2,3-Dihydroxynaphthalene as Indicator for the Complexometric Titration of Iron(III) with EDTA

2,3-Dihydroxynaphthalin als Indicator für die komplexometrische Titration von Eisen(III) mit ÄDTA

Best. von Eisen(III); Volumetrie; ÄDTA-Titration, 2,3-Dihydroxynaphthalin als Indicator.

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The present note describes the use of 2,3-dihydroxynaphthalene as a metal indicator for the complexometric titration of iron(III) with EDTA. The optimum conditions worked out are incorporated in the following *procedure*.

Adjust the pH of the acidic iron(III) solution containing 0.1 to 67.0 mg of iron(III) to about 2 with ammonia, add 10 ml of 1 M hydrochloric acid-sodium acetate buffer pH 5 (optimum range for the titration is 4.85–5.65) and 5–10 drops of 0.5% w/v ethanolic solution of 2,3-dihydroxynaphthalene, dilute to about 100 ml with distilled water and titrate against 0.010 to 0.050 M EDTA solution till a sharp colour change from purple to colourless is observed.

With higher amounts of iron(III), the end point is indicated by the disappearance of last traces of the purple colour leaving a clear yellow solution.

The relative standard deviation for the titration has been found to be in the range 0.8–1.5%.

The colour of the iron(III)-2,3-dihydroxynaphthalene complex is royal blue at pH 1–3, purple at

pH 4–6, and red above pH 7. As the titrations were found to be possible only in the range 4.85–5.65, end point was taken to be the disappearance of the purple colour.

With very large concentrations of iron(III) (80 mg and above), titrations became erratic probably because of the larger concentrations of acetate ions required to keep iron(III) in solution.

Interferences. The following ions do not cause any interference even when present in quantities up to 100 times that of iron: bromide, borate, tartrate, citrate, fluoride, zinc, cadmium, calcium, barium, strontium, manganese(II), magnesium and beryllium.

The following ions can be tolerated in the amounts given in the parentheses, together with the masking agent if any, for 5.85 mg of iron(III): aluminium(III) (100 mg, citrate), antimony(III) (50 mg, tartrate), bismuth(III) (50 mg, tartrate or thiourea with end point as disappearance of traces of purple colour), cobalt(II) (50 mg, tartrate), zirconium(IV) (100 mg, tartrate, citrate), titanium(IV) (10 mg, tartrate), phosphate (50 mg), thiosulphate (100 mg) and iodide (50 mg, thiosulphate).

The following ions interfere seriously: cyanide, sulphocyanide, oxalate, nickel(II), vanadium(IV), vanadate, molybdate, tungstate, uranyl and copper(II).

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Microdetermination of Formic, Oxalic and Acetic Acids in Their Mixture by Selective Oxidation

Mikrobestimmung von Ameisen-, Oxal- und Essigsäure im Gemisch durch selektive Oxidation

Mikrobest. von Ameisensäure; Oxalsäure; Essigsäure im Gemisch, selektive Oxidation mit Ag(III) und Cu(III).

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In the present paper, formic, oxalic and acetic acids have been determined in their mixture by selective oxidation with Ag(III) and Cu(III) reagents (see also our earlier publication on oxalic, formic and tartaric acids [2]).

Ag(III) oxidises only formic acid under specific conditions. Diperioato cuprate(III) solution oxidises only formic and oxalic acids under verified conditions, whereas ditellurato cuprate(III) solution oxidises formic, oxalic as well as acetic acids. Thus by the use of these three oxidants we can determine the three acids separately in their mixture.

Materials

Ditellurato Argentate(III) and Ditellurato Cuprate(III) Solutions were prepared and standardised as described earlier [3 and 1, resp.].

Diperioato Cuprate(III) Solution. Copper sulphate (3.54 g), potassium periodate (6.80 g), potassium persulphate (2.20 g) and potassium hydroxide (9.00 g) were added to 250 ml of water. The mixture was shaken thoroughly and then heated on a hot plate for about quarter to one hour. The mixture is then cooled and filtered through a sintered glass crucible (porosity 4) and the clear filtrate is