Method OIV-MA-AS313-05A   Type IV method

Tartaric acid

1. Principle

Tartaric acid is precipitated in the form of calcium (±)tartrate and determined gravimetrically. This determination may be completed using a volumetric procedure for comparison. The conditions for precipitation (pH, total volume used, concentrations of precipitating ions) are such that precipitation of the calcium (±)tartrate is complete whereas the calcium D(–) tartrate remains in solution.

When *meta*-tartaric acid has been added to the wine, which causes the precipitation of the calcium (±)tartrate to be incomplete, it must first be hydrolyzed.

2. Method

2.1. *Gravimetric method*

2.1.1. Reagents

- Calcium acetate solution containing 10 g of calcium per liter:

Calcium carbonate, $CaCO_3$	25 g
Acetic acid, glacial, CH_3COOH (ρ_{20}= 1.05 g/mL)	40 mL
Water to	1000 mL

- Calcium (±)tartrate, crystallized: $CaC_4O_6H_4 \cdot 4H_2O$.

  Place 20 mL of L(+) tartaric acid solution, 5 g/L, into a 400 mL beaker.

  Add 20 mL of ammonium D(–) tartrate solution, 6.126 g/L, and 6 mL of calcium acetate solution containing 10 g of calcium per liter.

  Allow to stand for two hours to precipitate. Collect the precipitate in a sintered glass crucible of porosity No 4, and wash it three times with about 30 mL of distilled water. Dry to constant weight in the oven at 70°C. Using the quantities of reagent indicated above, about 340 mg of crystallized calcium (±) tartrate is obtained. Store in a stoppered bottle.

- Precipitation solution (pH 4.75):

D(–) ammonium tartrate ....….	150 mg
Calcium acetate solution, 10 g calcium/L	8.8 mL
Water to ....…......…....…	1000 mL

  Dissolve the D(-) ammonium tartrate in 900 mL water; add 8.8 mL calcium acetate solution and make up to 1000 mL. Since calcium (±)tartrate is

slightly soluble in this solution, add 5 mg of calcium (±)tartrate per liter, stir for 12 hours and filter.

Note: The precipitation solution may also be prepared from D(-) tartaric acid.

D(–) tartaric acid ....……………. 122 mg
Ammonium hydroxide solution (ρ_{20}= 0.97 g/mL), 25 % (*v/v*) ..... 0.3 mL

Dissolve the D(–) tartaric acid, add the ammonium hydroxide solution and make up to about 900 mL; add 8.8 mL of calcium acetate solution, make up to a liter and adjust the pH to 4.75 with acetic acid. Since calcium (±)tartrate is slightly soluble in this solution, add 5 mg of calcium (±)tartrate per liter, stir for 12 hours and filter.

2.1.2. Procedure

— Wines with no added *meta*-tartaric acid

Place 500 mL of precipitation solution and 10 mL of wine into a 600 mL beaker. Mix and initiate precipitation by rubbing the sides of the vessel with the tip of a glass rod. Leave to precipitate for 12 hours (overnight).

Filter the liquid and precipitate through a weighed sintered glass crucible of porosity No. 4 fitted on a clean vacuum flask. Rinse the vessel in which precipitation took place with the filtrate to ensure that all precipitate is transferred.

Dry to constant weight in an oven at 70°C. Weigh. Let *p* be the weight of crystallized calcium (±)tartrate, $CaC_4O_6H_4 \cdot 4H_2O$, obtained.

— Wines to which *meta*-tartaric acid has been added.

When analyzing wines to which *meta*-tartaric acid has been or is suspected of having been added, proceed by first hydrolyzing this acid as follows:

Place 10 mL of wine and 0.4 mL of glacial acetic acid, CH_3COOH, (ρ_{20}= 1.05 g/mL) into a 50 mL conical flask. Place a reflux condenser on top of the flask and boil for 30 min. Allow to cool and then transfer the solution in the conical flask to a 600 mL beaker. Rinse the flask twice using 5 mL of water each time and then continue as described above.

Meta-Tartaric acid is calculated and included as tartaric acid in the final result.

2.1.3. Expression of results

One molecule of calcium (±)tartrate corresponds to half a molecule of L(+) tartaric acid in the wine.

- The quantity of tartaric acid per liter of wine, expressed in milliequivalents, is equal to:

$$384.5 \, p.$$

It is quoted to one decimal place.

- The quantity of tartaric acid per liter of wine, expressed in grams of tartaric acid, is equal to:

$$28.84\ p.$$

It is quoted to one decimal place.

- The quantity of tartaric acid per liter of wine, expressed in grams of potassium tartrate, is equal to:

$$36.15\ p.$$

It is quoted to one decimal place.

2.2. *Comparative volumetric analysis*

2.2.1. Reagents

- Hydrochloric acid (ρ_{20} = 1.18 to 1.19 g/mL) diluted 1:5 with distilled water
- EDTA solution, 0.05 M:

    EDTA (ethylenediaminetetraacetic acid disodium salt)........ 18.61 g
    Water to .... 1000 mL

- Sodium hydroxide solution, 40% (*m/v*):

    Sodium hydroxide, NaOH .... 40 g
    Water to .... 100 mL

- Complexometric indicator: 1% (*m/m*)

  2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthylazo)

    -3-naphthoic acid .... 1 g
    Sodium sulfate, Na_2SO_4 (anhydrous) .... 100 g

2.2.2. Procedure

After weighing, replace the sintered glass crucible containing the precipitate of calcium (±)tartrate on the vacuum flask and dissolve the precipitate with 10 mL of dilute hydrochloric acid. Wash the sintered glass crucible with 50 mL of distilled water.

Add 5 mL 40% sodium hydroxide solution and about 30 mg of indicator. Titrate with EDTA solution, 0.05 M. Let the number of mL used be *n*.

2.2.3. Expression of results

- The quantity of tartaric acid per liter of wine, expressed in milliequivalents, is equal to:

$$5\ n.$$

It is quoted to one decimal place.

- The quantity of tartaric acid per liter of wine, expressed in grams of tartaric acid, is equal to:

$$0.375 \, n.$$

It is quoted to one decimal place.

- The quantity of tartaric acid per liter of wine, expressed in grams of potassium acid tartrate, is equal to:

$$0.470 \, n.$$

It is quoted to one decimal place.

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