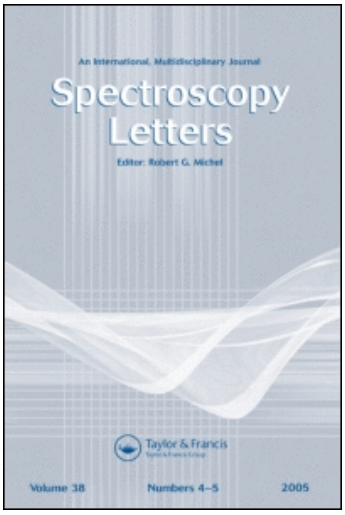

Spectroscopy Letters
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597299

Spectrochemical Determination of Microimpurities in Mercury

T. Oreshkov[a]; G. Gospodinov[a]
[a] Central Research Laboratory, Higher Institute of Chemical Technology, Burgas, Bulgaria

To cite this Article Oreshkov, T. and Gospodinov, G.(1990) 'Spectrochemical Determination of Microimpurities in Mercury', Spectroscopy Letters, 23: 9, 1195 — 1201
To link to this Article: DOI: 10.1080/00387019008054493
URL: http://dx.doi.org/10.1080/00387019008054493

SPECTROSCOPY LETTERS, 23(9), 1195-1201 (1990)

SPECTROCHEMICAL DETERMINATION OF MICROIMPURITIES IN MERCURY

Key words: Mercury, Mercury (II) iodide, spectrochemical analysis.

T. Oreshkov and G. Gospodinov

Higher Institute of Chemical Technology, Central Research Laboratory, 8010 Burgas, Bulgaria.

ABSTRACT

Mercury together with the microimpurities in it had been turned into iodides in advance in a vacuum ampule in the presence of collector - spectrally pure carbon powder, containing 2 per cent Li (as Li_2CO_3). The obtained mercury (II) iodide was separated from the microimpurities by sublimation at 100°C and the collector with the microimpurities was subjected to spectral analysis. The method allows the determination of 20 elements in mercury withim detection limits of 10^{-5} to 10^{-7}% and relative standard deviation 0.08 to 0.25. The method can be applied for the analysis of mercury (II) iodide, too.

INTRODUCTION

For lowering the detection limits in the analyses of mercury, physico-chemical methods for preliminary concentration of the microimpurities are often used. There are methods described for separating mercury from the microimpurities in it by distillation of mercury in the form of mercury (II) oxide in oxygen medium [1-3]. The main disadvantage of these methods is the fact that small amounts of mercury remain with the impurities and when evaporating the sample from the electrode, the toxic mercury vapours pollute the spectral apparatus and the environment.

There are also spectrochemical methods known to use extractive separation and concentration of microimpurities from mercury [4-10]. These methods require the use of large amounts of particularly pure chemical reagents. Additional pollution of the sample is also possible because of repeated dissolving and extraction. Together with mercury, some of the microimpurities are extracted partially or completely, and the concentrate obtained usually contains some amount of mercury, too.

The aim of the present study was to work out a spectrochemical method for determination of microimpurities in mercury. The method has to be easy to carry out, has to give reproducible results withim detection limits comparable to those of the best known methods and to exclude pollution with mercury.

EXPERIMENTAL

Concentration of Microimpurities

Mercury together with the microimpurities elements present in it were turned into iodides in advance. Iodine - superpure was used, purified in advance by a triple sublimation through filtering glasscloth. The synthesis of iodides took place in a two-section quartz ampule 150 mm long and 8 mm in diameter. At one end of the ampule there was an enlargement 16 mm in diameter and 30 mm long. The ampule had been washed with aqua regia and rinsed with deionized water. In the enlarged end of the well washed and dried ampule 10 g of mercury and 12.7 g of iodine were placed and covered with 100 mg of collector - spectrally pure carbon powder containing 2% Li (as Li_2CO_3). The end of the ampule with the sample was put into a Dewar flask filled with liquid nitrogen. The ampule was vacuumed and sealed and then put into a horizontal tunnel furnace. The furnace was heated at a rate of 1.6° per minute up to $200^{\circ}C$, at which temperature mercury reacted with iodine and changed completely to mercury (II) iodide for 6 hours. The mercury (II) iodide obtained was separated from the collector by sublimation. For the purpose, the end of the ampule with the sample was put into the furnace at $100^{\circ}C$. Mercury (II) iodide sublimated and condensed in the cool end of the ampule, which was outside the furnace, at room temperature. Then the ampule was cut, the

collector containing the microimpurities was taken out and subjected to spectral analysis. The concentration coefficient of the sample was $C_{smple} = 100$.

The method can be applied for the analysis of mercury (II) iodide, too. For the purpose, 10 g of mercury (II) iodide were placed in a quartz ampule and covered with 100 mg of collector. The ampule was vacuumed and sealed. Mercury (II) iodide was separated from the collector by sublimation, as was described above.

Preparation of Standard Specimens

As a base for preparing the standard specimens spectrally pure carbon powder, containing 2 per cent Li (as Li_2CO_3), was used. There were a main standard specimen, containing 0.1% Cu, Ag, Mn, Fe, Ca, Mg, Co, Bi, Pb, Cr, Cd, Au, Zn, Sn, Ni, Tl, Sb, Al, In and Ga, introduced in the form of their respective iodides. The standard specimens with elements content $1x10^{-2}$, $3x10^{-3}$, $1x10^{-3}$, $3x10^{-4}$, $1x10^{-4}$, $3x10^{-5}$ and $1x10^{-5}$ %, prepared by consecutive dilution of the standard specimens with pure base.

Conditions of Spectral Analysis

For the analysis carbon electrodes type RW I (Ringsdorff, BRD) with diameter of the crater 4.0 mm and depth 3.5 mm were used. The upper electrode was sharpened in the form of a truncated cone, 2.0 mm in diameter at the top. Two electrodes of each standard specimen and of

each investigated sample were burned in an AC arc with a current intensity of 16 A. Spectra were photographed with a ISP-30 (USSR) spectrograph having a three-lens illumination system. The width of the spectrograph slit was 0.012 mm, and of the intermediate diaphragm 3.2 mm. The discharge gap was 2.0 mm and the exposure time 90 sec. The spectra were registered on spectral photographic plates Blau Hart WU-2 (ORWO, DDR). The calibration graphs were built in log I/log C coordinates, taking into account the backround correction to the spectral lines. The limits of detection of a particular elements, achieved by this method, determined by the Kaiser criterion with $P = 0.997$, where P is the probability, and reproducibility of the analysis results are given in Table 1.

Losses During the Concentration of Microimpurities

The possible losses of some microimpurities during the sublimation of mercury (II) iodine were studied experimentally. For this purpose, particularly pure mercury (II) iodide was obtained by a triple consecutive sublimation at 100°C, using spectrally pure carbon powder and filtering glasscloth. 22.7 g of superpure mercury (II) iodide were mixed with 100 mg of the standard specimen, containing 1×10^{-3}% of the elements. The mixture was put in an ampule and treated in the described way. For Cu, Ag, Mn, Fe, Ca, Mg, Co, Bi, Pb, Cr, Cd, Au, Zn, Sn, Ni and Tl the results were withim the error limits of the method, which shows a practical lack of losses.

TABLE 1

Limits of detection and reproducibility of results in the analysis of mercury and mercury (II) iodide

Element and analytical line, nm	Limit of detection, %	Relative standard deviation, S_r
Cu I 327.40	1×10^{-7}	0.14
Ag I 328.07	1×10^{-7}	0.22
Mn I 279.48	3×10^{-7}	0.18
Fe I 302.06	5×10^{-7}	0.09
Ca II 317.93	1×10^{-5}	0.17
Mg I 285.21	1×10^{-7}	0.25
Co I 304.40	3×10^{-7}	0.11
Bi I 306.77	1×10^{-6}	0.17
Pb I 283.31	3×10^{-7}	0.18
Cr II 284.32	5×10^{-7}	0.12
Cd I 326.11	1×10^{-6}	0.14
Au I 267.60	5×10^{-7}	0.08
Zn I 334.50	3×10^{-5}	0.13
Sn I 284.00	1×10^{-7}	0.17
Ni I 305.08	5×10^{-7}	0.10
Tl I 276.79	1×10^{-6}	0.23
Sb I 259.81	1×10^{-7}	0.15
Al I 309.27	5×10^{-7}	0.19
In I 303.94	3×10^{-7}	0.25
Ga I 294.36	3×10^{-7}	0.24

Partial losses were established for Sb, Al, In and Ga. They can be multiplying the results of the analysis by the respective coefficients of losses during sublimation of mercury (II) iodide. It was established experimentally that these coefficients are 1.8 for Sb and Al, and 1.5 for In and Ga.

REFERENCES

1. Mandelshtam, S.L., Semenov, N.N., Turovtseva, Z.M., J. Anal. Chem. (USSR), 11, 9, (1956).

2. Zaidel, A.N., Kalitevski, N.I., Lipis, L.V., Tchaika, M.P., Belyaev, Yu.I., J. Anal. Chem. (USSR), 11, 21, (1956).

3. Gorbatcheva, A.M., Grinzaid, E.L., Trudy Leningradsk. Polytechn. Instituta (USSR), 201, 77, (1959).

4. Bock, R., Kusch, H., Bock, E., Z. Anal. Chem., 138, 167, (1953).

5. Morris, D.F.C., Williams,J.M., Talanta, 9, 623,(1962).

6. Jackwerth, E., Z. Anal. Chem., 206, 269, (1964).

7. Tiptsova, V.G., Malkina, E.I., Anisimova, Z.A., J. Anal. Chem. (USSR), 21, 459, (1966).

8. Tiptsova - Iakovleva, V.G., Dvortsan, A.G., J. Anal. Chem. (USSR), 24, 1141, (1969).

9. Tchanisheva, T.A., Yudelevitch, I.G., Mihailov, V.A., "Novie metodi instrumentalnogo analiza materialov", Moskwa, 35, (1979).

10. Kulish, N.G., Tshanisheva, T.A., Verevkin, G.V., Yudelevitch, I.G., Visokotchistie veshtestva (USSR), 1, 178, (1989).

Date Received: 06/25/90
Date Accepted: 07/28/90