BULLETIN OF THE CHEMICAL SOCIETY OF JAPAN, VOL. 46, 3595—3596 (1973)

Indirect Determination of Tantalum by Atomic Absorption Spectrophotometry

Tsutomu Matsuo, Junichi Shida, and Seiichi Kudo
Department of Applied Chemistry, Faculty of Engineering, Yamagata University, Jonan-4, Yonezawa, Yamagata 992

(Received May 22, 1973)

Reports have recently been given on indirect determination of arsenate,$^{1)}$ germanate,$^{2,3)}$ phosphate,$^{4,5)}$ silicate,$^{5)}$ and niobate,$^{6)}$ based on the extraction of heteropolymolybdate with the organic extractant and subsequent measurement of the molybdenum contained in the extract by atomic absorption spectrometry. Guyon *et al.* reported the spectrophotometric methods for determination of niobium$^{7)}$ and tantalum,$^{8)}$ in which heteropolymolybdate complex was used. The authors investigated an indirect determination of tantalum by atomic absorption spectrometry. The essential steps in this method are; formation of molybdotantalate, extraction of the molybdotantalate with methyl isobutyl ketone (MIBK) and measurement of molybdenum by aspirating the MIBK extract directly into the burner of the atomic absorption spectrophotometer.

Experimental

Apparatus. Atomic absorption spectrometric measurements were carried out with a Hitachi model 207 atomic absorption spectrophotometer equipped with a molybdenum hollow cathode lamp (Hitachi, HLA-3) as a light source at 3133 Å molybdenum line. Lamp current was 15 mA. An air-acetylene flame was used with flow rates 13 l/min and 3 l/min.

Standard Tantalum Solution (1.0×10^{-2} *M*). Dissolve 0.1809 g of tantalum metal (99.7%) in hydrofluoric acid containing a small amount of nitric acid and evaporate it to dryness. Add 5 ml of hydrofluoric acid and 5 ml of sulfuric acid, and heat until fumes of the sulfuric acid are evolved. After cooling, dilute to 100 ml with distilled water.

Procedure. Transfer 10 ml of 0.2 M ammonium molybdate solution to a 100 ml separatory funnel, add 2 ml of 6 M hydrochloric acid and swirl to mix. Add known amounts of tantalum solution and dilute with distilled water to 20 ml. After standing the solution 15 min, add 20 ml of MIBK and shake vigorously for 5 min. Separate the layers, wash the MIBK extract three times with 25 ml portions of 0.6 M hydrochloric acid and measure molybdenum by aspirating the MIBK extract of molybdotantalate into the burner of the atomic absorption spectrophotometer. A blank containing all the reagents except the tantalum sample is prepared by a similar procedure.

Results

Acidity for Extraction of Molybdotantalate. The optimum acidity of aqueous solution for the extraction of molybdotantalate with MIBK was examined. The results are shown in Fig. 1. The acidity range 0.5—0.7 M hydrochloric acid was found to be suitable, the absorbance value being constant in this range.

Effect of Molybdate Concentration. The effect of ammonium molybdate for the extraction of molybdotantalate was studied. The results are shown in Fig. 2. A constant absorbance value was obtained when 9 ml of 0.2 M ammonium molybdate was added.

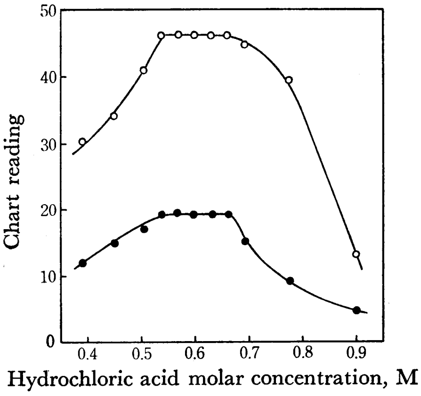

Fig. 1. Effect of acidity on extraction of heteropolymolybdates.
—○— Ta^{5+}; 2×10^{-5}M, —●— Nb^{5+}; 4×10^{-5}M

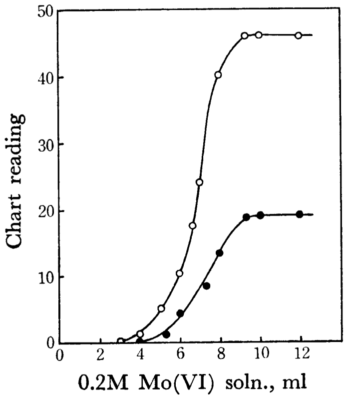

Fig. 2. Effect of amounts of 0.2M Mo(VI) soln.
—○— Ta^{5+}; 2×10^{-5}M —●— Nb^{5+}; 4×10^{-5}M

---

1) Y. Yamamoto, N. Kumamaru, Y. Hayashi, A. Kanke, and A. Matsui, *Bunseki Kagaku*, **21**, 379 (1972).

2) R. Jakubiec and D. F. Boltz, *Anal. Chem.*, **41**, 78 (1969).

3) T. Matsuo, J. Shida, and S. Kudo, *Bunseki Kagaku*, **22**, 1009 (1973).

4) G. F. Kirkbright, A. M. Smith, and T. S. West, *Analyst* (London), **92**, 411 (1967).

5) T. R. Hurford and D. F. Boltz, *Anal. Chem.*, **40**, 379 (1968).

6) G. F. Kirkbright, A. M. Smith, and T. S. West, *Analyst* (London), **93**, 292 (1968).

7) J. C. Guyon, G. W. Wallace, Jr., and M. G. Mellon, *Anal. Chem.*, **34**, 640 (1962).

8) J. C. Guyon, *Anal. Chim. Acta*, **30**, 395 (1964).

In the determination of tantalum, 10 ml of molybdate solution was used.

Washing of the MIBK-Extract. Molybdotantalate was extracted into MIBK with a trace of excess molybdate.[2,3] To remove the trace of excess molybdate, washing of the MIBK extract with hydrochloric acid was examined. Washing three times with 25 ml portions of 0.6 M hydrochloric acid solution was sufficient to remove the excess molybdate. Shaking time for washing was 3 min.

Effect of Standing Time. The rate of formation of heteropolymolybdate was relatively slow at room temperature. The effect of standing time prior to extraction of molybdotantalate was examined. Fifteen minutes was found to be the best for quantitative extraction.

Stability of Extract. No change in absorbance value was observed for at least two hours in the MIBK extract.

Calibration Curve. The calibration curve of absorbance value *versus* concentration of tantalum was linear for 2.5×10^{-6}—2.0×10^{-5} M of tantalum.

Effect of Diverse Ions. An atomic absorption spectrometric method for the determination of niobium based on the formation of molybdoniobophosphate was reported by Kirkbright *et al.*[6] Niobium also forms molybdoniobate, which should be removed before the tantalum determination. It was found that niobium could be determined by the proposed method (Figs. 1 and 2) and that the linear relationship was obtained in the niobium concentration range 1.0×10^{-5}—5.0×10^{-5} M. The sensitivity of molybdoniobate is about five times less than that of molybdotantalate. Heteropolymolybdates which give positive errors are arsenate, germanate, phosphate and silicate.