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REACTION OF TELLURIUM WITH PHENYLACETYLENE: A REINVESTIGATION

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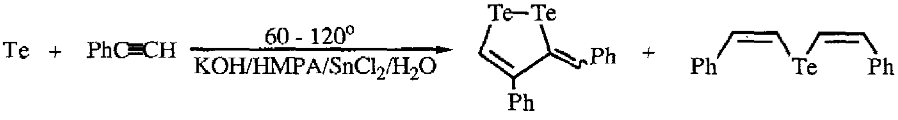
Summary: The reaction of powdered tellurium with phenylacetylene under strongly basic conditions yields cis and trans -2, 6- diphenyl-1, 4-ditellurafulvenes, rather than Z and E isomers of a five-membered cyclic ditelluride, as recently reported.

Our recent interest in the chemistry of cyclic disulphides and ditellurides[1] prompted us to reinvestigate the recently reported synthesis of **1**.[2]



1

The reaction, in our hands, yielded orange crystalline plates (acetone) with a melting point (215-216°), 55-56° higher than the reported value. The UV/Vis (dichloromethane) spectrum of the molecule did not exhibit an expected absorption band around $\lambda = 400$nm, a characteristic absorption for theTe-Te chromophore.[3] The mass spectral data, however, supported the proposed molecular formula of $C_{16}H_{12}Te_2$ with a molecular ion at m/e = 464 (Te^{130}) having the expected isotope pattern for Te_2. The NMR (DMSO-d_6) chemical shifts were reproducible and indicated the presence of two isomers in 1:1 ratio.

Cyclic voltammetry measurements showed an irreversible anodic wave $E_{pa} = 724$mV and a cathodic wave at $E_{pc} = -68$mV (CH_2Cl_2, 200 mV/S, vs (Ag/AgCl). The cathodic wave could probably result from the dimerization of the radical cation.

In order to interpret the contradictory results, we compared the spectral data (NMR, MS and C.V.) with the authentic samples of **2** and **3** which also have the same molecular formula and have been reported earlier from the protonation of sodium phenylethynyltellurolate with trifluoroacetic acid and ethereal hydrochloric acid respectively.[4,5]

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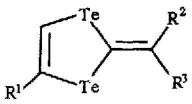

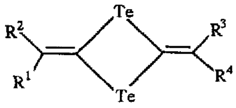

2a: R^1=R^3=Ph, R^2=H
2b: R^1=R^2=Ph, R^3=H

3a: R^1=R^3=Ph, R^2=R^4=H
3b: R^1=R^4=Ph, R^1=R^3=H

The NMR and C.V. data were identical with cis and trans isomers of 2. The tlc analysis of these compounds further confirmed that 1 was indeed a (1:1) mixture of 2a and 2b and not the Z and E isomers of 3-benzylidine-4-phenyl-1, 2-ditellurole, as claimed by Potapov et al. Thus, a simple cyclic five-membered ditelluride still remains unknown, though these exist when fused with aromatic rings.[6]

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References:

1 H. B. Singh and F. Wudl, unpublished results.

2 V.A. Potapov, N.K. Gusarova, SS.V. Amosova, A.S. Kashik and B.A. Trofimov, Sulfur Lett., 4, 13, (1985).

3 R.T. Mehdi and J.D. Miller, J. Chem. Soc. Dalton Trans. 1071 (1983).

4 M.V. Lakshmikantham, M.P. Cava, M. Albeck, L. Engman, F. Wudl and E. Ahron-Shaalom, J. Chem. Soc. Chem. Comm., 828 (1981); M.V. Lakshmikantham, M.D. Cava, M. Albeck, L. Engman, P. Carroll, J. Bergman and F. Wudl, Tetrahedron Lett., 22, 4199 (1981); F. Wudl and E. Ahron-Shalom, J. Am. Chem. Soc., 104 1154 (1982).

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

6 J. Meinwald, D. Dauplaise, F. Wudl and J.J. Hauser, J. Am. Chem. Soc. 99, 255 (1977).

7 Characterization date for cis and trans-2, 6-diphenyl-1, 4-ditellurafulvenes: m.p. 215-216, yield = 15%, λ max = 443.1 and 362.8; MS m/e = 464 (14.8%, M^+), 260 (31%, Te_2^+)103 (100%).

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