Journal of Organometallic Chemistry, 51 (1973) 237–239

TELLURIUM ANALYSIS IN ORGANOTELLURIUM COMPOUNDS BY ATOMIC ABSORPTION SPECTROSCOPY

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(Received August 16th, 1972)

SUMMARY

The application of atomic absorption spectroscopy to the determination of tellurium in organotellurium compounds is described. The samples are decomposed using a mixture of concentrated nitric and perchloric acids and an aqueous solution of telluric acid is used as the standard.

INTRODUCTION

The analysis of tellurium in alkyltellurium compounds has been achieved by Drew and Porter[1] using a gravimetric method. The new and more general gravimetric method developed by Tsao[2] and the volumetric method of Kruse *et al.*[3] for the analysis of tellurium in organotellurium compounds suffered from complexity of the procedure and the need for sufficiently large quantities of the material, respectively.

The desirability of a less time-consuming and more reliable method led us to explore the possibility of employing the versatile tool atomic absorption spectroscopy. This method has been successfully used for the analysis of tellurium in its alloys[4,5]. However, a modified procedure in the preparation of solutions is necessary for the application of this technique for organotellurium compounds.

Various organotellurium compounds of the type $RTeX_3$ (R = Ph, p-MeC_6H_4, p-$MeOC_6H_4$, p-$EtOC_6H_4$ and p-$PhOC_6H_4$; X = Cl, Br and I), (p-$EtOC_6H_4$)ClTeO and $[(p\text{-}EtOC_6H_4)TeCl_4]^-[C_5H_5NH]^+$ were studied in order to evaluate the applicability of the procedure.

EXPERIMENTAL

Instrument

The Perkin–Elmer Model 303 Atomic Absorption Spectrophotometer equipped with a readout unit was used. The tellurium hollow-cathode lamp was operated at 40 mA. An air/acetylene flame of flow rate 9 units was used. The absorptions were measured at 214 nm.

Reagents

Telluric acid (AR, H_6TeO_6), concentrated nitric acid (AR, sp.gr. 1.42), con-

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centrated perchloric acid (AR, 71–72%) and concentrated hydrochloric acid (AR).

The stock solution containing 1000 µg Te/ml was prepared by dissolving telluric acid 1.7951 g in distilled water containing hydrochloric acid (10 ml). The standard solutions for calibration were obtained by appropriate dilution of the stock solution within the range 6–20 µg Te/ml.

Procedure

A mixture of concentrated nitric acid (3–4 ml) and concentrated perchloric acid (3–4 ml) was added to the accurately weighed sample (15–50 mg) contained in a beaker (50 ml or 100 ml). The mixture was carefully heated on a hot plate and the acids were allowed to evaporate slowly (about $1\frac{1}{2}$ h) in an efficient fume-cupboard. The white residue obtained was dissolved in warm distilled water to which concentrated hydrochloric acid (about 0.5 ml) was added. The solution was cooled and made up in a volumetric flask (50 ml or 100 ml). The solution was diluted again within the range 7–18 µg Te/ml. The % absorption of the dilute solution was then determined and the concentration read off from the calibration graph. The calibration graph was obtained from freshly prepared standard solutions before each set of determinations.

The results obtained are given in Table 1.

TABLE 1

DETERMINATION OF TELLURIUM IN ORGANOTELLURIUM COMPOUNDS[a]

Compounds	*Analysis of Te found (calcd.) (%)*
$(p\text{-MeC}_6\mathrm{H}_4)\mathrm{TeCl}_3$	39.16 (39.25)
$(p\text{-MeC}_6\mathrm{H}_4)\mathrm{TeBr}_3$	27.52 (27.83)
$(p\text{-MeC}_6\mathrm{H}_4)\mathrm{TeI}_3$	20.96 (21.28)
$(p\text{-EtOC}_6\mathrm{H}_4)\mathrm{TeCl}_3$	35.60 (35.90)
$(p\text{-EtOC}_6\mathrm{H}_4)\mathrm{TeBr}_3$	26.47 (26.12)
$(p\text{-EtOC}_6\mathrm{H}_4)\mathrm{TeI}_3$	20.73 (20.23)
PhTeBr_3	28.30 (28.70)
PhTeI_3	21.58 (21.80)
$(p\text{-MeOC}_6\mathrm{H}_4)\mathrm{TeCl}_3$	37.40 (37.40)
$(p\text{-MeOC}_6\mathrm{H}_4)\mathrm{TeBr}_3$	27.37 (26.89)
$(p\text{-MeOC}_6\mathrm{H}_4)\mathrm{TeI}_3$	20.27 (20.27)
$(p\text{-PhOC}_6\mathrm{H}_4)\mathrm{TeCl}_3$	31.51 (31.64)
$(p\text{-EtOC}_6\mathrm{H}_4)\mathrm{TeOCl}$	42.85 (42.53)
$[(p\text{-EtOC}_6\mathrm{H}_4)\mathrm{TeCl}_4]^- [\mathrm{C}_5\mathrm{H}_6\mathrm{N}]^+$	26.73 (27.20)

[a] All the compounds gave satisfactory C and H analysis.

DISCUSSION

The successful application of atomic absorption spectroscopy for the determination of tellurium in organotellurium compounds is clearly indicated by the results as shown in Table 1. The modified procedure has been found suitable for a wide variety of compounds. This method has been found to be very reliable and less time-consuming than those previously reported. It also has the great advantage that several determinations can be carried out at the same time. The amount of sample required is only 25–50 mg, and this could be reduced further for compounds containing a higher percentage of tellurium.

The preparation of the stock solution using telluric acid[6] was found to be more convenient than the recommended procedure[7] using tellurium metal.

ACKNOWLEDGEMENTS

The author wishes to thank the Colombo Plan for a scholarship, and Chiengmai University (Thailand) for the study leave.

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