | 0.22 | 0.22 |
| 4 | 0.30 | 0.26 | 0.19 | 0.20 |
| 5 | 0.28 | 0.24 | 0.17 | 0.19 |





Thermo Scientific™ Orion Star™ A223 Dissolved Oxygen Portable Meter Kit

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## Summary

Using an Orion Star A223 Dissolved Oxygen (DO) portable meter with an optical dissolved oxygen sensor enables wineries to continually produce high quality wines. Because the optical DO sensor allows the wine to be measured directly from the tank, dissolved oxygen measurements can be made with speed and accuracy. The speed, accuracy and precision of the optical DO sensor is equivalent or superior to current DO measurement techniques.

To purchase an Orion Star A223 DO portable meter, Orion Optical DO Sensor and other related products, please contact your local equipment distributor and reference the part numbers listed below.

### Ordering Information

| Product | Cat. No. |
|---|---|
| **Portable Meters** | |
| Thermo Scientific Orion Star A223 Dissolved Oxygen Portable Meter | STARA2230 |
| Thermo Scientific Orion Star A223 Dissolved Oxygen Portable Meter Kit with Optical DO Sensor, Portable Meter Armor, Field Case and USB Computer Cable | STARA2235 |
| Thermo Scientific Orion Star A326 pH/DO Portable Meter Kit with ROSS Ultra Low Maintenance Gel pH/ATC Electrode, Optical DO Sensor, Portable Meter Armor, Field Case, Calibration Solutions and USB Computer Cable | STARA3265 |
| Thermo Scientific Orion Star A329 pH/ISE/Conductivity/DO Portable Meter Kit with ROSS Ultra Low Maintenance Gel pH/ATC Electrode, Conductivity Sensor, Optical DO Sensor, Portable Meter Armor, Field Case, Calibration Solutions and USB Computer Cable | STARA3295 |
| **Optical DO Sensors** | |
| Thermo Scientific Orion Optical DO Sensor with 3 Meter Cable | 087010MD |
| Thermo Scientific Orion Optical DO Sensor with 6 Meter Cable | 087020MD |
| Thermo Scientific Orion Optical DO Sensor with 10 Meter Cable | 087030MD |
| Thermo Scientific Orion Optical DO Sensor with 15 Meter Cable | 087050MD |
| Thermo Scientific Orion Optical DO Sensor with 30 Meter Cable | 087100MD |
| **Accessories** | |
| Calibration Sleeve for Optical DO Sensors | 087003 |
| Stainless Steel Protective Sensor Guard for Optical DO Sensors | 087002 |
| RS232 Computer Cable | 1010053 |

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APPLICATION NOTE



# Measuring the dissolved oxygen of wine in the bottle

**Key Words:** Wine quality, dissolved oxygen, DO sensor, optical sensor, portable meter, beverage testing.

## Goal

The following application note describes how to reliably measure the oxygen content of wine directly in the bottle, using a Thermo Scientific™ Orion™ Optical Dissolved Oxygen Sensor with automatic temperature compensation and a Thermo Scientific™ Orion Star™ A223 Dissolved Oxygen (DO) Portable Meter.

## Introduction

A bottle of wine's oxygen content has a great effect on its quality, stability and longevity. This is why monitoring and controlling the oxygen incorporation at different stages of the wine-making and bottling process is becoming a growing concern for wineries. Although oxygen is a part of the wine's natural aging process, adverse levels can cause discoloration to white wines and flavor degradation to both white and red varietals. To ensure the consumer is getting the highest quality product, measuring the concentration of molecular oxygen of wine after bottling is very important. By using an Orion Optical Dissolved Oxygen Sensor and an Orion Star A223 Dissolved Oxygen (DO) Portable Meter, reliable oxygen measurements can be made directly in the bottles of wine.

## Equipment

- Orion Star A223 Dissolved Oxygen (DO) Portable Meter Kit – includes optical DO sensor, portable meter armor, field case and USB computer cable (Cat. No. STARA2235) or
- Orion Star A223 DO Portable Meter (Cat. No. STARA2230) or equivalent Orion portable DO meter
- Optical DO Sensor – includes calibration sleeve and stainless steel sensor guard (Cat. No. 087010MD)
- Silicone tubing

## Solutions

- Deionized water (DI)

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## Luminescence-Based Dissolved Oxygen Method

The oxygen content of wine must be monitored throughout the wine-making process. Using the optical DO sensor with built-in automatic temperature compensation and a portable meter, reliable measurements can be directly in the wine bottle.

## DO Sensor Setup

Refer to the Optical DO Sensor User Guide for detailed assembly and preparation instructions for the optical DO sensor. Place DO sensor into the calibration sleeve. Remove the sponge from the bottom of the calibration sleeve. Moisten it with DI water, squeeze out excess water, and replace the sponge. Connect the optical DO sensor to the 9-pin MiniDIN input on the meter. Once assembled, the optical DO sensor can be used immediately.

## Meter Setup

Turn the meter on. The meter should automatically detect the type of DO sensor and update the measure type to optical DO. Access the setup menu and update the channel settings to the following, as needed:

- Measure Mode: Auto
- Measure Unit: mg/L
- Resolution: 0.01
- Read Type: Auto Read
- Baro Pressure: Auto
- Salinity Correct: Manual (0.0)

Update the instrument settings to the following, as needed:

- Export Data: On
- Data Log: On
- Date / Time: Set current date & time

## Sensor Performance Checks

A properly calibrated optical DO sensor should read between 98 and 102% saturation in the calibration sleeve. If not, recalibrate the sensor. The optical DO sensor should stabilize during calibration within 2 minutes when working properly. Make sure to thoroughly rinse and blot dry the optical DO sensor after measuring samples and before placing into the calibration sleeve (see Comments section). Refer to the optical DO sensor user manual if the sensor does not pass the performance checks.

## Sensor Rinsing, Soaking and Storage

After each sample measurement, rinse the optical DO sensor thoroughly with deionized water and blot the sensor dry with a lint-free cloth. For short term storage, overnight or between measurements, keep the optical DO sensor in the calibration sleeve or a biochemical oxygen demand (BOD) bottle with water-saturated air. For long-term storage, keep the optical DO sensor in the calibration sleeve.

## Sample Preparation and Preservation

No sample preparation required. Dissolved oxygen can be measured directly in the wine bottle.

## Calibration

If not already done, prepare the optical DO sensor according to the DO Sensor Setup procedure. Perform a water-saturated air (Air) calibration with the optical DO probe in the prepared calibration sleeve. A stable reading of 100.0 % saturation should be displayed within about two minutes.

## Analysis

Slide a ring of silicon tubing over the optical DO sensor, sliding it up the probe to just below the threads. For details see Notes below. Rinse the optical DO sensor with deionized water and blot excess rinse water off with a lint-free cloth. Place the optical DO sensor in the bottle. The silicone ring should make a seal with the bottle. Place the bottle on its side so the neck becomes flooded with wine, covering both the dissolved oxygen and temperature sensors on the optical DO probe. Initiate a reading using the Auto Read measurement mode by pressing the measure key on the meter keypad. For best results, take a second reading to ensure the dissolved oxygen measurement is fully stabilized, as it may take the optical DO sensor one to two minutes to fully stabilize in the wine sample. Use the second stable value for the oxygen content of the wine. Both readings will be saved in the meter data log.

## Comments

It is important to thoroughly clean the optical DO sensor after sample measurement. Rinse with deionized water and thoroughly blot all excess water with a lint free cloth several times before putting the sensor in the calibration sleeve. Rinsing following the completion of all sample measurements should take 5 to 10 minutes. The Orion meter data log collects up to 1000 measurement sets with time and date stamp and the non-volatile meter memory preserves data, even with loss of power. Download Orion Star Com software to facilitate the transfer of the data log from the meter to a computer at www.thermofisher.com/orionsoftware. Use the Orion Star Com software to export data to a Microsoft® Excel® spreadsheet or as a comma separated value file (.csv) or print data to a network or local printer.

### Quality Control (QC)

Recommended QC procedures may include: calibration, check of the thermistor (temperature sensor) response against a calibrated NIST-traceable thermometer, and measurement of a zero DO solution, such as 5% sodium sulfite.

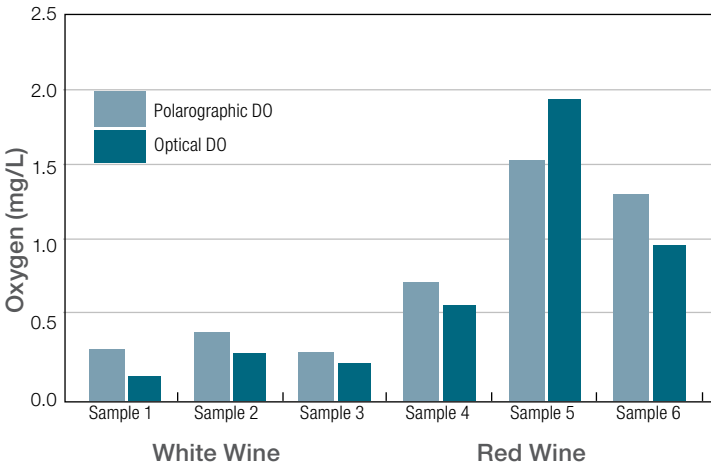
## Notes

- Silicone tubing is necessary to make a seal so the bottle can be laid on its side, immersing the probe and temperature sensor in the sample while keeping all of the wine in and ambient oxygen out of the bottle. The optical DO sensor and temperature sensor are not immersed in the sample if the bottle is sitting upright. Use a piece of soft silicone tubing with an inner diameter of 1/2 to 5/8 inch and outer diameter of 5/8 to 3/4 inch with a wall thickness of 1/8 inch. Cut a ring of tubing that is 1/4 to 1/2 inch wide. Before measuring the wine sample in the bottle, slide the ring onto the probe and push it up to just below the threads.
- Keeping the calibration sleeve clean and free from water or sample droplets is essential to getting good calibration and read back values in water-saturated air. Rinse the optical DO sensor thoroughly with deionized water and wipe excess water with a lint-free cloth prior to putting the sensor in the calibration sleeve.
- Optical DO sensors do not require stirring or a sample stream for accurate measurements. The speed, accuracy, and precision of the optical DO sensor are equivalent or superior to the traditional polarographic sensor measurement.
- If readings are slow or inconsistent, ensure the temperature sensor is completely submerged in the sample.
- If the temperature sensor is not in the sample, the DO readings will be incorrect.
- The optical DO cap must be replaced every 365 days. The remaining optical DO cap life can be viewed in the channel setup menu. The meter will display an error message when the optical DO cap needs to be replaced.

## Results

**Dissolved Oxygen Readings in Wine Bottles**

| Oxygen (mg/L) | | | | |
|---|---|---|---|---|
| | White Wine | | Red Wine | |
| | Optical | Polarographic | Optical | Polarographic |
| Sample 1 | 0.35 | 0.17 | 0.80 | 0.64 |
| Sample 2 | 0.46 | 0.32 | 1.61 | 2.01 |
| Sample 3 | 0.34 | 0.26 | 1.39 | 1.04 |
| Temperature (°C) | 21.5 | 21 | 21.3 | 21.1 |




Thermo Scientific™ Orion™ Optical DO Sensor

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## Summary

Using an Orion Star A223 Dissolved Oxygen (DO) Portable Meter with an optical DO sensor enables wineries to continually produce high quality wines. Since the optical DO sensor allows the wine to be measured directly in the bottle, dissolved oxygen measurements can be made with speed and accuracy. The speed, accuracy and precision of the optical DO sensor is equivalent or superior to current DO measurement techniques.

To purchase an Orion Star A223 DO Portable Meter, Orion Optical DO Sensor and other related products, please contact your local equipment distributor and reference the part numbers listed below.


**Thermo Scientific™ Orion Star™ A223 Dissolved Oxygen Portable Meter Kit**

### Ordering Information

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|---|---|
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| RS232 Computer Cable | 1010053 |

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