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 (ρ_{20} = 1.05 g/mL)	40 mL
Water to	1000 mL

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

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 (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 □ 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, (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 (□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: 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.

Bibliography

- KLING A., *Bull. Soc. Chim.*, 1910, 7, 567.
- KLING A., FLORENTIN D., *Ibid*, 1912, 11, 886.
- SEMICHON L., FLANZY M., *Ann. Fals. Fraudes*, 1933, 26, 404.
- PEYNAUD E., *Ibid*, 1936, 29, 260.
- PATO M., *Bull. O.I.V.*, 1944, 17, no, 161, 59, no, 162, 64.
- POUX C., *Ann. Fals. Fraudes*, 1949, 42, 439.
- PEYNAUD E., *Bull. Soc. Chim. Biol.*, 1951, 18, 911; *Ref. Z. Lebensmit. Forsch.* , 1953, 97, 142.
- JAULMES P., BRUN Mme S., VASSAL Mlle M., *Trav. Soc, Pharm.*, Montpellier, 1961, 21, 46-51.
- JAULMES P., BRUN Mme S., CABANIS J.C., *Bull. O.I.V.*, 1969, nos 462-463, 932.