Vorteile des Verfahrens. 1. Nach einer einmaligen Zusammenstellung ist das Gefäß ununterbrochen zum Messen bereit. 2. Minimaler Quecksilberverbrauch zur Herstellung des Quecksilberbodens. 3. Einfache und schnelle Durchführung der Messungen. 4. Große Zeitersparnis.

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Titrimetric Microdetermination of Zinc with EDTA Using 1,5-Di- β -Naphthylthiocarbazon (HNDZ) as an Extractive Indicator

Titrimetrische Mikrobestimmung von Zink mit ÄDTA unter Verwendung von 1,5-Di- β -Naphthylthiocarbazon als Extraktionsindikator

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Received December 22, 1971

1,5-Di- β -naphthylthiocarbazon has been found to form a violet complex instantaneously with zinc, which is extractable in chloroform and carbon tetrachloride. The colour reaction is highly sensitive. Accurate results for the microdetermination of zinc are obtained in the pH range 3.80 to 4.50. pH 4.0 has been selected for subsequent studies. One drop of indicator solution containing 5 mg of HDNZ in 100 ml of chloroform is sufficient to cause a sharp change of colour within a very short interval of time. A too large amount of the indicator results in a too dark colour.

The following diverse ions did not hinder the microdetermination of zinc when present in large excess: thiosulphate, borate, thiocyanate, chloride, bromide, iodide, tartrate, phosphate, sulphate, sulphite, chromate, fluoride, oxalate, manganese, calcium, barium, strontium, magnesium and copper (thiosulphate used as masking agent).

The accuracy and sensitivity proposed by the present method is very high as compared with dithizone (sensitivity being $0.0007 \mu\text{g Zn/cm}^2$).

Recommended Procedure. Pipette 10 ml of 1×10^{-2} M EDTA solution into a ground-glass flask, buffer to pH 4.0 by means of ammonium acetate/acetic acid followed by the addition of one drop of indicator solution (5 mg per 100 ml of chloro-

form). Titrate with zinc solution (1×10^{-2} M) taken from a microburette. Proceed slowly till the colour of the indicator turns from green to violet. Shaking is very necessary after each addition of zinc. In subsequent titrations the indicator solution is added just before the end point. From the titrimetric values the accuracy is determined by calculating percent error by the method of Ringbom [1].

Acknowledgement. One of the authors (SKS) is thankful to the Council of Scientific and Industrial Research for the appointment of Pool Officer.

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Precipitation of Chromium Oxinate from Homogeneous Solutions

Fällung von Chromoxinat aus homogener Lösung

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Received November 8, 1971/Revised February 21, 1972

It is known that chromium and oxine form a complex of definite composition [1], but systematic investigation on the analytical applications of the complex does not seem to have been made so far. As the technique of precipitation from homogeneous solution is an improved method of gravimetric analysis and gives more reliable results, this technique has been employed in our experiments.

Procedure for Precipitation of the Complex

Preliminary investigations showed that chromium(III) can be quantitatively precipitated as its oxinate complex by following any of the four homogeneous precipitation procedures given below:

a) *Precipitation by Urea Hydrolysis* [3]¹. The solutions containing about 10 mg of chromium(III), 8 ml of 2% oxine

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1 Before we could communicate the results of our investigations [4], a method for the precipitation of chromium oxinate by the urea hydrolysis method was reported [3]. The procedure was included in the present communication as some more studies were made on the precipitate obtained by following this procedure.