Journal of Organometallic Chemistry, 118 (1976) 55—58
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A RAPID PROCEDURE FOR THE DETERMINATION OF THE TELLURIUM CONTENT OF ORGANOTELLURIUM COMPOUNDS

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(Received March 22nd, 1976)

Summary

A rapid micro-analytical procedure for the analysis of tellurium in organotellurium compounds is described. The compounds are decomposed using the conventional oxygen flask method followed by treatment with aqueous hydrogen peroxide/hydrochloric acid solution. The tellurium content of the resulting solution is determined by atomic absorption spectrophotometry. If the analyses are carried out batch wise i.e. 6—8 samples with each standardisation of the instrument, the total analysis time is about ½ hour per sample.

Thavornyutikarn [1] has reviewed some of the methods which have been published for the determination of the tellurium content of organotellurium compounds. These gravimetric and titrimetric methods [2—4,] are time-consuming and invariably need large sample weights. Thavornyutikarn [1] also described a new procedure applicable to aryltellurium halides in which smaller sample weights (15—50 mg) were decomposed with a mixture of 3—4 ml of nitric acid and 3—4 ml of perchloric acid. The decomposition procedure takes about one and a half hours and an efficient fume cupboard is required. The white residue obtained is subsequently dissolved in hydrochloric acid, the solution made up to a convenient volume in a volumetric flask and the absorption of this solution measured by atomic absorption spectrophotometry.

A large number of tellurium compounds are prepared in our laboratory and there is a need for a reliable, reproducible method which is not time consuming. The oxygen flask method [5—10] is a well known procedure for the fast decomposition of organic materials and so it was decided to investigate its applicability to a wide range of organotellurium compounds. The aqueous solutions so obtained would be free from high concentrations of mineral acid and would give an ideal matrix for subsequent examination by atomic absorption spectro-

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photometry. The decomposition of organoselenium compounds by oxygen flask followed by analysis by the molecular emission cavity technique (MECA) has been recommended by Belcher et al. and the results compared with those obtained by atomic absorption [11]. Gubser [12] however states that selenium attacks the platinum gauze and this leads to inaccurate results.

For our study on application of the oxygen flask technique to tellurium containing compounds a wide range of organotellurium compounds were prepared. The list of selected compounds consisted of diaryl tellurides, diaryltellurium dihalides, aryltellurium trihalides, aryltellurium trihalide complexes with sulphur donor ligands and tellurium tetrahalide complexes. The analytical results obtained are given in the experimental section.

Experimental

Reagents. "Analar" telluric acid (purity 99.5%) used as a standard was obtained from Hopkins and Williams Ltd, and hydrogen peroxide (50%) was obtained from Fisons Ltd.

All tellurium compounds were also analysed for carbon and hydrogen and gave satisfactory analyses.

Standard Te solution. The stock solution of 1000 μg Te ml^{-1} was prepared by dissolving telluric acid 1.7951 g in distilled water containing conc.hydrochloric acid (10.0 ml). The calibration curve was obtained by dilution of this solution to give standards within the range 5—50 μg Te ml^{-1}.

Procedure. The accurately weighed sample (5—10 mg) contained in a wrapped filter paper is fixed into a hinge of a platinum gauze and then ignited in a 500 ml flask which has previously been flushed with oxygen, and which contains 15.0 ml of water, and 1.0 ml of hydrogen peroxide and 1.0 ml of hydrochloric acid. After ignition, the flask is shaken until the decomposition products are absorbed in the aqueous peroxide/hydrochloric acid solution which is transferred to a volumetric flask and the volume made up to 50.0 ml with distilled water. The percentage absorption of this solution or a suitable further dilution is determined by atomic absorption. We used a Perkin—Elmer 303 instrument equipped with a Perkin—Elmer recorder (Model No. 56), and a Fischer—Porter Burner regulator (air flow rate 9, acetylene 7—8). The absorptions were measured at 214.2 nm using an Activion tellurium hollow cathode lamp operated at 10 mA.

The platinum gauze may be used for about 10 combustions but then becomes brittle.

Results

Required values are in parentheses: $C_6H_5TeCl_3$ 41.15 (41.04); $C_6H_5TeBr_3$ 28.72 (28.72); p-$CH_3C_6H_4TeCl_3$ 39.02 (39.27); p-$CH_3C_6H_4TeBr_3$ 29.97 (27.84); p-$C_2H_5OC_6H_4TeBr_3$ 26.10 (26.13); p-$C_6H_5OC_6H_4TeCl_3$ 31.46 (31.65); p-C_6H_5-$OC_6H_4TeBr_3$ 23.72 (23.78); $(CH_3OC_6H_4)_2TeCl_2$ 30.91 (30.93); $(C_2H_5OC_6H_4)_2$-$TeCl_2$ 28.89 (28.96); $(CH_3OC_6H_4)_2Te$ 37.14 (37.35); $(C_2H_5OC_6H_4)_2Te$ 34.46

(34.52); $TeCl_4$tmdto * 28.95 (28.65); $TeBr_4$tmdto 20.39 (20.47); $C_6H_5TeCl_3$tmdto 26.15 (26.20); $C_6H_5TeBr_3$tmdto 20.01 (20.57); p-$CH_3C_6H_4TeCl_3$tmdto 25.16 (25.47); p-$CH_3C_6H_4TeBr_3$tmdto 20.03 (20.12); p-$CH_3OC_6H_4TeCl_3$tmdto 24.85 (24.68); p-$CH_3OC_6H_4TeBr_3$tmdto 19.69 (19.62); p-$C_2H_5OC_6H_4TeCl_3$tmdto 24.20 (24.03); p-$C_2H_5OC_6H_4TeBr_3$tmdto 19.02 (19.21); p-$C_6H_5OC_6H_4$-$TeCl_3$tmdto 22.16 (22.04); p-$C_6H_5OC_6H_4TeBr_3$tmdto 17.45 (17.91); p-C_6H_5-$SC_6H_4TeCl_3$tmdto 21.10 (21.45); $C_6H_5TeCl_3$tedto ** 23.32 (23.50); $C_6H_5TeBr_3$tedto 19.08 (18.87); p-$CH_3C_6H_4TeCl_3$tedto 22.84 (22.91); p-$CH_3C_6H_4TeBr_3$tedto 18.35 (18.48); p-$C_6H_5OC_6H_4TeCl_3$tedto 20.09 (20.03); p-$C_6H_5OC_6H_4$-$TeBr_3$tedto 16.34 (16.61); p-$C_6H_5SC_6H_4TeCl_3$tedto 19.91 (19.54); p-$C_6H_5SC_6$-H_4TeBr_3tedto 16.72 (16.27); p-$BrC_6H_4TeBr_3$tedto 16.95 (16.89); $Te(ttz)_2Cl_4$ *** 25.29 (25.16); $Te(ttz)_2Br_4$ 18.48 (18.62); $Te(ttz)_2I_4$ 14.76 (14.61); $Te(ttz)_4Cl_2$ H_2O 18.22 (18.43); $Te(ttz)_3Br_4$ 16.20 (15.87); p-$CH_3OC_6H_4Te(ttz)_4Br_3$ 13.38 (13.42); p-$C_2H_5OC_6H_4Te(ttz)Cl_3$ 26.54 (26.92); p-$C_6H_5OC_6H_4Te(ttz)_2Cl_3$ 19.50 (19.91).

Standard deviation was calculated from results for p-$C_6H_5OC_6H_4TeCl_3$tedto: C, 41.86; H, 5.02; N, 4.46%. Analysis found: Te, 20.10, 19.88, 19.88, 19.98, 19.89, 19.83, 19.70, 19.87, 19.96%. $C_{28}H_{29}Cl_3N_2S_2Te$ calcd.: C, 42.17; H, 4.67; N, 4.47; Te, 20.03%.

Discussion and conclusions

The analysis of the tellurium content of organotellurium compounds by their decomposition using the oxygen flask technique followed by atomic absorption spectrophotometry has been found to be successful for a wide variety of tellurium compounds containing from between 13—40% Te. The only class of compound not completely oxidised by the method was diaryl ditelluride.

A standard deviation of 0.11, coefficient of variation of 0.0055 and confidence limit for 95% of 19.90 ± 0.07 was obtained for $C_6H_5OC_6H_4TeCl_3$tedto analysed nine times. For the 41 other samples a standard deviation of 0.23 was obtained.

Acknowledgements

The authors wish to thank Miss Helen J. Couper for help with certain stages of the analyses, and Mr. Arnold Collett for the statistical interpretation.

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* tmdto = *N,N,N',N'*-Tetramethyldithiooxamide

** tedto = *N,N,N',N'*- Tetraethyldithiooxamide

*** ttz = Thiazolidine-2-thione

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