Kurze Mitteilungen

Photometric Determination of Mercury(II) with 4-(2-Pyridylazo)-resorcinol

Photometrische Bestimmung von Quecksilber(II) mit 4-(2-Pyridylazo)-resorcin

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Mercury reacts with 4-(2-pyridylazo)-resorcinol (PAR) forming an orange-red chelate at pH 3.1–3.3. Studies of the complex formation at various pH values indicate that the complex has maximum absorption at pH 3.1–3.3. The complex is less soluble in water, but solubility is enhanced by using mixtures of water-dioxane, water-iso-propylalcohol, water-tert.-butylalcohol etc. The complex is stable for at least 6 h. Measurements are taken after 1 min. Composition of the complex has been found to be 1:1. Beer's law is obeyed in the range of 0.8 to 7.0 μg of Hg/ml. Molar absorptivity is 20059 (absorption maximum 540 nm). Sensitivity (Sandell) is 0.01 $\mu\text{g} \times \text{cm}^{-2}$ and relative standard deviation $\pm 1.25\%$. The interference of about 35 ions has been studied with solutions containing 60 μg of Hg. The following tolerance limits were found (μg): Ir(III) 17; Be(II), Ce(III) 500; Cr(VI), Mo(VI), citrate, oxalate, malonate, HPO_4^{2-} , AsO_4^{3-} , SCN^- , $\text{S}_2\text{O}_3^{2-}$, CH_3COO^- 1000; Ca(II), Sr(II), Ba(II), W(VI), Al(III), Se(IV), Te(IV) 2500. As CN^- , EDTA^{2-} and F^- interfere seriously, it has been impossible to eliminate most of these metal ions and preliminary separation has to be carried out. The following ions show strong interferences: Mn(II), Co(II), Ni(II), Cu(II), Pb(II), Fe(III), V(IV), Th(IV), U(VI).

Recommended Procedure. The pH of an aliquot of mercuric nitrate solution taken is adjusted to 3.1–3.3 using HNO_3 -NaOH. To this solutions are added 10 ml of tert. butylalcohol and 2 ml of 0.1% PAR, made to 25 ml and shaken thoroughly. Absorbance is measured after an interval of 1 min.

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1-(2-Quinolylazo)-2-phenanthrol (QAP) as a Visual Indicator for the Determination of Mercury(II) with EDTA

1-(2-Chinolylazo)-2-phenanthrol als visueller Indikator zur Bestimmung von Quecksilber(II) mit ÄDTA

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1-(2-Quinolylazo)-2-phenanthrol (QAP) has been found to be a suitable indicator for the determination of mercury with EDTA. The pH range within which the titration can be carried out accurately is fairly wide (6.0 to 10.0), and also the temperature range (20 to 65°C). Subsequent titrations were, therefore, carried out at room temperature ($\sim 30^\circ\text{C}$) and at pH 7.0. Two drops of 0.01 M QAP in 1,4-dioxane are sufficient to cause a sharp change of colour at the end point within a very short interval of time. Large amounts of the indicator also give accurate results, but then the colour of the solution becomes very dark.

The sensitivity of the proposed method is higher than that obtained by 1-(2-pyridylazo)-2-naphthol (α -PAN) (0.001 $\mu\text{g}/\text{cm}^2$ and 0.0017 $\mu\text{g}/\text{cm}^2$ for $\log I_0/I$ 0.001) and the minimum amount of mercury that can be determined accurately by this method is 1×10^{-4} g.

The following foreign ions can be tolerated in the amounts (mg) given in parentheses when present with 1.0 mg of mercury: chloride (50), bromide (50), fluoride (200), acetate (800), iodate (110), thiourea (450), thiosulphate (75), thiocyanate (60), oxalate (50), nitrite (600), borate (20), phosphate (20), manganese (10), palladium (10) (masked with thiourea), aluminium (10) (masked with fluoride), and 20 mg each of calcium, barium, strontium, silver, and magnesium. Serious interferences are caused by copper, cobalt, nickel, zinc, cadmium, cyanide, and iodide.

Procedure for Titration. There are two ways in which mercury can be determined by titration with EDTA.

1. Direct Titration. Adjust the pH of 5.0 ml of 0.001 M mercury solution to 7.0 by adding 10.0 ml of sodium acetate/acetic acid buffer followed by the addition of 2 drops of 0.01 M QAP solution (0.349 g/100 ml in freshly distilled 1,4-dioxane). Titrate this solution slowly with standard