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## Potentiometric and Visual Titration of Trivalent Cerium with Potassium Permanganate

K. A. Idriss, I. M. Issa, and Y. M. Temerk  
Chem. Dept., Fac. of Science, Assiut University, Egypt  
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*Potentiometrische und visuelle Titration von Cer(III) mit Kaliumpermanganat*

Best. von Cer(III) mit Permanganat; Volumetric; potent., visuell.

A new method is described for the accurate determination of trivalent cerium. It is based on the oxidation of Ce(III) with  $\text{KMnO}_4$  in sulphuric acid medium in the presence of fluoride, required for preventing a sluggish reaction and the formation of insoluble oxides of manganese. A given Mn reagent solution can be titrated with the Ce containing sample solution either visually or potentiometrically. 21–54 mg of Ce(III) can be determined with average errors of 0.7% and 0.3%, respectively, in this way. The oxidation of Ce with permanganate followed by back-titrating the excess oxidant with monovalent thallium solution yields also accurate results and is applicable to a-

Dr. K. A. Idriss, Chem. Dept., Fac. of Science, Assiut University, Assiut, Egypt

mounts of 0.85–34 mg of Ce with an average error of 0.5%. Optimum experimental conditions are incorporated in the below given working directions.

*Equipment.* The e.m.f. of the titration cell was measured by a direct reading millivoltmeter of the Radiometer (28 Mb) type, using a Pt electrode with a saturated calomel electrode as reference half-cell.

*Direct Titration.* A volume of  $\text{KMnO}_4$  solution containing 1.5–6 mg of Mn is placed in a titration cell and mixed with the required volume of 1 M  $\text{H}_2\text{SO}_4$  to give an acidity of 0.02–0.06 N and also with 50 ml of 2% NaF solution. The mixture is diluted to 100 ml with twice distilled water, heated to  $\sim 40^\circ\text{C}$  and then titrated with the Ce containing sample solution. If visual end-point is desired, an appropriate amount of 0.25 M  $\text{CuSO}_4$  solution is to be added.

*Indirect Titration.* To a known excess of permanganate solution placed in a titration cell, 1 M sulphuric acid and 2% NaF are added so that the solution becomes 0.04 N with respect to  $\text{H}_2\text{SO}_4$  and 1% in NaF. The sample solution is then added dropwise while continuously shaking the mixture. The mixture is left to stand for 10–15 min followed by raising the acidity to about 1.2 N  $\text{H}_2\text{SO}_4$  and back-titrating the excess with standard thalious sulphate solution at  $35\text{--}40^\circ\text{C}$ .

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## Extraction and Spectrophotometric Determination of Nickel with Potassium Ethyl Xanthate

P. K. Paria and S. K. Majumdar  
Dept. of Chemistry, North Bengal University, Darjeeling, India  
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*Extraktion und spektralphotometrische Bestimmung von Nickel mit Kaliumäthylxanthat*

Best. von Nickel mit Äthylxanthat; Spektralphotometrie

Nickel forms a yellow brown precipitate with potassium ethyl xanthate, which can be extracted into chloroform and be evaluated spectrophotometrically. A method for the rapid determination of this element has been worked out accordingly. The absorption maximum of the complex is at 415 and 475 nm, the first one being recommended for measurement. Beer's law is obeyed over a concentration range of 3–16  $\mu\text{g}$  of Ni/ml. The molar absorptivity of the complex (composition metal:reagent = 1:2) is  $2.9 \times 10^3 \text{ l mole}^{-1} \text{ cm}^{-1}$  at 415 nm, sensitivity according to

Sandell is 0.02  $\mu\text{g}$  of Ni/ml. Optimum pH-range is 4–9. The colour is stable for up to 48 h. Results are accurate within  $\pm 1.5\%$ . Total time required is 20 to 25 min.

*Interferences.* Iron(III) should be masked by fluoride. Hg(II), Cd(II) and Ag(I) interfere by turbidities. Co(II) should be extracted before by thiocyanate into isoamyl alcohol. Bi(III) and Cu(II) interfere by their characteristic colours. High results are obtained in the presence of Pd(II), Mn(II) and Ru(III). Os(VIII) forms a black precipitate. EDTA prevents the extraction of nickel.

*Working Procedure.* Adjust the pH of the sample solution (1 ml) to 4–9 by addition of an appropriate buffer solution, add 1 ml of 0.5% aqueous solution of potassium ethyl xanthate, keep for 5 min, add 10 ml of chloroform, shake for 10 min, allow to settle for 5 min, make up the extract to 25 ml with chloroform and measure the absorbance at 415 nm against a reagent blank. Take the results from a calibration curve prepared in similar manner.

Dr. P. K. Paria, Dept. of Chemistry, North Bengal University, Raja-Rammohunpur, Distr. Darjeeling, India