OIV-MA-AS313-21 Determination of the presence of metatartaric acid

Type IV method

1. Introduction

Metatartaric acid added to the wine to avoid tartaric precipitation is traditionally proportioned by the difference between the total tartaric acid following hot hydrolysis of metatartaric acid and natural tartaric acid preceding hydrolysis. However, taking into account the precision of the determination of tartaric acid, traces of metatartaric acid are not detectable by this method, and the additive, which is not accepted in certain countries, must therefore be characterised using a more specific method.

2. Scope

Wines likely to contain traces of metatartaric acid.

3. Principle

In relatively acid mediums, metatartaric acid forms an insoluble precipitate with cadmium acetate; it is the only one of all the elements present in must and wine to give such a precipitate .

Note: Tartaric acid is also precipitated with cadmium acetate, but only in the presence of an alcohol content greater than 25% vol. The precipitate redissolves in water, unlike the precipitate obtained with metatartaric acid.

The cadmium precipitate of metatartaric acid breaks down by heating with sodium hydroxide and releases tartaric acid. The latter produces a specific orange colour with ammonium metavanadate.

4. Reagents

4.1. Cadmium acetate solution at 5 p.100

4.1.1. Dihydrated cadmium acetate at 98%

4.1.2. Pure acetic acid

4.1.3. Distilled or demineralized water

4.1.4. Cadmium acetate solution: dissolve 5 g of cadmium acetate (4.1.1) in 99 mL of water (4.1.3) add 1 mL of pure acetic acid (4.1.2)

4.2. Sodium hydroxide 1M

4.3. Sulfuric acid 1M

4.4. Solution of ammonium metavanadate 2% w/v

4.4.1. Ammonium metavanadate

4.4.2. Trihydrated sodium acetate at 99%

4.4.3. Sodium acetate solution at 27 p. 100: dissolve 478 g of sodium acetate (4.4.2) in 1 liter of water (4.1.3)

4.4.4. Solution of ammonium metavanadate: dissolve 10 g of ammonium metavanadate (4.4.1) in 150 mL of sodium hydroxide 1 M (4.2) add 200 of the sodium acetate solution at 27 p. 100 (4.4.3) and fill to 500 mL with water (4.1.3)

4.4.5. Ethanol at 96% vol.

5. Apparatus

5.1. Centrifuge with a rotor capable of housing 50-mL bottles

5.2. Spectrometer capable of operating in the visible spectrum and of housing cuvets with an optical thickness of 1 cm.

6. Operating method

6.1. Centrifuge 50 mL of wine for 10 minutes at 11000 rpm

6.2. Take 40 mL of limpid wine using a test-tube and place the sample in a centrifuge flask

6.3. Add 5 mL of ethanol at 96% vol (4.5)

6.4. Add 5 mL of the cadmium acetate solution (4.1.4)

6.5. Mix and leave to rest for 10 minutes

6.6. Centrifuge for 10 minutes at 11000 rpm

6.7. Decant by completely reversing the flask (once) and throw away the supernatant.

In the presence of metatartaric acid, a lamellate precipitate is formed at the bottom of the tube.

In the absence of any precipitate, the sample will be regarded as free from metatartaric acid. In the contrary case, or if the presence of a light precipitate is to be established with certainty, proceed as follows:

6.8. Wash the precipitate once with 10 mL of water (4.1.3) in the form of an energetic jet towards the bottom of the tube in order to detach the precipitate from the bottom

6.9. Add 2 mL of cadmium acetate solution (4.1.4)

6.10. Centrifuge at 11000 rpm for 10 minutes then throw away the supernatant by completely reversing the tube (once)

6.11. After adding one mL of sodium hydroxide 1M (4.2), plunge the tube to be centrifuged for 5 minutes in a water bath at 100° C

6.12. After cooling, add 1 mL of sulfuric acid 1M (4.3) and 1 mL of ammonium metavanadate solution (4.4.4)

6.13. Wait 15 minutes

6.14. Centrifuge for 10 minutes at 11000 rpm

6.15. Pour the supernatant into a spectrophotometer tank and measure the absorbance at 530 nm, after determining the zero point with water (4.1.3)

$i.e\ Abs_E$

Standard. In parallel, produce a standard comprising the same wine as that analyzed but heated beforehand for 2.5 minutes using a microwave generator set to maximum power or with a water bath at 100° C for 5 minutes.

$i.e.\ Abs_T$

7. Calculation

The presence of metatartaric acid in the wine is established when, at 530 nm:

$$Abs_E - Abs_T > 0.050$$