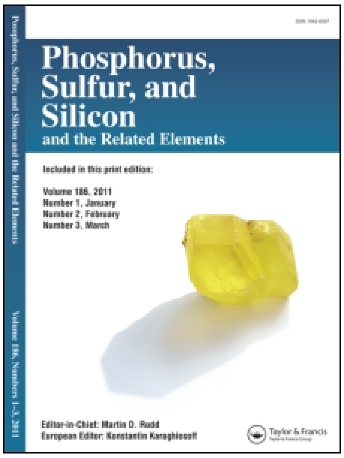

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# A STUDY OF 4,4-DIMETHYL 1,2-DITELLUROLANE

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The synthesis, x-ray crystal structure and solid -state nmr spectroscopy of 4,4-dimethyl 1,2-ditellurolane are reported.

Keywords: Tellurium, X-ray structure, $^{125}$Te solid-state nmr.

## INTRODUCTION

It has been known for some time that 1,2-diselenolane[1] and 1,2-ditellurolane[2] can be readily synthesised in solution but that they appear to occur only as dimers or polymers in the solid state. Recentl the spiro-derivative of 1,2-ditellurolane has been synthesised[3,4] and its x-ray structure determined by Lakshmikantham et al. [3], revealing the presence of the monomer. The presence of the oxetane ring was assumed to stabilise the 1,2-ditellurolane through a buttressing effect.

We have now succeeded in preparing 4,4-dimethyl 1,2-ditellurolane as a blue-black monomeric crystalline solid and have obtained its x-ray crystal structure. It is apparent that the oxetang ring is not required to stabilise the monomer. However, on exposure to visible light the blue-black solid becomes orange-red, consistent with dimerisation or polymerisation. This process has been followed in the solid-state by $^{13}C$ and $^{125}Te$ CP-MAS nmr spectroscopy.

## Experimental

1,3-dibromopropane was reacted with $K_2Te_2$ in DMF for 10 hr at 90°C. Following addition of $H_2O$, and subsequent work-up, blue- black plate-shaped crystals were obtained in 50% yield from hexane.

For the x-ray structural analysis, data was collected at 200K on an Euraf Nonius CAD 4F diffractometer. The data was analysed using standard programs[5,6]. The molecular structure is illustrated in Figure 1 and the unit cell packing in Figure 2.

Solid-state nmr spectra were obtained on a home-built spectrometer operating at 3.5 tesla ($^{13}C$ 37.56 MHz, $^{125}Te$ 47.13 MHz). Spectra were excited by Hartmann-Hahn cross-polarisation and the same field was used for proton decoupling. Spinning speeds were 3 to 3.6 KHz.

## Results and Discussion

The x-ray crystal structure of 4,4-dimethyl 1,2-ditellurolane shows the intramolecular bond-angles and bond-lengths within the five-membered ring to be very similar to those previously reported for the spiro derivative. Thus the Te-Te bond distance here is 274.69 pm and in the spiro derivative 274.1 pm; the C-Te-Te bond angles of 85.94° and 87.64° compare with those of 85.8° and 87.6° reported earlier. The C(1)-C(2)-

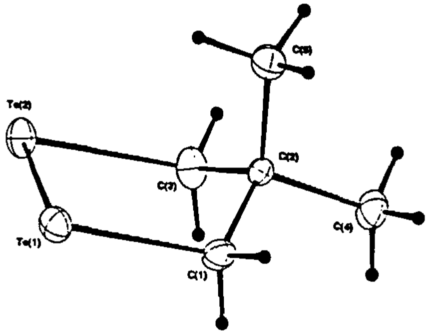

FIGURE 1 The molecular structure

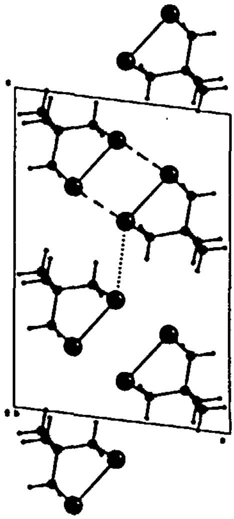

FIGURE 2 The unit cell

C(3) bond-angle of 110.16° compares with 112.9° in the spiro compound where the oxetane ring constrains the bond-angle about the quaternary carbon.

In visible light, a blue-black microcrystalline sample of 4,4-dimethyl 1,2-ditellurolane turned orange-red over a period of 60 days. This transformation was followed by CP-MAS nmr spectroscopy. The $^{13}C$ spectra show that the resonance at 49 ppm due to the quaternary carbon in the blue-black compound shifts to ca. 39 ppm, overlapping with the methylene and methyl resonances in the orange-red form. The $^{125}Te$ spectra show the presence of two resonances at 131.5 ppm and 112.9 ppm in the blue-black form, and four resonances at 264, 226, 184 and 144 ppm in the orange-red compound.

These data, together with the change in colour of the compound, are consistent with a light-induced solid-state dimerisation or polymerisation. From the x-ray crystal structure, the shortest intermolecular Te-Te contact is 345.6 pm and association through these contacts would lead to a chain-like polymer (the light dots in Figure 2). However, the unit cell shows two adjacent molecules with their Te-Te linkages suitably aligned for dimerisation and where the Te-Te contacts are 406 pm (the heavy dashes in Figure 2).

On dissolving the orange-red compound in benzene, a deep blue solution was obtained, and the blue-black solid resulting on removing the solvent yielded $^{13}C$ and $^{125}Te$ CP-MAS spectra identical to the parent 4,4-dimethyl 1,2-ditellurolane. It is apparent that the dimerisation or polymerisation observed in the solid-state is reversed on dissolving up the solid in benzene.

## References

[1.] G.T. Morgan and F.H. Burstall, J. Chem. Soc., 1497 (1930).
[2.] G. Merkel, H. Berge and P. Jeroschewski, J. Prakt. Chem., **326**, 467 (1984).
[3.] M.V.. Lakshmikantham, M.P. Cava, W.H.H. Günther, P.N. Nugara, K.A. Belmore, J.L. Atwood and P. Craig, J. Amer. Chem. Soc., **115**, 885 (1993).
[4.] R. Lok, W.H.H. Günther and J.P. Freedman, U.S. 4, 861, 703; 1989. Chem. Abstr. **112**, 148983 (1990).
[5.] E.J. Gabe, V. LePage, J.-P. Charland and P.S. White, J. Appl. Cryst., **22**, 384 (1989).
[6.] D.J. Watkins, J.R. Carruthers and P.W. Betteridge, Crystals (Chemical Crystallography Laboratory, University of Oxford, 1995).