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Diagnostic/Screening Test for Volatile Sulfur Compounds in Wine

Bench trial and Remediation Techniques

Hydrogen sulfide (H_2S), mercaptans and disulfides are all forms of volatile sulfur containing compounds (VSC). Table 1. lists their aroma and threshold found in wine. Before proceeding treatment, it is important to recognize and identify which VSC are present in order to proceed with the best remediation technique. Different VSC have varying aroma and varying winemaking treatments to remediate/remove them. A simple sensory screening trial outlined in Table 2. will help determine the most efficient treatment strategy. Unlike H_2S and mercaptans, disulfides cannot react with copper sulfate ($CuSO_4$), and require ascorbic acid to return them into their treatable mercaptan form. For more information refer to [Newsletter Reduction: How to Prevent and Treat It](#).

Table 1. Main Sulfur Off-Aroma Compounds in Wine

Sulfur compound		Aroma	Threshold
Hydrogen sulfide (H_2S)		Rotten eggs, sewer gas	0.5 ppb
Mercaptans	Methyl mercaptan	Rotten cabbage	1 ppb
	Ethyl mercaptan	Burnt match, earthy	0.02 – 2 ppb
Disulfides	Dimethyl disulfide (DMDS)	Onions, cooked cabbage	15 – 30 ppb
	Diethyl Disulfide (DEDS)	Burnt rubber, garlic	4 ppb

Table 2. Recognizing and Identifying Sulfur Off-Aromas

Control	**Copper sulfate** (2 g/hL of copper)	**EnartisTan ELEVAGE** 2 g/hL	**Ascorbic acid** (5 g/hL) + **EnartisTan ELEVAGE** (2 g/hL)	Interpretation
Sulfur off-aroma	**Off-odor disappears**	Off-odor is still there	Off-odor is still there	**H_2S**
	Off-odor disappears	**Off-odor disappears**	Off-odor is still there	**Mercaptans**
	Off-odor is still there	Off-odor is still there	**Off-odor disappears**	**Disulfides**

Enartis Tannins for VSC Remediation

The above is achieved to the best of our knowledge and experience.
The industrial application of the advice provided does not imply any responsibility on the part of our company.

Revision: January 2021

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Table 3. VSC Treatment Options and Associated Risks

Identified Sulfur Compound	Remediation Options	Associated Risk
Hydrogen Sulfide (H_2S)	Aeration/ Volatilization (Oxygen, Nitrogen)	✓ Detrimental oxidation ✓ Loss of desirable wine aroma
	Copper Sulfate ($CuSO_4$)	✓ Cupric haze ✓ Catalytic oxidation ✓ Removal of desirable aroma (thiols)
Mercaptans	Ellagic/Condensed Tannins: EnartisTan Elevage, EnartisTan Max Nature, EnartisTan SLI, EnartisTan Cœur de Chêne	✓ Subtle sensory modification
	Copper Sulfate ($CuSO_4$)	✓ Cupric haze ✓ Catalytic oxidation ✓ Removal of desirable aroma (thiols)
Disulfides	Reductive conditions (ascorbic acid and SO_2) with EnartisTan Elevage	✓ Oxidation due to ascorbic acid addition and issuficent SO_2 levels ✓ Subtle sensory modification
	Reductive conditions (ascorbic acid and SO_2) with $CuSO_4$.	✓ Cupric haze ✓ Catalytic oxidation ✓ Removal of desirable aroma (thiols)

VSC Remediation: Tannin and Copper Addition Trial

***IMPORTANT NOTE**: Before running copper addition trials it is recommended to proceed with the diagnostic test proposed in Table 2 to recognize and identify the VSC responsible for the reduction in wine. Copper treatment is only effective with H_2S and some mercaptans.*

Copper (Cu) is commonly used in the treatment of reductive characters. It reacts with H_2S and certain mercaptans but does not react with disulfides. These reactions may require the addition of copper in excess, which can detrimentally affect fruity volatile thiols, decreasing wine quality and complexity. Cu has the ability to catalyze oxidation reactions, leading to the formation of disulfides, premature ageing, and the potential for cupric haze with residual Cu levels higher than 0.5 mg/L. Recent studies have shown that, copper-sulfide complexes are not readily removed by racking and can even pass through some types of filtration. Moreover, these complexes can recycle bound sulfur compounds over time, revealing sulfur off-aromas post bottling. Copper is a heavy metal and levels in wine are regulated due to human health concerns. For these reasons, Enartis recommends the use of the listed enological tannins as a safe, low risk alternative to $CuSO_4$ treatment. If $CuSO_4$ treatment is determined to be the best course of action, determine the lowest dosage possible and apply metal removing fining agents such as with Claril HM or Stabyl Met to remove residual copper-sulfide complexes to reduce the associated risks with Cu treatment.

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Equipment

- 100 mL or 120 mL bottles with caps
- 1 mL serological pipet
- Wine glasses
- Watch glasses
- Eppendorf pipettor (optional) or 1 mL pipette
- Pipet safety bulb

Copper Reagent Forms and Conversions

Cupric Sulfate Forms	Commerical Dosage	Copper mg/L (PPM)
Cupric Sulfate 5 Hydride crystal $CuSO_4 \cdot 5\ H_2O$ MW = 249.686 Cu = 25.47%	1.0 g/L	254.7
	1.0 g/gal	67.29
	1#/1000 gal	30.55
1% $CuSO_4$ Solution	1 mL/L	2.57
	1 mL/gal	0.67
	0.15 mL/gal	0.1
10% $CuSO_4$ Solution	15 mL/1000 gal	0.1

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Bench Trial Solutions and Procedure

Cupric Sulfate % Solution	Bench Trial Dosage	Copper mg/L (PPM)
0.5% $CuSO_4$	0.1 mL/120 mL	1.05
0.1% $CuSO_4$	0.1 mL/120 mL	0.20
0.05% $CuSO_4$	0.1 mL/120 mL	0.10
0.0025% $CuSO_4$	0.1 mL/120 mL	0.05

For more information about bench trial solution preparation see the ENARTIS COPPER SOLUTION CALCULATOR

Procedure

1. Decant wine sample into desired number of treatments with at least one control (no treatment). We recommend using 120mL bottles for the trial (bench trial dosage is listed with 120 mL sample volume).

2. Label each bottle with increments of copper dosage (ppm) as well as other enological tannin treatments.
   - Example: +0.1ppm, +0.2ppm +0.3ppm +0.4ppm +0.5ppm for $CuSO_4$, 0.5 – 5.0 g/hL dosage range for tannins.

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- Enartis tannins for mercaptan scavenging: EnartisTan SLI, EnartisTan Élevage, EnartisTan Max Nature, EnartisTan Cœur de Chêne .

3. Homogenize each bottle, top off sample volume headspace, and tightly close. Let stand for 24 -48 hours.

4. Carefully decant the sample off the $CuSO_4$ into wine glasses, immediately cover with watch glasses. Code the samples and evaluate for improvement in VSC off odors (Ideally blind, with multiple tasters).
- Reference SULFIDE SENSORY THRESHOLDS AND AROMA DESCRIPTIONS and rate each treatment to determine the optimum treatment.
- If VSC off-aroma persists and disulfides are likely present, consider adding addition of ascorbic acid at 30-50 ppm to help break the disulfide bonds, at least 30 mintues prior to the addition of copper and tannin. Make sure the wine is protected with at least 0.5 ppm of molecular SO_2.

5. After determining the addition of $CuSO_4$ and treating the wine, follow up with Claril HM or Stabyl Met to remove residual copper-sulfide complexes.
- Copper-sulfide complexes are not readily removed by racking and can pass through some types of filtration media. These compounds recycle bound sulfur compounds over time, revealing sulfur off-aromas as well as potential cupric haze post bottling. Removing these complexes and excess copper is crucial in mitigating their reappearance as well as for human health and regualtory purposes.
- See TIPS AND BEST PRACTICES FOR CONDUCTING CLARIL HM AND STABYL MET BENCH TRIALS

6. If there is still VSC off-aromas after performing the trial
- If there is no improvment with the applied dosage range of $CuSO_4$, additions with ascorbic acid and higher dosage treatments (within the legal addition rate) should be evaluated.
- Some VSC, like disulfides, will not react with the copper, the addition of ascorbic acid prior to treatment with copper can help break some of these bonds, but caution should be taken as ascorbic acid can further oxidize these compounds if molecular SO_2 is lower than 0.5 ppm.
- Some C6, methoxypyrazines and other off-aromas can be confused with VSC aroma.

7. Learn more about managing and treating reduction with our online free educational content below
- Enartis Newsletter Reduction: How to Prevent and Treat It
- Winemaker Trial: Use of PVI/PVP to Remove Copper and Aluminum to Avoid Sulfide Formation
- Webinar: How to Avoid and Treat Reductive Wines

Copper Legal Limit

TTB 27 CFR § 24.246 and 27 CFR § 24.250 Wine Treating Material**:**
- The quantity of copper sulfate added (calculated as copper) must not exceed 6 parts copper per million parts of wine (6.0 mg/L). The residual level of copper in the finished wine must not exceed 1 part per million (1 mg/L). 21 CFR 184.1261 (GRAS).

For more information, call our Technical Winemaking Specialist at (707) 838-6312

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