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# Heterocyclic Hydroxamic Acids. VI. Microdetermination of Cerium(IV) with *N-p*-Chlorophenyl-2-furohydroxamic Acid

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## NOTE

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## Abstract

*N-p*-Chlorophenyl-2-furohydroxamic acid is presented as the most sensitive and selective of the hydroxamic acids reported so far for the separation-spectrophotometric determination of cerium(IV). Cerium was determined in lanthanum oxide.

## INTRODUCTION

During an ongoing-program (*1–4*) of studies on the physicochemical properties and analytical applications of heterocyclic hydroxamic acids, *N-p*-chlorophenyl-2-furohydroxamic acid was found to be the most sensitive and selective of the hydroxamic acids reported so far (*5–8*) for the separation–colorimetric determination of Ce(IV). The results of the investigations are presented.

## EXPERIMENTAL

CHFA was prepared following the general method of Tandon and Bhattacharyya (*9*). All other chemicals were of reagent grade. Conductivity water was used throughout. The metal ion solutions were standardized with the help of appropriate methods (*10*). The pH adjustments were carried out with a Radiometer pH meter PHM-29. Spectra were recorded on a Perkin-Elmer 402 spectrophotometer, and measurements at a constant wavelength were done with a SF-4 (USSR) spectrophotometer.

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### Procedure for Separation and Determination

The pH of a cerium solution containing 4–40 μg of Ce(IV) was adjusted between 7.8–10, and 5 mL of reagent solution (0.5% w/v) in ethanol was added with stirring. The resulting precipitate was equilibrated with 5 mL of chloroform for 2 min and the chloroform extract was separated. The extraction was repeated with a fresh 5 mL of chloroform, and the mother liquor was washed with two 2 mL portions of chloroform to recover any trapped droplets of the extract. The extracts and washings were combined, diluted to 25 mL, and the absorbance was measured at 460 nm against the reagent solution as the blank. A calibration curve was set up accordingly.

## RESULTS AND DISCUSSION

The chloroform extract has $\lambda_{\text{Max}}$ at 460 nm ($\varepsilon = 6.0 \times 10^3$ L mol$^{-1}$ cm$^{-1}$). Beer's law is obeyed in the range of 2–20 ppm of Ce(IV). The sensitivity of the method, as per Sandell's definition, (*11*), is 0.02 ppm. The method tolerates Au(III), Zn(II), and Hg(II) when present in 20-fold the amount of Ce(IV). Mg(II), Li(I), K(I), and Na(I) are tolerated when present in 30-fold the amount of Ce(IV). Halides, nitrate, sulfate, acetate, and carbonate are tolerated in a 100-fold excess. Tartrate, oxalate, and citrate, which tend to reduce Ce(IV) to Ce(III), interfere but their interference can be checked by oxidizing them with nitric acid prior to precipitation of Ce(IV).

The interference from V(V), Ti(IV), Fe(III), Cu(II), Ni(II), Co(II), and U(VI) can be eliminated by prior extraction of these ions by 0.1 *M* solutions of CFHA in chloroform from solutions maintained at 4–8 *M* HCl [for Ti(IV) and V(V)]; pH = 0.1[for Fe(III)]; pH = 4.2 for [Cu(II)]; pH = 5.0 [for U(VI)]; and pH = 5.5 [for Ni(II) and Co(II)].

### Determination of Cerium in Lanthanum Oxide

Lanthanum oxide, 100 mg, containing 0.5% cerium dioxide was dissolved in 1 *N* sulfuric acid (25 mL). A 25% w/v ammonium chloride solution (5 mL) was added, followed by the reagent solution (2 mL). The contents were diluted to 50 mL and, after adjusting the pH to 4.5 ± 0.5, were transferred to a 100 mL separatory funnel. The contents were twice equilibrated with 10 mL portions of chloroform. The organic phase was discarded. The pH of the aqueous phase was raised to ~9, and CFHA (0.75 g) dissolved in ethanol (5 mL) was added. Cerium was then determined as detailed above.

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