Synergetic Extraction and Spectrophotometric Determination of Iron(III) in Steel

Synergetische Extraktion und spektralphotometrische Bestimmung von Eisen(III) in Stahl

Best. von Eisen(III) in Stahl; Spektralphotometrie; Extraktion mit Acetylaceton und Pyridin

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A simple and rapid procedure has been worked out for the determination of iron(III) in alloy steels comprising extraction into benzene with acetylacetone in combination with pyridine from acetate medium and spectrophotometric measurement. The optimal pH-range is between 3 and 5.5. Sensitivity according to Sandell is $0.13 \mu\text{g}/\text{cm}^2$ at 430 nm. The complex formed is stable for 48 h. Beer's law is obeyed from 5 to 60 μg of Fe(III)/10 ml. A large

number of foreign ions can be tolerated even in > 20 fold amounts. Cu^{2+} , UO_2^{2+} and Sn^{2+} can be tolerated in the ratio 1 to 2, whereas osmium, tellurite and EDTA inhibit the extraction. A main advantage of the method is the non-interference of Co^{2+} , Ni^{2+} and Zn^{2+} .

Procedure. Dissolve about 0.05 g of the steel sample in 9 ml of conc. sulphuric acid and 50 ml of water, add 10 ml of conc. nitric acid, heat almost to dryness and then dilute to 100 ml. Take an aliquot of the solution, reduce trivalent iron with 2 ml of aqueous 10% hydroxylamine hydrochloride solution, adjust the pH to about 5 by sodium acetate/acetic acid buffer and shake for 2 min with 10 ml of extracting solution (mixture of equal volumes of 1 M pyridine solution in benzene and of 0.6 M acetylacetone solution in benzene). After separation of the phases measure the absorbance of the organic one against benzene at 430 nm. Prepare a calibration curve by the same procedure.

The entire operation takes about 40 min. In analysing a steel with 90.471% of iron deviations of $\pm 0.007\%$ were found.

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A Carrier Distillation Method for the Estimation of Non-Rare Earth Impurities in Y_2O_3 without Using an External Atmosphere

Träger-Destillationsverfahren zur Bestimmung von Nicht-SE-Verunreinigungen in Y_2O_3 ohne äußere Atmosphäre

Best. von Verunreinigungen in Yttriumoxid; Spektrometrie, Emission; Träger-Destillation

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When analysis of the non-rare earth impurities in Y_2O_3 is required, its complex dc arc spectrum makes it necessary either to chemically separate out the impurities and analyse the residue spectrographically or use the carrier distillation technique to suppress the matrix spectrum. The latter approach was adopted by Muntz [1] and also by Osumi *et al.* [2] and in both cases neck type preformed electrodes in conjunction

with an external atmosphere consisting of a mixture of argon and oxygen were used.

Our experiments with different types of electrodes and atmospheres proved that when the type of electrode (S-14)¹ employed by Muntz is used the carrier action is obtained only when that mixture of argon and oxygen is used. But even in open air the matrix spectrum and its accompanying background is almost completely suppressed when S-3¹ electrode is used in place of S-14. The use of this electrode also completely eliminates the problem of ejection of samples from the arc encountered by Muntz with most of the carriers.

Different carriers were also tried and it was found that a mixture of NaF, AgCl and graphite (1:4:5) gave a good overall sensitivity for all the elements. The ratio of AgCl to NaF was chosen in such a way that NaF is effective for the first half of the exposure time of 35 sec. The disappearance of sodium from the arc was marked by the change of colour of the arc and the sudden rise of voltage drop across the arc gap.

Experimental

Procedure. Y_2O_3 is mixed with carrier in the ratio 3:1 and ground in an agate mortar. 35 mg of this mixture are loaded in a S-3 graphite electrode which is used as the anode and

¹ ASTM designation.