

2. Kolthoff, I. M., Furman, N. H.: Potentiometric titrations, 2nd Ed., p. 159. New York: John Wiley 1931.
3. Lal, S., Christian, G. D.: Anal. Chem. **43**, 410 (1971).

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Determination of Tungsten with a Permselective Membrane Electrode

Bestimmung von Wolfram
mit einer permselektiven Membranelektrode

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Smith and Bradbury [3], originally, reported the preparation of silver tungstate, a pale yellow precipitate, obtained by intermixing solutions of sodium tungstate and silver nitrate. The salt, Ag_2WO_4 , is sparingly soluble in water, soluble in ammonia, nitric acid and is decomposed by heat and by treatment with several other reagents. Hence, the method did not gain acclaim as a suitable gravimetric method for the determination of tungsten. Recently, Shivahare [2] described the potentiometric determination of tungsten as Ag_2WO_4 using a silver wire as the indicator electrode.

The present communication embodies the potentiometric study of the silver nitrate-sodium tungstate reaction with the aid of a permselective membrane. The plastic membrane, impregnated with dithizone was used as the indicator electrode. Though the response of the membrane toward silver was non-Nernstian, yet it monitored the relative changes of Ag^+ ions accurately.

Details of the electrode construction are described elsewhere [1]. All the chemicals used were reagent grade and their solutions were prepared in deionized distilled water. 25 ml of silver nitrate solution were taken every time in a 50 ml beaker and standard sodium tungstate solution was added from the microburette. With the addition of sodium tungstate, the e.m.f. increased to a certain limit and then fell sharply yielding very small changes after the end point. An average of six determinations yielded 23.16 mg of W when 22.98 mg of W were originally present.

Titration in the reverse order could not give reproducible results as on the addition of a few drops of silver nitrate solution, the e.m.f. values tremendously changed from negative to high positive

values. Titrations in presence of 25% dioxane and 25% ethanol did not give sharp inflection points. Titrations in presence of sodium acetate, ammonium hydroxide and adjusting pH to 7 were not successful presumably due to diverse anions and cations present in the solution.

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References

1. Lal, S., Christian, G. D.: Anal. Chem. **43**, 410 (1971).
2. Shivahare, G. C.: Naturwissenschaften **52**, 157 (1965); cf. Z. Anal. Chem. **230**, 387 (1967).
3. Smith, E. F., Bradbury, R. H.: Chem. Ber. **24**, 2930 (1891).

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Photometric Determination of Vanadium(V), Titanium(IV), Manganese(II) and Iron(III) with N-Hydroxysuccinamic Acid (Di-Potassium Salt)

Photometrische Bestimmung von Vanadium(V), Titan(IV),
Mangan(II) und Eisen(III) mit dem Dikaliumsalz
der N-Hydroxysuccinamidsäure

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Hydroxamic acids ($\text{R}-\text{CO}-\text{NH}-\text{OH}$) have been widely investigated in the past 15 years for the determination of a number of metal ions. Bass and Yoe [1] have recently surveyed the reactions of 33 hydroxamic acids and 3 N-substituted hydroxamic acids. We have examined a new hydroxamic acid, namely, di-potassium salt of N-hydroxysuccinamic acid ($\text{KOOOC}-\text{CH}_2-\text{CH}_2-\text{CO}-\text{NH}-\text{OK}$). It has the advantage of being water-soluble and giving water-soluble metal complexes, thus offering a suitable method for the photometric determination of metal ions like vanadium(V), titanium(IV), manganese(II) and iron(III).

Reactions with Metal Ions. The reagent develops an orange colour with V^{5+} (pH 2.0–3.5) and Fe^{3+} (pH 4.0–8.9), intense yellow with Ti^{4+} (pH 0.8 to