2.10 Determination of nitrogen

Recommended procedures

Procedure for macrodetermination (Method A)

Place the amount of test substance specified in the monograph in a 200-mL long-necked flask, add 1 g of a mixture composed of 10 parts of potassium sulfate R or anhydrous sodium sulfate R and 1 part of copper(II) sulfate R, followed by nitrogen-free sulfuric acid (~1760 g/l) TS, using the quantities of the substance and of sulfuric acid specified in the monograph. Heat the mixture over a small flame until a clear green solution is obtained and boil gently for a further 30 minutes unless otherwise specified in the monograph; precautions should be taken to prevent the upper part of the flask from becoming overheated. Cool, dilute to 75-85 mL with water, using suitable precautions, and add a piece of granulated zinc R and a solution of 15 g of sodium hydroxide R and 2 g of sodium thiosulfate R in 25 mL of water. The quantity of sodium hydroxide should be increased, if necessary, to ensure that, before distillation, the mixture is strongly alkaline. Immediately connect the flask to a distillation apparatus, mix the contents, distil the liberated ammonia into 16 mL of boric acid (50 g/l) TS, and titrate with sulfuric acid (0.05 mol/l) VS using methyl red/ethanol TS as indicator. Repeat the operation without the substance being tested; the difference between the titrations represents the ammonia liberated by the substance being tested. Each mL of sulfuric acid (0.05 mol/l) VS is equivalent to 1.401 mg of N.

Procedure for microdetermination (Method B)

Place the amount of test substance specified in the monograph in a suitable digestion tube, add 3 drops of copper(II) sulfate (190 g/l) TS and 1 mL of nitrogen-free sulfuric acid (~1760 g/l) TS and boil gently for 10 minutes; cool; add 1 g of anhydrous sodium sulfate R and 10 mg of selenium R, boil gently for 1 hour and cool. Transfer the contents of the digestion tube to (or attach it to) an ammonia microdistillation apparatus, add 6 mL of sodium hydroxide (~400 g/l) TS and pass steam through the flask; distil for 7 minutes, collecting the distillate in a mixture of 5 mL of boric acid (50 g/l) TS, 5 mL of water, and 1 drop of methyl red/methylthioninium chloride TS and titrate with hydrochloric acid (0.015 mol/l) VS. Repeat the operation without the substance being tested; the difference between the titrations represents the ammonia liberated by the substance being tested. Each mL of hydrochloric acid (0.015 mol/l) VS is equivalent to 0.210 mg of N.