7.13 Determination of urea - Distillation method

Principle

Suspension of water of the test portion in the presence of de-colorant. Addition of Carrez I and Carrez II solutions. Stirring of the suspension, then filtration. Addition to the filtrate of 4-dimethyl-amino-benzaldehyde (DMAB) and spectrometric measurement at 420 nm of the absorbance of the solution thus obtained.

Reagents

- Active carbon - Which does not absorb urea.
- 4-Dimethyl-Amino-Benzaldehyde (4-DMAB) - Dissolve 1.6 g of 4-DMAB in 100 ml of 96 per cent (v/v) ethanol, add 10 ml of concentrated hydrochloric acid (P_{20}=1.19 g/ml) and mix.
- Carrez I Solution - Dissolve in water 24 g of zinc acetate dehydrate ($Zn(CH_3COO)_2.2H_2O$) and 3 g of glacial acetic acid. Make up to 100 ml with water and mix.
- Carrez II Solution - Dissolve in water 10.6 g of potassium hexacynoferrate (II) trihydrate (potassium ferrocyanide trihydrate) ($K_4(Fe(CN)_6).3H_2O$). Make up to 100 ml with water and mix.
- Urea - Standard solution corresponding to 1 g of urea per litre.

Apparatus

- Rotary shaker
- Spectrometer

Procedure

Weigh, to the nearest 1 mg, about 2 g of the test sample.

Preparation of the test solution

Transfer the test portion together with 1 g of the active carbon into a 500 ml volumetric flask. Add 400 ml of water, 5 ml of the Carrez I solution and 5 ml of the Carrez II solution. Mix for 30 min in a rotary shaker (Fig. 7.12). Make up to the mark with water homogenize and filter. If the filtrate is coloured, increase the quantity of active carbon.

Colour development

Transfer, by means of a pipette, 5 ml of the

Fig. 7.12 Rotary shaker

clear colourless filtrate into a test tube and add, by means of a pipette, 5 ml of the 4-DMAB soluti on.

Mix and leave to stand for 15 min in a water bath controlled at 20ºC .

Blank test

Carry out a blank test in parallel with the determination using the same procedure and the same quantities of all reagents, but omitting the test portion.

Preparation of the calibration graph

Pipette into a series of five 100 ml volumetric flasks 10, 20, 40, 50 and 80 ml of the urea standard solution. Make each flask up to the mark with water. One millilitre of the standard solution contains 100, 200, 400, 500 and 800 µg of urea, respectively.

Pipette into a series of five test tubes 5 ml of each of these solutions (one dilution per test tube). Add to each test tube, by means of a pipette, 5 ml of the 4-DMAB solution and homogenize. Transfer the solution to spectrometer cells and measure their absorbance at 420 nm, using the spectrometer against a compensation solution containing 5 ml of 4- DMAB and 5 ml of water.

Plot the calibration graph, with the absorbance value on the ordinate and corresponding concentrations of urea, in micrograms per millilitre.

Transfer the solution to a spectrometer cell and measure its absorbance at 420 nm, using the spectrometer, against the blank test.

Calculati on

$$\text{Urea content, (percentage by mass)} = \frac{C}{20 \times m}$$

Where,

C = is the urea content, in micrograms per ml of the filtrate of the test solution, determined from the calibration graph.

m = is the mass, in grams, of the test portion.

Reference: IS 13399 : 1992