

A Convenient Synthesis of Telluronium Ylides

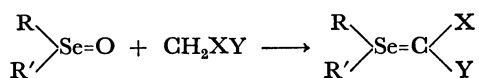
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Synopsis. Several telluronium ylides were readily prepared by mixing the corresponding telluroxides and a variety of active methylene compounds in CHCl_3 at ambient temperature or under reflux. Arylselenonium ylides could also be obtained in a similar way.

We recently reported a convenient procedure to prepare several alkyl and arylalkyl selenonium ylides by treating the corresponding selenoxides and active methylene compounds at room temperature.¹⁾ Mean-



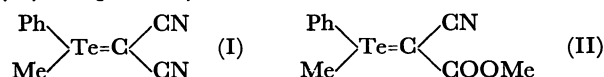
X, Y = CN or COOMe

while, only a few publications have been available on the general synthesis of telluronium ylides. Sadekov *et al.* reported that diaryl tellurium dichlorides gave the ylides when allowed to react with 5,5-dimethyl-1,3-cyclohexanedione (dimedone) in the presence of base in refluxing benzene.²⁾

In this article it is described that the method previously developed by us for the synthesis of selenonium ylides can be applied successfully to the preparation of telluronium analogs starting from telluroxides which are easily available.

Results and Discussion

A typical reaction is as follows: phenyl methyl telluroxide was treated with a 3-fold excess of malonitrile and methyl cyanoacetate in CHCl_3 at ambient temperature. After 12 h, evaporation of the solvent afforded solids of phenylmethyltelluronium dicyanomethylide (I) and cyano(methoxycarbonyl)methylide (II), respectively.



As was observed in the IR spectrum of the corresponding selenonium dicyanomethylide,^{1,3)} the cyano groups in the telluronium dicyanomethylide (I) also exhibited two absorption bands with comparable intensity at 2120 and 2160 cm^{-1} . The IR bands due to the cyano and carbonyl groups in II shift to lower wavelengths as compared to those of methyl cyanoacetate.

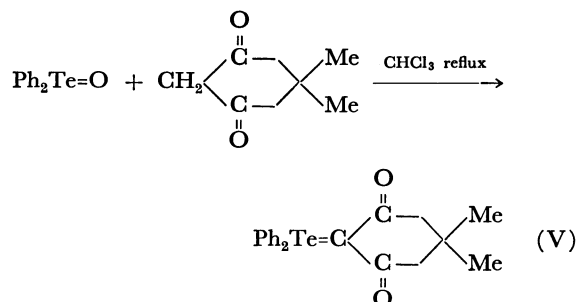
Meanwhile, when diphenyl telluroxide was utilized as a telluroxide, any reaction did not take place at room temperature. However, upon refluxing a chloroform solution of the oxide and an equimolar amount of methylene compounds for 10 h, the formation of

TABLE I. PRODUCT YIELDS AND PHYSICAL PROPERTIES OF TELLURONIUM AND SELENIUM YLIDES

Ylide	Yield %	Mp °C	IR(KBr) ν/cm^{-1}	NMR in CDCl_3 δ/ppm^b
$\text{PhMeTe}=\text{C}(\text{CN})_2$ (I)	82	148—149	2160, 2120	7.20—8.00(5H, m), 2.23(3H, s)
$\text{PhMeTe}=\text{C}(\text{CN})(\text{COOMe})$ (II)	80	123—125	2150, 1620	7.28—8.00(5H, m), 3.79(3H, s), 2.30(3H, s)
$\text{PhMeTe}=\text{C}(\text{CN})(\text{COOEt})$ (III)	75	131—132	2150, 1620	7.12—8.05(5H, m), 3.72—4.14(2H, q), 2.33(3H, s), 0.97—1.12(3H, t)
$\text{Ph}_2\text{Te}=\text{C}(\text{CN})(\text{COOMe})$ (IV)	78	a)	2130, 1610	7.1—7.6(10H, m), 3.65(3H, s)
$\text{Ph}_2\text{Te}=\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{C} \\ \parallel \\ \text{O} \end{array} \begin{array}{c} \text{Me} \\ \diagup \\ \text{C} \\ \diagdown \\ \text{Me} \end{array}$ (V)	100	162—163	1524	7.40—7.80(10H, m), 2.37(4H, s), 1.10(6H, s)
$\text{Ph}_2\text{Se}=\text{C}(\text{CN})(\text{COOMe})$ (VI)	56	a)	2160, 1649	7.58(10H, s), 3.75(3H, s)
$\text{ToI}_2\text{Se}=\text{C} \begin{array}{c} \text{O} \\ \parallel \\ \text{C} \\ \parallel \\ \text{O} \end{array} \begin{array}{c} \text{Me} \\ \diagup \\ \text{C} \\ \diagdown \\ \text{Me} \end{array}$ (VII)	100	128—129	1560	7.20—7.60(8H, m), 2.38(10H, s), 1.09(6H, s)

a) Oily and viscous materials. b) s: singlet; q: quartet; m: multiplet.

the ylides was attained in excellent yields. For instance, dimedone upon treatment with diphenyl telluroxide, afforded quantitatively the corresponding ylide (V).



The yields and physical data of these ylides are summarized in Table 1.

In a similar way, the attempt to prepare diaryl-selenonium counterparts, which were not obtained previously by the reaction at room temperature, can be realized in good yields.

The method described here is a useful and convenient alternative for the preparation of telluronium ylides, since it involves merely mixing reagents under mild conditions and only simple work-up of the reaction mixture as compared with that of Sadekov *et al.*²⁾

Experimental

Preparation of Ylides. A mixture of phenyl methyl

telluroxide (1 mmol, 235 mg) and a 3-fold excess of malononitrile in 20 ml of CHCl_3 was kept standing at room temperature for 12 h. After evaporation of the solvent, the resulting solid was recrystallized from CHCl_3 -ether, giving 82% of phenylmethyltellurium dicyanomethylide (I): Found: C, 41.86; H, 2.88; N, 9.68%. Calcd for $\text{C}_{10}\text{H}_8\text{N}_2\text{Te}$: C, 42.31, H, 2.82; N, 9.87%. Similarly, the corresponding cyano-(methoxycarbonyl)methylide (II) was prepared: Found: C, 41.29; H, 3.80; N, 4.71%. Calcd for $\text{C}_{11}\text{H}_{11}\text{NO}_2\text{Te}$: C, 41.62; H, 3.51; N, 4.46%. III: Found: C, 43.44; H, 3.86, N, 4.29%. Calcd for $\text{C}_{12}\text{H}_{13}\text{NO}_2\text{Te}$: C, 43.56, H, 3.93; N, 4.23%.

Compounds (IV—VII) were prepared by refluxing a CHCl_3 solution of diaryl telluroxides (1 mmol) and one equivalent of methylene compounds for 15 h. After the solvent was stripped off, V and VII were purified by recrystallization from benzene-hexane and ether, respectively. V: Found: C, 57.05; H, 4.78%. Calcd for $\text{C}_{20}\text{H}_{20}\text{O}_8\text{Te}$: C, 57.18, H, 4.80%. VII: Found: C, 65.89; H, 5.91%. Calcd for $\text{C}_{22}\text{H}_{24}\text{O}_2\text{Se}$: C, 66.14; H, 6.06%. IV and VI were purified by precipitation with ether from their CHCl_3 solutions.

References

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