



POTASSIUM BITARTRATE STABILIZATION OF WINES

Learning Outcomes: *Bitartrate stability and determining the optimum method(s) to use require and understanding of time, energy, water, water composition, waste and recovery, and the impact on wine sensory features. The reader will understand the practical considerations influencing crystal formation, bitartrate stabilization and evolution of wines*

Chapter Outline

BZ correct

Potassium Bitartrate Instability

Complexing Factors

Electrodialysis

Addition Compounds for Potassium Bitartrate Stabilization

Fining and Bitartrate Stability

Determining Potassium Bitartrate Stability

Cellar Considerations for Contact Seeding

Section 1.

Wine instability can be classified as one or more of the following:

- microbiological
- protein

- tartrate
- red wine oxidative and color
- white wine oxidative and color
- metals

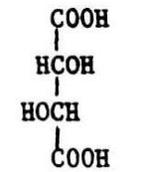
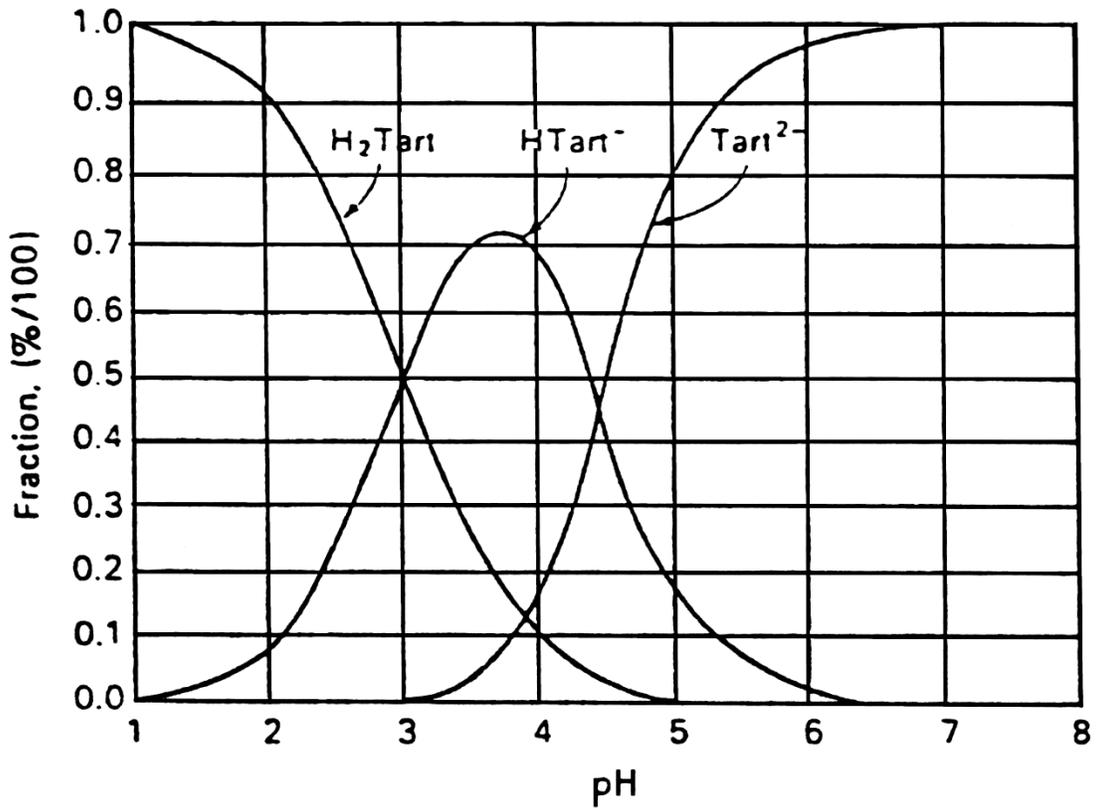
The problems of producing stable, highly palatable products are of concern to every vintner. The days when wineries can release wines to the public with physical instabilities, and still compete, have passed. Therefore, it is essential that every vintner understand the parameters affecting wine stabilities, and operate to obtain product stability while maximizing palatability.

Bitartrate Instability

Tartaric acid (H_2T) and its salts, potassium bitartrate (KHT) and calcium tartrate (CaT), are normal constituents of juice and wines and important to stability. The formation of crystalline deposits is a consequence of wine chemical and physical properties as well as aging. In that crystalline deposits generally do not meet with consumer acceptance, most winemakers strive to reduce the potential for post-bottling formation.

The tartaric acid content of grape must/juice ranges from 2.0 to 10 g/L and varies by region, variety, maturity, soil, and viticultural practices. In grapes and wine, tartaric acid ionizes to yield bitartrate and tartrate. Depending on pH, the ratios of $H_2T/HT^-/T^{2-}$ can vary greatly and thus significantly influence the potential for precipitation of insoluble salts. The relative distribution of each component as a function of pH is seen in the following figure:

Figure 1. Tartaric Acid and Ionized Species as a Function of pH. Source: Fritz and Schenck (1974)



Tartaric Acid (H₂T)



Partially-Dissociated Tartaric Acid (HT⁻)



Completely-Dissociated Tartaric Acid (T⁼)



Potassium Bitartrate (KHT)

Potassium bitartrate (KHT) is believed to be produced after véraison (beginning of ripening) with the movement of potassium into the fruit. During ripening, the amount of undissociated tartaric acid decreases as the mono and dibasic potassium salts are formed. Simultaneously, tartaric acid is diluted at a rate depending on the variety and climatic conditions as berry size increases.

Additionally, the tartrate content is also influenced by location, cultural practices, and the state of maturity.

Potassium uptake in the vine is dependent on factors such as rootstock, soil type and depth, irrigation treatment, etc. Thus, both tartrate and potassium contents differ widely with variety, region, climate, and viticultural practices. Both tartrate and potassium concentrations vary widely. The level of potassium in grape must ranges from 600 mg/L to over 2,500 mg/L. For example, it is not uncommon for Virginia produced fruit to have a potassium content of 2500 mg/L. As a consequence, winemakers handles juice and wines that have large variations in KHT concentrations.

Although potassium bitartrate is partially soluble in grape juice, alcohol and low temperature decrease its solubility, resulting in a supersaturated solution and subsequent precipitation by the end of fermentation and during cellaring.

Calcium is present in wine at levels of as low as 6 to 165+ mg/L (Amerine and Ough 1980) and may complex with tartrate (T^{2-}) and oxalate ($C_2O_4^{2-}$) anions to form crystalline precipitates. Several sources contribute to increased calcium in wine, including:

- soil practices, such as liming
- fermentation or storage in concrete tanks
- use of calcium-containing fining material and filter pads.
- $CaCO_3$ is used in deacidification or where "plastering" is used for adjusting the acidity of shemat material (sherry base wine)

When calcium tartrate instabilities are found, they usually appear from 4 to 7 months after fermentation. Calcium tartrate can represent a problem due to its temperature independence (relative to KHT) and the difficulties in predicting instability. Because of changes in processing technology, CaT is generally not as significant a problem in the United States as it once was.

All wines differ in their “holding,” or retention, capacity for tartrate salts in solution. If the holding capacity is exceeded, these salts will precipitate, resulting in the formation of "tartrate casse." Solubility of potassium bitartrate is dependent primarily upon:

- alcohol content
- pH
- temperature
- interactive effects of various cations and anions.

Although KHT is soluble in grape juice, the production of alcohol during fermentation lowers the solubility and gives rise to a supersaturated solution of KHT. In wine, the following equilibrium exists:

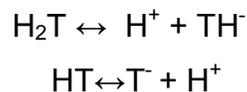


Figure 1 shows the distribution of tartrate species in wine as a function of pH. The percentage of tartrate present as potassium bitartrate (KHT) is maximal at pH 3.7, and, if other factors permit, precipitation will be greatest at this point.

- Any wine treatment causing changes in pH, such as blending, occurrence of a malolactic fermentation, acid addition, etc., may affect subsequent bitartrate precipitation.
- This is a primary reason why potassium bitartrate stability must be checked following all cellaring activities, just prior to bottling.

Potassium bitartrate stability has been traditionally accomplished by chilling, ion exchange, or both. A number of relatively new methods to obtain potassium bitartrate stability have been employed or suggested including:

- electrodialysis (Postel et al., 1977)
- reverse osmosis (Wucherpfennig, 1978)
- crystal-flow™ (Riese, 1980)
- contact seeding

- addition products such as carboxymethyl cellulose (CMC)

Of these processes, filtration, Crystal-flow™, and contact seeding have been the most successful commercially (Ewart, 1984).

Crystal Formation

In conventional cold stabilization procedures (chill-proofing), wines are chilled to a temperature designed to decrease potassium bitartrate (KHT) solubility, which optimally results in precipitation. The most important variables affecting the precipitation of potassium bitartrate during chilling are the following:

- the concentration of the reactants, specifically tartaric acid
- the availability of foci or nuclei for crystal growth
- the solubility of the potassium bitartrate (KHT) formed

Perin (1977) determined the following relationship for the temperature needed for KHT precipitation:

$$\text{Temperature } (-^{\circ}\text{C}) = \left(\frac{\% \text{ alcohol}}{2} \right) - 1$$

KHT precipitation occurs in two phases. The induction phase is when the level of KHT in solution increases due to chilling. This is followed by the crystallization phase, where crystal growth and development occurs. The rate of precipitation of potassium bitartrate at low temperatures is more rapid in table than in dessert wines, and more rapid in white than red wines (Marsh and Guymon, 1959).

During conventional chill-proofing, precipitation is usually rapid during the first 12 days, and then the rate of KHT precipitation diminishes considerably. This reduction is due to a decreased level of KHT saturation in solution. Temperature fluctuations during cold stabilization can have a significant effect in reducing the rate of KHT precipitation due to changes in the speed of nuclei formation. Without crystal nuclei formation, crystal growth and subsequent precipitation cannot occur.

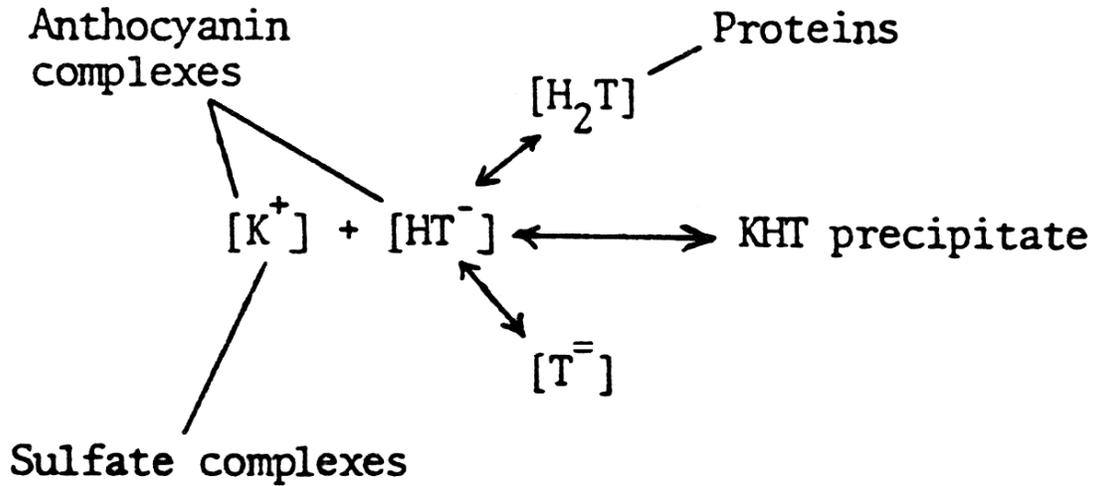
Simply opening the cellar doors in the winter, although cost effective, may not provide optimal temperatures for KHT precipitation or product palatability in certain wines. The increased absorption of oxygen into the wine at low temperatures over longer periods, and the subsequent oxidation of wine components may make alternatives to conventional cold stabilization desirable.

Complexing Factors and Tartrate Precipitation

"Complexing factors" can greatly affect potassium bitartrate formation and precipitation (see Figure 2). It is thought that wine is able to support a supersaturated solution of KHT because a percentage of the tartrate and potassium ions are complexed with wine constituents and, therefore, not precipitated. Metals, sulfates, proteins, gums, polyphenols, etc., can form complexes with free tartaric acid and potassium ions, thus inhibiting the formation of KHT (Pilone et al., 1965).

These complexes formed are mainly between polyphenols and tartaric acid in red wines, and proteins and tartaric acid in whites. The binding of free tartaric acid prevents potassium bitartrate formation. In a study of white Bordeaux wines, Peynaud et al. (1964) found sulfate to be the most important factor in stability, besides free potassium or tartrate. This would appear to be due to the complexing ability of sulfate with potassium (Chlebek and Lister, 1966).

Figure 2. Potassium Bitartrate Equilibria and the Interaction of "Complexing Factors"



Almost one-half of the sulfate in white wines and 100% of the sulfate in red wines is hypothesized to complex free potassium as K_2SO_4 or KSO_4^- . The differences in the extent of tartrate complexes formed and their "holding power" to prevent precipitation depends upon the wine in question.

Pigments of red wines are often involved in complexes with tartaric acid (Balakian and Berg, 1968). As wine oxidizes and pigment polymerization occurs, the holding capacity for tartaric acid diminishes, often resulting in delayed precipitation of potassium bitartrate. Additionally, cultivar, growing region and season may affect potassium bitartrate holding capacity (Boulton, 1980). Each wine, because of its unique composition, will achieve an equilibrium of KHT solubility under imposed temperature conditions.

Pilone and Berg, (1965) and Balakian and Berg, (1968) suggested the importance of colloids in potassium bitartrate stability. Pectins and other polysaccharides, such as glucanes from *Botrytis cinerea*, inhibit crystallization. This is the result of these compounds adsorbing onto the surface of the crystal and preventing further crystal growth.

Occasionally, winemakers choose to add complexing agents or inhibitors including yeast mannoproteins, metatartaric acid, carboxymethylcellulose, and gums to help prevent KHT formation.

Addition Compounds for Potassium Bitartrate Stabilization

Occasionally, winemakers choose to add complexing factors or inhibitors to prevent potassium bitartrate formation. In theory, the right inhibitor in the proper concentrations could reduce the need for traditional cold stabilization, contact seeding, or ion exchange by preventing the formation of potassium bitartrate crystals.

Certain colloidal compounds found naturally in wine and some addition products inhibit the precipitation of KHT. These colloids are absorbed on the surfaces of crystal nuclei, inhibiting crystal growth. Suitable inhibitors include carbonate mixtures, yeast mannoproteins (Mannostab®), metatartaric acid, sodium carboxymethylcellulose, tannins, pectins and gum. Generally, the mechanism of inhibition is the same for each of these, an interference in the formation and growth of potassium bitartrate crystals. Addition products that are not natural constituents of wine add to what critics refer to as “industrial winemaking”. This speaks to the subject of authenticity and *terrior*. For a discussion on this subject, see section title Great Wines.

Koldone is a proprietary product used to help obtain potassium bitartrate stability. It is produced by mixing calcium carbonate and L-(+)-tartaric acid in a specific ratio. The addition of Koldone into a wine causes the added calcium carbonate to react with the wine's tartaric acid to form an insoluble calcium tartrate. Precipitation is aided by calcium tartrate crystals present in Koldone, which act to seed the wine. Thus, the addition of Koldone to a wine causes it to become supersaturated with respect to tartrate, and this is precipitated rapidly from solution. The tartaric acid concentration can be reduced to a low-enough level where cold stability is achieved (Clark et al. 1985). Several other proprietary compounds designed to aid potassium bitartrate stability are available

Mannostab (a proprietary mannoprotein) is made from yeast cell walls. It is naturally present in wine and inhibits tartrate crystal formation by preventing the growth of certain

crystal faces, so the crystal only grows in a certain orientation, preventing it from precipitating. Filtration at bottling, if done too tightly, can retain colloids and Mannostab and render the treatment useless.

Metatartaric acid. Perhaps the best known inhibitor of potassium bitartrate crystal formation is metatartaric acid, the hemipolyactide of tartaric acid. This is an approved wine additive in certain countries. Metatartaric acid is produced by heating tartaric acid at 170°C for about 120 hours. It has been reported that 50 to 100 mg/L protects young wines from potassium bitartrate precipitation, even when they are stored at low temperatures for several months (Peynaud and Guimberteau, 1961). Cold water should be used for dissolving the metatartaric acid, to avoid its hydrolysis (Peynaud, 1984).

Inhibition occurs due to the coating by metatartaric acid of the crystalline tartrate nuclei, which prevents their growth (Peynaud 1984). After being added to wine, metatartaric acid is slowly hydrolyzed to tartaric acid, with a corresponding loss of activity. Its period of effectiveness is a function of the storage temperature of the treated wine. Peynaud (1984) found that wines stored at 0°C were inhibited from KHT precipitation for several years, but that metatartaric acid disappeared after two months in wines stored at 25°C. Therefore, metatartaric acid would have its greatest use in wines that are to be consumed rather quickly.

Carboxymethyl cellulose. Carboxymethyl cellulose (CMC) is another inhibitor of potassium bitartrate precipitation (Cantarelli 1963). CMC has been a permissible food additive for some time and has been authorized for use in wine by the International Organization of Vine and Wine (OIV). It is used in the food industry as a modifier and emulsion stabilizer. CMC is basically cellulose polymers with various side changes, resulting in a negatively charged particle. CMC products differ in polymer length and substitutions. Those generally used widely in the food industry are not suitable for wines.

- CMC may be an alternative for cold stabilization for potassium bitartrate (KHT) stabilization. CMC functions by physically limiting nucleation and crystal growth, impacting the crystal shape and thus slowing down crystal formation. There is no change in wine pH, tartaric acid (TA), or sensory characteristics, unlike cold stabilization.

CMC products for the wine industry have the following attributes and limitations:

- Overall economics: By avoiding cold stabilization there is a energy savings. For example, sources of energy consumption in the winery include those listed below. Lowering the energy required for cold stabilization may be an important sustainability issue.
 - refrigeration: 40-60%
 - pumps, fans, drives: 10-35%
 - lighting: 8-20%
 - compressed air: 3-10%
 - packaging and bottling: 8-30%
 - other consumptions: 3-15%
- Ease of use: CMC is relatively straightforward to use

CMC does have some important limitations:

- Limited duration: CMC competes with potassium and bitartrate ions preventing them from attaching to the crystal face. It should be noted that many of our wines in VA have a higher K (potassium) levels than the wines reported in the popular trade journal articles. Added complexing factors lowers the effectiveness of this or any product designed to limited nucleation and crystal growth.
- Shelf life: CMC products for wine have a limited shelf life.
- Wine must be ready to bottle before using CMC: Clarity/filterability (nephelometric turbidity units NTU) and stability occur prior to addition. Following addition, no modifications such as blending, acid adjustments, etc can be made.
- Mainly limited to white wines: CMC are not recommended for use in red wines.

- CMC can interact with proteins: A wine to be treated with CMC for KHT
- stabilization must be relatively protein-free. This includes lysozyme.

Tannin. Additionally, purified apple pectin and tannin can inhibit crystal formation. One gram/L of tannin is said to strongly inhibit precipitation of tartrates (Wucherpfennig and Ratzka, 1967).

The short term effectiveness of addition products can be assessed by destabilization of a treated wine with dissolved KHT. By then measuring the change in electrical conductivity we can tell you if the wine is stable.

Cold Stabilization and Complexing Factors

Rather than adding inhibitors, it is common to attempt to remove or reduce the complexing factors, so that potassium bitartrate precipitation and stabilization can occur prior to bottling. Consequently, there is an intimate relationship between wine fining and potassium bitartrate stabilization. For example, it is known that condensed polyphenols interfere with tartrate precipitation (Amerine and Joslyn, 1970). This suggests that removal of a portion of these polyphenols with certain fining agents prior to cold stabilization could enhance potassium bitartrate precipitation (Zoecklein 1988).

Cold stabilization procedures (chill-proofing) cause precipitation of potassium bitartrate crystals and proteins. The proteins of white wines can exhibit a holding capacity for tartaric acid, thus inhibiting KHT formation (Pilone and Berg, 1965). White wines contain relatively large insoluble proteins. As the phenols of white wines oxidize, they polymerize, and soon bind to and co-precipitate with proteins. This precipitation affects the tartrate holding capacity.

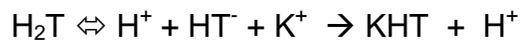
- Bentonite fining may decrease the tartrate holding capacity by reducing both proteins and, secondarily, phenolics (Berg and Akiyoshi 1971).
Addition of 30 g/hL (2.5 pounds per 1,000 gallons) or more of bentonite

has been found to reduce concentration product (CP) values of dry white wines from 15 to 18% and CP values in dry red wines from 25 to 32% (Berg et al. 1968). (see Concentration Product, below).

- Tartrate precipitation may impact wine pH and, secondarily, protein stability. In wines below pH 3.65, chill-proofing causes a downward shift in pH that may enhance protein precipitation.
- Some winemakers elect to bentonite fine during bitartrate stabilization which allows KHT crystals to help compact bentonite lees.
- Reducing sugars are also known to impact tartrate stability. Berg (1960) reported 20% higher CP values in sweet sherries (6°B) when compared with sherries at 1° B. Further, final tartrate deposition took 23% longer in the higher sugar group.

BITARTRATE STABILIZATION AND CHANGES IN TITRATABLE ACIDITY AND pH

Wines with initial pH values below 3.65 show reductions in pH and titratable acidity (TA) during cold stabilization because of the generation of one free proton per molecule of KHT precipitated:



The pH may drop by as much as 0.2 pH units with a corresponding decrease in TA of up to 2 g/L.

By comparison, KHT precipitation in wines with pH values above 3.65 results in higher pH levels and corresponding decreases in TA. This is the result of removal of one proton per tartrate anion precipitated:



- If acidulation is desired, it is best to do so before beginning cold stabilization.

Determining Potassium Bitartrate Stability

There is significant variation in cellar activities employed to reach stability. Additionally, there is significant variation in the methods by which KHT stability is measured and defined.

Table 1 shows the multitude of procedures used to determine cold stability by 18 surveyed wineries. The fact that there is no industry standard for determination of cold stability is readily apparent from the survey. This is not surprising, since stability is a relative term that can, and is, defined differently by different producers.

Table 1. Cold Stability Tests Performed by 18 Different Wineries

Winery	Test
A	Filter sample. 0.65 μm membrane. Seed sample with KHT. 36 h at 25°F with mixing, analyze for tartrate.
B	30 days at 30°F and CP.
C	14 days at 32-34°F. Examine cold at 7 and 14 days.
D	Not done – wines stable due to prolonged aging in cool cellar.
E	Filter thru 0.45 μm membrane and test 2 samples: A. 12 h frozen. 12 h at RT. Examine daily. B. 72 h at 38-42°F. 12 h at RT. Examine daily.
F	CP
G	96 h at 18°F. Examine. 24 h at RT. Examine. Deposits formed at 18°F should redissolve in 24 h at RT.
H	Filter sample thru 0.45 μm membrane. A. 16 h at 10°F. Thaw at RT. Examine. B. 16 h at 15°F. Thaw at RT. Examine.
J	3-4 wk at 35°F. Examine cold and RT.
K	Wine sample is pad filtered and membrane filtered if needed. A. One aliquot – 48 h at 26°F. A 2nd aliquot – 48 h at 100°F, and both examined. B. Both aliquots of A then 48 h at 26°F and examined. C. Both of B then 48 h at RT and examined.
L	2 wk at 40°F. Examine.

M	Filter. 24 h at 25°F. Examine.
N	72-96 h at 40°F. Examine or freeze 12 h. Thaw, examine at RT.
P	Filter, 0.65 µm membrane. Freeze 24 h. Thaw, examine at RT.
Q	4 h at 12°F. Examine.
R	2 wk at 34°F. Examine cold.
S	Not done.
T	12 h freeze and/or 72 h at 28°F. Examine cold and at RT. Stability based upon amount of deposit.

Source: Cooke and Berg (1984). RT = room temperature.

Two traditional methods of determining potassium bitartrate stability are the determination of CPs (Concentration Product values) and the freeze or slush test.

The CP Test

The relationship between tartaric acid and potassium ions can be expressed quantitatively by using the Concentration Product (CP):

$$CP = (\text{moles/L K}^+) (\text{moles/L of total tartrates}) (\%HT^-)$$

Berg and Keefer (1958), using model water/alcohol solutions, were able to establish solubility levels for potassium bitartrate and, as a result, to calculate solubility product values. Where calculated “CP values” exceed the published values considered “safe” for the wine type under consideration, the wine may deposit tartrates. Conversely, if the calculated values are less than the identified safe levels, the wine is considered by some to be stable with respect to bitartrate precipitation.

Therefore, determination of CPs may be a useful guide to the winemaker in estimating the probability of bitartrate instabilities. Any determination of CP values requires preliminary analysis of alcohol, pH, potassium ion, and total

tartaric acid. CP tables are available in most wine texts, including Zoecklein et al. (1995).

The concentration product procedure does not allow for the complexing action of certain wine constituents that prevent crystal formation. This may be a particular problem for some wines.

Freeze Test

Another traditional method of measuring potassium bitartrate (KHT) stability is the freeze test. This procedure relies on the formation of crystals as the result of holding filtered wine samples at reduced temperatures for a specified time period. As indicated in Table 1, a wide variety of procedures is employed for this analysis. Often, one of the samples is frozen, and then thawed, to determine the development of bitartrate crystals and if those crystals return to solution. The absence of crystal formation is meant to indicate a potassium bitartrate-stable wine.

The freeze test is, in reality, a distorted crystallization-rate test (Boulton, 1983). As ice formation increases, there is an increase in the relative concentration of all species in the sample, including alcohol, thus enhancing nucleation and crystallization. It is difficult to accurately relate crystal formation in this concentrated wine sample with bitartrate potassium instability.

As such, the freeze test is essentially a crystal-rate test, rather than an actual stability test. Filtration of the laboratory sample will remove crystal nuclei, which will affect test results. Unless one provides seed crystals (or doesn't lab-filter the test sample), precipitation over the short time period of the test is a measure of the ability to form nuclei and precipitate.

Two wine samples may be equally unstable. However, upon filtration and storage at low temperature, one precipitates tartrate and is considered unstable, whereas

the second does not form nucleation sites at the same rate and is, by way of this test, considered stable. As stated, the presence of crystals depends upon the rate of crystallization and time. The relationship between the results of a freeze test and long-term bottle stability is, therefore, vague.

A more accurate determination of potassium bitartrate stability can be determined by seeding with potassium bitartrate. The oversaturated portion of tartaric acid and potassium is deposited onto the added crystals. There is a reduction in the tartaric acid, potassium, titratable acidity, and electrical conductivity. These changes can be measured to determine potassium bitartrate (KHT) stability.

Conductivity Testing

Dunsford and Boulton (1979) measured the electrical conductivity change in a KHT-seeded wine by the disappearance of free potassium ions (K^+) from solution. The change in electrical potential (conductivity) was measured with a standard conductivity meter. This "conductivity" test provides a final stable conductivity value that is specific for the wine being tested.

Complexing, or "fouling," factors that may be present, and possibly affect potassium bitartrate crystal formation and growth, are taken into account. The temperature at which the test is performed should be the lowest temperature which the wine is expected to encounter after bottling. During this test, conditions are created for rapid crystal growth if any supersaturation of KHT exists.

An electrical conductivity meter capable of measuring in the range of 100-1000 micro-siemens is required for this analysis. A siemens unit (formerly known as a mho) of electrical conductance is the reciprocal of the resistance in ohms. Models are also available which are combination conductivity, pH, and mV meters.

The Test Procedure. The test procedure outlined by Boulton (1983) is 100 ml of wine chilled to the desired test temperature, mixed, and the conductivity measured. One gram of powdered KHT is added, and the conductivity is noted at one- and two-minute intervals while constantly mixing. The measurement of conductivity is continued until the conductivity reading becomes stable. This, according to Boulton, usually takes less than 20-30 minutes.

The conductivity value is affected by temperature; therefore, the sample temperature must be constant throughout the test. If the temperature rises, the conductivity reading will increase, simply due to temperature effects.

Stability Criterion. The final conductivity value corresponds to that of the stable wine. It can be used for a comparison with samples taken from the winery during the full-scale stabilization treatment to test when stability has been reached.

The difference between the conductivity value before the powdered KHT was added and the final value is a measure of potential KHT instability. If this difference is less than 5% of the initial value, the original wine is considered to be stable. If it is greater than 5% of the initial value, the original wine is unstable and can be treated in the cellar by seeding. Some consider a dry red wine with a conductivity value below 1800 micro-siemens/cm, or a dry white wine below 1400-1600, to be stable.



POTASSIUM BITARTRATE STABILIZATION OF WINES

Section 2.

Cellar Considerations for Contact Seeding

Cooke and Berg (1984) reported that approximately half of the wineries they surveyed attempted potassium bitartrate stability by seeding with potassium bitartrate. The most important factors affecting potassium bitartrate stabilization by seeding are the following:

- quantity of KHT and crystal size
- agitation
- time and temperature

Quantity of KHT and Crystal Size

The volume of KHT required will depend somewhat on the wine in question, due to the variation in complexing factors. The amount of KHT added for seeding must always constitute an overload to create a supersaturated solution. Table 2 shows the relationship between the quantity of KHT added as seed and several wine components. Tartaric acid, potassium, and the concentration product (CP) value diminish with increased levels of added KHT. This diminution is clear at 4 g/L, then slows with more elevated quantities of KHT (Gumberteau et al., 1981). The range of efficiency has been determined to be between 30 and 150 μm

(Gumberteau et al. 1981). Rhein and Neradt (1979) suggest 4 g/L of 40 µm KHT powder as the optimal quantity and particle size.

Table 2. Influence of Quantity of Potassium Bitartrate (40 µm Size) Added at 0°C with Constant Agitation

Assay Conditions	Tartaric Acid (g/L)	Potassium (mg/L)	C.P. x 10 ⁵
Control	1.58	920	15.1
+ 1 g/L	1.11	808	9.3
+ 2 g/L	1.03	794	8.5
+ 4 g/L	0.93	765	7.6
+ 8 g/L	0.78	754	6.2

Source: Blouin et al. (1982)

Agitation

In the cellar, seeding stabilization should be conducted in a small tank (no more than 2000 gallons) where the stabilization temperature and mixing can be controlled. Crystal growth is dependent upon available surface area and, for this reason, constant agitation is essential. Effective seeding is based upon intense contact of the wine with the powdered KHT added. Table 3 shows the relationship between wine constituents and agitation during the seeding process. The potassium, tartaric acid, and CP values are lower in an agitated than a static environment.

Table 3. Influence of Agitation on White Wine Constituents During Contact Seeding at 0°C with 4 g/L KHT

Assay Conditions	Tartaric Acid (g/L)	Potassium (mg/L)	C.P. x 10 ⁵
Control	1.58	920	15.1
With agitation	1.17	805	9.8
Without agitation	1.38	870	12.5

Source: Blouin et al. (1982). Control wine is without seed addition.

Time and Temperature

When tartrate seeding is employed, the addition of a large surface area of powdered KHT eliminates the natural energy-consuming nuclei-induction phase and allows for immediate crystal growth. This procedure can be carried out at any temperature. When applying the seeding process, the treatment temperature is identical to the desired stability temperature. For example, many wine producers seed their white wines at 0°C and red wines at +5°C. Thus, if the stabilization procedure is correctly performed, wines held at or above these temperatures should remain stable with respect to potassium bitartrate precipitation.

During the first hour of contact seeding, there is a rapid reduction in tartaric acid, potassium, and the concentration product value. This reduction slows after the first hour, then levels off for most wines at the end of three hours (Blouin et al., 1982). For security, it is desirable to have the duration of contact be a minimum of 4 hours (Gumberteau et al., 1981). It has been suggested that using 40-µm KHT would allow stabilization in 90 minutes (Neradt 1979). Reduction of the seeding quantity can prolong the necessary stabilization period.

Filtration of the wine following contact seeding is essential. This should be performed at the seeding temperature to avoid resolubilization of potassium bitartrate crystals back into solution.

Table 4 shows a comparison of several wine parameters as affected by potassium bitartrate stabilization methods. Gilbert (1976) found the sugar-free extract in KHT-precipitated wines decreased 0.8 g/L, and total acid decreased 0.5 g/L.

Table 4. Sparkling Wine Cuvée Parameters Prior to and After Potassium Bitartrate Stabilization by Several Methods

Stabilization Method	ETOH (% vol)	Sugar-free Extract (g/L)	pH	Total Acid (g/L)	Tartaric Acid(g/L)	K ⁺ (mg/L)
Untreated	9.37	21.26	3.31	7.55	2.50	720
Contact	9.46	20.34	3.20	7.15	1.95	565
Chilling	9.43	21.74	3.23	7.25	2.50	715
Ion Exchange	9.35	20.74	3.26	7.45	2.40	360

Source: Neradt (1979)

Ribéreau-Gayon and Sudraud (1981) reported a comparison of potassium bitartrate stabilization techniques using 16 white wines and 11 reds. Each wine was pre-filtered, and potassium bitartrate stabilization was performed by conventional cold stabilization at -4°C for 14 and 21 days. The results were compared with the contact procedure. Eighty percent of the wines tested showed concentration product values lower for the wines treated by contact seeding. In all tests, contact seeding produced results at least equivalent to conventional cold stabilization.

KHT crystals can be reused after removal from the treated wine. After the crystals have been used repeatedly, they grow in size, decreasing the number of active nuclei and decreasing the effectiveness of the crystallization phase (Neradt 1979). Wet grinding of the crystals must be eventually done for optimal performance.

During potassium bitartrate formation, 1.0 g/L of tartaric acid combines with 0.26 g/L potassium ions (K^+) to form 1.26 g/L potassium bitartrate (KHT). By comparing tartaric acid levels before and after seeding, the wine may be regarded as stable if the difference in tartaric acid at the stabilizing temperature is 200 mg/L or less (SWK Machines Technical File, 1978).

Comparing potassium levels before and after seeding can be used to measure stability. The wine under question may be considered stable if the difference in potassium levels before and after seeding with KHT is 40 mg/L or less (SWK Machines Technical File, 1978).

Comparing the change in titratable acidity before and after seeding can be a gauge for the determination of potassium bitartrate stability. In this case, the wine can be considered stable if the difference in titratable acidity levels before and after seeding is not greater than 100 mg/L.

Ion Exchange: Although not regarded by many contemporary winemakers as a desirable practice in premium wine production, ion exchange has been successfully used to bring about KHT stabilization. With ion exchange, the cation concentration of a wine can be reduced by exchanging it with either H^+ (hydrogen cycle) or Na^+ (sodium cycle). Reduction in the concentration of K^+ directly results in a decrease in the solubility coefficient and hence decreases the likelihood of salt precipitation at low temperatures. Unlike chill-proofing or seeding, the concentration of tartaric acid (tartrates) does not change. Hence,

there are not the changes in TA and pH that may accompany stability by storage of the wine at low temperatures.

Refrigeration represents the most significant cost in winery operations; ranging from 40-60% of the budget depending upon time of the year. When coupled with the observation that even the most “cold-stable” wines may still throw precipitates, there is a good deal of interest in identifying methods for reducing costs.

Electrodialysis . Electrodialysis (ED) is a relatively new technology originally developed by the French National Agronomic Research Institute (INRA). An electromembrane process in which ions (K^+ , Ca^{++} , HT^-) are selectively transported across ion-permeable membranes from one solution (wine/juice) to another (brine) under the influence of a potential gradient. Multiple electrodialysis cells are arranged with alternating anion and cation exchange membranes forming electrodialysis cells or a stack. Electrodialysis can be used to remove potassium and calcium cations and tartrate anions from wine. An electric potential applied to an electrodes leads to the migration of the ions in solution across a membrane. The potassium and calcium cations migrate toward the cathode and the tartrate anions toward the anode. The membranes consist of a filter press module comprising a large number of parallel mounted cells (cationic membranes, spacers and anionic membranes. The even compartments receive the wine and the odd compartments the brine. The two fluids never come into contact and the membrane allows passage of ions between them. The cationic membranes are alternated with the anionic membranes and a small electric field is created. Potassium and calcium ions (cations) and tartrate ions (an anion) are extracted by the brine and thus prevent from forming crystals in the wine.

Benefits of ED include the following:

- Significantly lower energy usage/smaller carbon footprint: At the current relatively low cost of electricity in some regions this may not be incentive enough, but how long will costs stay so low?
- Reduced wine losses: Typical volume savings relative to conventional cold stabilization have been estimated at 0.5-1.5%.

- Just-in-time production: No waiting for chilling, holding and warming.
- EC can also be used to lower the calcium and chloride concentrations.
- Better pH control with less tartaric acid addition.
- Improved mouthfeel and flavor retention compared to traditionally cold-stabilized wines as a result of wine colloids not being removed.

There are some problems with ED including:

- Water usage: Typically the process uses water at a rate of about 10-15% of the volume of wine treated.
- Waste disposal: The tartrate removed in the ED waste stream has to go somewhere, as does the tartrate washed away in lees and tank washings.

Practical Summary of Winemaking Issues

- The most common tartaric stabilization technique is cold stabilization. Wine is cooled above its freezing point when crystals of tartrate form and settle. After a stability check the wine is then filtered. In a variation of cold stabilization, potassium hydrogen tartrate (cream of tartar) is added to the wine during cooling to seed crystal formation.
- Critics say these techniques are expensive and time-consuming. They require temperature changes and a good knowledge of the instability of the wine to treat. Most importantly, they are not completely reliable as crystals can still precipitate after treatment during storage and transportation. In addition, careful clarification is usually required before cooling and an additional step is needed to separate the crystals.
- Depending on the production methods, the amount of energy used to obtain cold stability may be substantial. Since the development of suitable ion exchange membranes electro dialysis has become a viable option along with newly formulated addition products such as CMC. Unlike traditional cold stabilization, these treatments have no impact on the concentrations of polyphenols, color, polysaccharides, amino acids and volatile compounds.



Study Questions

1. What is the relationship between cold stability and alcohol?
2. If two bitartrate stable wines are blended the resultant wine may not be stable-why?
3. The addition of cane sugar to a sweet wine may result in bitartrate instability-why?
4. Why is stability considered a relative term?
5. Contact seeding involves the same principle that we learned in grade school while making rock candy. What are the parameters to consider in contact seeding?
6. What is/are the relationships between protein stability and bitartrate stability?
7. CMC is now an important addition compound to help prevent bitartrate stabilization. Contrast the advantages and disadvantages of the use of this compound vs. electrical dialysis and conventional cold stabilization.

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