OIV-MA-AS315-06 Cyanide derivatives

Type II method

1. Principle

Free and total hydrocyanic acid is liberated by acid hydrolysis and separated by distillation. After reaction with chloramine T and pyridine, the glutaconic dialdehyde formed is determined by colorimetry, due to the blue coloration it gives with 1.3-dimethyl barbituric acid.

2. Equipment

2.1. Distillation apparatus: Use the distillation apparatus described for the determination of alcohol in wine.

2.2. Round-bottomed 500 mL flask with standard taper joint.

2.3. Water bath, thermostated at 20° C.

2.4. Spectrophotometer permitting the measurement of absorbance at a wavelength of 590 nm.

2.5. Glass cuvette or disposable cuvettes for one use only, with 20 mm optical path.

3. Reagents

3.1. Phosphoric acid (H_3PO_4) at 25 p. 100 (w/v)

3.2. Solution of chloramine T ($C_7H_7ClNNaO_2S.\ 3H_2O$) 3% (w/v)

3.3. Solution of 1,3-dimethylbarbituric acid: dissolve 3.658 g of 1,3-dimethylbarbituric acid ($C_6H_8N_2O_3$) in 15 mL of pyridine and 3 mL of hydrochloric acid (ρ_{20} = 1.19 g/mL) and bring to 50 mL with distilled water.

3.4. Potassium cyanide (KCN)

3.5. Solution of potassium iodide (KI) 10% (w/v)

3.6. Solution of silver nitrate ($AgNO_3$), 0.1 M

4. Procedure

1. Distillation:

In the 500 mL round-bottomed flask (2.2), place 25 mL of wine, 50 mL of distilled water, 1 mL of phosphoric acid (3.1) and some glass beads.

Immediately place the round-bottomed flask on the distillation apparatus. Collect the distillate through a delivery tube connected to a 50 mL volumetric flask containing 10 ml of water. The volumetric flask is immersed in an iced water bath. Collect 30-35 mL of distillate (a total of about 45 mL of liquid in the volumetric flask). Wash the delivery tube with a few milliliters of distilled water, bring the distillate to 20°C and dilute with distilled water to the mark.

4.2. Measurement:

Place 25 mL of distillate in a 50 mL glass-stoppered Erlenmeyer flask, add 1 mL of chloramine T solution (3.2) and stopper tightly. After exactly 60 seconds, add 3mL of 1,3-dimethylbarbituric acid solution (3.3), stopper tightly and let stand for 10 minutes. Then measure the absorbance relative to the reference blank (25 mL of distilled water instead of 25 mL of distillate) at a wavelength of 590 nm in cuvettes of 20 mm optical path.

5. Establishing the standard curve

1. *Argentimetric titration of potassium cyanide.*

In a 300 mL volumetric flask, dissolve about 0.2 g of KCN (3.4) precisely weighed in 100 mL of distilled water. Add 0.2 mL of potassium iodide solution (3.5) and titrate with the solution of 0.1 M silver nitrate (3.6) until obtaining a stable yellowish color.

In calculating the concentration of KCN in the sample, 1 mL of 0.1 M silver nitrate solution corresponds to 13.2 mg of KCN.

5.2. Standard Curve.

5.2.1. Preparation of the standard solutions:

Knowing the KCN concentration determined in accordance with 5.1, prepare a standard solution containing 30 mg/L of hydrocyanic acid (30 mg HCN = 72.3 mg of KCN). Dilute this solution to 1/10.

Introduce 1.0, 2.0, 3.0, 4.0, and 5.0 mL of the diluted standard solution in 100 mL volumetric flasks and bring to the mark with distilled water. The prepared standard solutions correspond to 30, 60, 90, and 150 µg/L of hydrocyanic acid, respectively.

5.2.2. Determination:

Using 25 mL of the solutions, continue as indicated above in 4.1 and 4.2.

The values obtained for the absorbance with these standard solutions, reported according to the corresponding levels of hydrocyanic acid, form a line passing through the origin.

6. Expression of the results

Hydrocyanic acid is expressed in micrograms per liter (µg/L) without decimal.

6.1. Calculation:

Determine the concentration of hydrocyanic acid from the standard curve. If a dilution was done, multiply the result by the dilution factor.

Repeatability (r) and Reproducibility (R)

White wine	r=3.1 µg/L	i.e. approximately 6% . X_i
	R= 12 µg/L	i.e. approximately 25% . X_i
Red wine	r=6.4 µg/L	i.e. approximately 8% . X_i
	R=23 µg/L	i.e. approximately 29% . X_i

Xi = average concentration of HCN in the wine.

Bibliography

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