CCA-644

545.82:546.811
Note

Spectrographic Trace Analysis of Tin in the Presence of Large Amounts of Iron

Š. Cerjan-Stefanović, S. Turina, and V. Marjanović

Institute of Inorganic and Analytical Chemistry, University of Zagreb, Zagreb, Yugoslavia

Received January 22, 1970

It is possible to lower the inferior limit of spectrographic trace analysis of Sn in iron and steel[1] if the Sn is separated by coprecipitation from the solution obtained by dissolving the sample in a mineral acid.

The coprecipitation of Sn^{2+} [2,3] was in these experiments tested with CdS which is formed in the investigated solution. The amount of Cd^{2+} must be known because the spectrographic lines of Cd were used as reference lines, *i. e.* as an internal standard.

EXPERIMENTAL

The sample — about 1 g or more — was dissolved in 20—25 ml of dilute sulphuric acid (1 : 4). After dissolution of the sample, a mixture containing 100 mg of Cd-acetate, 1 g of Na-acetate, 10 ml of acetic acid and 100 ml of distilled water was added. The CdS was precipitated by introducing H_2S into the solution.

After filtration and burning the precipitate, the residue was mixed with graphite powder (spectrographically pure) in the ratio about 1 : 1. With this powder the graphite electrode shown on the Fig. 1 a was filled. The counter electrode (graphite) is shown on Fig. 1 b.

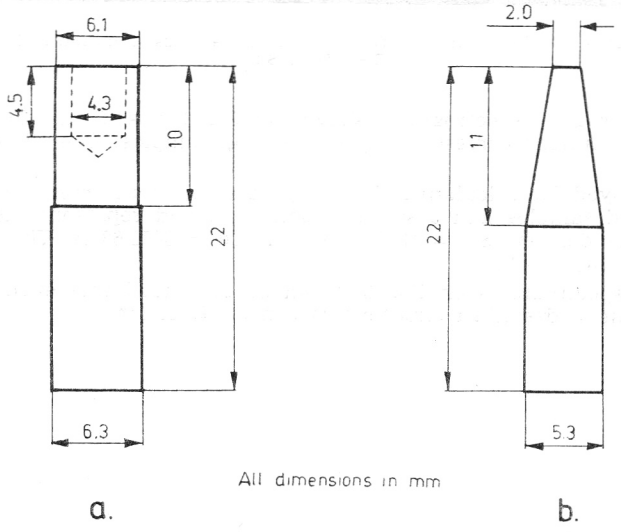

Fig. 1.*a*. Graphite electrode filled with a mixture of the graphite powder and the investigated precipitate; *b*. The counter electrode (graphite).

Spectrographic analyses were done on a »Zeiss Plangiter Spectrograph — PGS — 2«. The working conditions in performing spectrographic analyses were the following:

Excitation: Arc A. C. 4 Amp.

Time of excitation: 30 sec.

Slit: 0.03 mm

Registration: Microfilm (Fotokemika, Zagreb).

Density of Sn and Cd spectrographic lines on the obtained spectrograms was measured by means of a Zeiss-Schnellphotometer.

The experiments were carried out with standard samples and simulate solutions. The amounts of Sn in simulate solutions were equivalent to those in dissolved samples containing Sn in the range from 0.01 to 0.1%.

The spectrograms shown on Fig. 2 were obtained directly from the iron samples (A — 0.0035% Sn and B — 0.051% Sn). The Sn spectrographic lines were overlapped by the Fe lines and the measuring of Sn was impossible.

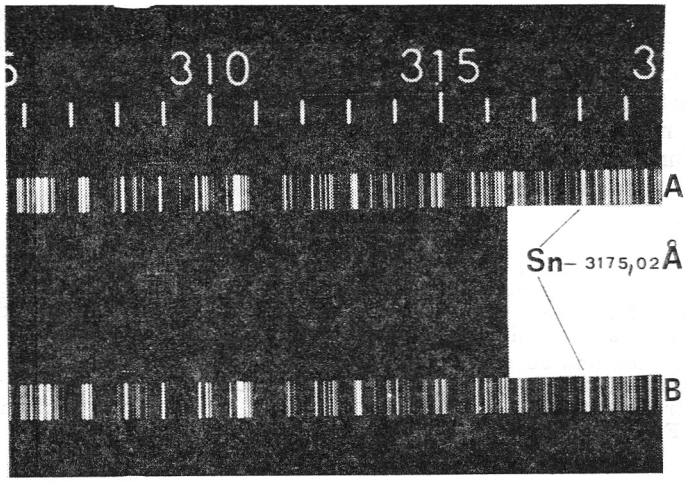

Fig. 2. Spectrograms obtained directly from the iron samples containing Sn (A—0.0035% Sn; B—0.0510% Sn).

The samples gave spectrograms shown on Fig. 3 pos. C and D were obtained from simulate solutions corresponding to the iron samples: C — 0.0015% and D — 0.005% Sn.

Data employed for calculating the standard error of the method[4] were obtained from 2 standard samples and 8 simulate solutions, and represent the difference of Sn and Cd lines density (Sn) = 3175.02 Å and (Cd) = 3082.68 Å. The standard error of the method was 10—12%.

From the spectrograms on Fig. 3 it can be concluded that even smaller quantities of Sn than in described experiments can be determined.

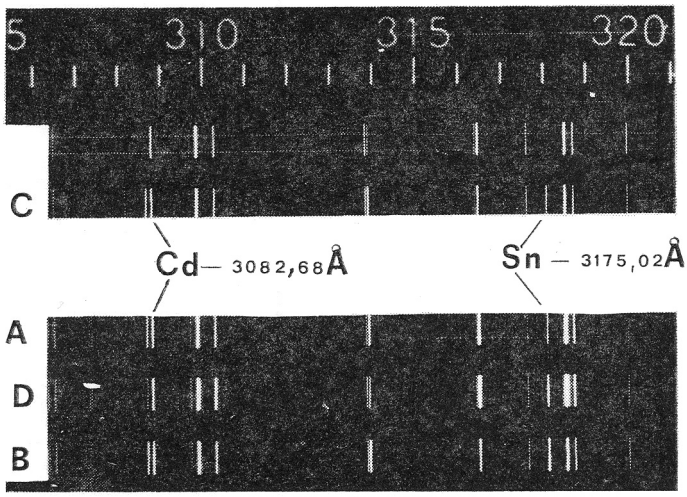

Fig. 3. Spectrograms obtained by the separation of Sn from the samples (A—0.0035% Sn; B—0.0510% Sn; C—0.0015% Sn and D—0.0050% Sn).

REFERENCES

1. R. V. Rilley, *Spectrochim. Acta* **4** (1950) 93.
2. C. Feldman, *Anal. Chem.* **21** (1949) 1211.
3. V. D. Pisarev, *Zavodsk. Lab.* **22** (1956) 462.
4. J. M. Lopez, *Spectrochim. Acta* **4** (1951) 265.

IZVOD

Spektrografsko određivanje tragova kositra u prisutnosti velikih količina željeza

Š. Cerjan-Stefanović, S. Turina i V. Marjanović

Donja granica spektrografskog određivanja tragova kositra u čeliku i željezu može se sniziti ako se prethodno kositar sutaloženjem odvoji iz otopljenog uzorka. Ispitano je sutaloženje kositra s kadmijevim sulfidom formiranim neposredno u ispitivanoj otopini. Kadmij, dodan u određenoj količini, služi kao interni standard.

Ovim postupkom omogućeno je određivanje kositra u 0,001%-tnoj koncentraciji, za red veličine nižoj od koncentracije kod direktne spektrografske analize.

INSTITUT ZA ANORGANSKU I ANALITIČKU KEMIJU
SVEUČILIŠTA U ZAGREBU

Primljeno 22. siječnja 1970.