Fining Agents

- What is the purpose of using a fining agent?
- How do I know what fining agents are permitted to be added to my juice or wine?
- What are some of the most commonly-used fining agents?
- What is the best way to apply fining agents?
- If required, which fining agent should I use?
- If I use fining agents to make my wine, what are my obligations with regard to adding 'allergen' statements on my wine label?

What is the purpose of using a fining agent?

The purpose of adding a fining agent preparation to wine is to soften or reduce its astringency and/or bitterness; remove proteins capable of haze formation; or reduce colour by the adsorption and precipitation of polymeric phenols and tannins. The fining agent reacts with wine components either chemically or physically, to form a new complex that can separate from the wine.

How do I know what fining agents are permitted to be added to my juice or wine?

You can search the AWRI's database of 'Permitted additives and processing aids' available on <u>here</u>. This database doesn't provide a complete list of all the countries to which Australia exports wine but those which have made available their winemaking regulations. For specific details on an additive or processing aid permitted in a country, please refer to the country's regulations. For further information, please contact the <u>AWRI helpdesk</u>.

What are some of the most commonly-used fining agents?

Some of the most commonly-used and permitted fining agents for wine are:

- <u>Gelatine</u>
- <u>Isinglass</u>
- Egg white (egg albumen)
- <u>Casein</u>
- <u>Skim milk</u>
- <u>Bentonite</u>
- <u>Carbon</u>
- Polyvinylpolypyrrolidone (PVPP)

Selecting the appropriate fining agent for a given wine or juice depends on many factors: the most important being the destination market of the wine and the wine style sought by the winemaker. It is advisable to trial several agents in the laboratory, before addition in the cellar, as their effect will vary with wine style.

What is the best way to apply fining agents?

Fining should be carried out only when necessary and using lower rather than higher addition rates, as it is possible to remove positive flavour characteristics. It is important, however, that sufficient fining agent is added when the prime purpose of fining is to achieve stability and/or to remove undesirable sensory characters. It is essential that identical components used in the cellar for the finished wine is replicated in the laboratory trials. A control, where no addition is made, should always be set up so that the sensory effect of the addition can be assessed.

If required, which fining agent should I use?

The addition of protein based fining agents to white wines might also affect protein stability. It is advisable to carry out a heat stability test after the fining operation is complete. It is also advised that fining agents be assessed for the presence of any taints or off-flavours prior to use See <u>screening tests for commonly</u> <u>used winemaking chemicals and processing aids</u>. Note however that some fining agents, such as bentonite, gelatine and isinglass do have their own unique aroma.

Table 1. Comparison of fining agents and their different capabilities



Carbon
Casein
Isinglass
Egg white
Isinglass
Egg white
Casein
Isinglass
Casein
Isinglass
Bentonite
Casein
Gelatine
Bentonite
Carbon
Egg white

 1 Most effective at the top of each column and least effective at the bottom

Modified table reproduced from Zoecklein (1987).

Table 2. Typical rates of additions of fining agents in white and red wine

Type of product	
Typical doses used	
(mg/L)	
Characteristics	
White wine	
Gelatine	

15 - 120
Good clarity. Effective in reducing bitter after-taste.
Isinglass
10 - 100
Good clarity. Intensifies yellow colour. Light flakes, bulky, settles slowly.
Casein
50 - 250
Good clarification. Treats and prevents oxidation. No over fining.
Bentonite
200 - 1000
Average clarification. Treats and prevents protein instability and reduces the likelihood of copper casse. Facilitates settling. Avoids over fining.
Carbon
50 - 2000
Removes off-odours. Effective in colour reduction (browning and pinking).
PVPP
100 - 800
Effective in colour reduction (browning and pinking). Reduces bitterness. At high rates can result in colour and flavour stripping.
Red wine
Gelatine
30 - 240
Very good fining agent for tannic wines. Affects only the most aggressive tannins. May make wine softer or thinner.
Egg white (egg albumen)
30 - 150
Very good fining agent for tannic wines with some age. Tends not to remove protective colloids.

Casein

50 - 250

Good clarification. Treats and prevents oxidation. No over fining.

Bentonite

200 - 500

Clarification of young wines. Eliminates colloidal colouring matter. Facilitates sedimentation of protein fining agents.

Carbon
50 - 2000
Removes off-odours. Effective in colour reduction (browning and pinking).
PVPP
100 - 450

Reduces bitterness. Brightens colour. At high rates can result in colour and flavour stripping.

Modified table reproduced from Ribreau-Gayon et al. (2000).

Gelatine

Gelatine is often added to white juice, and particularly pressings, to aid clarification and to reduce the level of phenolic compounds associated with bitterness, astringency and browning. It is added to red wine to reduce the level of phenolic compounds associated with excessive bitterness and astringency and might also remove some colour. Gelatine interacts mainly with larger polyphenolic compounds and sometimes it is added in conjunction with tannin to provide better clarification.

Of the proteinaceous fining agents, gelatine is the most aggressive and can easily result in over fining and colour removal. As gelatine preferentially binds with larger molecules it has a more dramatic effect on colour and tannin reduction in older wines as they contain a greater percentage of large polyphenols. Gelatine is occasionally used to help remove the harshness and colour of press juice prior to fermentation. In view of the fact that gelatine is a wine soluble and heat un-stable protein, residual protein might remain in the wine if an excessive amount is used, possibly increasing the risk of the wine throwing a protein haze.

• <u>Preparation of 1% w/v stock solution for laboratory fining</u> There are two types of gelatine available; powder and liquid forms. One that is commonly used is a commercially available liquid form. The percentage gelatine activity (normally around 30%) should be noted from the manufacturer's instructions. A stock solution of 1% w/v can be prepared by diluting the liquid form accordingly. To make a 1% w/v solution using gelatine powder, add 10 mL of 96% ethanol to 80 mL of distilled water. Add 1 g of gelatine to the solution. Gently stir while warming the solution, but do not allow the temperature to exceed 40°C. When the mixture is homogeneous; make to volume with distilled water in a 100 mL volumetric flask. Mix well but gently. This stock solution should be prepared fresh every few days.

- <u>Conducting and assessing a laboratory trial</u> One mL of the 1% w/v stock solution added to 100 mL of juice/wine represents an addition rate of 100 mg/L.Typical ranges to evaluate white juices and wines are 15 to 120 mg/L, whilst red wines are 30 to 240 mg/L. To calculate addition rates, access the <u>AWRI</u> <u>Fining Trial Calculator</u>. The appropriate rate is determined by <u>sensory evaluation</u>.
- <u>Addition in the cellar</u> The temperature of the wine should be about 10°C. The liquid form of gelatine can be added directly to the wine. Accurately measure out the required volume. Add the liquid to the wine slowly and with thorough but gentle mixing. Allow a few days for settling, then rack or earth filter.

Isinglass

Isinglass is a preparation of the protein collagen and is primarily used for clarifying white wines. It gives a brilliantly clear wine and has a less dramatic effect on the astringency and body of the wine compared to gelatine. Monomers and smaller polyphenolic compounds react easily with isinglass, which can aid in the removal of harsh taste sensations.

Isinglass is available in two forms; as sheet or flocculated isinglass. The flocculated form is easiest to work with because it does not have to be rinsed to remove fishy odours.

Isinglass is principally used in white wine fining to bring out or unmask fruit character without large changes in phenolic levels. It is less active towards condensed tannins than either gelatine or casein. Since condensed phenolics are principally responsible for astringency, isinglass has a less dramatic effect on the reduction of both wine astringency and body than most other protein fining agents. Also, isinglass has the added benefit of not requiring extensive counter-fining as compared with other proteinaceous fining agents.

Excess isinglass can impart a fishy odour to the wine and thus should always be preceded with an assessment of a laboratory fining trial. The lees produced, when using isinglass, tend to be light and fluffy, thus care needs to be taken not to disturb the lees on racking or filtering. In the same way as gelatine, excessive use of isinglass can result in residual protein remaining in the wine, which might increase the chance of a protein haze forming.

- <u>Preparation of 0.5% w/v stock solution for laboratory fining trials.</u> Dissolve 1 g of citric acid in about 80 mL of distilled water. Add 0.5 g of finely chopped isinglass to this solution. Disperse by gentle stirring for about 12 hours or overnight (do not heat). Make to volume with distilled water in a 100 mL volumetric flask. The preparation is a jelly-like solution. If the preparation is taking a long time to prepare or if the preparation is to be stored, then sulfur dioxide should be added at a concentration of 200 mg/L.
- <u>Conducting and assessing a laboratory trial.</u> One mL of the 0.5% w/v stock solution added to 100 mL of juice/wine represents an addition rate of 50 mg/L. Typical ranges to evaluate white wines are 10 to 100 mg/L and if used for light red wines 30 to 150 mg/L. To calculate addition rates, access the <u>AWRI</u> Fining Trial Calculator. The appropriate rate is determined by <u>sensory evaluation</u>.
- <u>Addition in the cellar.</u> The temperature of the wine should be about 10°C. Prepare a 0.5 % w/v solution by weighing out the required amount of isinglass and dissolving in an adequate volume of water. Mix and ensure the isinglass is well dispersed. Add this solution slowly and with thorough but gentle mixing to the wine. Allow a few days for settling, then rack or earth filter.

Egg white (egg albumen)

A solution of egg whites can be used to remove phenolic compounds associated with harsh astringency in red wines, as the protein binds with the larger polymeric material in the wine. The fining leads to a softening and improved suppleness in the wine. It is often carried out when the wine is in barrel or prior to bottling. The weight of an egg white in an average medium size egg is approximately 30 g, of which approximately 12 g is protein.

- <u>Preparation of a 10% w/v stock solution for laboratory fining trials</u> Break eggs and separate the white from the yolk. Weigh egg whites into a large beaker. Add 10 times this weight of distilled water which has been adjusted to pH 7 (using potassium carbonate) and containing 0.5 % potassium chloride. Dissolving can be facilitated by potassium chloride as it maintains the globulins in solution. Stir gently, but avoid foaming, until dissolved. Vigorous stirring will denature the proteins. The egg white solution must be prepared fresh and used on the same day. Egg white is also available in dried and frozen form, however fresh egg whites tend to give the best result.
- <u>Conducting and assessing a laboratory trial</u> One mL of the 10% w/v stock solution added to 100 mL of juice/wine represents an addition rate of 1000 mg/L. Typical ranges to evaluate red wines are 300 to 600 mg/L. To calculate addition rates, access the <u>AWRI Fining Trial Calculator</u>. The appropriate rate is determined by <u>sensory evaluation</u>. As a guide, the number of egg whites added to 225 L of wine can vary from 2 to 8.
- <u>Addition in the cellar</u> The temperature of the wine should be about 10°C. Combine the required number of egg whites in a suitable size beaker, add 10 times the weight of distilled water adjusted to pH 7 and stir gently. Note that potassium chloride is not an allowed additive and thus cannot be used in the preparation in the cellar. Add this mixture slowly and with thorough but gentle mixing to the wine. Allow about a week for settling, then rack or earth filter. A small amount of foam might appear on the top of the wine which can be skimmed off or gently stirred into the wine.

Casein

Casein is the principal protein in milk. It is used mainly for fining white wine and Sherries to reduce the level of phenolic compounds associated with bitterness and browning. It is softer than gelatine or isinglass but has limited clarifying action. Several types of casein fining agents are available including casein, potassium caseinate, mixtures of potassium caseinate with bentonite/silica and skim milk. The most commonly used casein fining agent is potassium caseinate.

Casein can be used as a substitute for carbon in de-colourising wines. Although it is not as effective in colour removal as carbon, casein avoids the oxidative degradation often associated with carbon.

- <u>Preparation of 1% w/v stock solution for laboratory fining trials</u> Dissolve 1 g of potassium caseinate in 100 mL of distilled water with stirring. Warm the solution, but do not exceed 40°C. Stirring might be required for several hours to completely mix the powder into solution. This solution should be used within a day or two. If using casein, the preparation needs to be made alkaline to make it dissolve: dissolve 1 g of casein into 100 mL of distilled water which has been adjusted to pH 8 by addition of potassium carbonate.
- <u>Conducting and assessing the laboratory trial</u> One mL of the 1% w/v stock solution added to 100 mL of wine represents an addition rate of 100 mg/L. Typical ranges to evaluate are 50 to 250 mg/L. To calculate addition rates, access the <u>AWRI Fining Trial Calculator</u>. The stock solution of casein flocculates immediately on addition to wine. Therefore, when adding the stock solution to the wine, it should be quickly blown in via a graduated pipette into the middle of the volume of wine and mixed immediately. The appropriate rate is determined by <u>sensory evaluation</u>.

• <u>Addition in the cellar</u> The temperature of the wine should be about 10°C. Accurately weigh out the required amount of potassium caseinate or casein and dissolve in a minimal volume of distilled water. Add the solution slowly to the wine and mix in immediately. Rack or earth filter after about a week.

Skim milk

The addition of skim milk (low fat) removes similar phenolic compounds to those removed by casein/ potassium caseinate but is less specific.

- <u>Preparation of stock solution for laboratory fining trials</u> Skim milk can be added as a solution prepared from a powder or by diluting with water. Prepare a stock solution from the powder by following the manufacturer's recommendations. Prepare a stock solution from liquid skim milk (low fat) by diluting it 1:1 with distilled water.
- <u>Conducting and assessing a laboratory trial</u> For powdered preparations, follow the supplier's recommendations. The preparation of the 1:1 liquid skim milk (low fat)/ distilled water is added at rates between 0.4 and 2.0 mL to 100 mL wine. To calculate addition rates, access the <u>AWRI Fining</u> <u>Trial Calculator</u>.
- <u>Addition in the cellar</u> The temperature of the wine should be about 10°C. Accurately measure out the required volume of milk. Add the milk slowly, and with thorough but gentle mixing, to the wine. Allow a few days for settling then rack or earth filter.

Bentonite

Bentonite is a type of very fine clay made of aluminium-silicate. It is distinct from other clays in that it is formed from volcanic ash. Bentonite is principally used to remove proteins from white wine and juice, as it is a negatively charged clay colloid and reacts with positively charged proteins, precipitating them from the wine. Use of bentonite in red wines should be limited because of its ability to reduce colour by adsorption of anthocyanins.

More information about protein stability and bentonite fining can be found in the following articles:

- Getting proactive about protein (full text pdf available)
- Protein stability tests and their effectiveness in predicting protein stability during storage and transport (AWRI staff publication, request from library)
- Predicting protein haze formation in white wines (AWRI staff publication, request from library)
- Further understanding of bentonite's impact on metals in wine (full text pdf available)

Because the positive charge on proteins is stronger at lower pH values, the effectiveness of bentonite is greater in wines with lower pH values. If it is intended to adjust the pH and titratable acidity of the wine, then this should be carried out prior to bentonite fining, since stability might be different under the new pH conditions.

- <u>Preparation of 5 % w/v stock solution for laboratory fining trials</u> Weigh 5 g bentonite into a dry 50 mL beaker. Measure approximately 85 mL of distilled water into a 250 mL glass beaker. Heat the water to approximately 60°C. While stirring, slowly sprinkle the bentonite into the water to disperse it thoroughly. When all the bentonite is added, allow the suspension to cool. During this period the bentonite will swell; normally the suspension is left to stand overnight. If the bentonite is not well dispersed, the suspension might need to be heated a second time. The suspension is then made to volume with distilled water in a 100 mL volumetric flask. Mix well.It is important to note that the same batch of bentonite should be used in both laboratory trials and the cellar. Additionally, the bentonite suspension should be prepared under conditions as similar as possible to those existing in the cellar; with respect to water, water temperature, composition, and time of rehydration before use.
- <u>Conducting and assessing the laboratory trial</u> One mL of the 5% w/v stock solution added to 100 mL of wine represents an addition rate of 500 mg/L. To calculate addition rates, access the <u>AWRI Fining</u> <u>Trial Calculator</u>. Conduct an initial trial covering a wide range of bentonite additions in order to ascertain the appropriate rate of fining to achieve protein stability. This should be done using 100 mL measuring cylinders and adding the bentonite with a graduated pipette. Having established the rate at which stability is achieved in this range of additions, a further trial should be conducted at narrower range. The appropriate rate is determined by the heat stability test (80°C for six hours). For more information about the heat stability test <u>click here</u>. If a haze is seen in the fined heated wine, it is not stable and more bentonite is required. A turbidimeter can be used for more objective comparison of the turbidity in the heated samples. In this case, wines that exhibit a turbidity increase of greater than a given criterion for Nephelometer Turbidity Units (NTU) after heating, as compared with the unheated control, can be considered to have failed the heat stability test. Some laboratories use a criterion of 0.5 NTU, but other practitioners in industry have indicated that a more reasonable criterion is 2.0 NTU.
- Addition in the cellar Prepare a 5 % w/v solution by weighing out the required amount of bentonite and slowly adding, with stirring, to an adequate volume of water which has been heated to about 60°C. The suspension is left to stand overnight and used the next day. Mix well before addition. It is important that the bentonite is thoroughly dispersed in the slurry. If the suspension settles out or forms lumps, break up the lumps, reheat and stir vigorously to re-suspend the bentonite. To reiterate, it is important that the same batch of bentonite is used in both the laboratory trials and in the cellar. Additionally, the bentonite suspension should be prepared under conditions as similar as possible to those existing in the cellar; with respect to water, water temperature, composition, and time of rehydration before use.
- <u>Different bentonite types</u> Sodium bentonite, calcium bentonite or combinations are common. Calcium bentonites often require higher addition rates but are easy to hydrate and prepare and also result in quite compact bentonite lees and thus less wine loss. Extraction of calcium is possible into the wine so care should be taken to avoid <u>calcium instabilities</u>. Other metals can also be transferred into wine from bentonite refer to the article <u>Further understanding of bentonite's impact on metals in wine</u> published in *Technical Review* for more information.
- <u>Timing of bentonite additions</u> Bentonite fining is best performed on the final wine blend, after fermentation and after all proteinaceous fining has been conducted. Some producers may trial bentonite additions towards the end of fermentation to aid in yeast settling and also to reduce the number of racking/clarification steps. An additional light bentonite fining may be required once the wine is blended or sweetened or if the wine is fined with proteinaceous fining agents at later stages. Bentonite addition to juice can be successful but note that juice contains many types of protein that will naturally precipitate out of solution during fermentation without the need for bentonite. Thus, much higher rates of bentonite are often required.

Carbon

Carbon can be used to remove off-flavours and odours from wine, to decrease browning or pinking in white wines and to remove colour from red wines. There are two forms of carbon: 'decolourising' carbon, normally marked KBB and 'deodourising', normally marked AAA. Carbon works well in combination with PVPP in both tasks. The compounds to be removed are physically adsorbed to the large surface area of the carbon particles. The adsorption rate on the carbon surface is typically very fast. Carbon is regarded as a severe and relatively non-specific fining agent and therefore should be used with care. Special care should be taken to avoid exposure to carbon: use eye protection in combination with breathing protection.

- <u>Preparation of 10 % w/v stock solution for laboratory fining trials</u> Add 10 mL of 96 % ethanol to about 80 mL of distilled water. Add 10 g of the appropriate carbon to this solution. Stir to prepare a thoroughly mixed slurry. Make to volume with distilled water in a 100 mL volumetric flask. Mix thoroughly.
- <u>Conducting and assessing a laboratory trial</u> One mL of the 10% w/v stock solution added to 100 mL of wine represents an addition rate of 1000 mg/L. Typical ranges to evaluate are 50 to 500 mg/L for odour removal and 100 to 2000 mg/L for colour removal. To calculate addition rates, access the <u>AWRI Fining Trial Calculator</u>. Make sure the carbon slurry is mixed before each sampling. After addition of the carbon slurry, each addition rate (wine plus carbon) should be mixed about every 10 minutes over a 1 hour period. After 1 hour, filter each solution. The appropriate fining rate can be determined by sensory evaluation when carbon is used for odour removal. When carbon is used to remove colour or decrease pinking and browning, selection of the appropriate fining rate can be aided by the results of spectrophotometer readings, as well as by sensory evaluation.
- <u>Addition in the cellar</u> Weigh out the required amount of carbon. It can be added to the wine by direct addition of the powder or as a slurry, with thorough mixing. Allow a few days for settling, then earth or pad filter.

Polyvinylpolypyrrolidone (PVPP)

PVPP is a synthetic polymer used to reduce the level of phenolic compounds associated with browning and astringency in white wine. It can also be used to remove pink colour and pinking precursor compounds in white wines. PVPP is practically insoluble in wine and absorbs the low molecular weight phenolics, especially anthocyanins and catechins. PVPP is a gentle fining agent which preserves wine aroma, unlike some other fining agents. When used for colour reduction in white wines, combining with carbon is more effective in many cases, as it helps with clarification of the carbon particles. PVPP is not commonly used in red wines, however, it can reduce bitterness and brighten the colour. In many cases PVPP can also reduce certain off-flavours and bitterness.

• <u>Preparation of 10 % w/v stock solution for laboratory fining trials</u> Add 10 mL of 96 % ethanol to about 80 mL of distilled water. Add 10 g of PVPP. Stir to prepare a thoroughly mixed slurry and make to volume with distilled water in a 100 mL volumetric flask. Mix thoroughly.

- <u>Conducting and assessing a laboratory trial</u> One mL of the 10% w/v stock solution added to 100 mL of wine represents an addition rate of 1000 mg/L. Typical ranges to evaluate are 100 to 800 mg/L for white wines and 100 to 450 mg/L for red wines. To calculate addition rates, access the <u>AWRI Fining</u> <u>Trial Calculator</u>. When used to reduce astringency, the appropriate fining rate is determined by <u>sensory</u> <u>evaluation</u>. When PVPP is used to remove brown colour and pink colour, selection of the appropriate fining rate can be aided by the results of spectrophotometer readings, as well as by visual examination.
- <u>Addition in the cellar</u> Accurately weigh out the required amount of PVPP and make a slurry with minimal volume of distilled water. Add the slurry slowly with mixing to the wine, allow a few days for settling, then earth or pad filter.

If I use fining agents to make my wine, what are my obligations with regard to adding 'allergen' statements on my wine label?

Australia was the first country to adopt allergen labelling for foods in December 2002. A new clause was included under Standard 1.2.3 Mandatory warning labels and advisory statements and declarations of the Australia New Zealand Food Standards Code, which states that the presence in a food of a potential allergen must be declared on the label. For wine, this means that the presence of added sulfites (such as SO₂/PMS) in concentrations of 10mg/kg or more (preservative); the proteinaceous processing aids casein and potassium caseinate, egg white (including lysozyme), milk and evaporated milk, must be declared on the label. The Food Standards Code was amended on 28 May 2009 exempting isinglass (fish) for labelling in wine and beer. For further information on allergen labelling in Australia please refer to the <u>Wine Australia</u> <u>Compliance Guide</u> or the Food Standards Australian New Zealand website <u>http://www.foodstandards.gov.au</u> and <u>previously published information by the AWRI</u>.

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