OIV-MA-AS313-02 Volatile acidity

Type I method

1. Definition

The volatile acidity is derived from the acids of the acetic series present in wine in the free state and combined as salts.

2. Principle

Carbon dioxide is first removed from the wine. Volatile acids are separated from the wine by steam distillation and titrated using standard sodium hydroxide.

The acidity of free and combined sulfur dioxide distilled under these conditions should be subtracted from the acidity of the distillate.

The acidity of any sorbic acid, which may have been added to the wine, must also be subtracted.

Note: Part of the salicylic acid used in some countries to stabilize the wines before analysis is present in the distillate. This must be determined and subtracted from the acidity. The method of determination is given in the Annex of this Chapter.

3. Apparatus

1. Steam distillation apparatus consisting of:

    - a steam generator; the steam must be free of carbon dioxide;
    - a flask with steam pipe;
    - a distillation column;
    - a condenser.

This equipment must pass the following three tests:

(a) Place 20 mL of boiled water in the flask. Collect 250 mL of the distillate and add to it 0.1 mL sodium hydroxide solution, 0.1 M, and two drops of phenolphthalein solution. The pink coloration must be stable for at least 10 sec (i.e. steam to be free of carbon dioxide).

(b) Place 20 mL acetic acid solution, 0.1 M, in the flask. Collect 250 mL of the distillate. Titrate with the sodium hydroxide solution, 0.1 M: the volume of the titer must be at least 19.9 mL (i.e. at least 99.5% of the acetic acid entrained with the steam).

(c) Place 20 mL lactic acid solution, 1 M, in the flask. Collect 250 mL of the distillate and titrate the acid with the sodium hydroxide solution, 0.1 M.

The volume of sodium hydroxide solution added must be less than or equal to 1.0 mL (i.e. not more than 0.5% of lactic acid is distilled).

Any apparatus or procedure which passes these tests satisfactorily fulfils the requirements of official international apparatus or procedures.

3.2. Water aspirator vacuum pump.

3.3. Vacuum flask.

4. Reagents

1. Tartaric acid, crystalline.
2. Sodium hydroxide solution, 0.1 M.
3. Phenolphthalein solution, 1%, in neutral alcohol, 96% (*m/v*).
4. Hydrochloric acid (ρ20 = 1.18 to 1.19 g/mL) diluted 1/4 with distilled water.
5. Iodine solution, 0.005 M.
6. Potassium iodide, crystalline
7. Starch solution, 5 g/L.

Mix 5 g of starch with about 500 mL of water. Bring to the boil, stirring continuously and boil for 10 min. Add 200 g sodium chloride. When cool, make up to one liter.

4.8. Saturated solution of sodium tetraborate,$Na_2B_4O_7.10H_2O$, about 55 g/L at 20°C.

4.9. Acetic acid, 0.1 M.

4.10. Lactic acid solution, 0.1 M

100 mL of lactic acid is diluted in 400 mL of water. This solution is heated in an evaporating dish over a boiling water bath for four hours, topping up the volume occasionally with distilled water. After cooling, make up to a liter. Titrate the lactic acid in 10 mL of this solution with 1 M sodium hydroxide solution. Adjust the solution to 1 M lactic acid (90 g/L).

5. Procedure

1. Preparation of sample**:** elimination of carbon dioxide. Place about 50 mL of wine in a

vacuum flask; apply vacuum to the flask with the water pump for one to two min while shaking continuously. Other CO_2 elimination systems may be used if the CO_2 elimination is guaranteed.

2. Steam distillation

Place 20 mL of wine, freed from carbon dioxide as in 5.1, into the flask. Add about 0.5 g of tartaric acid. Collect at least 250 mL of the distillate.

5.3. Titration

Titrate with the sodium hydroxide solution, (4.2), using two drops of phenolphthalein (4.3) as indicator. Let n mL be the volume of sodium hydroxide used.

Add four drops of the dilute hydrochloric acid (4.4), 2 mL starch solution (4.7) and a few crystals of potassium iodide (4.6). Titrate the free sulfur dioxide with the iodine solution, 0.005 M (4.5). Let n' mL be the volume used.

Add the saturated sodium tetraborate solution (4.8) until the pink coloration reappears. Titrate the combined sulfur dioxide with the iodine solution, 0.005 M (4.5). Let n'' mL be the volume used.

6. Expression of results

1. Method of calculation

The volatile acidity, expressed in milliequivalents per liter to one decimal place, is given by:

- $5 (n - 0.1 n' - 0.05 n'')$.

The volatile acidity, expressed in grams of sulfuric acid per liter to two decimal places, is given by:

- $0.245 (n - 0.1 n' - 0.05 n'')$.

The volatile acidity, expressed in grams of acetic acid per liter to two decimal places, is given by:

- $0.300 (n - 0.1 n' - 0.05 n'')$.

6.2. Repeatability (r)

- $r = 0.7$ meq/L
- $r = 0.03$ g sulfuric acid/L
- $r = 0.04$ g acetic acid/L.

3. Reproducibility (R) R = 1.3 meq/L

- R = 0.06 g sulfuric acid/L
- R = 0.08 g acetic acid/L.

4. Wine with sorbic acid present

Since 96% of sorbic acid is steam distilled with a distillate volume of 250 mL, its acidity must be subtracted from the volatile acidity, knowing that 100 mg of sorbic acid corresponds to an acidity of 0.89 milliequivalents or 0.053 g of acetic acid and knowing the concentration of sorbic acid in mg/L as determined by other methods.

Annex Determination of Salicylic Acid entrained in the distillate from the volatile acidity

1. Principle

After the determination of the volatile acidity and the correction for sulfur dioxide, the presence of salicylic acid is indicated, after acidification, by the violet coloration that appears when an iron (III) salt is added.

The determination of the salicylic acid entrained in the distillate with the volatile acidity is carried out on a second distillate having the same volume as that on which the determination of volatile acidity was carried out. In this distillate, the salicylic acid is determined by a comparative colorimetric method. It is subtracted from the acidity of the volatile acidity distillate.

2. Reagents

- Hydrochloric acid, HCl, (ρ_{20} = 1.18 to 1.19 g/L).
- Sodium thiosulfate solution, $Na_2S_2O_3.5H_2O$, 0.1 M.
- Iron (III) ammonium sulfate solution, $Fe_2(SO_4)_3(NH_4)SO_4.24H_2O$, 10% (*m/v*)
- Sodium salicylate solution, 0.01 M: containing 1.60 g/L sodium salicylate, Na $C_7H_5O_3$.

3. Procedure

3.1. Identification of salicylic acid in the volatile acidity distillate

Immediately after the determination of the volatile acidity and the correction for free and combined sulfur dioxide, introduce into a conical flask 0.5 mL hydrochloric acid, 3 mL of the sodium thiosulfate solution, 0.1 M, and 1 mL of the iron (III) ammonium sulfate solution. If salicylic acid is present, a violet coloration appears.

3.2. Determination of salicylic acid

On the above conical flask, indicate the volume of the distillate by a reference mark. Empty and rinse the flask. Subject a new test sample of 20 mL wine to steam distillation and collect the distillate in the conical flask up to the reference mark. Add 0.3 mL concentrated hydrochloric acid, and 1 mL of the iron (III) ammonium sulfate solution. The contents of the conical flask turn violet.

Into a conical flask identical to that carrying the reference mark, introduce distilled water up to the same level as that of the distillate. Add 0.3 mL concentrated hydrochloric acid and 1 mL of the iron (III) ammonium sulfate solution. From the burette run in the sodium salicylate solution, 0.01 M, until the violet coloration obtained has the same intensity as that of the conical flask containing the wine distillate.

Let n''' mL be the volume of solution added from the burette.

4. Correction to the volatile acidity

Subtract the volume 0.1 x n'''' mL from the volume n mL of sodium hydroxide solution, 0.1 M, used to titrate the acidity of the distillate during the determination of volatile acidity.

Bibliography

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