

Fining and Stabilisation

Richard Gibson



Author

Richard Gibson is the principal of Scorpex Wine Services, specialising in independent oenological consultancy. Prior to establishing Scorpex, Richard held senior roles with Southcorp Wines and gained wide experience in all facets of wine production, stabilisation and packaging.



Contents

Overview	STB-1
1 Materials added to wine – additives and process aids	STB-1
1.1 Definition	STB-1
1.2 Legal requirements	STB-1
1.3 Freedom from contamination	STB-2
1.4 Other issues	STB-2
2 Product losses, quality loss and waste	STB-3
3 Targeting fining and stabilisation operations	STB-3
4 Preparation and mixing	STB-3
5 Fining	STB-3
5.1 Bentonite	STB-3
5.2 Protein fining agents – gelatine, isinglass, casein, milk, egg white albumen, plant proteins	STB-4
5.3 Co-finings – tannin, silicon dioxide	STB-6
5.4 Chelating agents and protective colloids – phytates, citric acid, and gum arabic	STB-7
6 Stabilisation	STB-7
6.1 Potassium and calcium salts of tartaric acid	STB-8
6.2 Reaction and precipitation of reactants – refrigeration with or without crystal seeding	STB-8
6.3 Reduction of reactants by other means – ion exchange, electrodialysis	STB-9
6.4 Addition of crystallisation inhibitors	STB-10
6.5 Testing for calcium tartrate and potassium hydrogen tartrate stability	STB-10
6.6 Protein	STB-10
6.7 Testing for protein stability	STB-11
6.8 Microbial growth	STB-11
6.9 Alteration of the wine environment	STB-16
6.10 Oxidative degradation	STB-17

This work may be cited as:

R. Gibson, 2013, 'Fining and Stabilisation', *Australian Winemaking*, eds N. Bulleid and V. Jiranek,
online XXXXwww.trivinumpress.com.au/XXX



Copyright © Trivinum Press, 2013. All rights reserved.

Trivinum Press Pty Ltd, PO Box 7, Tanunda SA 5352

e: info@trivinumpress.com.au w: www.trivinumpress.com.au

Disclaimer

This work provides general advice, etc. For a full statement of copyright and disclaimer information visit www.trivinumpress.com.au/copyright etc **TO BE FINALISED**

Fining and Stabilisation

Richard Gibson

Overview

Newly made wine contains materials that may detract from wine appearance, aroma and flavour. These materials may be immediately apparent, such as haze or palate bitterness. The materials may also be present in precursor forms that only create a negative impact when wine is exposed to certain conditions. For example, residual grape protein molecules are not immediately obvious in newly made white wine. However, exposure of this wine to elevated temperatures, for even a short period, can cause the protein to denature and coagulate, and form a precipitate.

The processes of fining and stabilisation can enhance wine quality by removing, breaking down or binding these materials to ensure that wine is presented to the consumer with an appearance, aroma and flavour profile that matches target style and quality criteria. These processes may be applied to juice, fermenting wine or finished wine.

Fining and stabilisation often involve the addition of materials to juice or wine. The added materials bind with target compounds and precipitate. Clear product is then separated from the solid residue. Some stabilisation processes, such as chilling to remove excess potassium hydrogen tartrate, use physical conditions to induce a precipitate. The addition of materials to wine, the requirement to separate out a solids fraction and the targeted nature of fining and stabilisation operations introduce some important generic principles that should be observed whenever fining and stabilising are carried out.

1 Materials added to wine – additives and process aids

1.1 Definition

It is important to understand the difference between an additive and a process aid.

Additive – an additive is officially defined as a substance that is added to a food or beverage product, such as wine, that continues to carry out a technological function in the product when it is released to the market. For example, sulfur dioxide is added to wine during the production

process. After the wine is released to the market, the sulfur dioxide in the wine prevents oxidative degradation of compounds in the wine and inhibits yeast and mould growth. Therefore, sulfur dioxide is an additive to wine.

Process aid – a process aid is added to a food or beverage during production, but is then removed during the production process, prior to release to the market. For example, bentonite is added to white wine to remove protein. The bentonite is removed from wine prior to release to the market. Therefore, bentonite is a process aid. A process aid may also be added during manufacture and remain in the product after release to the market, without carrying out a technological function. Copper sulfate is an example of this type of process aid. When added to wine, the copper component reacts with hydrogen sulfide to form an insoluble precipitate, which can then be removed. Unreacted copper and all of the added sulfate may remain in solution when the wine is presented for sale. This material performs no technological function in the finished wine.

1.2 Legal requirements

The use of materials in wine that do not originate in grapes is closely regulated around the world. Care must be taken to ensure that only approved materials are used when fining and stabilisation processes are carried out.

The Australian and New Zealand Food Standards Code contains a list of additives and process aids allowed for the production of wine in Australia. The Food Standards Code reference is a positive list. This means that only those materials listed can be used during the production of wine in Australia. If the material is not on the list, it cannot be added to Australian wine.

Requirements for Australian wine sold in other countries vary depending on legislative arrangements negotiated with other countries. There are four basic philosophies in place.

Mutual acceptance

Under the mutual acceptance philosophy, countries agree to accept imported wine that is made according

to the domestic requirements of the exporting country. Through the World Wine Trade Group, Australia has a Mutual Acceptance Agreement on Oenological Practices in place with other signatory countries such as the USA, New Zealand, Chile, Canada and Argentina. Under this arrangement, Australian wine exported to the USA must meet Australian winemaking law. It does not have to meet the winemaking laws of the USA. The use of additives and process aids for fining and stabilisation are included in the definition of oenological practices.

Compliance with importing country regulations

Under these conditions, Australian wine must meet the domestic requirements of the importing country. For example, Australian wine sold in Japan must meet Japanese Food Law requirements for additives and process aids. Certain materials used in winemaking in Australia are not permitted by Japanese Food Law and must not be used in wines destined for the Japanese market.

Compromise between these philosophies

Annex 1 to Australia's agreement with the European Union on wine contains a positive list of practices and processes allowed for wines originating in Australia that are to be sold in EU markets. This list is a compromise between Australian regulations and the EU's domestic requirements. There are several requirements in this list that differ markedly from Australian requirements:

1. Maximum limits are stipulated for the amount of many of the materials that can be added to wine, or the maximum level that can be present in finished product. This philosophy is different from that found in Australian regulation. Australian requirements contain maximum limits for some additives and process aids that can present a health risk or quality issue when present in excess (such as sulfur dioxide and sorbic acid). In most cases, however, the levels of addition and final concentration of winemaking materials in wine in Australia are left up to the producer, who is expected to follow good manufacturing practice. The EU philosophy is quite different, and this philosophy is reflected in the requirements of Annex 1 to the agreement.
2. Manufacturing processes are regulated. For example, the list refers to processes such as ion exchange and filtering that are important in fining and stabilisation operations. Only those processes listed in the annex may be used for wines destined for the EU market. Australian legislation does not regulate processes.

One set of global rules

The Office International de la Vigne et du Vin (OIV) is the inter-governmental scientific and technical body that oversees global wine and vine matters. One area in which OIV exerts considerable influence is the review of the suitability

of additives, process aids and processes for winemaking. New materials and processes are considered in great detail. When a material or process passes final approval, relevant information is placed in the International Code of Oenological Practices, The International Oenological Codex and the compendium of International Methods of Analysis of Wine and Musts. These documents detail the purity standards and conditions of use for materials and parameters and restrictions that must be observed for processes. Analytical procedures for the detection and control of approved materials are also detailed.

Member countries are not bound to adopt OIV resolutions into domestic law. However, some countries and trading blocs (notably the EU) choose to do so.

1.3 Freedom from contamination

The purity of all materials added to wine is an area in which much care must be exercised. All additives and process aids listed in the Australian and New Zealand Food Standards Code must conform to the purity requirements listed in reference documents such as the Compendium of Food Additive Specifications, the Food Chemicals Codex and the International Pharmacopoeia. It is important that all materials used in wine conform to relevant requirements. This should be a basic component of any winery quality plan.

The issue of additive and process aid contamination by taint is another important area of focus. Fining and stabilisation materials such as bentonite can adsorb odours when stored before use. Organic materials such as gelatine and casein can undergo microbial degradation. Adding contaminated materials to wine can lead to loss of quality, with a concomitant downgrading of value and a requirement for further remedial processing, all of which are undesirable outcomes. It is good practice to ensure that a sensory check is carried out on each batch of all additives and process aids used in the winery. This can be done by adding an appropriate amount of material to a neutral white wine and performing a sensory evaluation in comparison with a control wine.

To minimise any risk of contamination and spoilage, fining and stabilisation materials should be kept in dry, ventilated locations and stored in sealed packages. It is good practice not to store fining and stabilisation materials on wooden pallets, which can harbour compounds that can easily be transferred to wine, such as chloroanisoles.

1.4 Other issues

Genetically Modified Organisms – many fining and stabilising materials are made from plant and animal sources (e.g. gelatine, isinglass) or through the conversion of plant or animal derived raw materials by organisms (e.g. citric acid, pectinolytic enzymes). At the present time, many major wine industry customers will not accept the use of materials in wine made from genetically modified plants or animal raw materials, or materials made through conversion

by genetically modified organisms. Gaining assurances from suppliers concerning the GM status of organisms used in the production of fining and stabilising materials is currently a key part of ensuring customer acceptance of Australian wine.

Allergen labelling – legislation recently introduced in Australia requires mandatory label declaration of the presence of certain material in foods when released to the market. These materials are potential allergens for sensitive members of the population. The list includes sulfur dioxide (when present at greater than 10 mg/L), as well as fish, egg and dairy products. Several materials used for the fining and stabilisation of wine are fish, egg or dairy products, and wineries must consider label declaration when these are used during the winemaking process.

2 Product losses, quality loss and waste

Many fining and stabilising processes involve the addition or creation of solid materials that must be separated from the treated liquid to complete the process. This process creates the potential for product losses, quality reduction through adsorption of aroma and flavour compounds, and environmental issues. In general, the following principles should be followed to minimise these impacts.

- Minimise the amount of fining material added.
- Use only enough material to achieve the desired effect.
- Ensure that maximum lees compaction occurs to optimise the quantity of clear product that can be obtained. When appropriate, fining and stabilisation operations should be carried out at the same time to reduce losses and the number of operations required.
- Ensure that any liquid or solid waste streams are processed to recover organic materials, such as alcohol and tartrate, prior to disposal to the environment.

3 Targeting fining and stabilisation operations

All fining and stabilisation operations should be carried out with a specific target outcome in mind. Each fining and stabilising step requires cellar labour and involves volume and, potentially, quality losses. Trial fining is extremely valuable, ensuring that an added compound will have the desired effect in wine. Where possible, pre-analysis of wine is helpful to confirm that fining or stabilising is actually necessary. Verification of fining action by post-treatment analysis and tasting is also valuable.

4 Preparation and mixing

Many fining and stabilisation processes require correct material preparation and product mixing processes. If these processes are ignored, materials are wasted, wine must be re-handled and loss of quality may result.

5 Fining

The process of fining involves the addition of an adsorptive

or reactive material that binds with or breaks down target solid, colloidal or dissolved components in juice or wine.

When binding takes place, the materials aggregate into solid particles and precipitate. Treated product can then be separated from the solids, using processes such as racking, centrifugation and filtration.

When breakdown occurs, the target component changes. Molecular size is reduced, and the component and its derivatives usually become more soluble and have less impact on wine or juice composition.

Some fining processes are carried out specifically to stabilise wine against undesirable change at some future stage. Other fining processes are carried out to effect a change in juice or wine composition that is immediately apparent.

The fining agents that will be considered in this section are:

- Bentonite
- Protein fining agents – gelatine, isinglass, casein, milk, egg white albumen, plant proteins
- Synthetic polymer fining agents – polyvinylpyrrolidone
- Polysaccharides – agar and alginates
- Carbon
- Co-finings – tannin, silicon dioxide
- Potassium ferrocyanide
- Copper
- Chelating agents and protective colloids – phytates, citric acid, gum arabic
- Enzymes – pectic enzyme preparations, urease, glucanase

5.1 Bentonite

Bentonite is a clay material of volcanic origin found in numerous source deposits around the world. It is commercially available in either calcium or sodium form. Some bentonites are activated by treatment with compounds to change the metallic cation species in the bentonite platelets. Bentonite particles consist of stacked layers or platelets of aluminium silicate. When bentonite swells in water, the platelets separate and a large number of negatively charged sites are exposed. When swollen bentonite is added to wine, these negatively charged sites react with positively charged molecules through electrostatic bonding. This reaction takes place within minutes when bentonite and wine or juice are well mixed.

The major function of bentonite in winemaking is the removal of heat-unstable protein from white wine. If this grape-derived protein is not removed it can precipitate from untreated wines under certain conditions, such as periods of elevated temperature, creating an undesirable haze or sediment. These unstable proteins are not usually found in red wines as they react with grape-derived phenolics and co-precipitate during the winemaking process.

varies, depending on the materials used in construction. Foil lined bags give high oxygen barrier performance, but the oxygen exclusion performance of the foil layer can be compromised by flex cracking during the transport of filled packs. Bags incorporating high barrier polymers are now available that give better transport performance. The shelf life of all wine in bag-in-box is limited by the gradual entry of oxygen through the pack materials. Smaller packs are more vulnerable to oxidation, owing to the higher surface area to volume ratio as the volume of the bag is reduced. To ensure that product quality is maintained during the distribution process, bag-in-box wines should be packaged on demand with sufficient SO₂ to provide a buffer against oxidation. They should be turned over rapidly in warehouses

Glass bottles allow no oxygen entry through the bottle walls. However, oxygen can enter wine in the bottle through and past bottle seals. Good corks provide an almost complete barrier to oxygen entry, especially when bottles are laid down or inverted. However, poor corks can allow rapid oxygen entry. (Reference) Unfortunately, it not possible to predict cork performance before use, and corks in the same batch can give inconsistent oxygen barrier performance, leading to variation in the oxidation status of a batch of bottled wine over time. The synthetic closures available to date allow a small but finite amount of oxygen entry into the bottle over time. This limits the shelf life of wine sealed with these closures to around two years. Screwcaps with a tin-faced wad will give very low oxygen entry into the bottle, but other wad types can allow rapid oxygen entry.

The treatment of the bottle headspace and the application of closures both have an impact on the oxidative stability of bottled wine.

When using inserted closures, dosing with inert gas and/or the application of vacuum prior to insertion of the closure can reduce the headspace oxygen content. Dosing with nitrogen must be followed by the application of vacuum, or high headspace pressures can result after closure insertion owing to the insolubility of nitrogen in wine, which can lead to leakage. Dosing with carbon dioxide can be used without vacuum as long as the closure in use can withstand the transient high pressure generated until the carbon dioxide compressed by insertion of the closure can dissolve in the wine.

The headspaces of bottles that are to be sealed with screw caps can be dosed with carbon dioxide or nitrogen to displace oxygen prior to cap application.

Inserted closures must be applied without cuts or creases to ensure that maximum protection against oxygen entry is maintained. Screw caps must be applied with the correct head pressure and thread roller tensions to ensure that sufficient wad compression has taken place to provide an effective oxygen barrier. (See also Davies 2011.)

Addition of antioxidants: Several antioxidant materials may be added to wine. The effectiveness of these

materials depends on their ability to react more quickly with oxidants than other wine components. Reaction with oxidants reduces the concentration of the antioxidants. If the concentration of antioxidants is reduced to low levels, further exposure to sources of oxidants such as air will result in oxidative degradation of wine components causing changes to colour, aroma and flavour.

The major antioxidants added to wine are sulfur dioxide (SO₂), ascorbic acid and erythorbic acid.

Sulfur dioxide has already been considered in some detail in the section on microbial stability. SO₂ acts as an antioxidant as well as an antimicrobial agent in wine. Oxygen (and/or the reaction products of oxygen and other compounds in wine) reacts with the free SO₂ component of wine, producing sulphate and reducing the free SO₂ level. Equilibrium reactions between the bound and free SO₂ fractions in wine ensure that the free SO₂ fraction is topped up to a certain extent. However, when the free SO₂ level reaches a sufficiently low point, oxidised characters can emerge. In a study of closure performance by the Australian Wine Research Institute (Godden et al. 2001), the critical level of free SO₂ below which oxidation characters became apparent was found to be 10 mg/L.

The level of SO₂ in wine at bottling can have a significant impact on the shelf life of packaged wine. Free SO₂ levels of 45–50 mg/L are often used in wines that are packaged in containers that allow oxygen ingress, such as bags used in bag in box packs. The use of lower levels of SO₂ simply reduces the time that elapses before oxidised characters become apparent in the wine. Care must be taken, however, not to exceed the legal limits for SO₂ concentration or the point at which SO₂ becomes apparent in the sensory characteristics of the wine at the earliest time at which consumers are likely to open the wine package.

The measurement of molecular SO₂ has little relevance to protection against oxidation. This factor must be considered when wine is packaged in containers or with closures that allow oxygen ingress. The use of sufficient levels of SO₂ at bottling to reach molecular SO₂ targets for control of microorganisms may not provide sufficient protection against oxygen ingress to achieve a reasonable commercial shelf life.

SO₂ can also react with other oxidants, such as hydrogen peroxide. This is a key factor in the use of ascorbic acid in wine. This will be considered further in the following section.

As discussed earlier, SO₂ interferes with the structure and function of some enzymes. These include the oxidative enzyme polyphenol oxidase, which is strongly inhibited by SO₂. The other oxidative enzyme found in grapes, laccase, is not inhibited by the levels of SO₂ usually used in winemaking.

Ascorbic acid is often added to white wine to provide more rapid oxygen scavenging than is found with the use of SO₂ alone. Ascorbic acid reacts more rapidly with oxygen

than SO₂, producing hydrogen peroxide as a by-product. This hydrogen peroxide then reacts quickly and completely with free SO₂. Erythorbic acid is an optical isomer of ascorbic acid and provides equivalent oxygen reaction performance.

Recent research findings (Skouroumounis et al. 2005) show that ascorbic acid assists in reducing the degree of oxidation in packaged wines. Additions of up to 100 mg/L of ascorbic or erythorbic acid are generally made.

Care must be taken to ensure that sufficient free SO₂ is present in wine when ascorbic or erythorbic acid is added. As noted above, the reaction of ascorbic acid with oxygen leads to the formation of hydrogen peroxide. This hydrogen peroxide can then oxidise other wine components when free SO₂ is not present.

Additions of ascorbic and erythorbic acid can be very effective in preventing oxidative pinking in white wines.

References and further reading

- Allen, M. (1988) *Oenology 3 Notes Riverina Murray Institute of Higher Education* 1988.
- Australian Wine Research Institute Annual Report (2003).
- Barnett, D. (1985) Sulphites in food: their chemistry and analysis. *Food Technology in Australia* 37, 11.
- Burroughs, L.F. Determining free sulphur dioxide in red wine. *American Journal of Enology and Viticulture* 26(1), 25–29.
- Clarke, A.C. and Scollary, G.R. (2002) Copper(II)-mediated oxidation of (+)-catechin in a model white wine system. *Australian Journal of Grape and Wine Research* 8, 186–195.
- Davies, M. (2013) *Australian Winemaking, Bottling*, online www.trivinumpress.com.au/BOT
- Earle, M.D. and Putt, G. (1984) Sorbates in food – a review. *Food Technology in New Zealand* 9, 29–35.
- Fialdes, E. (1990) Les charbons actif en oenologie. *Revue des Oenologues*, 54, 19–22.
- Gerbaux, V., Villa, A., Monamy, C. and Bertrand, A. (1997) Use of lysozyme to inhibit malolactic fermentation and to stabilize wine after malolactic fermentation. *American Journal of Enology and Viticulture* 48, 49–54.
- Godden, P., Francis, L., Field, J., Gishen, M., Coulter, A., Valent, P., Hoj, P. and Robinson, E. (2001) Wine bottle closures: physical characteristics and effect on composition and sensory properties of a Semillon wine. 1. Performance up to 20 months post bottling. *Australian Journal of Grape and Wine Research* 7, 64–105.
- Hahn, G.D. and Possman, P. (1977) Colloidal silicon dioxide as a fining agent for wine. *American Journal of Enology and Viticulture* 28, 108–112
- Jarvis, B. and Lea, A.G.H. (2000) Sulphite binding in ciders. *International Journal of Food Science and Technology* 35, 113–127.
- Lee, T.H. (1985) Protein instability: Nature, characterization and removal by bentonite. In: *Physical Stability of Wine*. T.H. Lee (Ed.) pp. 23–40. Australian Society of Viticulture and Oenology, Adelaide.
- McDonald, D. (1988) Gelatine in wine and juice fining. *Australian and New Zealand Wine Industry Journal* 3, 27–28.
- Nygaard, M., Petersen, L., Pilatte, E. and Lagarde, G. (2002) Prophylactic use of lysozyme to control indigenous lactic acid bacteria during alcoholic fermentation. Presentation to the 53rd Annual Meeting of the ASEV, Portland.
- Ough, C.S. and Crowell, E.A. (1979) Pectic enzyme treatment of white grapes: temperature, time and skin contact time factors. *American Journal of Enology and Viticulture* 30, 22–27.
- Rankine, B.C. (1962) New method for determining sulphur dioxide in wine. *Australian Wine Brewing and Spirit Review* 80(5), 14, 16.
- Rankine, B. (1987) The role of bentonite in winemaking. *Australian Grapegrower and Winemaker*, July.
- Rankine, B.C. (1989) *Making good wine*. Macmillan, South Melbourne. 374 pp.
- Rankine, B.C. and Pocock, K.F. (1970) Alkalimetric determination of sulphur dioxide in wine. *Australian Wine Brewing and Spirit Review* 88, 40–44.
- Schmidt, T. (1987) Potassium sorbate or sodium benzoate? *Wines and Vines*, November.
- Skouroumounis, G.K., Kwiatkowski, M.J., Francis, I.L., Oakey, H., Capone, D.L., Peng, Z., Duncan, B., Sefton, M.A., Waters, E.J. (2005) The influence of ascorbic acid on the composition, colour and flavour properties of a Riesling and wooded Chardonnay wine during five years' storage. *Australian Journal of Grape and Wine Research* 11, 355–368.
- Simpson, R.F., Bennett, S.B. and Miller, G.C. (1983) Oxidative pinking of white wines: a note on the influence of sulphur dioxide and ascorbic acid. *Food Technology in Australia* 35(1), 34–36.
- Somers, T.C. and Evans, M.E. (1977) Spectral evaluation of young red wines: anthocyanin equilibria, total phenolics, free and molecular SO₂, 'chemical age'. *Journal of the Science of Food and Agriculture* 28, 279–287.
- Zoecklein, B. (1991) Protein stability determination in juice and wine. Virginia Cooperative Extension, publication 463–015.

Further information on this author and chapter:

<http://www.scorpex.net>