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CYCLO-1,4-DIMETHYLENETETRATELLURIUM

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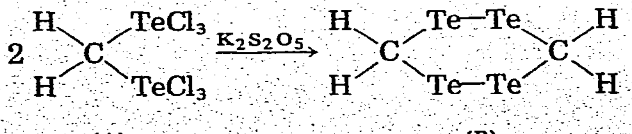
Summary

The mass spectrum and the Mössbauer spectrum provide evidence that the compound regarded previously as ditelluromethane, CH_2Te_2, is cyclo-1,4-dimethylenetetratellurium, $C_2H_4Te_4$.

Introduction

Tellurium tetrachloride (1 mol) reacts with acetic anhydride (2 mol) in boiling chloroform in air (4 h) with the evolution of hydrogen chloride and the precipitation of a grey mixture of bistrichlorotelluro(IV)acetic anhydride, $(Cl_3TeCH_2CO)_2O$, and trichlorotelluro(IV)acetic acid, $Cl_3TeCH_2CO_2H$. Reduction of the filtered solid by aqueous potassium metabisulphite gives red crystalline ditelluro(II)diacetic acid, $Te_2(CH_2CO_2H)_2$, m.p. 142°C, lit. [1] 144°C. Evaporation of chloroform from the filtrate gives colourless crystalline bistrichlorotelluro(IV)methane (A), m.p. 168—172°C, lit. [1] 173°C, which is reduced to a red-brown ditelluride, m.p. 54—102°C, lit. [1] 50—90°C, by potassium metabisulphite. It is proposed here that the reduction product is cyclo-1,4-dimethylenetetratellurium (B) rather than the strained three-membered cyclic ditelluromethane, CH_2Te_2 [1].



(A) (B)

TABLE 1

^{125}Te MÖSSBAUER DATA FOR $C_2H_4Te_2$

δ(^{125}Sb/Cu) (mm s^{-1})	Δ (mm s^{-1})	2 Γ (mm s^{-1})
0.27 0.06	9.33 0.11	6.33

Results and discussion

Cyclo-1,4-dimethylenetetratellurium was insufficiently soluble in common organic solvents for measurement of its molecular weight in solution. An attempt was made by the Department of Crystallography, Birkbeck College, to measure the unit cell dimensions using X-ray powder photography. The presence of powder rings confirmed that the sample was crystalline, but the overall pattern was insufficiently precise for successful indexing.

The mass spectrum, measured by the P.C.M.U., Harwell, showed the presence of ions corresponding to Te, Te_2, CH_2Te_2 and $C_2H_4Te_2$ in addition to a parent ion at *m/e* 409.7446 ($C_2H_4{}^{126}Te^{128}Te_2$ calcd.: 409.7439). A low intensity fragment pattern at higher molecular weight may correspond to the six-membered cyclic compound (B) which appears to eliminate tellurium under the conditions of mass spectrometry.

The ^{125}Te Mössbauer spectrum of crystalline cyclo-1,4-dimethylenetetratellurium confirms that the tellurium atoms are chemically equivalent, and that the sample was not contaminated by elemental tellurium. The Mössbauer parameters recorded in Table 1 are similar to those observed for diaryl ditellurides [2]. The small chemical isomer shift, δ, indicates low *s*-electron density at the tellurium nucleus, and the large quadrupole splitting, Δ, indicates an unsymmetrical electronic environment.

Cyclo-1,4-dimethylenetetratellurium was just sufficiently soluble in chloroform and dimethyl sulphoxide for ^{1}H NMR spectral measurements. The presence of one solvent-dependent singlet confirms that the protons are chemically equivalent. Comparisons of the chemical shifts with those of the precursor (A) illustrate the electronwithdrawing effects of chlorine (Table 2).

TABLE 2

^{1}H NMR CHEMICAL SHIFTS

Compound	Solvent	δ(CH_2) (ppm)
$CH_2(TeCl_3)_2$	$CDCl_3$	5.88
	$(CD_3)_2SO$	4.08
$C_2H_4Te_4$	$CDCl_3$	3.85
	$(CD_3)_2SO$	3.34

Experimental

Bistrichlorotelluro(IV)methane, m.p. 168—172° C, prepared by the method of Morgan and Drew [1] (Found: C, 2.69; H, 0.42; Cl, 44.46. $CH_2Cl_6Te_2$ calcd.: C, 2.49; H, 0.41; Cl, 44.17%) was reduced by cold aqueous potassium metabisulphite to give red-brown crystalline cyclo-1,4-dimethylenetetratellurium, m.p. 54—102° C (Found: C, 4.40; H, 0.62. $C_2H_4Te_4$ calcd.: C, 4.46; H, 0.74%).

The ^{125}Te Mössbauer spectrum was obtained from a ^{125}Sb/Cu source at 4.2 K using a modification of the apparatus described earlier [3].

References

1 G.T. Morgan and H.D.K. Drew, J. Chem. Soc., (1925) 531.
2 F.J. Berry, E.H. Kustan and B.C. Smith, J. Chem. Soc. Dalton Trans., (1975) 1323.
3 B.M. Cheyne and C.H.W. Jones, Can. J. Chem., 52 (1974) 564.