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cis-3,5-DIBENZYLIDENE-1,2,4-TRITELLUROLE, A NOVEL ORGANOTELLURIUM HETEROCYCLE

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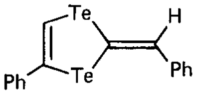
Summary: The reaction of sodium phenylethynyltellurolate with ethereal hydrogen chloride afforded, in addition to trans-2,4-dibenzylidene-1,3-ditelluretane, a product shown to be the title compound by an X-ray crystallographic analysis.

In 1979, Petrov et al[2] reported that the reaction of sodium phenylethynyltellurolate (1) with ethereal hydrogen chloride afforded a compound, mp 270-275°, which was assigned the 1,3-ditellurole structure 2 by analogy with known protonation reactions of arylethynylthiolate and arylethynylselenolate ions.[3,4] A very recent reinvestigation of this reaction gave a compound which was shown by X-ray analysis to be not 2, but rather trans-2,4-dibenzylidene-1,3-ditelluretane (3); the cis isomer (4) was also isolated.[5]

PhC≡CTeNa

1



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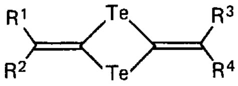
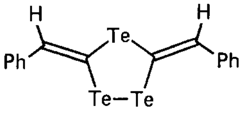


3: $R^1 = R^4 = H$; $R^2 = R^3 = Ph$
4: $R^1 = R^3 = H$; $R^2 = R^4 = Ph$



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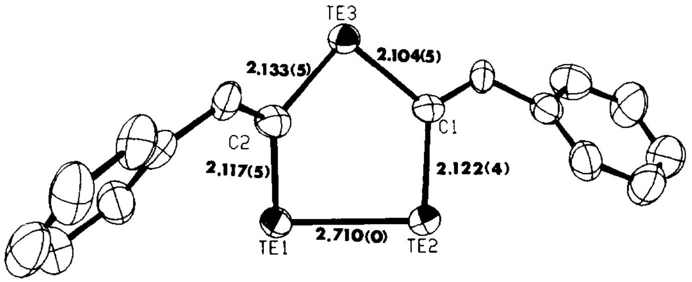
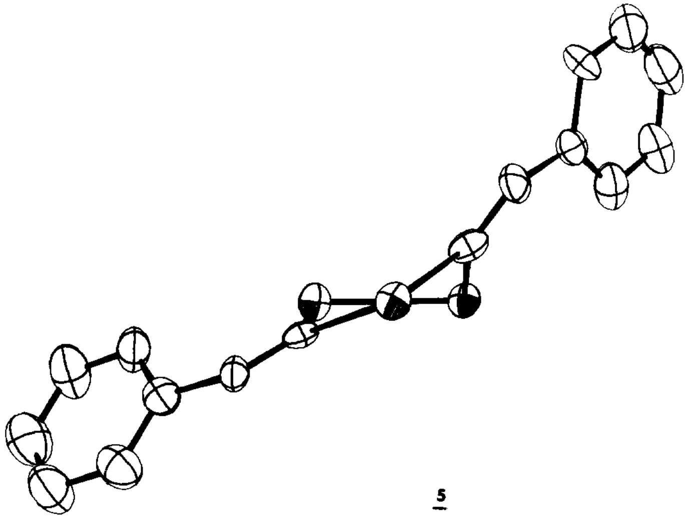
Our own independent reinvestigation of Petrov's reaction led to the formation of two crystalline products (2% each), the structures of which were determined by X-ray crystallographic analysis. The first of these, mp 265-270° C (dec) proved to be the 1,3-ditelluretane 3; our structural details were essentially in agreement with those reported by Bender et al,[5] and will not be repeated here. The second product, mp 208-210° C, formed red rhombs from dimethylformamide; mass spectrometry confirmed the composition $C_{16}H_{12}Te_3$, and an X-ray crystallographic analysis revealed that it has the unusual structure of cis-3,5-dibenzylidene-1,2,4-tritellurole (5).[6] Compound 5 which is the first example of a 1,2,4-tritellurole, was shown to have a non-planar heterocyclic system in which the ditelluride bond forms a dihedral angle of 35.9° with the other three ring atoms.

Ditelluretane 3 may be viewed as the cyclodimerization product of the transient telluroketene 6, while the tritellurole 5 is most likely formed via the addition of the phenylethynylditelluride ion (7) to telluroketene 6.





5

$PhCH{=}C{=}Te$

6

$PhC{\equiv}CTeTeNa$

7

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References and Footnotes

1. On leave from Bar-Ilan University, Ramat Gan, Israel.

2. M. L. Petrov, V. Z. Laishev and A. A. Petrov, Zh. Org. Khimii (English translation), 15, 2346 (1979).

3. R. Mayer, B. Hunger, R. Prousa and A. K. Muller, J. Prakt. Chem., 35, 294 (1967).

4. I. Lalezari, A. Shafiee and M. Yalpani, J. Org. Chem., 38, 338 (1973).

5. S. L. Bender, N. F. Haley and H. R. Luss, Tetrahedron Lett., 22, 1495 (1981).

6. Data were collected on a crystal of 5 (crystal size 0.35 mm x 0.37 mm x 0.05 mm) using a computer-controlled Enraf-Nonius CAD4 Diffractometer utilizing the ω-2Θ scan technique. Space group P2$_1$/c, Z = 4; unit cell dimensions: a = 14.585 (2) Å, b = 10.608 (4) Å, c = 10.397 (2) Å, B = 98.18 (3) Å; d (calcd) = 2.449 g/cm^{-3}, d (obsd) = 2.43 g/cm^{-3}. A total of 3335 reflections were collected using graphite-monochromated MoK$_\alpha$ radiation. Of these 2682 had $I > 3\sigma$ (I). The structure was solved and refined by standard methods to R = 0.053, R$_w$ = 0.079. An absorption correction was applied (μ = 56.4 cm^{-1}).

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