A General Method for the Synthesis of *Te*-Aryl Phosphorotellurate Triesters

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Received 15 April 2004

Dedicated to Professor Teruaki Mukaiyama on the occasion of the celebration of his 77th birthday

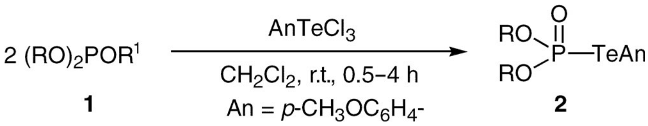
Abstract: A general and practical method for the synthesis of *Te*-aryl phosphorotellurates is described. A series of *Te*-aryl phosphorotellurates could be synthesized in good to excellent yields by the reaction of dialkyl, diaryl and trialkyl phosphites with arenetellurynyl and arenetellurenyl halides. A phenylphosphonotelluroate and a phosphorotelluramidate were also prepared by a similar procedure.

Key words: phosphorus, phosphorotellurates, tellurium

General methods for the synthesis of phosphate esters and phosphorochalcogenoates esters bearing thio and/or seleno substituents have been well established, and many kinds of such compounds have been prepared for both synthetic and biological studies.[1] In contrast, there are only two examples of the heavier analogues, i.e. phosphorotellurate esters, as unstable compounds, reported so far.[2] Both examples are the *Te*-alkyl esters, which were synthesized by alkylation of sodium or triethylammonium salts of phosphorotellurates. In spite of the growing utilities of organotellurium compounds in organic synthesis,[3] there is no other way for the preparation of phosphorotellurate esters and therefore only little was known about their reactivities, although the phosphorotellurate esters should provide a great interest on account of their reactivities. Herein we wish to report a general, and practical method for the preparation of *Te*-aryl phosphorotellurates by the reaction of phosphites with arenetellurynyl or arenetellurenyl halides. Syntheses of a phenylphosphonotellurate and a phosphorotelluramidate by a similar procedure are also described.

We have already reported tellurium tetrachloride-mediated oxidative phosphorylation of alcohols and thiols by trialkyl phosphites.[4,5] During the course of our mechanistic study of this reaction using *p*-methoxybenzenetellurynyl chloride instead of $TeCl_4$, we found that *Te*-*p*-methoxyphenyl *O*,*O*′-dialkyl phosphorotellurate was formed in concomitant with the phosphorylation of the alcohol. Then, the reaction condition was optimized for maximizing the yields of these rare phosphorotellurates.

In the presence of an excess triethylamine, *p*-methoxybenzenetellurynyl chloride ($AnTeCl_3$)[6] smoothly reacted with 2 equivalents of dialkyl or diaryl phosphite at room temperature to afford the corresponding *Te*-aryl *O*,*O*′-dialkyl(diaryl) phosphorotellurate in excellent yields (Scheme 1, Table 1, entries 1–5). Trialkyl phosphites also gave the phosphorotellurates in excellent yields via the Arbuzov-type dealkylation in the presence of a small amount of 2,6-lutidine instead of triethylamine (Table 1, entries 7–10). Although most of trialkyl phosphites successfully reacted without base to give the desired product in good yields, the addition of 2,6-lutidine was found to be effective to avoid deposition of elemental tellurium with decomposition of the product during the reaction, which sometimes occurred without base (Table 1, entry 6).



Scheme 1

The reactions using $AnTeCl_3$ required two molar equivalents of the phosphite. The fact shows that the Te(IV) reagent is first reduced to AnTeCl, and then this reactive Te(II) species react with another mole of phosphite, resulting in the formation of the phosphorotellurates. Based

Table 1 Synthesis of Phosphorotellurate **2** with Arenetellurynyl Chloride and Di- or Trialkylphosphites

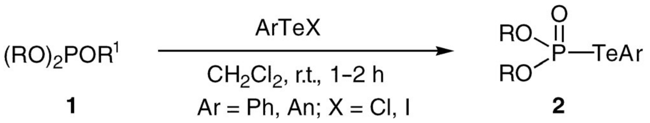
Entry	R	R[1]	Base[a]	Time (h)	Product	Yield (%)
1	Et	H	Et_3N	0.5	**2a**	86
2	*n*-Bu	H	Et_3N	1.0	**2b**	83
3	Bn	H	Et_3N	4.0	**2c**	95
4	Ph	H	Et_3N	2.0	**2d**	95
5	*o*-Xy[b]	H	Et_3N	1.0	**2e**	76
6	Me	Me	none	0.2	**2f**	77[c]
7	Me	Me	2,6-lutidine	1.0	**2f**	94
8	Et	Et	2,6-lutidine	0.7	**2a**	91
9	*n*-Bu	*n*-Bu	2,6-lutidine	0.7	**2b**	89
10	Bn	Bn	2,6-lutidine	1.0	**2c**	89

[a] Amount of base: Et_3N (3.3–3.6 equiv); 2,6-lutidine (0.5 equiv).
[b] *o*-Xy represents *o*-xylylenediyl.
[c] Elemental tellurium was deposited due to decomposition.

SYNTHESIS 2004, No. 9, pp 1481–1485
Advanced online publication: 26.05.2004
DOI: 10.1055/s-2004-822386; Art ID: C03004SS

on the consideration, we chose Te(II) species instead of $AnTeCl_3$. Arenetellurenyl halides[7a] (ArTeX, X = Cl, I) are Te(II) species of choice, which are easily prepared by the oxidation of diaryl ditellurides with halogen sources such as sulfuryl chloride or iodine. As expected, the reaction of ArTeX with an equimolar amount of phosphites gave the corresponding phosphorotellurates in good yields (Scheme 2, Table 2). This procedure provides a quite general and efficient route toward the phosphorotellurate triesters owing to wide availability of various diorganyl ditellurides by several methods.[7b]

Although phosphoroselenoate esters can be prepared by the reaction of phosphorochloridates with alkylselenide salt,[8] an investigation of tellurium version was not successful at all probably due to instability of the product under strong basic condition.



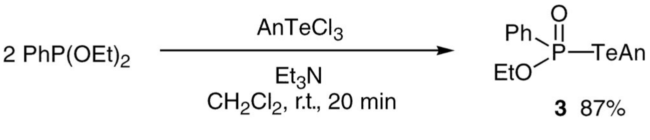
Scheme 2

Table 2 Synthesis of **2** from Arenetellurenyl Halides

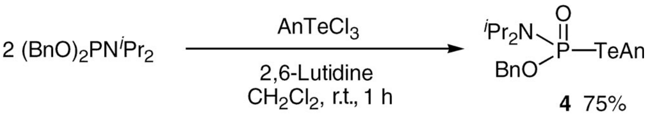
Entry	R	R^1	ArTeX[a]	Base[b]	Time (h)	Product	Yield (%)
1	Me	Me	AnTeCl	2,6-lutidine	2.0	**2f**	83
2	*n*-Bu	*n*-Bu	AnTeCl	2,6-lutidine	1.5	**2b**	80
3	Ph	H	AnTeCl	Et_3N[c]	1.0	**2d**	88
4	Et	H	PhTeI[d]	Et_3N	2.0	**2g**	92
5	Ph	H	PhTeI[d]	Et_3N	1.0	**2h**	88
6	Bn	H	PhTeI[e]	Et_3N	1.0	**2i**	95

[a] AnTeCl was prepared in situ from An_2Te_2 and SO_2Cl_2. PhTeI was prepared from Ph_2Te_2 and I_2 and used in situ or after isolation.
[b] Amount of base: 2,6-lutidine (0.5 equiv); Et_3N (1.2–1.4 equiv, except entry 3).
[c] Excess amount of Et_3N (3.5 equiv) was used.
[d] Isolated PhTeI was used.
[e] PhTeI was prepared and used in situ.

In addition to the phosphorotellurates, *Te*-aryl phenylphosphonotellurate and *Te*-aryl phosphorotelluramidate are successfully prepared by the same procedure from *O*,*O*′-diethyl phenylphosphonite and *O*,*O*′-dibenzyl *N*,*N*-diisopropyl phosphoramidite, respectively (Schemes 3 and 4). Contrary to the case of the phosphonite, *O*-ethyl diphenylphosphinite gave diaryl ditelluride quantitatively instead of the phosphinotellurate ester.



Scheme 3



Scheme 4

The phosphorotellurates and derivatives described here are stable enough to handle in air, and could be purified by a simple column chromatography on silica gel. They could be stored in solution for weeks without detectable decomposition. It is in contrast to the reported *Te*-alkyl phosphorotellurates that are known as too unstable and sensitive towards air and moisture.[2] The *Te*-aryl phosphorotellurates gradually turned orange to form the corresponding diaryl ditellurides and pyrophosphates when they are kept neat. Noteworthy is that a cyclic phosphorotellurate **2e**, isolated in a crystalline form, is quite stable even in solid state and did not decompose for months.

The structure of the phosphorotellurates was confirmed by ^1H, ^{13}C, ^{31}P, ^{125}Te NMR and FABMS data. Characteristic ^{125}Te-satellite peaks were observed in ^{31}P NMR spectra with large coupling constants ($^1J_{^{31}P\text{–}^{125}Te}$ = 1128–1552 Hz) that indicated the direct connection between the phosphorus and the tellurium atoms (Table 3). Similar large coupling constants were reported for a *Te*-alkyl phosphorotellurate and phosphoroselenoates. ^{125}Te NMR spectra showed doublet peaks with the same coupling constants observed in ^{31}P NMR spectra. A characteristic isotopic fingerprint around calculated molecular mass weights in their FABMS spectra also supported the expected structure.

Since only little is known about the reactivities of phosphorotellurates,[9] further study of the phosphorotellurates is now under investigation.

Phosphorotellurates 2 from $AnTeCl_3$; *Te*-*p*-Methoxyphenyl *O*,*O*′-Diethyl Phosphorotellurate (2a); Typical Procedure (Scheme 1)

To a stirred suspension of *p*-methoxybenzenetellurynyl chloride (37.3 mg, 0.11 mmol) in CH_2Cl_2 (1.0 mL) was added successively diethyl phosphite (34.5 mg, 0.25 mmol) and Et_3N (36.4 mg, 0.36 mmol) at r.t. under N_2. After stirring the mixture at r.t. until the phosphite had been almost consumed (monitored by TLC), H_2O was added in one portion. The mixture was extracted with EtOAc, washed successively with aq 1 N HCl, aq sat. $NaHCO_3$ and brine. The organic extract was dried ($MgSO_4$), evaporated to dryness, and the residue was purified by column chromatography on silica gel (EtOAc–*n*-hexane, 1:1) to afford **2a** (34.9 mg, 86%) as a colorless oil. When a trialkyl phosphite was used, 2,6-lutidine (ca. 0.5 equiv) was applied as base instead of Et_3N (Tables 1 and 3).

Phosphorotellurates 2 from ArTeX; *Te*-*p*-Methoxyphenyl *O*,*O*′-Diphenyl Phosphorotellurate (2d); Typical Procedure (Scheme 2)

To a stirred solution of bis(*p*-methoxyphenyl) ditelluride (39.7 mg, 0.085 mmol) in CH_2Cl_2 (1.0 mL) was added SO_2Cl_2 (13.7 mg, 0.10 mmol) at r.t. to prepare *p*-methoxybenzenetellurenyl chloride in situ. After the color of the reaction mixture turned orange to dark red (ca. 1 h), diphenyl phosphite (47.5 mg, 0.20 mmol) was added

Table 3 ^{1}H, ^{13}C, ^{31}P, ^{125}Te NMR Data for Compound **2a**–**i**, **3** and **4**

Product	^{1}H NMR (400 MHz, $\mathrm{CDCl_3}$) δ, J (Hz)	^{13}C{^{1}H} NMR (100 MHz, $\mathrm{CDCl_3}$) δ, J (Hz)	^{31}P{^{1}H} NMR (162 MHz, $\mathrm{CDCl_3}$) δ	^{125}Te{^{1}H} NMR (126 MHz, $\mathrm{CDCl_3}$) δ
2a	1.32 (t, 6 H, J = 7.2), 3.80 (s, 3 H), 4.05–4.21 (m, 4 H), 6.77–6.82 (m, 2 H), 7.72–7.76 (m, 2 H)	15.76 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 7.3), 55.14, 63.31 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 5.7), 97.68 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 7.9), 115.63 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.2), 141.80 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 3.4), 160.46 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.8)	–0.79 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1370 Hz)	362.6 (d, $^{1}J_{\mathrm{P\text{-}Te}}$ = 1370 Hz)
2b	0.90 (t, 6 H, J = 7.4), 1.35 (sext, 4 H, J = 7.4), 1.63 (quint, 4 H, J = 7.1), 3.80 (s, 3 H), 3.98–4.11 (m, 4 H), 6.79 (d, 2 H, J = 8.3), 7.74 (dd, $^{3}J_{\mathrm{H\text{-}H}}$ = 8.4, $^{4}J_{\mathrm{P\text{-}H}}$ = 1.2)	13.56, 18.73, 31.90 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 7.2), 55.16, 67.04 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.2), 97.67 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 7.7), 115.59 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.4), 141.82 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 3.7), 160.47 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.4)	–0.71 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1364 Hz)	362.5 (d, $^{1}J_{\mathrm{P\text{-}Te}}$ = 1364 Hz)
2c	3.76 (s, 3 H), 5.05 (dd, 2 H, J = 9.0, 11.8), 5.08 (d, 2 H, J = 8.6, 11.8), 6.69–6.71 (m, 2 H), 7.25–7.35 (m, 10 H), 7.62–7.66 (m, 2 H)	55.12, 68.52 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.0), 97.40 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 8.0), 115.60 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.2), 127.97, 128.38, 128.43, 135.32 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 7.8), 142.0 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 3.5), 160.54 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.4)	–1.53 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1418 Hz)	396.7 (d, $^{1}J_{\mathrm{P\text{-}Te}}$ = 1418 Hz)
2d	3.80 (s, 3 H), 6.73–6.75 (d, 2 H), 7.13–7.36 (m, 10 H), 7.55–7.58 (m, 2 H)	55.14, 97.16 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 8.0), 115.68 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.0), 120.93, 120.97, 125.43 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.0), 129.37, 129.71 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 1.0), 142.51 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 3.0), 150.17 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 8.0), 160.78 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.0)	–9.98 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1552 Hz)	445.9 (d, $^{1}J_{\mathrm{P\text{-}Te}}$ = 1552 Hz)
2e	3.79 (s, 3 H), 5.15 (dd, 2 H, $J_{\mathrm{H\text{-}H}}$ = 14.2, $J_{\mathrm{P\text{-}H}}$ = 17.0), 5.19 (dd, 2 H, $J_{\mathrm{H\text{-}H}}$ = 14.2, $J_{\mathrm{P\text{-}H}}$ = 21.4), 6.79–6.82 (m, 2 H), 7.15–7.20 (m, 2 H), 7.28–7.32 (m, 2 H), 7.82–7.85 (m, 2 H)	55.19, 68.12 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 8.0), 95.77 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 7.4), 115.91 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.3), 128.29, 128.71, 134.51, 142.39 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 3.4), 160.83 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.5)	3.80 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1438 Hz)	389.3 (d, $^{1}J_{\mathrm{P\text{-}Te}}$ = 1438 Hz)
2f	3.72 (d, 6 H, $^{3}J_{\mathrm{P\text{-}H}}$ = 13.2), 3.80 (s, 3 H), 6.80 (d, 2 H, J = 8.4), 7.74 (dd, J = 8.8, $^{4}J_{\mathrm{P\text{-}H}}$ = 1.4)	53.55 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 5.1), 55.15, 97.14 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 8.2), 115.74 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.3), 141.91 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 3.6), 160.58 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.6)	3.93 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1405 Hz)	331.7 (d, $^{1}J_{\mathrm{P\text{-}Te}}$ = 1405 Hz)
2g	1.31 (t, 6 H, $^{2}J_{\mathrm{H\text{-}H}}$ = 7.2), 4.05–4.21 (m, 4 H), 7.25 (t, 2 H, J = 7.2), 7.36 (t, 1 H, J = 6.0), 7.84 (d, 2 H, J = 8.0)	15.69 ($^{3}J_{\mathrm{P\text{-}C}}$ = 7.4), 63.41 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 5.5), 108.77 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 8.0), 128.82 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.2), 129.59 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 1.9), 139.83 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 4.0)	–0.57 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1343 Hz)	382.2 ($^{1}J_{\mathrm{P\text{-}Te}}$ = 1343 Hz)
2h	0.89 (t, 6 H, J = 7.6), 1.34 (sext, 4 H, J = 7.6), 1.63 (quint, 4 H, J = 7.6), 4.00–4.12 (m, 4 H), 7.22–7.28 (m, 2 H), 7.33–7.38 (m, 1 H), 7.82–7.84 (m, 2 H)	13.51, 18.68, 31.82 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 7.2), 67.12 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.2), 108.78 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 7.9), 128.76 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.2), 129.54 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 1.8), 139.81 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 4.0)	–0.40 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1339 Hz)	381.0 ($^{1}J_{\mathrm{P\text{-}Te}}$ = 1339 Hz)
2i	5.04 (d, 1 H, J = 12.0), 5.07 (d, 1 H, J = 11.6), 5.08 (d, 1 H, J = 12.0), 5.10 (d, 1 H, J = 11.6), 7.17 (t, 2 H, J = 7.6), 7.23–7.37 (m, 11 H), 7.73–7.78 (m, 2 H)	68.67 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.0), 108.52 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 8.1), 128.03, 128.44, 128.46, 128.95 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.4), 129.62 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 2.0), 135.21 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 7.9), 140.12 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 4.0)	–1.24 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1390 Hz)	413.3 ($^{1}J_{\mathrm{P\text{-}Te}}$ = 1390 Hz)
3	1.40 (t, 3 H, J = 7.1), 3.77 (s, 3 H), 4.20–4.37 (m, 2 H), 6.66–6.68 (m, 2 H), 7.33–7.57 (m, 7 H)	15.94 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 7.1), 55.10, 63.02 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.9), 99.04 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.0), 115.38 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 1.9), 127.98 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 14.3), 130.45 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 11.5), 132.18 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 3.4), 135.23 (d, $^{1}J_{\mathrm{P\text{-}C}}$ = 118.1), 142.31 (d, $^{4}J_{\mathrm{P\text{-}C}}$ = 2.6), 160.40 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.1)	22.7 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1129 Hz)	483.23 (d, $^{1}J_{\mathrm{P\text{-}Te}}$ = 1129 Hz)
4	1.10 (d, 6 H, J = 6.4), 1.19 (d, 6 H, J = 6.4), 3.51 (sept, 1 H, J = 6.8), 3.57 (sept, 1 H, J = 6.8), 3.78 (s, 3 H), 4.99 (dd, 1 H, $^{3}J_{\mathrm{P\text{-}H}}$ = 8.0, $^{2}J_{\mathrm{H\text{-}H}}$ = 12.0), 5.79 (dd, 1 H, $^{3}J_{\mathrm{P\text{-}H}}$ = 8.8, $^{2}J_{\mathrm{H\text{-}H}}$ = 12.0), 6.74 (d, 2 H, J = 8.7), 7.27–7.36 (m, 5 H), 7.78 (d, 2 H, J = 8.7)	21.67, 22.98, 46.65 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 5.8), 55.12, 66.51 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.9), 99.22 (d, $^{2}J_{\mathrm{P\text{-}C}}$ = 6.9), 115.30 (d, $^{3}J_{\mathrm{P\text{-}C}}$ = 2.0), 127.68, 128.02, 128.40, 136.33 (d, $^{3}J_{\mathrm{P\text{-}C}}{}^{4}$ = 8.0), 142.06 (d, $J_{\mathrm{P\text{-}C}}$ = 3.2), 160.17 (d, $^{5}J_{\mathrm{P\text{-}C}}$ = 2.2)	2.92 (with satellite doublet $^{1}J_{\mathrm{P\text{-}Te}}$ = 1148 Hz)	464.2 ($^{1}J_{\mathrm{P\text{-}Te}}$ = 1148 Hz)

and the resulting mixture was stirred for 1 h at r.t. Then H_2O was added, and the mixture was extracted with EtOAc, washed successively with aq sat. $NaHCO_3$, H_2O and brine. The organic extracts were dried ($MgSO_4$), evaporated, and the residue was purified by silica gel column chromatography (EtOAc–*n*-hexane, 1:4) to afford **2d** (74.5 mg, 88%). PhTeI was prepared similarly from diphenyl ditelluride and I_2. Procedure for the preparation of **2** from the isolated PhTeI was the same as that for $AnTeCl_3$ except for the amount of the phosphite used (Tables 2 and 3).

Te-*p*-Methoxyphenyl *O,O′*-Diethyl Phosphorotellurate (2a)

R_f = 0.45 (EtOAc–*n*-hexane, 1:2).

IR (neat): 3020, 1587, 1489, 1441, 1248, 1217, 1045, 908 cm^{-1}.

FABMS: *m/z* calcd for $C_{11}H_{17}PO_4Te$: 374; found: a maximum at 375 with tellurium isotopic fingerprint ranged between 366 and 376; 366 (8), 367 (12), 368 (14), 369 (29), 370 (55), 371 (56), 372 (63), 373 (92), 374 (71), 375 (100), 376 (15).

Te-*p*-Methoxyphenyl *O,O′*-Dibutyl Phosphorotellurate (2b)

R_f = 0.41 (EtOAc–*n*-Hexane, 1:2).

IR (neat): 3005, 2964, 2875, 1587, 1489, 1286, 1248, 1213, 1178, 1045, 978 cm^{-1}.

FABMS: *m/z* calcd for $C_{15}H_{25}PO_4Te$: 430; found: a maximum at 431 with tellurium isotopic fingerprint ranged between 422 and 432; 422 (5), 423 (10), 424 (10), 425 (29), 426 (57), 427 (61), 428 (66), 429 (95), 430 (75), 431 (100), 432 (17).

Te-*p*-Methoxyphenyl *O,O′*-Dibenzyl Phosphorotellurate (2c)

R_f = 0.31 (EtOAc–*n*-hexane, 1:3).

IR (neat): 3031, 1736, 1585, 1566, 1489, 1458, 1377, 1288, 1250, 1176, 987 cm^{-1}.

FABMS: *m/z* calcd for $C_{21}H_{21}PO_4Te$, 498; found: a maximum at 498 with tellurium isotopic fingerprint ranged between 490 and 500; 490 (7), 491 (11), 492 (17), 493 (31), 494 (66), 495 (57), 496 (87), 497 (87), 498 (100), 499 (95), 500 (22).

Te-*p*-Methoxyphenyl *O,O′*-Diphenyl Phosphorotellurate (2d)

R_f = 0.26 (EtOAc–*n*-hexane, 1:4).

FABMS: *m/z* calcd for $C_{19}H_{17}PO_4Te$, 470; found: a maximum at 471 with tellurium isotopic fingerprint ranged between 462 and 473; 462 (14), 463 (20), 464 (26), 465 (37), 466 (31), 467 (67), 468 (71), 469 (96), 470 (84), 471 (100), 472 (29), 473 (10).

Te-*p*-Methoxyphenyl *O,O′*-*o*-Xylylenediyl Phosphorotellurate (2e)

Mp 129.0–130.0 °C (EtOAc–hexane, dec.); R_f = 0.27 (EtOAc–*n*-hexane, 1:1).

IR (Nujol): 2927, 2854, 2719, 1716, 1581, 1458, 1377, 1265, 1173, 1002, 818 cm^{-1}.

FABMS: *m/z* calcd for $C_{15}H_{15}PO_4Te$: 420; found: a maximum at 421 with tellurium isotopic fingerprint ranged between 413 and 422; 413 (14), 414 (15), 415 (33), 416 (55), 417 (58), 418 (61), 419 (93), 420 (71), 421 (100), 422 (21).

Anal. Calcd for $C_{15}H_{15}PO_4Te$: C, 43.12; H, 3.62; Found: C, 43.08; H, 3.49.

Te-*p*-Methoxyphenyl *O,O′*-Dimethyl Phosphorotellurate (2f)

R_f = 0.40 (EtOAc–$CHCl_3$, 1:10).

IR (neat): 3020, 1587, 1489, 1288, 1248, 1217, 1178, 1020 cm^{-1}.

FABMS: *m/z* calcd for $C_9H_{13}PO_4Te$: 346; found: a maximum at 347 with tellurium isotopic fingerprint ranged between 339 and 348; 339 (10), 340 (14), 341 (26), 342 (54), 343 (58), 344 (56), 345 (95), 346 (61), 347 (100), 348 (11).

Te-Phenyl *O,O′*-Diethyl Phosphorotellurate (2g)

R_f = 0.20 (EtOAc–*n*-hexane, 1:4).

IR (neat): 3066, 2981, 2900, 1562, 1473, 1389, 1238, 1161, 1014, 964 cm^{-1}.

FABMS: *m/z* calcd for $C_{10}H_{15}PO_3Te$: 344; found: a maximum at 345 with tellurium isotopic fingerprint ranged between 336 and 346; 336 (5), 337 (11), 338 (11), 339 (24), 340 (42), 341 (59), 342 (39), 343 (96), 344 (45), 345 (100), 346 (13).

Te-Phenyl *O,O′*-Dibutyl Phosphorotellurate (2h)

R_f = 0.40 (EtOAc–*n*-hexane, 1:2).

IR (neat): 3055, 2958, 2873, 1574, 1466, 1381, 1242, 1149, 976, 833 cm^{-1}.

FABMS: *m/z* calcd for $C_{14}H_{23}PO_3Te$: 400; found: a maximum at 401 with tellurium isotopic fingerprint ranged between 393 and 403; 393 (11), 394 (10), 395 (24), 396 (41), 397 (61), 398 (38), 399 (96), 400 (46), 401 (100), 402 (17), 403 (6).

Te-Phenyl *O,O′*-Dibenzyl Phosphorotellurate (2i)

R_f 0.30 (EtOAc–*n*-hexane, 1:4).

IR (neat): 3062, 2950, 2885, 1574, 1454, 1377, 1238, 987 cm^{-1}.

FABMS: *m/z* calcd. for $C_{20}H_{19}PO_3Te$, 468; found: a maximum at 469 with tellurium isotopic fingerprint ranged between 460 and 471; 460 (10), 461 (19), 462 (20), 463 (32), 464 (51), 465 (65), 466 (48), 467 (94), 468 (58), 469 (100), 470 (29), 471 (17).

Te-*p*-Methoxyphenyl *O*-Ethyl Phenylphosphonotellurate (3)

R_f = 0.19 (EtOAc–*n*-hexane, 1:2).

IR (neat): 3059, 2981, 2835, 1585, 1566, 1489, 1458, 1439, 1389, 1288, 1250, 1219, 1176, 1111, 1022, 949, 822 cm^{-1}.

FABMS: *m/z* calcd for $C_{15}H_{17}PO_3Te$, 406; found: a maximum at 407 with tellurium isotopic fingerprint ranged between 399 and 408; 399 (9), 400 (10), 401 (24), 402 (45), 403 (59), 404 (46), 405 (93), 406 (55), 407 (100), 408 (17).

Te-*p*-Methoxyphenyl *N,N*-Diisopropyl *O*-Benzyl Phosphorotelluramidate (4)

R_f = 0.31 (EtOAc–*n*-hexane, 1:3).

IR (neat): 3062, 2966, 2885, 1585, 1489, 1365, 1246, 1157, 995, 821 cm^{-1}.

FABMS: *m/z* calcd for $C_{20}H_{28}PNO_3Te$, 491; found: a maximum at 492 with tellurium isotopic fingerprint ranged between 484 and 493; 484 (12), 485 (15), 486 (27), 487 (49), 488 (65), 489 (54), 490 (97), 491 (62), 492 (100), 493 (26).

Acknowledgment

The authors thank Venture Business Laboratory of Ehime University for their financial support. We also thank the Center for Cooperative Research and Development, Ehime University for measurement of mass spectra. This work was financially supported by the Fujisawa Foundation and Saneyoshi Scholarship Foundation.

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