# Free $SO_2$ Determination in Home Winemaking

By David J. Elliott
The Vineyard@Skydances Ranch

# Comparative $SO_2$ Tests

Test 2 Wines (Red & White) for free $SO_2$ Levels

Analyze Free $SO_2$ Using Home Kits and Laboratory Methods

Compare to Commercial Laboratory Results

# Wines for $SO_2$ Tests

# Free $SO_2$ Test Methods

## Home Kit Methods

- Titrets®
- Accuvin®

## Laboratory Methods

- “Ripper” Method
- “Modified Ripper” Method
  (Modified using $IO_3^{-}$)
- Aeration/Oxidation

# “Titrets®”


70470/70484
Inspected By: 1
CHEMetrics
Titrets®
for the determination of Sulfite in Wine
• Range 10-100 mg/L
• 10 Tests
• Catalog Number K-9610W
W510

# “Accuvin®”


www.ACCUVIN.com
A FRIEND of the ENVIRONMENT
Point of Use Tests
COMPLETE Instructions Inside
ACCUVIN, LLC
Expiration:
12/07
G19H
QUICK TESTS™
FREE SO2
for WINE, MUST, and JUICE
Monitor to
Inhibit Native Bacteria at crush
Inhibit Browning at crush
Inhibit Lactic Acid Bacteria and Malolactic Organisms after alcholic fermentation
Reduce enzymatic and chemical oxidation and control bacteria during aging and just prior to bottling
HIGH RANGE SUSPECTED?
Use Red Cap
0 20 40 50 60 75 90 110 130 ppm
If strip color is between 2 chart colors, read second row.
Lower Range? Use Green Cap
0 4 8 12 16 22 28 34 40 ppm
If strip color is between 2 chart colors, read second row.
Keep out of direct sunlight.
$21

# Laboratory Methods - “Chemistry”

Chemistry - Ripper Method

$$SO_2 + I_2 + 2H_2O \rightarrow SO4^{=} + 2I^{-} + 4H^{+}$$

# Laboratory Methods - “Ripper” Method (1)

**Procedure**:

Pipet 50 mL of the wine sample into a 250 mL erlenmeyer flask. Add 5 mL of a starch indicator Solution, 5 mL of 1:3 sulfuric acid. Rapidly titrate the free suflurous acid using 0.02 N iodine solution. The endpoint is the first darkening of the solution to a bluish color, which persists for 1-2 min. The Temperature should not exceed 20˚ C. For red wines place a strong source of yellow light so that the light Transmitted through the solution from the side, to make the end point more distinguishable.

Free $SO_2$ (mg/L) = (V) (N) (32) (1000) ÷v

Where V = Volume of Iodine Solution used in titration (mL)
N = normality of iodine solution
v = volume of wine sample (mL)

(1) M. A. Amerine and C. S.Ough, “Methods for Analysis of Musts and Wines”Wiley, 1980.,

# Laboratory Methods - “Modified Ripper” Method

**Procedure**:

Prepare a potassium iodide-starch solution by dissolving 10 g KI and 2.5 g starch with distilled water in A liter flask and bring to volume. Dissolve 0.11135 g of potassium iodate with 200 mL of 2 N sulfuric Acid and bring to volume in a 1-Liter flask with distilled water.

Add 10 mL of wine and 2.0 mL of iodide-starch solution to a 250 mL flask and titrate with the 0.0005203 M potassium iodate solution.

Free SO2 (mg/L) = 10 x V

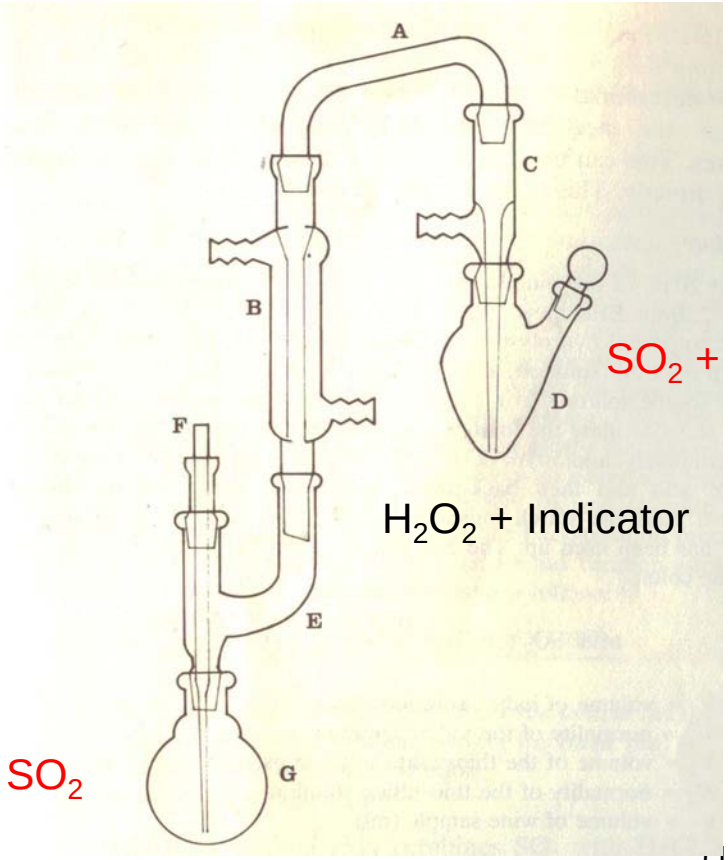
Where V = volume of wine used

If any other molarity or volumes are used, then

Free $SO_2$ (mg/L) = (V) (N) (32) (1000) ÷
Where V = Volume of KIO3 solution (mL)
N = normality KIO3 solution (1 M = 6 N)
v = volume of wine sample (mL)

(1) M. A. Amerine and C. S.Ough, “Methods for Analysis of Musts and Wines”Wiley, 1980.,

# Laboratory Methods - “Aeration / Oxidation” Method




$SO_2 + H_2O_2 \longrightarrow SO_4^{=} + 2H^+$

$H_2O_2$ + Indicator

$H_2SO_3 \longrightarrow H_2O + SO_2$

Wine + $H_3PO_4$

(1) M. A. Amerine and C. S.Ough, “Methods for Analysis of Musts and Wines”Wiley, 1980.,

# Laboratory Methods - “Aeration / Oxidation” Method

**Procedure**:

Prepare standardized 0.01 N sodium hydroxide. Re-standardize weekly. Keep protected with a soda lime tube. Dilute 1 mL of 30% hydrogen peroxide solution to 100 mL with water. Make up daily and store in refrigerator. Add 0.100 g methyl red and 0.050 methyl blue to a volumetric flask and make to 100 mL with 50 vol% ethanol. Dissolve 280 mL of phosphoric acid (90%) in a 1 Liter flask and bring to volume.

Pipette 10 mL of 0.3% hydrogen peroxide into the 50 mL pear shaped flask (D). Add 3 drops of indicator to turn The solution purple. Adjust the color to olive green with the addition of a few drops of 0.01 N sodium hydroxide. Connect vacuum line to adaptor C. Pipet 20 mL of wine and 10 mL of 25 vol% phosphoric acid into the round-Bottom flask G. The flask should be submerged into an ice bath to prevent dissociation of of bound SO2. Start Water in condenser B; this restrains volatile organic acids from distilling over. Turn on the vacuum and draw air Through the system at 1000 tp 1500 mL/min for 10 min. Remove the flask (D) and titrate the acid formed with the 0.01 N sodium hydroxide back to the olive green end point. It is advisable to run a blank, and if necessary make This correction by subtracting mL of sodium hydroxide required for the blank from the sample.

Free $SO_2$ (mg/L) = (V) (N) (32) (1000) ÷v

Where V = Volume of NaOH solution (mL)
N = normality NaOH solution
v = volume of wine sample (mL)

(1) M. A. Amerine and C. S.Ough, “Methods for Analysis of Musts and Wines”Wiley, 1980.,

If volumes and concentrations above are used, Free SO2 (mg/L) = 16 * V in mL

# “Home Winemaker” Kits

Costs:

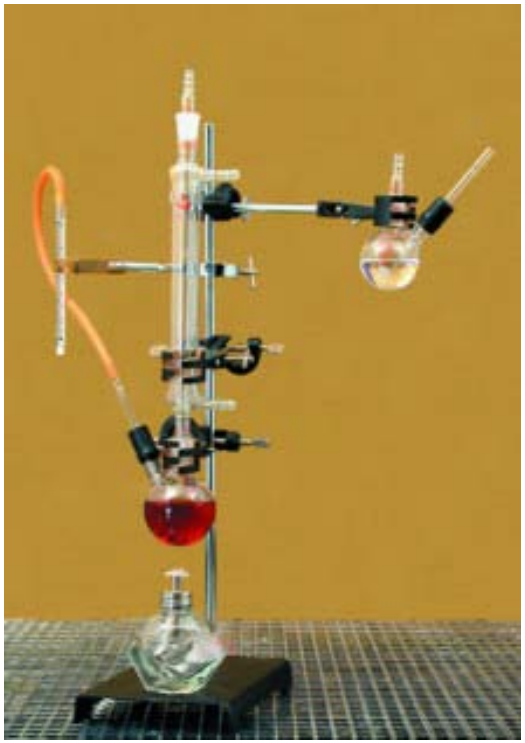
- Titrets® (~$1.25 - $2.00/Test)
- Accuvin® (~$2.00/Test)

# “Aeration / Oxidation System”
## (More Wine!)

Cost: $425/295

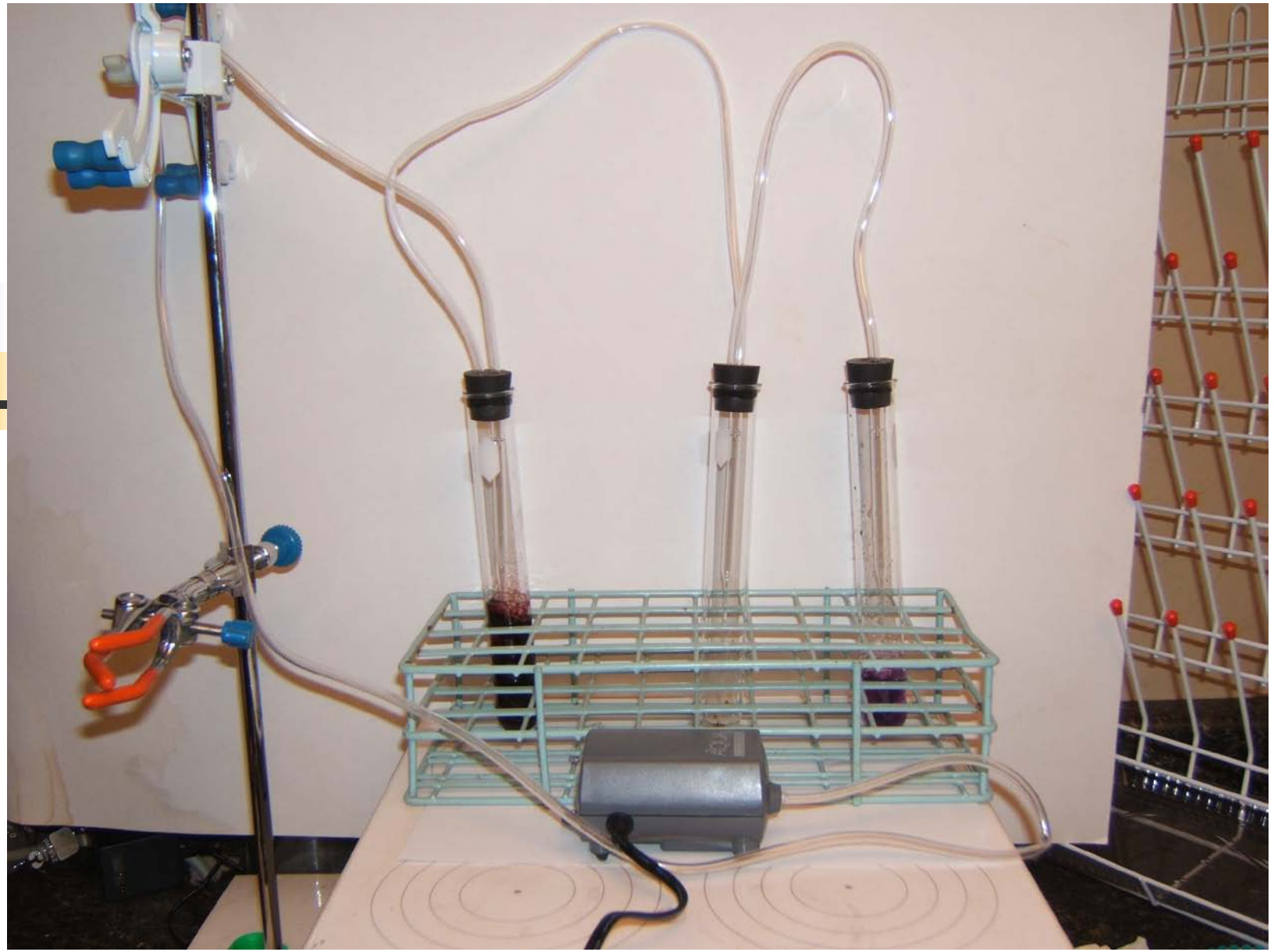
# “Aeration / Oxidation System” (Cynmar)

Cost: $495

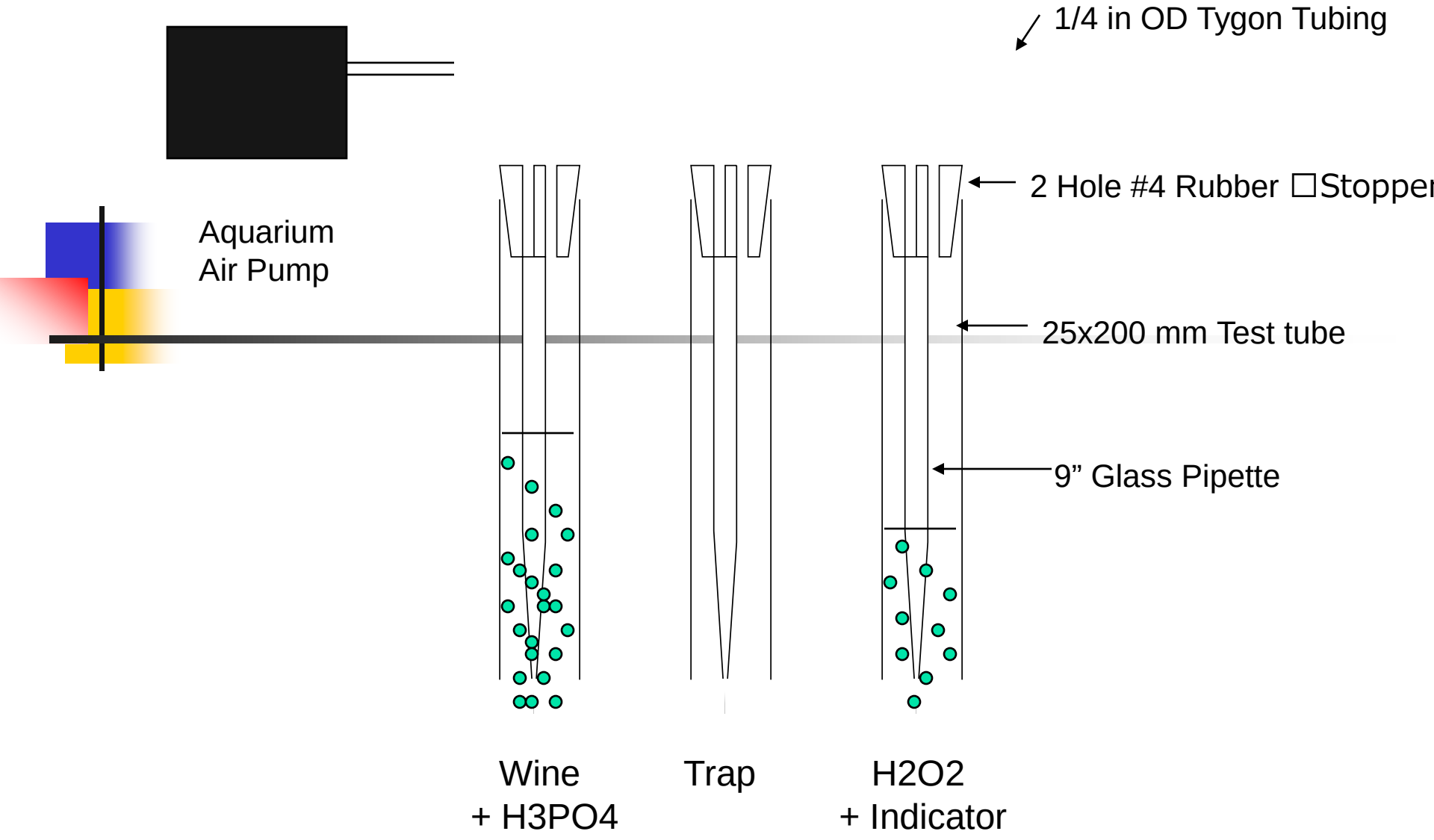


Cost: $215

# “Elliott” Aeration/Oxidation Apparatus



# “Elliott” Aeration/Oxidation Apparatus



# Costs for “Elliott” Aeration / Oxidation Apparatus

| Equipment | | Source | Cost |
|---|---|---|---|
| Aquarium Pump (1200 cc/min) | | Walmart | $7.32 |
| Test Tubes (24-25x200 mm) | (115-33521) | Cynmar | $28.00 |
| Rubber Stoppers (2-Hole #4, 1lb ~30) | (124-33666) | Cynmar | $5.25 |
| Disposable Pipettes (9” 250/box) | (132-24553) | Cynmar | $9.85 |
| Test Tube Rack (21 Tube, 30mm) | (150-25995) | Cynmar | $3.95 |
| | | Total | $54.37 |

| Reagents: | Source | Cost | Cost/Test |
|---|---|---|---|
| Phosphoric Acid | Cynmar/Wine! | $16.40 / 500 mL | $0.245 |
| Acid Indicator ($SO_2$) | Wine! | $4.50/ 1 oz | $0.03 |
| Hydrogen Peroxide (3%) | Walmart | $0.87 / 16 oz | $0.002 |
| NaOH | Cynmar | $10.15 / 1L | ~$0.01 |

# Sources for “Elliott” Aeration / Oxidation Apparatus & Supplies

Cynmar = www.cynmar.com

Wine! = www.morewinemaking.com

# Results of Comparative $SO_2$ Tests

**Chardonnay**

| | Home Tests | | | | | Lab Analysis * | | |
|---|---|---|---|---|---|---|---|---|
| **Method** | **Accuvin** | **Titrets** | **Ripper** | **Mod Ripper** | **Aer. / Ox.** | **Ripper** | **Aer. / Ox.** | **Flow Inj.** |
| **Trial 1** | 22 | 32 | 32 | 26 | 24 | | | |
| **Trial 2** | 28 | 34 | 30.7 | 25 | 16 | | | |
| **Trial 3** | 22 | 35 | 33.3 | 28 | 33.6 | | | |
| **Average** | **24.0** | **33.7** | **32.0** | **26.3** | **24.5** | **26.0** | **25.0** | **24.0** |

**Cabernet Sauvignon**

| | Home Tests | | | | | Lab Analysis * | | |
|---|---|---|---|---|---|---|---|---|
| **Method** | **Accuvin** | **Titrets** | **Ripper** | **Mod Ripper** | **Aer. / Ox.** | **Ripper** | **Aer. / Ox.** | **Flow Inj.** |
| **Trial 1** | 50 | 38 | 40.3 | 46 | 25.6 | | | |
| **Trial 2** | 60 | 51 | 41 | 48 | 27.2 | | | |
| **Trial 3** | 34 | 58 | 38.4 | 51 | 27.2 | | | |
| **Average** | **48.0** | **49.0** | **39.9** | **48.3** | **26.7** | **26.0** | **25.0** | **23.0** |

*** Performed by ETS Laboratories, 899 Adams Street, Suite A, St. Helena, CA 94574 www.etslabs.com**

# Performance of Comparative $SO_2$ Tests

| | Method | | | | |
|---|---|---|---|---|---|
| | Accuvin | Titrets | Ripper | Mod Ripper | Aer. /Ox. |
| Readibility* | 6 | 2.5 | 0.1 | 0.1 | 0.1 |
| Resolution * | 24% | 10% | 4% | 4% | 6% |
| Precision (Std. Dev White) | 3.5 | 1.5 | 1.3 | 1.5 | 8.8 |
| Precision (Std. Dev Red) | 13.1 | 10.1 | 1.3 | 2.5 | 0.9 |
| Bias (White) | 0% | 40% | 33% | 10% | 2% |
| Bias (Red) | 109% | 113% | 70% | 109% | 16% |

* at 25 ppm SO2

# Advantages/Disadvantages of Home $SO_2$ Tests

| Test | Advantages | Disadvantages |
|---|---|---|
| Accuvin® | Quick & Easy<br>No Equipment Required | Imprecise Endpoint<br>Low Precision |
| Titrets® | Quick & Easy<br>No Equipment Required<br>Good Precision & Accuracy for Whites | Easy to Overshoot Endpoint<br>High Bias for Red Wines |
| “Ripper” | Good Precision & Accuracy for Whites | Storage Stability of Reagents<br>High Bias for Red Wines<br>Requires “Exotic” Reagents |
| “Modified Ripper” | Good Precision & Accuracy for Whites<br>Storage Stability of Reagents | Requires “Exotic” Reagents<br>High Bias for Red Wines |
| Aeration / Oxidation | Low Bias for Red & White Wines<br>Good Precision & Accuracy for Red Wines | Need to Purchase Equipment<br>Need to Purchase Acids |