Zusammenfassung

Es wird eine dünnenschichtchromatographische Schnellmethode zur Trennung und zum Nachweis von Oestrone, Oestrone-3-methyläther, Oestradiol, Oestradiol-17-valerianat, Oestradiol-17-palmitat und Progesteron-(3,20)-dion beschrieben. Die Grenze der Nachweisbarkeit dieser Substanzen liegt zwischen $5 \times 10^{-2}$ und $5 \times 10^{-3} \mu g$.

Summary

Separation and identification of oestrone, oestrone-3-methyl-ether, oestradiol, oestradiol-17-valerianate, oestradiol-17-palmitate and pregnane-(3,20)-dione can be rapidly achieved by use of thin-layer chromatography. The limit of detection of these substances is in the range of $5 \times 10^{-2}$ to $5 \times 10^{-3} \mu g$.

Literatur


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Complexometric Estimation of Thorium

By

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Thorium forms a very stable 1:1 complex with EDTA (ethylenediaminetetra-acetic acid), the formation constant of the complex being 23.2. A number of methods utilizing this reaction have been developed for the complexometric determination of thorium. FURT and FORD recommended the use of the Alizarin S as indicator in the volumetric determination of thorium with EDTA between pH 2.3 to 3.4. BANERJEE used SPADNS [2-(p-sulphophenylazo)-1,8-dihydroxy-naphthalene-3,6-disulphonic acid] as indicator in the complexometric determination of thorium with EDTA at pH 3.04; the colour change at the end point being from
blue violet to scarlet red. Pyrocatechol violet was used as indicator in the titrimetric determination of thorium with EDTA by Sux and Malati. Köble, Péribi and Ems suggested Xylenol Orange as indicator in Th-EDTA titrations between pH 2.5 to 3.5. Various other workers have used PAN \[1,2(-pyridylazo)-2-naphthol\], Naphthyl Azoxine \[7-1-naphthylazo)-5, sulfo-8-quinolinol\], quinizarine derivatives, Omega Chrome Green BLL, sodium salt of o-carboxyphenylazochromotropic acid, azo dyes such as Ellamina Blue FFL, Stilbamido R \[di-NH4-4,4'-bis-(3,4-dihydroxyphenylazo)stilbene-2,2'-disulfonate\] or trisodium salt of sulphodi chlorohydroxydimethylfuchsone dicarboxylic acid as an indicator in the trilometric determination of thorium.

Singh et al. used a mixture of potassium thiocyanate and p-anisidine in equimolecular proportion as indicator in the complexometric determination of ferric iron with EDTA (disodium salt). As this mixture is very sensitive even to very small traces of iron, this prompted the authors to use a drop of ferric iron solution mixed with thiocyanate-p-anisidine as indicator in Th-EDTA titrations.

In the present investigation thorium in presence of some other metallic ions has been determined with EDTA (disodium salt) using a drop of 0.01 M ferric iron solution mixed with 0.5 ml of thiocyanate-p-anisidine as indicator. The end-point is marked by disappearance of the pink colour.

**Experimental**

*Estimation of Thorium in Presence of other Metallic Ions.* A known amount of thorium nitrate was taken into a conical flask. 20 ml of water, 0.5 ml of thiocyanate-p-anisidine indicator and one drop of 0.01 M ferric nitrate solution were added to it. The mixture was titrated against 0.01 M EDTA (disodium salt) solution and the colour of the reaction mixture changed from pink to colourless at the end-point. The titration was then performed in the presence of calcium chloride, barium chloride, strontium nitrate, magnesium chloride, manganese chloride, chromium nitrate, zinc chloride, zirconium sulphate, lead nitrate, silver nitrate, aluminium chloride, bismuth nitrate, disodium hydrogen phosphate, sodium fluoride or ammonium oxalate respectively. The pH of the reaction mixture remained between 3 to 4.5 during these titrations. All these titrations were also repeated using pyrocatechol violet as indicator.

For comparison some typical results obtained by using a drop of 0.01 M iron(III) solution mixed with thiocyanate-p-anisidine and pyrocatechol violet as indicators are given in the Table.

In the complexometric titrations of thorium with EDTA, a drop of 0.01 M iron(III) solution mixed with thiocyanate-p-anisidine indicator gives quantitative results which are comparable with those obtained by using pyrocatechol violet as an indicator. Thorium alone or in presence of Ca\(^{2+}\), Ba\(^{2+}\), Sr\(^{2+}\), Mg\(^{2+}\) or Mn\(^{2+}\) ions respectively can be determined volumetrically by titrating with EDTA (disodium salt) solution using a drop of 0.01 M iron(III) solution mixed with thiocyanate-p-anisidine as indicator.

All chemicals used in this investigation are of guaranteed purity.