Tetrahedron 67 (2011) 8763–8768

Contents lists available at SciVerse ScienceDirect

Tetrahedron

journal homepage: www.elsevier.com/locate/tet

An alternative approach for the synthesis of aryl–alkyl tellurides: reaction of aryl iodides with metal alkyltellurolates promoted by CuI

Márcio S. Silva *, João V. Comasseto

Instituto de Química, Universidade de São Paulo, Av. Prof. Lineu Prestes, 748, Caixa Postal 26077, 05508-000 São Paulo, SP, Brazil

ARTICLE INFO

Article history:
Received 24 July 2011
Received in revised form 1 September 2011
Accepted 6 September 2011
Available online 10 September 2011

Keywords:
Organotellurolate
Coupling reaction
Aryl–alkyl telluride
Copper iodide
Aryl iodide

ABSTRACT

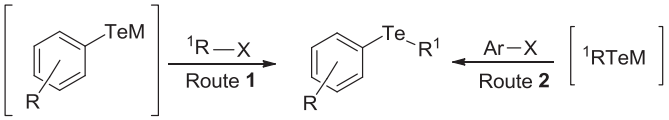
Aryl iodides react with metal organotellurolates in tetrahydrofuran/dimethylformamide in the presence of CuI (5 mol %) or CuI (5 mol %) and 1,10-phenanthroline (10 mol %) to afford the corresponding aryl–alkyl tellurides in good yields.

1. Introduction

The organic chemistry of tellurium is currently well developed, and several classes of organotellurides have found application as synthetic reagents,[1,2] particularly in the preparation of biologically active compounds.[3] Systematic investigations of the biological activities of organic derivatives of tellurium in different oxidation states have revealed interesting results.[4] For example, organotellurium (IV) compounds are potent inhibitors of cysteine proteases,[5] while organotellurium (II) compounds have been shown to be glutathione peroxidase mimetics.[4b] Moreover, a recent publication has claimed, with full justification, that the organic chemistry of tellurium must be considered an emerging area with great potential in the search for new and more potent drugs.[6] In the light of these comments, practical methods for the synthesis of organotellurium compounds are apposite, particularly those that avoid the manipulation of low molecular weight dialkylditellurides in the preparation of nucleophilic species of tellurium.

In recent years, the use of CuI in catalytic amounts to promote the coupling of aryl halides with substrates bearing different types of functional groups has been explored[7] and applied in the synthesis of structurally complex molecules.[7b] However, while CuI-promoted reactions that generate organosulfides and organoselenides are known, details concerning the analogous transformations to yield organotellurides are scarce.[8] Indeed, to the best of our knowledge, CuI-promoted reactions of metal organotellurolates with aryl halides have yet to be described.[9] The aim of the present study was, therefore, to explore the reactions of aryl iodides with metal organotellurolates in order to establish a novel and general route to aryl–alkyl tellurides.

Classical methods for the preparation of aryl–alkyl tellurides[1] involve the production of aryltellurolate anions through the insertion of elemental tellurium into an aryl organometallic, or by reduction of a diaryldiselenide and reaction of the metal aryltellurolate with an alkylating agent (Scheme 1, route 1).[1c] Both of these methods are incompatible with aryl rings bearing functional groups that are sensitive to reducing agents or to organometallics. In the present study, an inverse approach is adopted in which the alkyl moiety forms part of the metal organotellurolate generated under non-reducing conditions that are inert to a number of functional groups (Scheme 1, route 2).



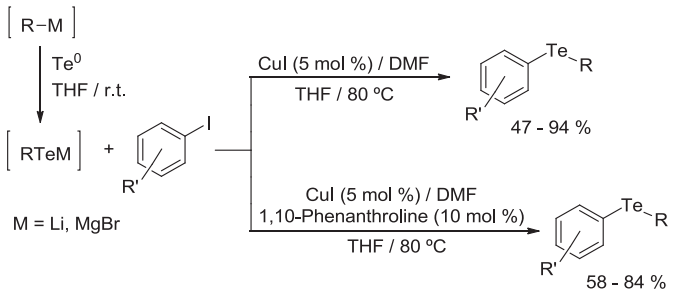
Scheme 1. General approaches to the synthesis of aryl–alkyl tellurides.

In this paper, we describe the preparation of aryl–alkyl tellurides in good yields by reaction of aryl iodides with lithium alkyltellurolates in dimethylformamide (DMF) and tetrahydrofuran

* Corresponding author. Tel.: +55 11 3091 2176; fax: +55 11 3815 5579; e-mail addresses: marciosasi@usp.br (M.S. Silva), jvcomass@iq.usp.br (J.V. Comasseto).

0040-4020/$ – see front matter © 2011 Elsevier Ltd. All rights reserved.
doi:10.1016/j.tet.2011.09.014

(THF) in the presence of 10 mol % 1,10-phenanthroline and/or 5 mol % CuI (Scheme 2).

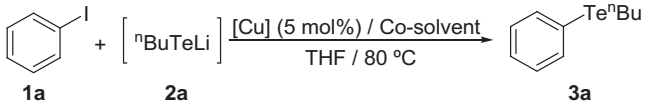


Scheme 2. Synthesis of aryl–alkyl tellurides by reaction of aryl iodides and lithium alkyltellurolates in the presence of CuI or CuI and 1,10-phenanthroline.

2. Results and discussion

Lithium organotellurolate **2** was prepared by the addition of the appropriate alkyl lithium to a suspension of elemental tellurium in THF at room temperature. The lithium organotellurolate (obtained as a clear yellow solution) was transferred via a cannula to a solution of the appropriate aryl iodide **1** and CuI (5 mol %) at room temperature. The yields of products **3** obtained after heating the reaction mixture at 80 °C (oil bath temperature) for the indicated times are presented in Tables 1–4. Yields were calculated on the basis of the quantity of product obtained after purification by column chromatography (CC) over silica gel, taking into account the amount of aryl iodide consumed during the reaction.

Table 1
Optimization of the reaction conditions

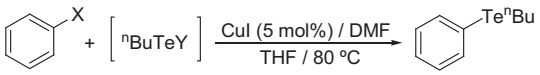


Entry	[Cu]	Co-solvent	Time[a] (h)	Yield of **3a**[b] (%)
1	—	—	24	25
2	CuI	—	24	48
3	CuI	—	6	40
4	—	DMSO	24	59
5	—	DMF	24	68
6	CuI	DMSO	6	85
7	CuI	Dioxane	6	56
8	CuI	CH_3CN	6	75
9	CuI	Toluene	12	52
10	CuI	DMF	6	94
11	CuBr	DMF	6	82
12	CuCl	DMF	12	38

[a] Reaction was monitored by TLC and GC.
[b] Isolated yield, after CC over silica gel, based upon phenyl iodide (**1a**) consumed.

Initially, control reactions were performed with THF, THF/dimethylsulfoxide (DMSO) or THF/DMF as solvent but in the absence of CuI (Table 1, entries 1,4 and 5), and in each case relatively low yields of **3a** were obtained after 24 h at 80 °C. Addition of 5 mol % of CuI to the reaction mixture improved the yields considerably irrespective of the solvent employed, thus showing that the presence of CuI is beneficial for yield enhancement. With the system CuI/DMF (Table 1, entry 10), the isolated yield of **3a** was 94% after 6 h at 80 °C. For comparison purposes, reactions involving the systems CuI/THF and CuI/DMSO were quenched after 6 h at 80 °C and the yields of **3a** obtained were 40 and 85%, respectively. These

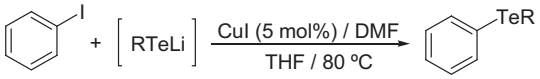
Table 2
Influence of aryl halide **1** and alkyl tellurium **2** on the course of the reaction



Entry	Ar–X (**1**)	^{n}BuTeY (**2**)	Time[a] (h)	Yield of **3a**[b] (%)
1	**1a**	^{n}BuTeMgBr **2b**	6	82
2	**1b**	^{n}BuTeLi **2a**	24	n.r.
3	**1c**	^{n}BuTeLi **2a**	24	n.r.
4	**1a**	$(^n$BuTe$)_2$ **2c**	24	29

[a] Reaction was monitored by TLC and GC.
[b] Isolated yield, after CC over silica gel, based upon phenyl iodide (**1a**) consumed.

Table 3
Influence of the lithium organotellurolate **2** on the course of the reaction

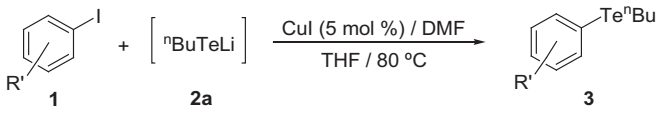


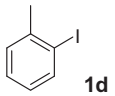
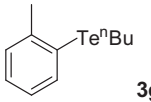
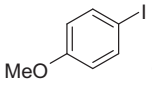
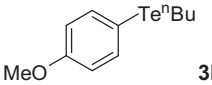
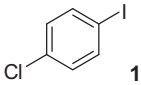
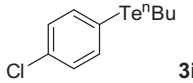
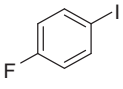
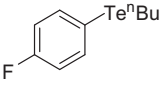
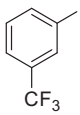
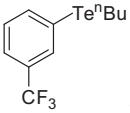
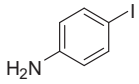
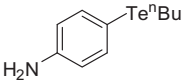
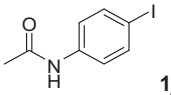
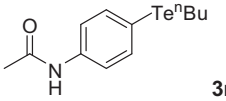
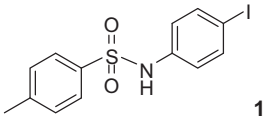
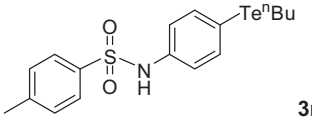
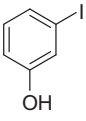
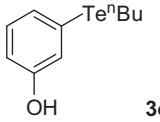
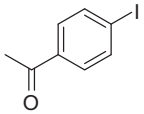
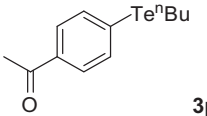
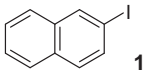
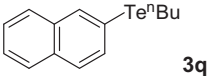
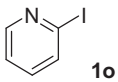
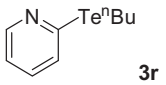
Entry	RTeLi (**2**)	Product (**3**)	Time (h)	Yield of **3**[a] (%)
1	MeTeLi **2d**	**3b**	8	80
2	^{s}BuTeLi **2e**	**3c**	6	84
3	^{t}BuTeLi **2f**	**3d**	6	75
4	PhTeLi **2g**	**3e**	12	51[b]
5	*p*MeOPhTeLi **2h**	**3f**	12	37[b]

[a] Isolated yield, after CC over silica gel, based upon phenyl iodide (**1a**) consumed.
[b] The phenyl iodide **1a** was recovered.

reactions were monitored by TLC and GC, and in each case, unreacted phenyl iodide could be detected after 6 h at 80 °C. All of the reactions were performed in triplicate and the yields did not change significantly (±5%) from run to run. Solvents other than THF, DMSO and DMF were tested together with different copper salts,

Table 4
Reaction of aryl iodides with lithium *n*-butyltellurolate using the system CuI/DMF[a]



Entry	ArI (**1**)	Product (**3**)	Time(h)	Yield of **3**[b] (%)
1	 **1d**	 **3g**	6	90
2	 **1e**	 **3h**	6	88
3	 **1f**	 **3i**	12	68
4	 **1g**	 **3j**	12	55
5	 **1h**	 **3k**	15	47
6	 **1i**	 **3l**	6	85
7	 **1j**	 **3m**	7	77
8	 **1k**	 **3n**	7	75
9	 **1l**	 **3o**	6	83
10	 **1m**	 **3p**	24	n.r.
11	 **1n**	 **3q**	6	84
12	 **1o**	 **3r**	10	56[c]

[a] Reaction conditions: aryl iodide **1** (1 mmol), lithium *n*-butyltellurolate (**2a**) (2 mmol), CuI (5 mol %) and DMF (1 mL) in THF (5 mL) at 80 °C under nitrogen.
[b] Isolated yields.
[c] A non-catalyzed substitution reaction of 2-iodopyridine (**1o**) with ^{n}BuTeLi (**2a**) has been reported previously.[10]

but in all cases inferior yields of **3a** were obtained as shown in Table 1.

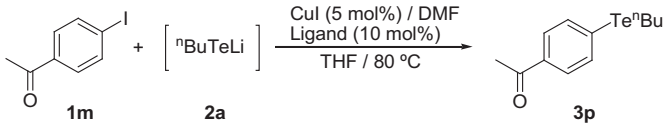
In view of its efficiency, the CuI/DMF system was adopted as standard in order to investigate the effects of the structures of the aryl halide **1** and the organotellurium **2** on products and yields. As shown in Table 2, reaction of ^{n}BuTeMgBr (**2b**) with phenyl iodide (**1a**) gave a yield of **3a** that was slightly lower than that observed in the reaction with ^{n}BuTeLi (**2a**) (cf. Table 1, entry 10 and Table 2, entry 1). The reaction of phenyl iodide (**1a**) with dibutylditelluride (**2c**) under the same conditions gave **3a** in only 29% yield based upon consumption of **1a** (Table 2, entry 4). In contrast, the reaction of ^{n}BuTeLi (**2a**) with phenyl bromide (**1b**) or phenyl chloride (**1c**) (Table 2, entries 2 and 3, respectively) did not afford the desired product **3a**.

Table 3 shows the influence of the nature of lithium organotellurolate **2** on products and yields. Use of lithium methyl-, *sec*-butyl and *tert*-butyltellurolate under the standard conditions afforded products in good yields (Table 3, entries 1–3). Lithium aryltellurolates (**2g** and **h**) did not react with phenyl iodide (**1a**) (Table 3, entries 4 and 5), but rather the product comprised the diaryltelluride (**3e** and **f**) corresponding to the lithium aryltellurolate and was contaminated with the diarylditelluride.

Finally, the influence of the nature of the aryl iodide **1** on product yield was investigated. The results obtained by reaction of lithium *n*-butyltellurolate (**2a**) with aryl iodides **1** bearing different functional groups are shown in Table 4, from which it can be observed that the reaction is compatible with several functionalities and affords good yields in most cases (Table 4, entries 1, 2 and 6–11). However, the presence of electron withdrawing groups seems to be detrimental to the success of the reaction, and for *p*-iodoacetophenone (**1m**) (Table 4, entry 10) no product was observed.

It is well known that the presence of additives in the medium can improve the yield of coupling reactions that are promoted by CuI.[7] On this basis, the CuI-promoted reaction between lithium *n*-butyltellurolate (**2a**) and *p*-iodoacetophenone (**1m**) was repeated in the presence of diverse additives. The desired product, *p*-butyltelluroacetophenone (**3p**), was formed in variable yields but the highest was afforded with 1,10-phenanthroline (Table 5, entry 2). In view of these results, the reactions with *p*-chloroiodobenzene (**3i**) (Table 6, entry 1), *p*-fluoroiodobenzene (**3j**) (Table 6, entry 2) and *m*-CF_3-iodobenzene (**3k**) (Table 6, entry 3) were performed in the presence of 1,10-phenanthroline, generating the products and yields shown in Table 6. In all cases yields were improved in comparison with those observed in the absence of additives (cf. Table 4, entries 3, 4 and 5, respectively).

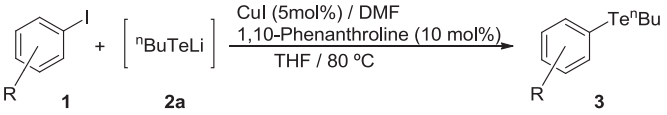
Table 5
Influence of the presence of ligands on reaction yields

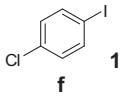
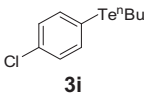
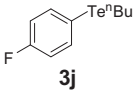
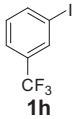
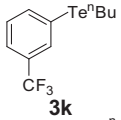
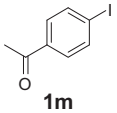
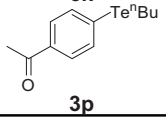


Entry	Ligand (10 mol %)	Time[a] (h)	Yield of **3p**[b] (%)
1	None	24	0
2	1,10-Phenanthroline	12	62
3	L-Proline	12	56
4	Glycol	24	28
5	Pyridine	15	45

[a] Reaction was monitored by TLC and GC.
[b] Isolated yield after CC over silica gel.

Table 6
Reaction of aryl iodides **1** with lithium *n*-butyltellurolate (**2a**) using the system CuI/1,10-phenanthroline/DMF



Entry	ArI	Product	Time[a] (h)	Yield of **3**[b] (%)
1	 **f**	 **3i**	12	84
2	 **1g**	 **3j**	12	69
3	 **1h**	 **3k**	24	58
4	 **1m**	 **3p**	12	62

[a] Reaction was monitored by TLC and GC.
[b] Isolated yield after CC over silica gel.

3. Conclusions

A mild and practical method has been developed by which to prepare functionalized aryl–alkyl tellurides under conditions that are compatible with a number of functionalities. It is of interest to note that the prepared compounds did not emit unpleasant odours and, when free of solvent, could be handled safely in the presence of light and air. Moreover, the compounds, when stored under refrigeration, remained stable for several months with no perceptible decomposition.

4. Experimental

4.1. General experimental methods

Reactions between metal organotellurolates and aryl iodides were carried out using dry solvents in anhydrous conditions under nitrogen. Standard syringe techniques were applied in the transfer of dry solvents, and some air-sensitive reagents were introduced into reaction vessels through rubber septa. THF was distilled from sodium benzophenone prior to use. DMF was distilled from CaH_2 and stored under 4 Å molecular sieves.

Reactions were monitored by TLC analysis on layers of silica gel 60 F_{254} (Merck) eluted with hexane/ethyl acetate (9:1) and visualized under UV light and/or by treatment with 5% *p*-anisaldehyde in 10% H_2SO_4 and heat. GC analyses were performed using a Shimadzu model 2014 instrument (column DB1 30×0.32 mm id×df=0.25 μm, flame ionization detector and chromatographic conditions: 60–230 °C—injector 240 °C—sample volume 1 μL—pressure 44.0 kPa). Flash CC was carried out over silica gel with particle size 230–400 mesh (Merck). IR spectra were measured using a Bomem MB 100/FT-IR spectrophotometer with samples dissolved in $CHCl_3$ solution: wavenumbers are reported in cm^{-1}. NMR spectra were recorded on Varian INOVA 200 (200 MHz) or 300 (300 MHz) instruments with samples dissolved

in $CDCl_3$. Chemical shifts are reported in δ (ppm) relative to TMS standard for ^1H and to $CDCl_3$ for ^{13}C NMR: coupling constants (J) are reported in hertz. Mass spectra (MS) were determined using a Shimadzu model CG-17A/QP5050A mass spectrometer and high resolution (HR) MS were recorded on a Bruker Daltonics MicroTOF.

4.2. Preparation of lithium organotellurolates

The appropriate organolithium reagent (2.0 mmol) in hexane was added dropwise to a suspension of elemental tellurium (0.255 g, 2 mmol) in 5 mL of dry THF maintained under nitrogen in a two necked 50 mL round bottomed flask equipped with a magnetic stirrer and rubber septa. A clear solution was formed after 5 min of stirring at room temperature.

4.3. General procedure for the reaction of lithium organotellurolates with aryl iodides promoted by CuI or CuI plus ligand

Lithium organotellurolate **2** (2 mmol; prepared as in Section 4.2) in 5 mL of dry THF was added to aryl iodide **1** (1.0 mmol), CuI (0.01g, 5 mol %) and dry DMF (1 mL) maintained under nitrogen in a two necked 50 mL round bottomed flask equipped with a magnetic stirrer, a reflux condenser and a rubber septum. The reaction mixture was heated at 80 °C (water bath temperature) and the progress of the reaction was monitored by TLC and GC. When consumption of **1** had ceased, the reaction mixture was quenched at room temperature by addition of saturated aqueous NH_4Cl solution (10 mL) and subsequently extracted with EtOAc (3×10 mL). The combined organic layers were washed with aqueous NaCl (10 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by CC over silica gel eluting with hexane/EtOAc mixtures with a gradual increase in polarity to afford products **3** with yields shown in Tables 1–4.

Reactions promoted by CuI plus ligand were carried out using an identical procedure but with the addition of 10 mol % of the appropriate ligand to the reaction mixture. The yields of products **3** are shown in Tables 5 and 6.

*4.3.1. n-Butyl(phenyl)tellane (**3a**).* Yellow oil; yield 0.248 g (94%); ^1H NMR (300 MHz, $CDCl_3$) (δ, ppm): 7.78–7.81 (m, 2H), 7.25–7.37 (m, 3H), 2.99 (t, J=7.5 Hz, 2H), 1.87 (quin, J=7.2 Hz, 2H), 1.48 (app sext, J=7.5 Hz, 2H), 0.98 (t, J=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm): 138.4, 129.3, 127.6, 112.1, 34.2, 25.3, 13.6, 8.7; GC/MS m/z (relative intensity): 264 (43) [M^+], 208 (32), 77 (100), 57 (59), 41 (62), 43 (69); CAS No 32343-98-9.[11]

*4.3.2. n-Butyl(o-tolyl)tellane (**3g**).* Yellow oil; yield 0.250 g (90%); ^1H NMR (300 MHz, $CDCl_3$) (δ, ppm): 7.65–7.68 (m, 1H), 7.17–7.27 (m, 2H), 7.02–7.08 (m, 1H), 2.93 (t, J=7.5 Hz, 2H), 2.47 (s, 3H), 1.83 (quin, J=7.2 Hz, 2H), 1.46 (app sext, J=7.2 Hz, 2H), 0.96 (t, J=7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm): 142.3, 136.7, 129.1, 127.5, 126.5, 116.6, 33.7, 26.5, 25.3, 13.5, 7.6; CG/MS m/z (relative intensity): 278 (29) [M^+], 91 (100), 57 (29), 41 (35); CAS No 874147-99-6.[11]

*4.3.3. n-Butyl(4-methoxyphenyl)tellane (**3h**).* Yellow oil; yield 0.259 g (88%); ^1H NMR (300 MHz, $CDCl_3$) (δ, ppm): 7.67 (d, J=13.2 Hz, 2H), 6.75 (d, J=13.2 Hz, 2H), 2.82 (t, J=11.1 Hz, 2H), 1.73 (quin, J=10.5 Hz, 2H), 1.39 (app sext, J=11.1 Hz, 2H), 0.88 (t, J=10.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm): 159.6, 140.8, 115.0, 100.5, 55.1, 33.8, 24.9, 13.4, 8.7; CAS No 32343-98-9.[11]

*4.3.4. n-Butyl(4-chlorophenyl)tellane (**3i**).* Yellow oil; yield 0.202 g (68%); ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.62 (d, J=8.6 Hz, 2H), 7.16 (d, J=8.6 Hz, 2H), 2.89 (t, J=7.4 Hz, 2H), 1.76 (quin, J=7.2 Hz, 2H), 1.38 (app sext, J=7.6 Hz, 2H), 0.90 (t, J=7.4 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 139.7, 134.0, 129.3, 109.2, 33.8, 25.0, 13.3, 8.9; CAS No 874148-10-4.[11]

*4.3.5. n-Butyl(4-fluorophenyl)tellane (**3j**).* Yellow oil; yield 0.155 g (55%); ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.69 (dd, J=8.6 and 5.6 Hz, 1H), 6.90 (t, J=9.0 Hz, 3H), 2.87 (t, J=7.4 Hz, 2H), 1.75 (quin, J=7.0 Hz, 2H), 1.37 (app sext, J=7.4 Hz, 2H), 0.89 (t, J=7.0 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 162.3 (^1J=246.05 Hz), 140.8 (^3J=7.45 Hz), 116.4 (^2J=20.65 Hz), 105.1 (^4J=3.55 Hz), 33.8, 25.0, 13.4, 8.9; HRMS (ESI) m/z: calcd for $C_{10}H_{13}FTe$: 282.0063; found: 283.0094 (+H); CAS No 1159428-50-8.[12]

*4.3.6. n-Butyl(3-(trifluoromethyl)phenyl)tellane (**3k**).* Yellow oil; yield 0.156 g (47%); ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.85–7.93 (m, 2H), 7.49–7.53 (m, 1H), 7.29 (t, J=7.6 Hz, 1H), 2.96 (t, J=7.4 Hz, 2H), 1.80 (quin, J=7.2 Hz, 2H), 1.42 (app sext, J=7.4 Hz, 2H), 0.91 (t, J=7.2 Hz, 3H); CAS No 113556-16-4.[13]

*4.3.7. 4-(Butyltellanyl)benzenamine (**3l**).* Yellow oil; yield 0.237 g (85%); IR ($CHCl_3$ solution) (ν_{max}, cm^{-1}): 3352, 2956, 1488, 616; ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.55 (d, J=8.4 Hz, 2H), 6.54 (d, J=8.6 Hz, 2H), 3.7 (br s, 2H), 2.78 (t, J=7.4 Hz, 2H), 1.72 (quin, J=7.0 Hz, 2H), 1.36 (app sext, J=7.4 Hz, 2H), 0.88 (t, J=7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 146.3, 141.1, 116.0, 97.4, 33.8, 24.9, 13.4, 8.7; GC/MS m/z (relative intensity): 279 (23) [M^+], 222 (35), 93 (100), 57 (24), 41 (37); HRMS (ESI) m/z: calcd for $C_{10}H_{15}NTe$: 279.02667; found: 280.0349 (+H).

*4.3.8. N-(4-(Butyltellanyl)phenyl)acetamide (**3m**).* Yellow oil; yield 0.247 g (77%); IR ($CHCl_3$ solution) (ν_{max}, cm^{-1}): 3299, 2956, 1668, 821, 510; ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.96 (br s, 1H), 7.64 (d, J=8.4 Hz, 2H), 7.36 (d, J=8.6 Hz, 2H), 2.86 (t, J=7.6 Hz, 2H), 2.16 (s, 3H), 1.75 (quin, J=7.0 Hz, 2H), 1.37 (app sext, J=7.6 Hz, 2H), 0.88 (t, J=7.4 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 168.7, 139.4, 137.6, 120.6, 105.7, 33.8, 24.9, 24.4, 13.3, 8.7; GC/MS m/z (relative intensity): 321 (7) [M^+], 222 (11), 93 (54), 57 (39), 41 (100); HRMS (ESI) m/z: calcd for $C_{12}H_{17}NOTe$: 321.0372; found: 344.0270 (+Na).

*4.3.9. N-(4-(Butyltellanyl)phenyl)-4-benzenesulfonamide(**3n**).* Yellow oil; yield 0.325 g (75%); IR ($CHCl_3$ solution) (ν_{max}, cm^{-1}): 3256, 2922, 1159, 665; ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.70 (d, J=8.2 Hz, 2H), 7.54 (d, J=8.6 Hz, 2H), 7.21 (d, J=8.0 Hz, 2H), 6.93 (d, J=8.6 Hz, 2H), 2.82 (t, J=7.4 Hz, 2H), 2.36 (s, 3H), 1.71 (quin, J=7.0 Hz, 2H), 1.34 (app sext, J=7.4 Hz, 2H), 0.86 (t, J=7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 143.9, 139.4, 136.2, 135.8, 129.7, 127.2, 121.7, 107.1, 33.7, 24.9, 21.5, 13.3, 8.7; GC/MS m/z (relative intensity): 278 (38) [M^+–Tosyl], 91 (100), 57 (42), 41 (58); HRMS (ESI) m/z: calcd for $C_{17}H_{21}NO_2Ste$: 433.0355; found: 456.0247 (+Na).

*4.3.10. 4-(Butyltellanyl)phenol (**3o**).* Yellow oil; yield 0.232 g (83%); IR ($CHCl_3$ solution) (υ_{max}, cm^{-1}): 3375, 2957, 1575, 854; ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.12–7.20 (m, 2H), 6.29 (tap, J=7.8 Hz, 1H), 6.64–6.69 (m, 1H), 4.88 (brs, 1H), 2.84 (t, J=7.4 Hz, 2H), 1.72 (quin, J=7.0 Hz, 2H), 1.32 (app sext, J=7.4 Hz, 2H), 0.83 (t, J=7.4 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 155.5, 130.4, 130.1, 124.6, 114.6, 112.8, 33.9, 25.1, 13.4, 8.5; GC/MS m/z (relative intensity): 278 (23) [M^+], 94 (100), 57 (30), 41 (26); HRMS (ESI) m/z: calcd for $C_{10}H_{14}OTe$: 280.01069; found: 303.0041 (+Na).

*4.3.11. 1-(4-(Butyltellany)phenyl)ethanone (**3p**).* Yellow oil; yield 0.190 g (62%); ^1H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.67–7.61 (m, 4H), 2.88 (t, J=7.2 Hz, 2H), 2.48 (s, 3H), 1.73 (q, J=8.1 Hz, 2H), 1.33 (app sext, J=7.6 Hz, 2H), 0.83 (t, J=7.5 Hz, 3H); ^{13}C NMR (50 MHz,

$CDCl_3$) (δ, ppm): 197.7, 136.7, 135.9, 128.6, 121.0, 33.8, 26.7, 25.2, 13.5, 8.8; CAS No 874148-02-4.[11]

*4.3.12. Butyl(naphthalen-2-yl)tellane (**3q**).* Yellow oil; yield 0.264 g (84%); ^{1}H NMR (200 MHz, $CDCl_3$) (δ, ppm): 8.20 (br s, 1H), 7.61–7.81 (m, 4H), 7.40–7.50 (m, 2H), 2.97 (t, J=7.6 Hz, 2H), 1.80 (quin, J=7.2 Hz, 2H), 1.39 (app sext, J=7.4 Hz, 1H), 0.89 (t, J=7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 137.6, 135.0, 134.1, 132.4, 128.1, 127.7, 127.1, 126.2, 126.1, 109.2, 33.9, 25.0, 13.4, 8.6; CAS No 95849-65-3.[14]

*4.3.13. 2-(Butyltellanyl)pyridine (**3r**).* Yellow oil; yield 0.148 g (56%); ^{1}H NMR (200 MHz, $CDCl_3$) (δ, ppm): 8.48 (dq, J=5.0 and 1.0 Hz, 1H), 7.48 (dt, J=7.8 and 1.0 Hz, 1H), 7.32 (dt, J=7.4 and 2.0 Hz, 1H), 7.01 (ddd, J=7.4, 6.2 and 1.4 Hz, 1H), 3.14 (t, J=7.4 Hz, 2H), 1.90 (quin, J=7.2 Hz, 2H), 1.43 (app sext, J=7.4 Hz, 2H), 0.93 (t, J=7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 150.7, 141.2, 135.3, 131.6, 120.9, 33.9, 25.2, 13.4, 9.3; CAS No 181647-39-2.[10]

*4.3.14. Methyl(phenyl)tellane (**3b**).* Yellow oil; yield 0.178 g (80%); ^{1}H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.05–7.59 (m, 2H), 7.27–7.40 (m, 1H), 7.11–7.17 (m, 2H), 2.12 (s, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 136.7, 129.1, 128.7, 127.1, −16.6; GC/MS m/z (relative intensity): 222 (59) [M^+], 207 (52), 77 (100), 51 (57); CAS No 161063-16-7.[15]

*4.3.15. sec-Butyl(phenyl)tellane (**3c**).* Yellow oil; yield 0.222 g (84%); ^{1}H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.78–7.81 (m, 2H), 7.17–7.34 (m, 3H), 3.40 (app sext, J=7.0 Hz, 1H), 1.69 (quin, J=9.0 Hz, 2H), 1.61 (d, J=7.2 Hz, 3H), 0.98 (t, J=7.2 Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 140.3, 129.1, 127.9, 111.6, 32.6, 26.1, 24.1, 14.1; GC/MS m/z (relative intensity): 264 (36) [M^+], 208 (68), 77 (100), 57 (71), 41 (46); CAS No 83817-37-2.[16]

*4.3.16. tert-Butyl(phenyl)tellane (**3d**).* Yellow oil; yield 0.198 g (75%); ^{1}H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.88–7.92 (m, 2H), 7.22–7.42 (m, 3H), 1.59 (s, 9H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 142.1, 128.9, 128.4, 112.8, 35.5, 29.1; GC/MS m/z (relative intensity): 264 (20) [M^+], 208 (33), 77 (71), 57 (100), 41 (39); HRMS (ESI) m/z: calcd for $C_{10}H_{14}Te$: 264.01577; found: 265,0160 (+H); CAS No 83817-38-3.[17]

*4.3.17. Diphenyltellane (**3e**).* Yellow oil; yield 0.158 g (56%); ^{1}H NMR (300 MHz, $CDCl_3$) (δ, ppm): 7.73–7.77 (m, 4H), 7.23–7.35 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) (δ, ppm): 138.1, 129.6, 127.9, 114.8; CAS No 298680-10-0.[18]

*4.3.18. Bis(4-methoxyphenyl)tellane (**3f**).* Yellow oil; yield 0.151 g (44%); ^{1}H NMR (200 MHz, $CDCl_3$) (δ, ppm): 7.62 (d, J=6.6 Hz, 4H), 6.75 (d, J=6.6 Hz, 4H), 3.76 (s, 6H); ^{13}C NMR (50 MHz, $CDCl_3$) (δ, ppm): 159.6, 139.7, 115.4, 104.3, 55.2; CAS No 4456-34-2.[19]

Acknowledgements

The authors wish to thank FAPESP, CNPq and CAPES for financial support.

Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tet.2011.09.014.

References and notes

1. (a) Irgolic, K. C. Organotellurium compounds In. *Houben-weyl Methods of Organic Chemistry*; Klaman, R., Ed.; Georg Thieme: Stuttgart, 1990; Vol. E12b; (b) Comasseto, J. V.; Clososki, G. C.; Cunha, R. L. O. R. Tellurium In. *Comprehensive Organometallic Chemistry III*; Crabtree, R. H., Mingos, D. M. P., Eds.; Elsevier: Amsterdam, 2007; Vol. 9, pp 587–648; (c) Petragnani, N.; Stefani, H. A. *Tellurium in Organic Synthesis, Second Edition*; Academic: London, 2007.
2. For reviews see: (a) Comasseto, J. V.; Barrientos-Astigarraga, R. R. *Aldrichimica Acta* **2000**, *33*, 66–78; (b) Zeni, G.; Lüdtke, D. S.; Panatieri, R. B.; Braga, A. L. *Chem. Rev.* **2006**, *106*, 1032–1076.
3. For reviews see: (a) Wendler, E. P.; Dos Santos, A. A. *Synlett* **2009**, 1034–1040; (b) Princival, J. L.; Dos Santos, A. A.; Comasseto, J. V. *J. Braz. Chem. Soc.* **2010**, *21*, 2042–2054; (c) For a recent example see: Ferrarini, R. S.; Dos Santos, A. A.; Comasseto, J. V. *Tetrahedron Lett.* **2010**, *51*, 6843–6846.
4. For reviews see: (a) Nogueira, C. W.; Zeni, G.; Rocha, J. B. T. *Chem. Rev.* **2004**, *104*, 6255–6285; (b) Cunha, R. L. O. R.; Gouvea, I. E.; Juliano, L. *An. Acad. Bras. Cienc.* **2009**, *81*, 393–407; (c) Alberto, E. E.; Nascimento, V.; Braga, A. L. *J. Braz. Chem. Soc.* **2010**, *21*, 2032–2041.
5. (a) Albeck, A.; Weitman, H.; Sredni, B.; Albeck, M. *J. Inorg. Chem.* **1998**, *37*, 1704–1712; (b) Cunha, R. L. O. R.; Urano, M. E.; Chagas, J. R.; Almeida, P. C.; Bincoletto, C.; Tersariol, I. L. S.; Comasseto, J. V. *Bioorg. Med. Chem. Lett.* **2005**, *15*, 755–760.
6. Ba, L. A.; Döring, M.; Jamier, V.; Jacob, C. *Org. Biomol. Chem.* **2010**, *8*, 4203–4216.
7. For reviews see: (a) Ma, D.; Cai, Q. *Acc. Chem. Res.* **2008**, *41*, 1450–1460; (b) Evano, G.; Blanchard, N.; Toumi, M. *Chem. Rev.* **2008**, *108*, 3054–3131; (c) For a recent example see: Huang, Y.-B.; Yang, C.-T.; Yi, J.; Deng, X.-J.; Fu, Y.; Liu, L. *J. Org. Chem.* **2011**, *76*, 800–810; (d) Niu, L.-F.; Cai, Y.; Liang, C.; Hui, X.-P.; Xu, P.-F. *Tetrahedron* **2011**, *67*, 2878–2881.
8. For a review see: Beletskaya, I. P.; Ananikov, V. P. *Chem. Rev.* **2011**, *111*, 1596–1636.
9. Suzuki, H.; Abe, H.; Ohmasa, N.; Osuka, A. *Chem. Lett.* **1981**, 1115–1116 This paper describes the formation of diaryltellurides by heating a mixture of nitroiodoarenes and sodium benzenetellurolate (prepared by reduction of diphenylditelluride with $NaBH_4$ in hexamethylphosphoramide) at 90 °C in the presence of stoichiometric amounts of CuI. In most cases the yields obtained were only moderate (65–53%), and when DMF or DMSO were used as solvents, the expected products were not formed.
10. Kondo, Y.; Shilai, M.; Ushiyama, M.; Sakamoto, T. *J. Chem. Soc., Perkin Trans. 1* **1996**, 1781–1782.
11. Cella, R.; Cunha, R. L. O. R.; Reis, A. E. S.; Pimenta, D. C.; Klitzke, C. F.; Stefani, H. A. *J. Org. Chem.* **2006**, *71*, 244–250.
12. Singh, F. V.; Milagre, H. M. S.; Eberlin, M. N.; Stefani, H. A. *Tetrahedron Lett.* **2009**, *50*, 2312–2316.
13. Comasseto, J. V.; Lang, E. S.; Ferreira, J. T. B.; Simonelli, F.; Correia, V. R. *J. Organomet. Chem.* **1987**, *364*, 329–340.
14. Cunha, R. L. O. R.; Omori, A. T.; Castelani, P.; Toledo, F. T.; Comasseto, J. V. *J. Organomet. Chem.* **2004**, *683*, 3631–3636.
15. Khurana, J. M.; Sharma, V.; Chack, S. A. *Tetrahedron* **2007**, *63*, 966–969.
16. Cougnon, F.; Feray, L.; Bazin, S.; Bertrand, M. *Tetrahedron* **2007**, *63*, 11959–11964.
17. Tamago, S.; Iida, K.; Yoshida, J. *Tetrahedron Lett.* **2001**, *42*, 5061–5064.
18. Taniguchi, N. *J. Org. Chem.* **2007**, *72*, 1241–1245.
19. Alves, D.; Pena, J. M.; Vieira, A. S.; Botteselle, G. V.; Guadagnin, R. C.; Stefani, H. A. *J. Braz. Chem. Soc.* **2009**, *20*, 988–992.