

A Hatchman ②

MAKING GOOD WINE

A MANUAL OF WINEMAKING PRACTICE
FOR AUSTRALIA AND NEW ZEALAND

BRYCE RANKINE

WITH AN INTRODUCTION BY
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Other important points are the amount of contamination of bentonite with metals, particularly iron, freedom from foreign odours or taints, the ability of the bentonite to form a stable suspension in water, and the method of addition. To ensure rapid mixing it is best to add the bentonite suspension on the suction side of a pump circulating the wine in a vessel. Bentonite treatment is a mixing problem, and this method of addition has been found to be an effective way of rapidly mixing the aqueous bentonite suspension into the wine.

The dosage required depends largely on the wine, the type of bentonite and its method of preparation. Consequently, a laboratory fining trial is necessary. Usually a fining rate of 0.3 to 0.8 grams per litre is adequate, but some wines made from protein-rich grapes, such as Muscat Gordo Blanco, may need a higher addition. A fining rate of 0.5 grams per litre, for example, corresponds to an addition of 5 litres of a 10 per cent smooth suspension in water to 1,000 litres of wine.

Addition of a complementary fining, such as gelatin, assists in sedimenting the bentonite. Protein removal occurs quickly in wine. If mixing is rapid, the protein is removed in the first few minutes and the remaining time is spent waiting for the bentonite to settle. In fact it is possible to fine wine with bentonite in-line if it is effectively mixed. The wine can then be centrifuged or filtered and stability achieved quickly.

The use of bentonite is decreasing, due to the high cost of its recovery, reduction (usually) in aroma and flavour, a small increase in the sodium content of the treated wine (which could influence the export of the wine to the European Economic Community, where low sodium is specified), and the availability of other methods of clarification.

To the present, protein-splitting enzymes or proteases have not been completely effective in protein removal in wine, but when protease becomes available which can operate rapidly and economically at the acidity levels of white table wines and without untoward side effects, this should result in reduced usage of bentonite.

We must be careful not to regard protein in wine as only one constituent. Recent findings with Muscat Gordo Blanco juice indicate many protein fractions, ranging in molecular weight from 31,000 to more than 500,000. The isoelectric points of the various fractions are similarly wide, ranging from 3.7 to 9.5, although most of the protein fractions lie within the narrow pH range of 3.7 to 4.7.

Cold-stabilisation

Young wine is usually saturated with potassium bitartrate and sometimes calcium tartrate, although the latter is not usually present in sufficient concentration to reach saturation unless the calcium content of the wine is high. When the wine is cooled the solubility limit of potassium bitartrate is exceeded and some comes out of solution as a crystalline deposit. When this happens to white wine in the bottle the deposit is unsightly and may be mistaken by the uninitiated for broken glass. Customers expect such wines to be free from deposit and, consequently, its prevention is essential.

Potassium bitartrate is present in the grapes, partially precipitates as argols during fermentation because of its lower solubility in alcohol, and is present in the young wine as a saturated or even a super-saturated solution in excess of its normal solubility. If the wine is cooled some potassium bitartrate slowly precipitates on the walls and floor of the storage vessel as argols (impure crystals of potassium bitartrate with pigment from the wine), until the saturated equilibrium at that temperature is reached. This slow precipitation can take weeks or sometimes months to reach the new equilibrium set up by the cooler temperature. If the wine is further cooled more potassium bitartrate precipitates until a further new equilibrium is reached. Calcium tartrate, if present, is also involved in this precipitation, but at a slower rate because its solubility is less influenced by temperature.

Several factors influence the rate of tartrate precipitation, viz:

- nucleation or the number of tiny nuclei on which the crystals form and grow;
- diffusion, or the rate at which the dissolved potassium bitartrate contacts the growing crystals;
- crystal surface growth or the rate at which cold stabilisation occurs;
- the grape variety.

The amount of potassium bitartrate which will remain in solution depends on the following factors:

- Alcohol content—the higher the alcohol level, the less soluble is potassium bitartrate. This is the reason why masses of argols or impure potassium bitartrate are deposited during fermentation.
- Temperature—the cooler the temperature, the less soluble is the bitartrate.

- pH—at pH 3.6 or thereabouts, depending on the alcohol content, the proportion of the sparingly soluble potassium bitartrate in relation to the soluble tartaric acid and potassium tartrate is greatest. As the pH moves away from 3.6 in either direction the proportion of potassium bitartrate becomes less, although the total tartrate content remains the same, and the likelihood of precipitation becomes less. Because of this pH effect two cold-stable wines can result in an unstable blend. Therefore, the blend must be cold-stabilised and not the individual components.
- Potassium (and calcium) content—the addition of potassium or calcium renders the wine more cold-unstable.

The normal procedure for cold-stabilising wine before bottling to avoid unsightly precipitation of tartrate crystals in the bottle has been to chill the wine through a heat-exchanger to near its freezing point in the temperature range of about minus 2 to plus 2°C, store in an insulated vessel for between one and three weeks, then filter or rack while still cold. In very cold areas the same result can be achieved by opening the cellar doors to chill the wine naturally over winter.

Advances in technology have resulted in important changes to this cold-stabilisation process, particularly through a greater understanding of the principles of nucleation involved in the precipitation of potassium bitartrate in wine—the main compound involved in cold instability. The most important at present is the contact process.

Stabilisation by contact process

This process is an important development in the rapid cold stabilisation of wines, and by virtue of saving considerable time has advantages over the traditional prolonged cold storage of wine. German researchers in a joint venture between the Seitz-Werke and the Henkel companies have found that wine can be cold-stabilised rapidly by stirring in contact with finely powdered potassium bitartrate crystals for 30 to 60 minutes at approximately 0 to minus 2°C. Potassium bitartrate present in the wine as a saturated solution at normal temperature crystallises on to the added crystals, which serve as the source of nucleation. The deposited crystals are removed, dried, ground and reused. The stabilised wine is then warmed against incoming wine in a heat-exchanger, and will be stable at temperatures down to that at which it was treated.

The small crystal size (less than about 40 microns and about the same particle size as finely powdered icing sugar) is necessary to expose sufficient surface area to act as the nucleation sites on which the potassium bitartrate in the wine can crystallise quickly.

potassium bitartrate crystals can be used for seeding—other sharp-edged crystals do not produce the desired result. It is necessary to add a large number of very small crystals in order to provide sufficient surface area. In fact, 4 grams of finely powdered potassium bitartrate crystals (the recommended dosage per litre) has a crystal surface area of approximately 3 square metres. Crystallisation is rapid and complete in an hour or less in the stirred suspension, because the potassium bitartrate molecules in solution need only to travel a calculated average of 0.3 millimetres before encountering a crystal face on which to grow.

Furthermore, polymeric inhibitors of nucleation, which slow down the rate of conventional cold-stabilisation, have relatively little effect on the stabilisation time required by the contact process. The avoidance of this problem means that stabilisation by the contact process is more predictable. The deposited potassium bitartrate is reused by separating, drying and grinding, and the balance is available for sale as a winery by-product (cream of tartar) if a demand exists for it. Stabilisation is best carried out after final blending before bottling.

Cold-stabilisation of red wines by the contact process usually involves a brief preliminary chilling to remove cold-unstable pigments and tannins, otherwise gross contamination of the crop of potassium bitartrate crystals will occur. The length of time of pre-chilling depends on the wine and may require only 24 hours.

The contact process has considerable economic advantage, particularly for white wines, because the wine needs only to be cooled to the desired temperature for an hour or so, enabling continuous cold-stabilisation to be carried out with this process. For batch treatment the successive operations are as follows: chill the filtered wine against out-going wine to the desired stabilisation temperature, add 4 grams per litre of finely powdered potassium bitartrate crystals, mix for approximately one hour, and separate the deposited crystals by centrifugation.

Stabilisation by filtration

Another process employs stabilisation by filtration of the chilled wine using a filter containing a bed of potassium bitartrate crystals, with a lower rate of seeding, such as 1 gram per litre, in the incoming wine. The crystals grow in the filter as the potassium bitartrate in the chilled wine deposits on the preformed crystals. The wine may be manipulated through the filter if required.

Stabilisation by crystal flow

Further process is crystal flow developed by the Alfa Laval company,

in which tiny potassium bitartrate and ice crystals are generated in the wine by chilling in a scraped-surface heat-exchanger (as distinct from swept-surface), at about the freezing point of the wine. Three steps are involved:

- 1 Cooling the wine to its freezing point with agitation to induce formation of tartrate crystal nuclei.
- 2 Growth of the crystals in the suspension of ice and tartrate crystals.
- 3 Melting the ice crystals in a plate heat-exchanger to allow the tartrate crystals to be removed by filtration or racking.

The temperature to which the wine is chilled depends on the alcohol and solute composition and is usually about minus 5°C. The crystals act as nuclei for further crystal growth, without having to add seeding crystals, and the plant is easy to run as it is continuous and self-regulating, as distinct from the seeding process. This process relies on having a scraped-surface heat-exchanger, which is not a normal item of winery equipment at present. However, the normal swept-surface ultracooler can be modified to operate as a scraped-surface cooler.

The question may be asked as to which of these processes is the 'best' under our conditions, and it is likely that all three will find a place in our industry. Slow chilling and prolonged cold storage of wine to allow cold-stabilisation to take place is still being used, for the joint reasons of retaining quality and achieving cold-stability in the process. However, these recent developments are expected to save time as well as to lead to continuous operation. Quality-control tests for checking cold-stability are set out in Chapter 21.

Refrigeration requirements for cold-stabilisation

If the wine is to be cold-stabilised by initially chilling it through a heat-exchanger, the chilling requirement in kilowatts is the product of the wine flow rate in litres per hour and the required temperature drop in °C, divided by 850. To this must be added the minor heat loads of the electrical power of the wine pump and the heat gain from the surroundings, both calculated using the same procedures as set out in the section on must cooling in Chapter 5.

If the wine is to be cooled in a tank, the chilling load in kilowatts is the product of the volume of wine in kilolitres, the required temperature drop in °C, and 1.15, divided by the time in hours required to cool the wine. If the tank is insulated with 50 millimetres of polystyrene, the heat gain from the surroundings in kilowatts is the tank surface area (walls and top) in square metres divided by 75. Alternatively, if the tank is uninsulated but inside an enclosed non-cool-

winery, the heat gain is the tank area divided by 8. If the tanks are uninsulated but housed in an uninsulated building, the heat gains from the surroundings, fans, people, lights, etc. can be calculated as set out in the section on fermentation cooling in Chapter 8.

Prevention of tartrate precipitation by ion-exchange

Ion-exchange treatment for prevention of potassium bitartrate and calcium tartrate precipitation in wine was developed in Australia and has been in use since 1955, and its application has been of considerable benefit to the wine industry. It employs the same resin and equipment as is used for acidification of must and wine, and Chapter 7 should also be referred to.

An ion-exchange resin is an insoluble gel matrix containing labile ions capable of exchanging with ions in the surrounding liquid, without physical change taking place in the structure of the resin. The resins are divided into two broad groups, cation and anion exchangers, which can be further subdivided into weakly acid or basic and strongly acid or basic, according to their chemical groupings. The resins used in Australian wineries are highly acidic unfunctional cation-exchangers with a polystyrene base and sulphonic-acid groups which contain exchangeable hydrogen atoms. The resins are in the form of little amber-coloured beads, which are more easily handled than the irregularly shaped granules of some other types of resin. They are also resistant to chemical attack.

From the viewpoint of understanding the process of prevention of potassium bitartrate deposition, wine may be regarded as a saturated solution of potassium bitartrate in dilute ethyl alcohol, with other cations, mainly calcium, magnesium and sodium; and anions, mainly malate, succinate, lactate and the inorganic anions. Wine can be stabilised by ion-exchange in three ways:

- By replacing potassium with sodium with a cation-exchange resin in the sodium cycle, forming the soluble sodium bitartrate.
- By replacing the tartrate anion with hydroxyl or other anion with an anion-exchange resin.

By replacing potassium and tartrate respectively with hydrogen and hydroxyl by a cation and an anion resin, in effect exchanging portion or all of the potassium bitartrate for water.

In practice, the sodium cycle is the most suitable because wine can be stabilised without affecting its flavour and with only slight reduction in acidity. It increases the sodium content of the wine, which

may be undesirable for people on a low-sodium diet. If desired, the acidity of the wine can be increased by preparing the resin in the mixed sodium and hydrogen form. This is desirable when treating low-acid dessert wines, since the acidity of the wine can be increased to any desired level concurrently with prevention of potassium-bitartrate precipitation. The resin can also be prepared in the magnesium, or mixed magnesium and sodium form.

When considering the sodium exchange, in which wine passes down through a column of a cation exchange resin in the sodium form so that the potassium in the wine is replaced with sodium from the resin, care must be taken not to oversimplify the process. The resin has a greater affinity for the divalent cations, calcium and magnesium, than for potassium, and as the wine flow progresses the resin column takes up calcium and magnesium preferentially as a top zone, below which is the potassium zone while the remainder of the resin column is in the sodium form.

As the wine continues to pass down, these zones in the resin also extend although far more slowly than the wine, until at the breakthrough point where no more sodium remains on the resin, the potassium zone reaches the bottom of the resin column. Potassium then begins to be released from the resin back into the wine, due to its continued replacement on the resin by calcium and magnesium. If the wine flow continues the situation alters markedly. The resin is then in the combined potassium, calcium and magnesium form and further wine flow will have the effect of displacing potassium from the resin column, so that the effluent wine again becomes prone to potassium bitartrate precipitation.

Method of operation

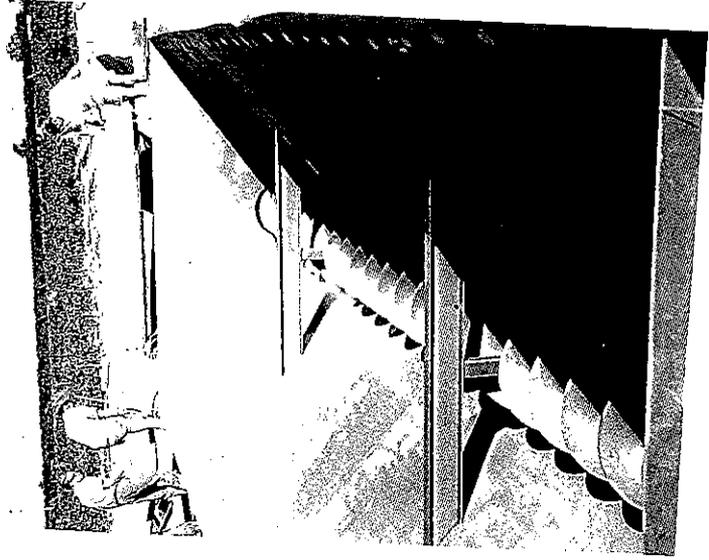
The recommended flow rates and related details for column operation are set out below. For proper operation of the column a flowmeter or gauged vessels are essential.

1 Regeneration: 2.5 resin volumes of 10 per cent sodium chloride brine are flowed up through the resin bed in about 20 minutes. The regeneration rate corresponds to 25 kilograms of sodium chloride per 100 litres of resin, and for a column containing 800 litres of resin, for example, 126 kilograms of salt dissolved in 1200 litres of water are required. In the past, down-flow regeneration was carried out but is now being replaced by the more efficient up-flow.

2 Washing: About 6 resin volumes of clean wash water are required and this should flow down through the resin bed in about 30 minutes until free from salt. A check with silver nitrate solution will indicate whether any salt remains by forming a white silver chloride precipitate.



Measuring the temperature of a grape load. (S. Smith & Sons Pty Ltd, Angaston, SA)



Grape receipt at Renmark Growers Cooperative

Rain water is best, but if this is in short supply then mains water can be used after passage through the resin, which then acts as a conventional water softener. Untreated mains water can contain appreciable quantities of calcium and magnesium, which will reduce the efficiency of the resin.

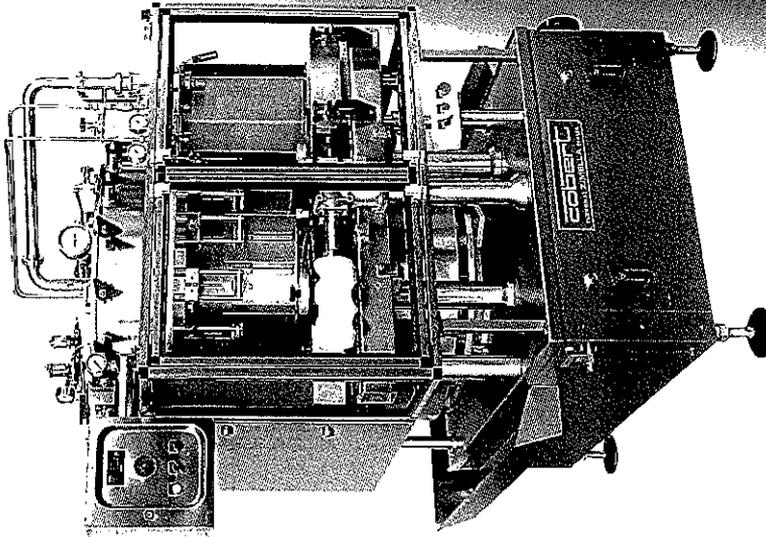
3 Wine flow: 15 resin volumes per hour are flowed down through the resin until the breakthrough point occurs. This is usually after about 30 resin volumes have been treated, depending on the content of potassium, calcium, and magnesium in the wine. The wine flow is complete in about 2 to 3 hours. A knowledge of the cation content of wines likely to be encountered is important in understanding the reason why the resin does not treat the same quantity of different wines. The range of values of the major cations in Australian wines in milligrams per litre is shown in the following table.

Wine type	Potassium	Sodium	Calcium	Magnesium	Sum of cations
White table	390-1520	12-276	38-112	55-115	495-2023
Red table	730-3000	9-276	40-112	68-180	847-3568
White dessert	702-1560	58-253	30-70	49-114	839-2027
Red dessert	780-1870	70-276	32-78	73-132	955-2356

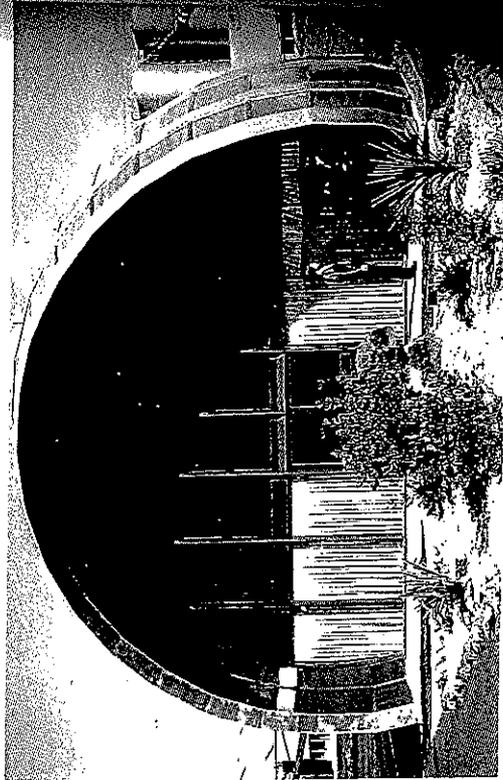
In order to avoid diluting the wine on initial entry to the column, it is best to drain the wash water to waste and then introduce the wine from the bottom until the column is full and the air removed, the wine reverse the flow so that the wine enters at the top of the column, then upward flow of wine should not be used because channeling occurs resulting in inefficient exchange. To avoid aeration all wine movements should be carried out under inert gas.

It is important to be able to measure the breakthrough point when the resin becomes exhausted of its sodium ions. Since the process principally involves exchange of two similar cations, potassium and sodium, there is no obvious change in acidity or taste at the end of the run, and the progress of the treatment cannot be observed by acidity measurements. The pH of the wine usually rises slightly during the course of the exchange (up to 0.05 pH unit) due to replacement of some of the hydrogen ions in the wine with sodium.

The most desirable method of detecting the breakthrough point is to measure the potassium content of the effluent wine at intervals, so that the breakthrough point can be determined accurately. The measurements can be made quickly with a flame photometer, or by measuring the potassium content chemically with an appropriate



Cobert 16-head counter-pressure filler with pre-evacuation of bottles and self-levelling (J.B. MacMahon Pty Ltd, Adelaide)



'Cask Room' for public relations functions (McWilliams Wines Pty Ltd, Hanwood, NSW)

reagent, such as cobalt-nitrite. The flame photometer is rapid and accurate, and should be regarded as part of the capital outlay of setting up an ion-exchange plant. It is possible to determine the stage at which potassium bitartrate precipitation reappears in the effluent wine by refrigerating samples taken at intervals, but such a procedure cannot be carried out immediately and does not give an indication of the stage at which the breakthrough point occurs.

4 Backwashing: This loosens and resets the bed, and also washes out any particulate matter that may have been introduced during the previous cycle. About 12 resin volumes of water are required and should be passed up through the resin in about 30 minutes, and until the effluent is quite clear. The wine should always be clear before treatment and the resin should not be used as a filter, since a surface coating of colloidal and suspended constituents will reduce the exchange capacity of the resin as well as reduce the flow rate.

Size of ion-exchange columns. When ion-exchange treatment was first introduced the columns used were almost all less than 50 centimetres in diameter, and were eventually found to be too small as wineries treated a larger proportion of their output. Current practice is to build columns with larger diameters, such as one metre, containing about 1000 litres of resin. Assuming a minimum throughput of 30 resin volumes to the breakthrough point, 1000 litres of resin will treat about 30,000 litres of wine, which is a useful size for many wineries and does away with the need for back-blending. The depth of resin in the column should be at least 75 centimetres, otherwise channelling and inefficient exchange will occur.

Inefficiency of batch regeneration. One maker of ion-exchange columns produced a unit mounted on trunnions so that it could be rotated end-over-end in a vertical plane, with the object of mixing the resin with the brine to achieve regeneration as a batch process. This is an inefficient method, because to obtain the most efficient regeneration the brine must pass through a stationary resin bed whereby an efficient exchange takes place with the released cations—potassium, calcium and magnesium—passing to waste with the brine effluent. If the regeneration process is carried out batch-wise the regenerant actually becomes a mixture of sodium, potassium, calcium and magnesium—the last three being released from the resin—and the final charge on the resin is a mixture of the four cations in an equilibrium determined by their concentrations and relative affinities for the resin.

Effect on taste of the wine. Many tests have been carried out on the effect of ion-exchange treatment on the taste of the wine, with the consensus being that the taste of dessert wines is not detectably

altered but that of delicate table wines may be. Some winemakers treat their entire make of dessert and table wines, whereas others do not ion-exchange their white table wines. It is unlikely that a trained taster could detect whether a wine has been treated without having the control as a comparison.

Danger of over-running the column and effect of blending. The danger of over-running the column should be stressed. The treated wine up to the stage of breakthrough cannot precipitate potassium bitartrate under any conditions, and can be blended judiciously with a proportion of untreated wine if desired. However, if the column is over-run well past the stage where potassium begins to be released from the resin back into the wine, then the wine will again precipitate potassium bitartrate, and this cannot be corrected by a further passage through regenerated resin since the wine is now high in sodium and the second exchange treatment is ineffective. It should be remembered that an exchange of ions continues as long as wine is passed through the resin, until the resin is saturated with calcium and magnesium.

The danger of over-running the column is related to a situation which can easily occur—viz. the purchase of a parcel of treated wine, perhaps in ignorance that it has been treated, and blending this with other wine which has not been treated to produce a composite blend for bottling. If the final blend is unstable due to the moiety of untreated wine, it may not be able to be stabilised by a subsequent ion-exchange treatment because the wine already contains a considerable amount of sodium from the first treatment. In such cases, it is advisable to ensure that the proposed components of a blend are both ion exchanged before blending.

Storage of resin in the column. Preservation of the resin in the column during the period when it is not in use can present a problem, since removal of the residue of sugar left after treating dessert wines is difficult and the moist resin may grow mould or become stagnant. Various methods have been used, such as storage in brine, sulphurous acid or diluted spirit. The resin should be in the regenerated hydrogen form, and most winemakers prefer to use brine storage although it has a slight corrosive action on stainless steel. Sulphurous-acid storage has the advantage of acting as a mild acid regenerant and removes the metal ions bound to the resin. New resin may impart an initial off-flavour to the wine, and this is best prevented by soaking the resin in fortified wine for a few days and then regenerating it with warm dilute hydrochloric acid before brine regeneration.

Restoration of resin after prolonged use. After long use the resin

may partially lose its exchange capacity and progressively treat less wine per regeneration. The reason for this is due to absorption by the resin of a range of polymeric compounds—proteins, tannins, pectins and the like—which reduce the number of exchange sites available. These compounds, together with metals, are not adequately removed by brine regeneration and more drastic action is necessary. The best treatment is to soak the resin in sodium hypochlorite solution containing about 3 per cent available chlorine which will restore its colour and activity. Soaking in hydrogen peroxide is another treatment. If this does not fully restore the resin then a treatment with chromic acid (after removing the resin from the column) will rid the resin of contaminants, but this acid is corrosive and difficult to handle. Cation-exchange resins are resistant to oxidation and can withstand fairly drastic treatment without damage.

Measurement of exchange capacity of resin. The method of checking the exchange capacity of the resin is simple and is given here so that winemakers can test their own resin.

- 1 Dry the resin in a thin layer in an oven at 105°C for an hour. Weigh accurately 1 gram slurry with water and place in a vertical glass tube about 6 millimetres internal diameter, with the resin supported on a pad of glass wool. The resin bed will be approximately 10 centimetres high. Fit a rubber tube and constricting clip to the bottom of the glass tube so that the flow of liquid down through the resin can be controlled, and mount the tube vertically in a retort stand. Ensure that the resin is free from air bubbles and covered with liquid throughout the determination.

- 2 Pass down through the resin 10 millilitres of dilute hydrochloric acid (1 part acid and 3 parts water) with the flow rate adjusted so that this takes about 20 minutes. Then pass down distilled water, at a faster rate if desired, until the effluent is less acid than pH 4. Pass down through the resin 10 millilitres of 20 per cent sodium chloride solution in about 20 minutes, followed by 10 millilitres of distilled water, collecting the effluent in a beaker. Add approximately 50 millilitres of distilled water and a few drops of phenol-phthalate indicator to the beaker and titrate with standard sodium hydroxide either normal or tenth normal. The exchange capacity of the resin in milli-equivalents per dry gram is simply the number of millilitres of normal sodium hydroxide, or one-tenth the number of millilitres of tenth normal used in the titration.

Resins from different makers vary somewhat in their exchange capacities, and the makers' specifications should be referred to. New resins usually have an exchange capacity of between 4 and 8 milli-equivalents per dry gram.

Removal of trace metals from wine. Copper and iron are present in wine in both ionised and bound forms. The ionised metals are removed by ion-exchange but those bound as non-ionised complexes to the organic acids in wine are not. The extent of complexing is less as the pH falls and this is the reason why metal removal is less effective with acid wines, and best when the resin is used in the hydrogen form for acidification. Ion-exchange treatment does not bring about a significant reduction in the content of lead in wines. It has been found that metals removed from wine tend to accumulate on the resin and are poorly removed by brine regeneration. It is desirable to give the resin a regeneration with acid at intervals to remove these metals.

Comparison with refrigeration. The advantage of ion-exchange treatment is its low cost compared with refrigeration, usually less than 10 per cent. The final result of both treatments is the same as far as prevention of potassium bitartrate deposition is concerned, although refrigeration is claimed to stabilise wine against other cold-unstable materials, which are little affected by ion-exchange, although sometimes claimed that refrigeration improves a wine, although the danger of oxidation after refrigeration is well known. Also, refrigeration has other uses, such as cooling fermentations during vintage, and ion-exchange may be used in conjunction with refrigeration, for example, when a winemaker does not wish to ion-exchange a delicate white table wine, while the remainder of the wines are ion-exchanged.

In recent years sodium ion-exchange has decreased in use, because wineries have installed refrigeration for temperature control during vintage and then used this later for cold stabilisation. In addition, if the wine is to be exported some countries have a maximum limit for sodium, which precludes the use of sodium ion-exchange for such wines.

Metatartaric acid

One of the valuable recent developments in oenology has been in new methods of preventing potassium bitartrate precipitation when wine is chilled. Metatartaric acid falls into this category—it is a nucleation inhibitor meaning that it prevents crystals from forming. It was first used in Europe in 1955.

When finely ground tartaric acid powder is heated in an oven at 200°C it melts and undergoes a transformation involving release of water vapour, dehydration, esterification and polymerisation. The result is an amorphous deliquescent (absorbs moisture from the air)