Kurze Mitteilungen

Spectrographic Determination of Boron and Silicon in Uranium Tetrafluoride

Spektralanalytische Bestimmung von Bor und Silicium in Urantetrafluorid

Best. von Bor und Silicium in Urantetrafluorid; Spektralanalyse; ZnO-Zusatz

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For the spectrographic determination of impurity elements in UF_4 , samples are usually converted into U_3O_8 through pyrohydrolysis and the ordinary carrier distillation methods of U analysis can be used [3]. This procedure is not feasible, however, for impurity elements which form highly volatile fluorides such as B and Si, that are lost during the pyrolytic transformation. Methods of direct spectrographic analysis of UF_4 either with no carrier [1] or with Al_2O_3 to suppress the volatilization of U [2] have been described in the literature. With the former method we have obtained a poor reproducibility for Si because U spectrum appears before Si is completely volatilized and in the latter one the limit of detection of B is 0.5 ppm. Since in our case a lower limit was necessary (0.2 ppm), a number of materials were considered to delay U volatilization and improve sensitivity. The use of ZnO has been found to be optimum.

With the use of ZnO the volatilization times of B and Si remain practically unchanged (~ 10 sec) while the volatilization of U is delayed and U intensity becomes lower.

Experimental

Apparatus and excitation parameters are as follows:

Electrodes	Scribner type (Ultra Carbon 1990 and 1992)
Analytical gap	4 mm
Charge	100 mg tamped and pierced with a venting tool
Excitation unit	Jarrell-Ash Varisource, DC arc 10 A
Spectrograph	Hilger Littrow automatic large, quartz optics
Illumination	Arc image focused on slit by 64 mm focal length lens
Wavelength range	2200–2800 Å
Slit	22 μ wide and 1 mm high
Preburn	None
Exposure	10 sec

Emulsion	Kodak S.A. No. 1
Developer	Kodak D-19b, 21°C, 4 min
Fixer	Kodak F-5, 21°C, 4 min
Plate calibration	Two-step method, iron arc exposure
Microphotometer	Jarrell-Ash digital comparator densitometer
Analytical lines	B 2497.7 Å Si 2435.2 Å

When employing a UF_4 sample containing 0.3 ppm B and 15 ppm Si a ZnO concentration of 35% has been found to be most suitable. DC arc currents lower than 10 A delay the excitation of U, but also decrease unsatisfactorily the impurity line intensities.

The detection limits are 0.1 ppm B and 5 ppm Si, the latter caused by residual impurity in the UF_4 matrix. With a sample containing 0.3 ppm B and 15 ppm Si, the standard deviations obtained by 20 repetitive measurements are $\pm 9.9\%$ and $\pm 9.6\%$, respectively. Linear calibration curves are obtained from 0.1–2 ppm B and 5–100 ppm Si.

A research was also undertaken concerning the reactions in the anode cavity, accounting for the delay in the volatilization of uranium when ZnO is added. Summed up the results revealed that the formation of UO_2 and a fluorine, uranium and zinc compound takes place, both less volatile than UF_4 . This allows the uranium intensity in the uranium volatilization curve to be lower and the complete uranium volatilization to take a longer time.

References

1. National Lead Company of Ohio, Ed., Analytical chemistry manual of the feed materials production center, Report TID-7022, Vol. II, December 1964.
2. Podobnik, B., Spenko, M.: Anal. Chim. Acta **34**, 294 (1966).
3. Scribner, B. F., Mullin, H. R.: J. Res. Nat. Bur. Std. **37**, 379 (1946).

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Berichtigung

F. Lux und R. Zeisler:
Instrumentelle Multi-Element-Aktivierungsanalyse von biologischem Gewebe und ihre Anwendung zur Metallose-Untersuchung.
Diese Z. **261**, 314–328 (1972).

In der letzten Zeile der rechten Spalte auf S. 318 muß es richtig heißen:
„... ein Kobaltgehalt von 50 ppm vorgetäuscht ...“