2.7 Nitrite titration

Nitrite titration is a titration method used particularly for the assay of primary aromatic amines.

The apparatus usually used in an electrometric procedure for nitrite titration is composed of an open titration vessel containing two platinum electrodes connected to a suitable circuit. The electrodes should have a potential difference of 50-100 mV. The circuit should include a device for measuring current with a sensitivity of 0.1 to 1 nA, usually with an indicating needle. The titration vessel should be provided with a suitable mechanical or magnetic stirring device, or a stream of nitrogen passing through the solution may be used to mix this solution. Electrodes made of platinum wire 0.5 mm in diameter and about 20 mm long are suitable. Before each use, the electrodes should be cleaned by immersing them for a few seconds in boiling nitric acid (~1000 g/l) TS, to which about 1 mg/mL of ferric chloride R has previously been added, and then thoroughly rinsing them with water.

Recommended procedure

Place 20 mL of hydrochloric acid (~250 g/l) TS and 50 mL of water in the titration vessel, add the quantity of the test substance and, if indicated, a catalyst, as specified in the monograph and stir to dissolve; cool to about 15 °C and titrate slowly with sodium nitrite (0.1 mol/l) VS, placing the burette tip below the surface of the solution. During the addition of the titrant, stir the solution continuously and gently, without pulling a vortex of air under the surface, and maintain the temperature of the solution at about 15 °C.

When the titration is within 1 mL of the estimated end-point, add the titrant in 0.1 mL portions, allowing not less than 1 minute to elapse before adding subsequent portions. Initially, the needle of the measuring device deflects at every addition of reagent and then returns to its original position. No deflection is observed when the end-point of the determination is reached.