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Synthesis of 2-diphenylphosphinoyl-3,5-diaryl-3,4-dihydro-2*H*-telluropyrans by reaction of chalcones with bis[(diphenylphosphinoyl)methyl]telluride

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ABSTRACT

The NaH-promoted tandem Michael addition/intramolecular Wittig–Horner reaction of bis[(diphenylphosphinoyl) methyl]telluride with chalcones stereoselectively afforded *trans*-2-diphenylphosphinoyl-3,5-diaryl-3,4-dihydro-2*H*-telluropyran derivatives in 51–72% yield, under mild conditions.

Tellurium compounds have become the key components of new materials with potential technological applications, because of their distinguishing features and rich chemistry. They also represent a variety of versatile and useful reagents for organic synthesis. In addition, the development of a large number of tellurium-based synthetic methods which took place in the recent years has made significant contributions to synthetic organic chemistry.[1]

The six-membered unsaturated heterocycles containing tellurium have drawn attention as catalysts, as well as photosensitizers, photoconducting materials and chemotherapeutic agents.[2]

Baum et al. reported the syntheses of the fluorinated 3,6-dihydro-2*H*-telluropyrans **1a,b** by [4+2] cycloaddition between perfluoroalkyl trimethylstannyl tellurides and 1,3-butadiene (Fig. 1).[3] Telluropyrylium dyes exemplified by **2** and **3**, which show electronic absorptions at lower energies than other dyes, have been synthesized and tested with respect to the generation of the singlet oxygen, as potential photochemotherapeutic agents in photodynamic therapy.[4] These compounds were found to be active in animal studies.[5]

In addition, telluropyrylium dyes and their derivatives have also been proposed as optical recording media[6] and catalysts for solar energy storage via photochemical-thermal generation of hydrogen peroxide,[7] and their basic hydrolysis was studied under aerobic and anaerobic conditions.[8]

$\Delta^{4,4'}$-4-Telluropyranyl-4*H*-telluropyrans and their congener [4,4′]bitellurochromenylidene **4**, have been prepared as part of mixed-stack organic metal complexes research, in order to study their properties as π-electron donors and organic conductors.[9] Furthermore, the preparation of carbene complexes (**5**) exhibiting interesting nonlinear optical properties[10] and other chalcogenides bearing the 4*H*-2,6-diphenyl telluropyran motif, including **6**,[11]

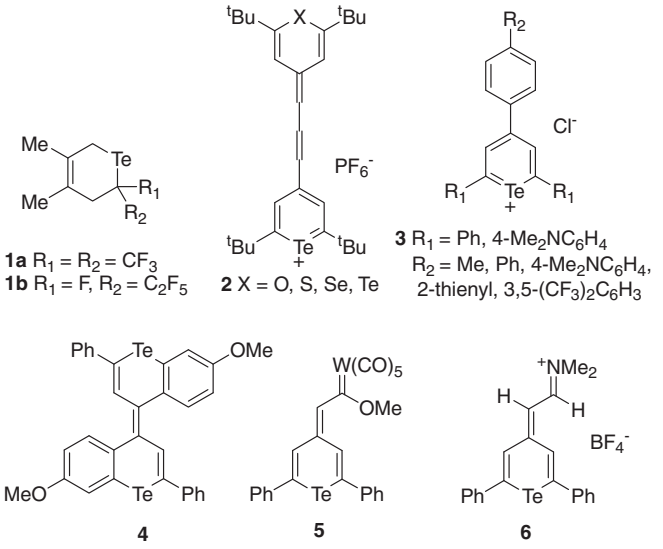

Figure 1. Chemical structures of some representative six-membered unsaturated heterocyclics containing tellurium.

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have been described. On the other hand, Sashida et al. synthesized isotellurochromenes observing the equilibrium between 1-benzyl-2-benzotelluropyrylium salts and the corresponding 1-benzylidene derivatives, resulting from β-hydrogen elimination.[12]

Tandem reactions are powerful methodologies for efficiently synthesizing complex molecules, whereby several carbon–carbon or carbon–heteroatom bonds can be constructed in one pot without isolating the intermediates. Advantageously, these reactions often proceed stereoselectively and with high regioselectivity[13]; therefore, they have shown to be valuable for the stereoselective synthesis of multifunctionalized cyclic and polycyclic compounds.[14]

Vinylic tellurides are a useful class of tellurium derivatives, the Wittig or Wittig–Horner reaction being one of the most useful methods for their synthesis.[15] In this respect, we[16] and others[17] have reported a convenient means for accessing vinylic tellurides employing this methodology.

Recently, we have described the Wittig–Horner reaction of bis[(diphenylphosphinoyl)methyl] chalcogenides with carbonyl compounds, including aldehydes and ketones, to afford symmetrical and unsymmetrical bis-vinylic chalcogenides.[18] We have also disclosed the tandem Michael addition/intramolecular Wittig–Horner reaction of bis[(diphenylphosphinoyl)methyl]sulfide with chalcones to afford 3,4-dihydro-2*H*-thiopyran derivatives.[19] As a continuation of this work, here we report the outcome of the reaction of chalcones with the analogous bis[(diphenylphosphinoyl)methyl] telluride (**1**).

The telluride reagent **1** was prepared in three steps and 71% overall yield from diphenylphosphane, following the previously reported procedure,[18] as a slightly unstable solid, which slowly decomposes at room temperature and in chloroform solution.

As shown in Table 1, when the reaction was run in DMF for 24 h at room temperature, with 1.2 equiv of 3-(2-chlorophenyl)-1-(4-chlorophenyl)-propenone as model chalcone and 2 equiv of NaH as a base, an extensive decomposition of the starting tellurium reagent took place, and no cyclized product could be isolated (entry 1). On the other hand, the starting telluride proved to be insoluble in toluene and scarcely soluble in Et_2O.

However, to our satisfaction, when the reaction was carried out in a 1:1 mixture of Et_2O and THF for 24 h at room temperature, 21% of the product could be obtained, as a yellow solid (entry 2). Despite telluropyrans and telluropyrylium salts having been previously accessed by various means, this represented the first entry to 3,5-diaryl-3,4-dihydro-2*H*-telluropyrans.

Performing the transformation in THF with 3 equiv of NaH as a base furnished 51% of cyclized product (entry 3). Increasing the amount of chalcone and reducing the quantity of NaH produced small improvements in the yield, while submitting the reaction to reflux promptly gave decomposition of the starting telluride, furnishing only 49% of the product after 1 h (entry 5). Further lowering the amount of NaH to 2.0 equiv gave an optimized 60% yield of the cyclized product, after performing the transformation at room temperature (entry 6).

With the optimal reaction conditions in hand, we then examined the tandem reaction of a spectrum of chalcones. The transformation demonstrated to be general and eight telluropyrans were obtained, in 51–72% yield,[20] as detailed in Table 2.[21]

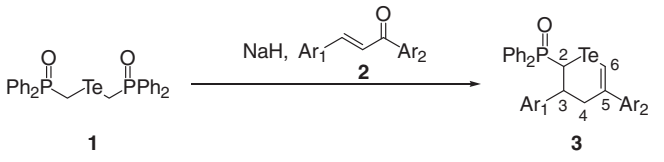
It was found that depending on the reaction conditions, chalcones react with phosphonates either in a Michael or a Wittig–Horner fashion.[22] By analogy with our previously reported synthesis of 3,5-diaryl-3,4-dihydro-2*H*-thiopyrans, formation of the telluropyrans **3a–h** may be rationalized as being the result of a sequence of reactions comprising deprotonation of **1**, followed by the ylide-initiated Michael addition of the resulting anion to the enone **2**.[18]

The intermediate enolate could then mediate deprotonation of the remaining methylene group attached to both tellurium and phosphorous, by proton migration, favored by the higher acidity of the protons associated to this methylene group.[23] A final intramolecular Wittig–Horner reaction of the phosphinoyl carbanion, would then be triggered, leading to the cyclized product after loss of diphenylphosphinate. A quite similar mechanism seems to be operating in the regioselective synthesis of dihydropyridines by reaction of *N*-vinylic phosphazenes with α,β-unsaturated ketones and in the recently described organocatalytic asymmetric tandem Michael addition–Wittig reaction leading to cyclohexenones.[24]

The ^1H and ^{13}C NMR spectra of the telluropyrans indicated that they were formed as single isomers. By analogy with our previous findings on the synthesis of *trans*-2-diphenyl phosphinoyl-3,5-diaryl-3,4-dihydro-2*H*-thio pyrans,[19] assuming a half-chair conformation and taking into account the magnitude of the coupling constants between H-2 and H-3 ($J \cong 6.1$–6.9 Hz), a trans configuration was proposed for the heterocycles.[25]

This stereochemistry could be established during the Michael addition stage, where the intermediate enolate could decompose back to the reactants, providing a means to yield the product which

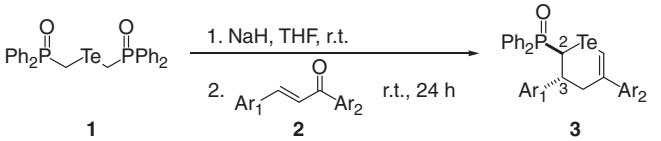
Table 1
Optimization of the reaction conditions for the synthesis of the 3,4-dihydro-2*H*-telluropyrans (**3**)



Run No.	Chalcone[a] (equiv)	NaH (equiv)	Solvent	Temperature (°C)	Reaction time (h)	Yield (%)
1	1.2	2.0	DMF	rt	24	—[b]
2	1.2	2.0	THF/ Et_2O[c]	rt	24	21
3	1.2	3.0	THF	rt	24	51
4	1.5	2.3	THF	rt	24	57
5	1.2	2.8	THF	Reflux	1	49
6	1.2	2.0	THF	rt	24	60

[a] Ar_1 = 2-Cl–C_6H_4; Ar_2 = 4-Cl–C_6H_4.
[b] Extensive decomposition of the tellurium reagent **1** takes place.
[c] Starting materials are poorly soluble in Et_2O; a 1:1 THF/Et_2O mixture was employed.

Table 2
Sodium hydride-promoted synthesis of 2-diphenylphosphinoyl-3,5-(diaryl)-3,4-dihydro-2*H*-telluropyrans (**3**) from bis[(diphenylphosphinoyl)methyl]telluride (**1**) and 1,3-diaryl-propenones (chalcones, **2**)



Entry No.	Compd No.	Ar_1	Ar_2	Yield (%)
1	**3a**	C_6H_5	C_6H_5	63
2	**3b**	C_6H_5	4-Me–C_6H_4	51
3	**3c**	C_6H_5	4-Cl–C_6H_4	52
4	**3d**	4-Cl–C_6H_4	C_6H_5	57
5	**3e**	4-Me–C_6H_4	4-Me–C_6H_4	60
6	**3f**	4-MeO–C_6H_4	4-MeO–C_6H_4	52
7	**3g**	4-Me–C_6H_4	4-MeO–C_6H_4	72
8	**3h**	2-Cl–C_6H_4	4-Cl–C_6H_4	60

minimizes steric interactions between the bulkiest substituents.[26] Alternatively, it could be the result of the deprotonation–reprotonation of the acidic methinic proton H-2, leading to the most stable diastereomer; partial deuteration of H-2 was found upon quenching a mixture of NaH and a 2-diphenyl phosphinoyl-3,5-diaryl-3,4-dihydro-2*H*-thio pyran, previously heated in THF.[19]

In conclusion, it was demonstrated that the reaction of bis[(diphenylphosphinoyl)methyl]telluride with variously substituted chalcones proceeds with high diastereoselectivity, under mild conditions, providing *trans*-2-diphenylphosphinoyl-3,5-diaryl-3,4-dihydro-2*H*-telluro-pyrans via a tandem Michael addition/intramolecular Wittig–Horner reaction. This stereoselective approach represents the first strategy to access these kinds of polysubstituted 3,4-dihydro-2*H*-telluropyran derivatives.

Acknowledgments

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20. *General procedure*: NaH (60%, 40 mg, 1.0 mmol) was added at room temperature to a solution of bis[(diphenyl phosphinoyl)methyl]telluride (0.50 mmol) in anhydrous THF (10 mL), stirred under an argon atmosphere. After 20 min, the appropriate chalcone (0.6 mmol, 1.2 equiv) was added and the yellow reaction mixture was further stirred for 24 h at room temperature. Then, saturated aqueous NH_4Cl (15 mL) was added and the mixture was extracted with CH_2Cl_2 (3 × 15 mL). The combined organic extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by column chromatography (hexanes–EtOAc–CH_2Cl_2, 5:3:2).
21. *Spectral data of selected compounds*: *trans*-2-Diphenyl phosphinoyl-3,5-(diphenyl)-3,4-dihydro-2*H*-telluropyran (**3a**): Yield: 63%. Mp 138–139 °C (dec). IR (KBr): ν = 524, 696, 1190 and 1438 cm^{-1}. ^1H NMR ($CDCl_3$, 200 MHz): δ = 2.86–3.06 (m, 2H), 3.32–3.48 (m, 1H), 4.67 (dd, J = 1.7 and 6.4 Hz, 1H), 7.01–7.49 (m, 16H), 7.19 (s, 1H), 7.60–7.70 (m, 2H) and 7.74–7.84 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 22.7 (d, $J_{P\text{-}C}$ = 66.3 Hz), 41.7, 44.4 (d, $J_{P\text{-}C}$ = 5.2 Hz), 101.1 (d, $J_{P\text{-}C}$ = 1.3 Hz), 125.6, 126.7, 126.9, 127.3, 128.1, 128.3 (d, $J_{P\text{-}C}$ = 11.4 Hz), 128.4, 128.6 (d, $J_{P\text{-}C}$ = 11.6 Hz), 130.7 (d, $J_{P\text{-}C}$ = 8.7 Hz), 131.2 (d, $J_{P\text{-}C}$ = 2.8 Hz), 131.3 (d, $J_{P\text{-}C}$ = 8.9 Hz), 131.6 (d, $J_{P\text{-}C}$ = 96.6 Hz), 131.8 (d, $J_{P\text{-}C}$ = 2.6 Hz), 133.6 (d, $J_{P\text{-}C}$ = 101.9 Hz), 143.0, 144.2 (d, $J_{P\text{-}C}$ = 6.8 Hz) and 145.5 ppm. GC–MS: m/z (%) = 550 (10) [M^+], 348 (18), 219 (50), 218 (100), 217 (35), 202 (62), 201 (68), 115 (99) and 91 (55). *trans*-2-Diphenylphosphinoyl-5-(4-methylphenyl)-3-phenyl-3,4-dihydro-2*H*-telluropyran (**3b**): Yield: 51%. Mp 140–141 °C (dec). ^1H NMR ($CDCl_3$, 400 MHz): δ = 2.26 (s, 3H), 2.91 (dd, J = 7.4 and 14.9 Hz, 1H), 2.99 (dd, J = 1.8 and 14.9 Hz, 1H), 3.33–3.41 (m, 1H), 4.65 (dd, J = 1.8 and 6.5 Hz, 1H), 6.94 (d, J = 8.2 Hz, 2H), 7.00 (d, J = 8.2 Hz, 2H), 7.05–7.09 (m, 5H), 7.15 (s, 1H), 7.24–7.52 (m, 6H), 7.62–7.67 (m, 2H) and 7.76–7.81 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 21.0, 22.6 (d, $J_{P\text{-}C}$ = 66.5 Hz), 41.7, 44.5 (d, $J_{P\text{-}C}$ = 5.2 Hz), 100.0, 125.6, 126.7, 127.4, 128.3 (d, $J_{P\text{-}C}$ = 11.6 Hz), 128.4, 128.6 (d, $J_{P\text{-}C}$ = 11.5 Hz), 128.8, 130.7 (d, $J_{P\text{-}C}$ = 8.6 Hz), 131.3 (d, $J_{P\text{-}C}$ = 8.9 Hz), 131.5 (d, $J_{P\text{-}C}$ = 95.5 Hz), 131.9 (d, $J_{P\text{-}C}$ = 2.4 Hz), 133.5 (d, $J_{P\text{-}C}$ = 102.0 Hz), 136.7, 140.2, 144.3 (d, $J_{P\text{-}C}$ = 6.8 Hz) and 145.5 ppm. GC–MS: m/z (%) = 564 (8) [M^+], 362 (16), 233 (35), 232 (100), 201 (40), 130 (31), 129 (43), 115 (38) and 91 (26). Anal. Calcd for $C_{30}H_{27}OPTe$: C, 64.10; H, 4.84. Found: C, 63.95; H, 5.24. *trans*-2-Diphenylphosphinoyl-5-(4-chloro phenyl)-3-phenyl-3,4-dihydro-2*H*-telluropyran (**3c**): Yield: 52%. Mp 150–151 °C (dec). ^1H NMR ($CDCl_3$, 400 MHz): δ = 2.90 (dd, J = 6.9 and 15.0 Hz, 1H), 2.96 (dd, J = 1.9 and 15.0 Hz, 1H), 3.36–3.44 (m, 1H), 4.64 (dd, J = 1.9 and 6.1 Hz, 1H), 6.94 (d, J = 8.5 Hz, 2H), 7.04–7.11 (m, 5H), 7.13 (d, J = 8.5 Hz, 2H), 7.21 (s, 1H), 7.27–7.53 (m, 6H), 7.63–7.68 (m, 2H) and 7.76–7.81 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 22.7 (d, $J_{P\text{-}C}$ = 66.4 Hz), 41.6, 44.2 (d, $J_{P\text{-}C}$ = 4.6 Hz), 102.0 (d, $J_{P\text{-}C}$ = 1.4 Hz), 126.8, 126.9, 127.3, 128.2, 128.4 (d, $J_{P\text{-}C}$ = 11.7 Hz), 128.5, 128.7 (d, $J_{P\text{-}C}$ = 11.6 Hz), 130.7 (d, $J_{P\text{-}C}$ = 8.7 Hz), 131.3 (d, $J_{P\text{-}C}$ = 8.8 Hz), 131.4 (d, $J_{P\text{-}C}$ = 96.5 Hz), 132.0 (d, $J_{P\text{-}C}$ = 2.5 Hz), 132.8, 133.4 (d, $J_{P\text{-}C}$ = 101.9 Hz), 141.5, 144.0 (d, $J_{P\text{-}C}$ = 7.5 Hz) and 144.2 ppm. GC–MS: m/z (%) = 584 (8) [M^+], 252 (58), 215 (39), 202 (100), 201 (86), 139 (29), 125 (32), 115 (62), 91 (25) and 77 (52). Anal. Calcd for $C_{29}H_{24}ClOPTe$: C, 59.79; H, 4.15. Found: C, 59.51; H, 4.38. *trans*-2-Diphenyl phosphinoyl-3,5-bis(4-methoxyphenyl)-3,4-dihydro-2*H*-telluropyran (**3f**): Yield: 52%. Mp 126–127 °C (dec). ^1H NMR ($CDCl_3$, 400 MHz): δ = 2.87 (dd, J = 7.6 and 14.9 Hz, 1H), 2.96 (dd, J = 1.7 and 14.9 Hz, 1H), 3.30–3.38 (m, 1H), 3.70 (s, 3H), 3.74 (s, 3H), 4.62 (dd, J = 1.7 and 6.9 Hz, 1H), 6.60 (d, J = 8.7 Hz, 2H), 6.73 (d, J = 8.7 Hz, 2H), 6.98 (d, J = 8.7 Hz, 2H), 7.02 (d, J = 8.7 Hz, 2H), 7.05 (s, 1H), 7.24–7.53 (m, 6H), 7.60–7.69 (m, 2H) and 7.76–7.80 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 23.0 (d, $J_{P\text{-}C}$ = 66.5 Hz), 41.0, 44.5 (d, $J_{P\text{-}C}$ = 5.6 Hz), 55.1, 55.2, 98.6, 113.5, 113.8, 126.8, 128.2 (d, $J_{P\text{-}C}$ = 11.5 Hz), 128.4, 128.6 (d, $J_{P\text{-}C}$ = 11.6 Hz), 130.6 (d, $J_{P\text{-}C}$ = 8.6 Hz), 131.1 (d, $J_{P\text{-}C}$ = 2.4 Hz), 131.3 (d, $J_{P\text{-}C}$ = 8.9 Hz), 131.8 (d, $J_{P\text{-}C}$ = 96.4 Hz), 131.8 (d, $J_{P\text{-}C}$ = 2.5 Hz), 133.7 (d, $J_{P\text{-}C}$ = 101.8 Hz), 135.9, 136.4 (d, $J_{P\text{-}C}$ = 6.6 Hz), 145.2, 158.2 and 158.7 ppm. GC–MS: m/z (%) = 610 (7) [M^+], 408 (22), 406 (20), 278 (100), 202 (17), 201 (33), 146 (44), 121 (36) and 77 (25). HRMS: m/z calcd for $C_{31}H_{29}O_3PTe$: 610.0917. Found: 610.1010.
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