gens bei der Spurenanalyse solcher Reinstmetalle rechnen, auf die dieses Verfahren anwendbar ist.

Bisherige Ergebnisse unserer Arbeiten lassen erkennen, daß neben dem Normalpotential der Elemente in starkem Maße die Fähigkeit zur Amalgambildung eine Rolle für den Erfolg bei der Spurenanreicherung spielt. Wichtige Faktoren scheinen die Löslichkeit von Matrix und Spur in Quecksilber sowie die Benetzbarkeit der Elemente mit diesem Metall zu sein. Auch die Bildung von Mehrmetall-Amalgamen sowie von Mischphasen bei Matrix und Spur sind offensichtlich wichtig für die Frage, ob ein Element angereichert wird oder nicht.

Es besteht eine begründete Aussicht, daß wie in Aluminium, Cadmium und Zink Spurenverunreinigungen auch in den Reinstmetallen Blei, Wismut, Gallium, Indium, Zinn und anderen durch partielles Lösen der Matrix angereichert werden können. Detaillierte Ergebnisse unserer Untersuchungen sowie Arbeitsvorschriften für die Anreicherung und Bestimmung von Spurenelementen in Reinstmetallen, auf die dieses Verfahren anwendbar ist, werden wir in Kürze an dieser Stelle veröffentlichen.

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Direct Thermometric Determination of Calcium Based on the Substitution Reaction between Ca and Mg-EDTA

Direkte thermometrische Bestimmung von Calcium auf Grund der Substitutionsreaktion zwischen Ca und Mg-ÄDTA

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Received April 15, 1971

The principle of thermometric determination which has been proposed by Sajó and coworkers for the analysis of various materials has now been applied to the determination of Ca using the reaction between Ca and Mg-EDTA. Investigations have shown that this reaction produces a heat effect which is by 25% higher than that of the reaction with H_2 -EDTA. Furthermore, it offers the advantage of the heat of mixing being extraordinarily low and being completely eliminated by varying the pH. Linear relationship was obtained between heat of reaction and concentration. Selectivity of the method can be achieved by careful selection of the reaction conditions. A

neutral pH-value causes precipitation of trivalent and tetravalent ions as hydroxides, which do not react with the Mg-EDTA reagent. The high ammonium salt concentration employed prevents precipitation of Mn^{2+} and Mg^{2+} as hydroxides. Mn^{2+} may be eliminated as MnS , Mg^{2+} as oxinate.

Procedure. Dissolve the sample (containing not more than 400 mg of CaO) in 50 ml of acid mixture (100 ml of conc. HCl + 100 ml of conc. HNO_3 + 700 ml of water), add 50 ml of 20% NH_4Cl solution and neutralize to a faint odour of ammonia. (Filtration of precipitated R_2O_3 compounds is not required.) Fill a 10 ml immersion pipette with Mg-EDTA reagent (325 g of Na_2 -EDTA + 220 g of $MgSO_4 \cdot 7 H_2O$; neutralized with ~ 150 ml of 25% ammonia and diluted to 1 l) and carry on the determination according to the general working procedure described by Sajó [1].

Insoluble matter is treated as follows: Melt ~ 1 g of the sample in a silver crucible with 8 g of KOH, dissolve in water, acidify with 25 ml of conc. HCl, boil and neutralize with conc. ammonia to a faint odour. Proceed as described above.

Different samples of cements, clinkers, limestones, dolomites, slags and glass have been analyzed according to this method and for comparison also according to the classical procedure. Results obtained were in close agreement.

Reference

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1,5-Di- β -naphthylthiocarbazone as an Extractive Indicator for the Determination of Lead with EDTA

1,5-Di- β -naphthylthiocarbazon als Extraktionsindikator für die Bestimmung von Blei mit ÄDTA

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Received April 30, 1971

In the course of studies concerning reactions of metal ions with 1,5- β -naphthylthiocarbazone (HDNZ) it has been found that lead forms a pink complex with this compound, which is extractable into chloroform and carbon tetrachloride. The colour reaction is extremely sensitive.

Optimum pH range for different concentrations have been calculated from stability and reaction constants and have been verified by experiment. Other variables have been investigated and optimum conditions worked out.