OIV-MA-AS313-05A Tartaric acid

Type IV method

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 (±) 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 ($□_{□□}$= 1.05 g/mL)	40 mL
Water to	1000 mL

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

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 (□□□= 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 □ be the weight of crystallized calcium (±)tartrate, $CaC_4O_6H_4\ 4H_2O$, 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, (□□□= 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 (□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

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: $5n$.

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: $0.375n$.

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: $0.470n$.

It is quoted to one decimal place.

Bibliography

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