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, CaCO₃ ................................................. 25 g
  Acetic acid, glacial, CH₃COOH (ρ₂₀= 1.05 g/mL) ............... 40 mL
  Water to ................................................................. 1000 mL
- Calcium (±)tartrate, crystallized: CaC₄O₆H₄•4H₂O.
  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 \(\rho_{20} = 0.97 \, \text{g/mL}\), 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, \( \text{CaC}_4\text{O}_6\text{H}_4\cdot 4\text{H}_2\text{O} \), 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, \( \text{CH}_3\text{COOH}, \rho_{20} = 1.05 \, \text{g/mL} \) 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:
  \[ 28.84 \text{ p} \]
  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:
  \[ 36.15 \text{ p} \]
  It is quoted to one decimal place.

2.2. Comparative volumetric analysis

2.2.1. Reagents

- Hydrochloric acid (\( \rho_{20} = 1.18 \) 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\(_2\)SO\(_4\) (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:
  \[ 5 \text{ } n \]
  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:
  
  \[ 0.375 \times n \]
  
  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:
  
  \[ 0.470 \times n \]
  
  It is quoted to one decimal place.

**BIBLIOGRAPHY**


