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Syntheses and crystal structures of compounds containing short Te–N bonds

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Abstract

The reaction of tellurium(IV)chloride with *N*-trimethylsilyl-*P*-triphenyliminophosphorane in a molar ratio of 1:2 gives $(Ph_3P{=}N)_2TeCl_2$. A similar procedure with $RTeCl_3$ (R = *p*-$MeOC_6H_4$) gives the monosubstituted product $Ph_3P{=}NTe(R)Cl_2$. X-Ray diffraction studies show that both compounds contain short Te–N bonds, with lengths of ca. 192 pm. Additional tellurium–nitrogen compounds have been obtained from reactions of $RTeCl_3$ with $Me_2S({=}O){=}NSiMe_3$, $(Me_3SiN{=})_2S$, and $Me_3SiN(Me)C({=}O)N(m\text{-}CF_3C_6H_4)SiMe_3$, which give $Me_2S({=}O){=}NTe(R)Cl_2$, $Me_3SiN{=}S{=}NTe(R)Cl_2$, and $\overline{MeNC({=}O)N(m\text{-}CF_3C_6H_4)}Te(R)Cl$, respectively.

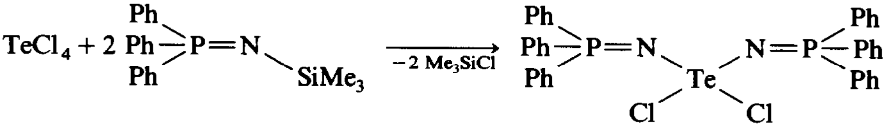
Introduction

Compound containing Te–N bonds have been known for only about 15 years, but such compounds have increasingly studied in the past decade [1]. Recently we reported the synthesis of the first tellurium nitride stable at room temperature [2]. No compounds with real Te–N double bonds have yet been made; the only compound that formally contains a Te–N double bond, synthesised by Naddaka et al., was shown by an X-ray diffraction study to have more of a dipolar character [3].

We report here two new tellurium(IV) phosphorane iminato complexes containing short Te–N bonds. These compounds may be models for possible electronic stabilisation of Te–N double bonds. We also describe the syntheses of some further *N*-derivatives of *p*-methoxyphenyltellurium trichloride.

Results

N-Trimethylsilyl-*P*-triphenyliminophosphorane, **1**, was been shown to be a useful ligand for stabilising metal–nitrogen bonds [4]. We previously reported that reaction of **1** with tellurium(IV) chloride in a molar ratio of 1:1 gives the dimeric species $(Ph_3P{=}NTeCl_3)_2$, which contains a planar Te_2N_2 ring [5]. However, the reaction of tellurium(IV) chloride with two equivalents of **1** has now been shown to give the disubstituted product **2** (yield 56%).

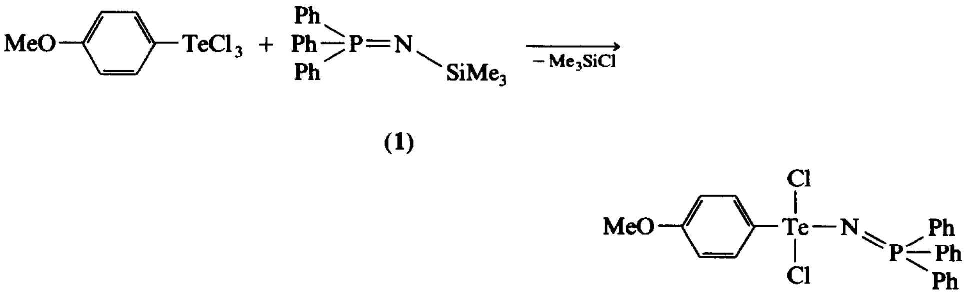


(1) (2)

The IR spectrum of compound **2** shows the typical absorptions associated with the symmetric and antisymmetric stretching modes of the Te–N–P unit at 520 and 1100 cm^{-1}, respectively. The mass spectrum shows an ion at *m/z* 717 which was identified as $[M - Cl]^+$ by analysis of the isotope pattern. The ^{31}P NMR spectrum exhibits a singlet at δ 21.8 ppm, a value close to that for the P atoms in $(Ph_3P{=}N)_2WF_4$ (δ 23.6 ppm) [6].

Single crystals suitable for an X-ray structural study were obtained by recrystallisation from pyridine. Atomic coordinates are given in Table 1 and selected bond lengths and angles in Table 2. The molecular structure is shown in Fig. 1. Compound **2** has a distorted tetrahedral coordination sphere around the Te atom. The mean Te–N bond distance is 191.8 pm, the mean P–N bond length is 158.7 pm, and the mean Te–N–P angle is 127.4°. Te–Cl bonds have a mean length of ca. 260 pm. In the Cl–Te–Cl unit the two Cl atoms are bent away from a linear arrangement (the Cl–Te–Cl angle is 162.7°) towards the lone-pair of the Te atom would be expected to lie. The N–Te–N angle is 99.1°. The geometry of **2** resembles that of the analogous Se compound $(Ph_3P{=}N)_2SeCl_2$ [7].

Reaction of *p*-methoxyphenyltelluriumtrichloride ($RTeCl_3$) with **1** in toluene at 80°C gave **3** in 68% yield.



(1)

(3)

The IR spectrum of **3** shows a number of absorptions due to the *p*-methoxyphenyl substituent. Absorptions of the symmetric and antisymmetric stretching vibrations of the Te–N–P unit are found at 690 and 1110 cm^{-1}, respectively. The mass spectrum shows the $[M - Cl]^+$ ion at *m/z* 548, with the expected isotope pattern. The ^1H NMR spectrum of **3** shows the expected signals from the MeO group (δ 3.83 ppm) and the various aromatic protons. The ^{31}P NMR spectrum contains a singlet at δ 30.2 ppm, a value similar to that found for $(Ph_3P{=}NTeCl_3)_2$ (δ 36.3 ppm) [5]. Comparison of the ^{31}P NMR chemical shifts for the three known triphenylphosphorane iminato complexes of Te reveals a marked downfield shift in the sequence **2**, **3** and $(Ph_3P{=}NTeCl_3)_2$, indicating decreasing electron density at the P atoms in this series.

Table 1

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients (pm^2 $\times 10^{-1}$) for **2**

	x	y	z	U_{eq} [a]
Te(1)	3978(1)	4165(1)	2500	33(1)
Cl(1)	5154(1)	4743(1)	2674(1)	57(1)
Cl(2)	2940(1)	2787(1)	2262(1)	57(1)
N(1)	3711(2)	5187(4)	3418(2)	37(1)
N(2)	3834(2)	5727(3)	1751(2)	38(1)
P(1)	3920(1)	5583(1)	837(1)	33(1)
P(2)	3537(1)	6905(1)	3527(1)	34(1)
C(11)	3959(2)	3690(4)	464(2)	41(1)
C(12)	3427(3)	2935(5)	256(3)	67(2)
C(13)	3464(4)	1403(7)	48(3)	96(3)
C(14)	4008(5)	685(7)	71(4)	101(4)
C(15)	4542(4)	1433(7)	273(4)	95(3)
C(16)	4519(3)	2966(5)	475(3)	61(2)
C(21)	4614(2)	6534(4)	509(2)	36(1)
C(22)	4966(2)	7285(5)	1059(3)	46(1)
C(23)	5484(2)	8087(5)	818(3)	55(2)
C(24)	5637(2)	8142(6)	52(3)	60(2)
C(25)	5299(2)	7387(5)	−493(3)	57(2)
C(26)	4783(2)	6562(5)	−272(2)	48(2)
C(31)	3269(2)	6515(4)	378(2)	39(1)
C(32)	2843(2)	7222(5)	839(3)	49(1)
C(33)	2351(2)	7984(6)	498(3)	67(2)
C(34)	2298(2)	8034(6)	−296(3)	66(2)
C(35)	2718(3)	7325(5)	−755(3)	69(2)
C(36)	3208(2)	6558(5)	−415(2)	55(2)
C(41)	4073(2)	8275(4)	3143(2)	39(1)
C(42)	4641(2)	8371(5)	3500(3)	52(2)
C(43)	5088(2)	9380(6)	3227(3)	70(2)
C(44)	4956(3)	10285(5)	2607(3)	69(2)
C(45)	4400(3)	10169(5)	2245(3)	65(2)
C(46)	3953(2)	9164(4)	2498(5)	52(1)
C(51)	2767(2)	7369(4)	3180(2)	40(1)
C(52)	2374(2)	6201(5)	3021(3)	52(2)
C(53)	1774(2)	6453(6)	2775(3)	71(2)
C(54)	1570(2)	7904(6)	2678(3)	76(2)
C(55)	1953(2)	9077(6)	2841(3)	72(2)
C(56)	2558(2)	8838(5)	3081(3)	56(2)
C(61)	3532(2)	7187(4)	4572(2)	35(1)
C(62)	3277(2)	8483(5)	4904(3)	51(2)
C(63)	3278(2)	8665(6)	5702(3)	61(2)
C(64)	3547(2)	7608(5)	6179(3)	59(2)
C(65)	3806(2)	6351(6)	5850(3)	58(2)
C(66)	3804(2)	6145(5)	5060(2)	47(1)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Recrystallisation of compound **3** from toluene gave colorless plates suitable for an X-ray structural determination. Atomic coordinates are given in Table 3 and selected bond lengths and angles in Table 4. The molecular structure, shown in Fig. 2, is clearly related to that of **2**. Both compounds have a distorted tetrahedral symmetry at the Te atoms. The Te–N bond distance in **3** (191.8 pm) is comparable

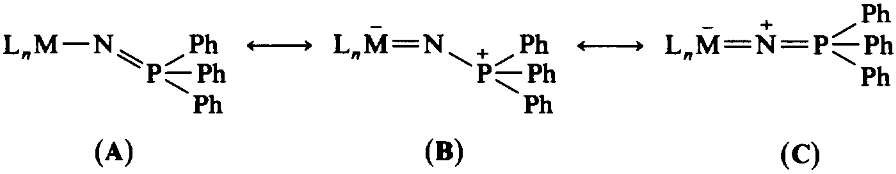
Table 2

Selected bond lengths (pm) and angles (°) in 2

Te(1)-N(1)	191.2(2)	Te(1)-N(1)-P(2)	128.9(2)
Te(1)-N(2)	192.3(3)	Te(1)-N(2)-P(1)	125.8(2)
N(1)-P(2)	158.8(3)	N(1)-Te(1)-N(2)	99.1(1)
N(2)-P(1)	158.6(4)	Cl(1)-Te(1)-N(1)	96.1(1)
Te(1)-Cl(1)	261.4(1)	Cl(1)-Te(1)-N(2)	95.3(1)
Te(1)-Cl(2)	259.2(1)	Cl(2)-Te(1)-N(1)	95.4(1)
		Cl(2)-Te(1)-N(1)	95.6(1)
		Cl(1)-Te(1)-Cl(2)	162.7(1)

with that in 2. The Te–N–P angle (122.4°) in 3 is substantially smaller than that in 2. The average Te–Cl bond length is 255.5 pm. The arrangement of the Cl–Te–Cl unit is very similar to that in 2; the Cl atoms are bent away from the *p*-methoxyphenyl and triphenyliminophosphoranato ligands (Cl–Te–Cl angle 172.7°).

Short Te–N bond lengths were observed in the dimer $(Ph_3P{=}NTeCl_3)_2$ (199.6 pm), but 2 and 3 show the shortest Te–N bond distances so far observed. The bonds have a partial double bond character and may be represented by the following resonance forms [4].



Both compounds have Te–N–P bond angles of approximately 120 to 130° and so the resonance structures A and B seem to be dominant.

The Te–Cl bond lengths are longer than those for the non-bridging Cl atoms in tellurium(IV) chloride (mean 231.1 pm) [8] and p-$C_2H_5OC_6H_4TeCl_3$ (mean 239.6

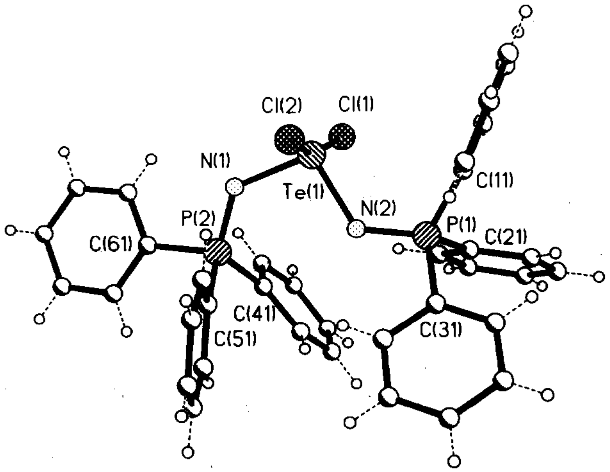


Fig. 1. Molecular structure of 2.

Table 3

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($pm^2 \times 10^{-1}$) for **3**·toluene

	x	y	z	U_{eq} [a]
Te	6697(1)	5674(1)	8393(1)	37(1)
Cl(1)	8782(1)	4517(1)	7246(1)	66(1)
Cl(2)	4400(1)	6632(1)	9650(1)	53(1)
N	7164(2)	7079(2)	7472(2)	45(1)
P	8199(1)	7827(1)	7609(1)	36(1)
C(11)	9551(3)	8135(2)	6304(2)	41(1)
C(12)	9678(3)	7566(3)	5465(2)	62(1)
C(13)	10745(4)	7745(3)	4473(3)	79(2)
C(14)	11686(3)	8489(3)	4319(3)	74(1)
C(15)	11559(3)	9058(3)	5135(3)	72(2)
C(16)	10496(3)	8885(3)	6129(2)	58(1)
C(21)	9066(3)	7219(2)	8587(2)	40(1)
C(22)	10474(3)	6817(2)	8280(2)	53(1)
C(23)	11104(3)	6369(3)	9051(3)	70(2)
C(24)	10328(4)	6303(3)	10136(3)	72(2)
C(25)	8910(4)	6670(3)	10459(2)	69(2)
C(26)	8284(3)	7126(3)	9694(2)	58(1)
C(31)	7258(3)	9140(2)	8060(2)	39(1)
C(32)	7807(3)	9826(2)	8479(3)	57(1)
C(33)	7036(3)	10798(2)	8840(3)	64(1)
C(34)	5721(3)	11100(2)	8791(2)	59(1)
C(35)	5158(3)	10439(2)	8382(3)	61(1)
C(36)	5915(3)	9453(2)	8018(2)	52(1)
C(41)	5503(3)	5281(2)	7553(2)	39(1)
C(42)	4890(3)	6103(2)	6948(2)	49(1)
C(43)	4069(3)	5833(2)	6420(2)	53(1)
C(44)	3861(3)	4720(3)	6526(2)	51(1)
C(45)	4498(3)	3885(3)	7126(2)	61(1)
C(46)	5311(3)	4157(2)	7639(2)	55(1)
O	3067(2)	4342(2)	6066(2)	74(1)
C(44A)	2360(3)	5146(3)	5447(3)	80(2)
C(1)	7170(5)	−967(4)	3441(4)	97(2)
C(2)	6781(5)	165(4)	3170(4)	100(2)
C(3)	7455(6)	733(4)	2169(5)	114(3)
C(4)	8539(6)	199(6)	1415(5)	120(3)
C(5)	8927(5)	−925(5)	1712(5)	120(3)
C(6)	8269(5)	−1507(4)	2694(4)	105(2)
C(1A)	6409(6)	−1598(5)	4524(4)	149(4)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 4

Selected bond lengths (pm) and angles (°) in **3**

Te–N	191.8(2)	Te–N–P	122.4(1)
N–P	159.7(3)	N–Te–C(41)	95.2(1)
Te–Cl(1)	252.1(1)	N–T–Cl(1)	94.5(1)
Te–Cl(2)	258.9(1)	N–Te–Cl(2)	92.0(1)
Te–Cl(41)	210.2(3)	Cl(1)–Te–C(41)	88.4(1)
		Cl(2)–Te–C(41)	87.8(1)
		Cl(1)–Te–Cl(2)	172.7(1)

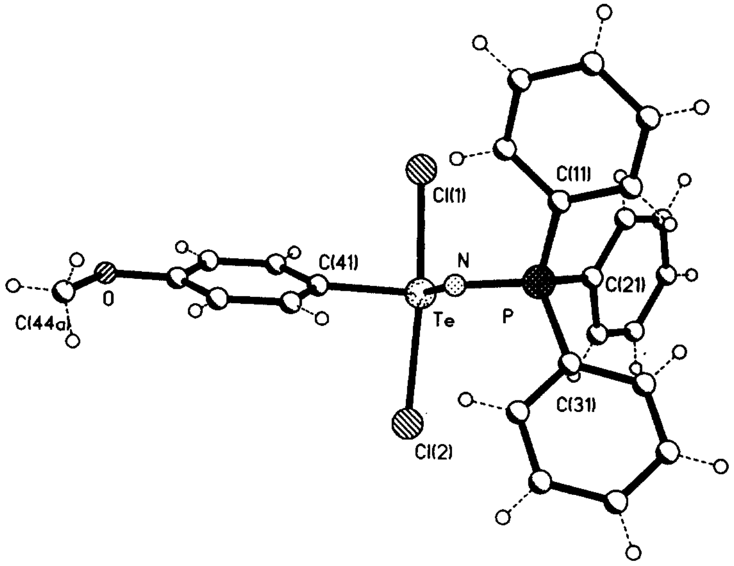


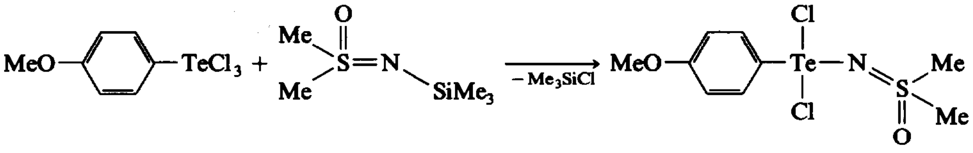
Fig. 2. Molecular structure of 3.

pm) [9]. In addition the Cl atoms in both cases are bent away from a linear arrangement towards the position in which the Te lone pair is expected to lie, though this effect is smaller in **3** than in **2**. In terms of the VSEPR model, this means that the steric demand of the lone pair is smaller than that of the two ligands, which could indicate a stronger *s*-character of this lone pair.

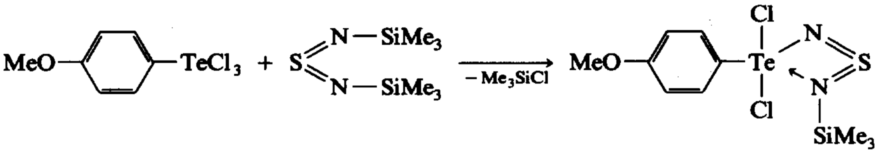
Preparations of additional Te–N compounds

Only a few *N*-derivatives of monosubstituted tellurium(IV) compounds have been synthesised up to now. Some were obtained by Miller et al. who treated aryltellurium trichlorides (aryl = C_6H_5, *p*-$MeOC_6H_4$, *p*-$C_6H_5C_6H_4$) with *N*-trimethylsilyldialkylamines and obtained very unstable monosubstituted compounds [10]. We therefore tried to generate this type of molecules using electronically stabilising substituents.

Reactions of $RTeCl_3$ with *S*,*S*-dimethyl-*N*-trimethylsilylsulfoximine (**4**) and *N*,*N*′-bis(trimethylsilyl)sulfurdiimide (**5**) yielded the monosubstituted products **6** and **7**:

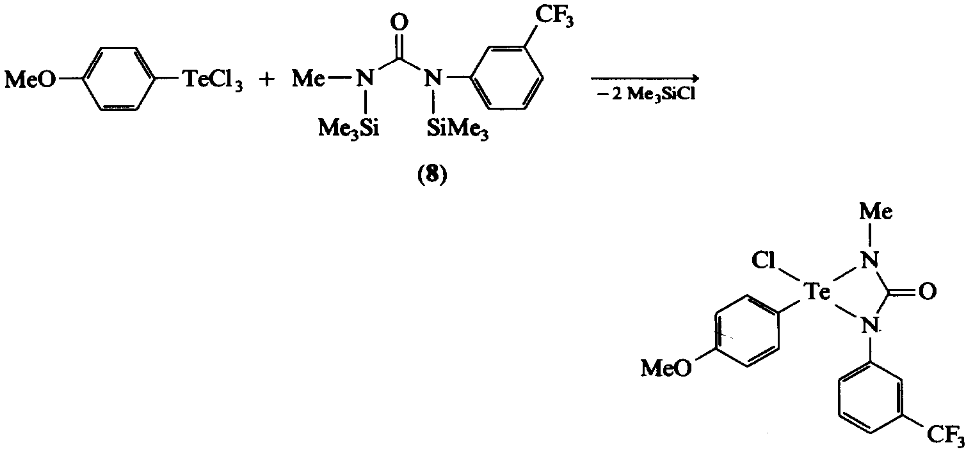


(4) (6)



(5) (7)

Under similar conditions *N*-(3-trifluormethyl)-*N*′-methyl-*N*,*N*′-(bistrimethylsilyl)urea, **8**, reacts to give the four-membered ring species **9**:



(8)

(9)

In addition to the absorptions of the *p*-methyoxyphenyl ligand in the IR spectrum of **6**, there is a broad band at 1210 cm^{-1} due to the SO and SN bonds. The ^{1}H NMR spectrum shows the signal for the Me_2S groups at δ 3.30, and that for the MeO group at δ 3.86 ppm. The aromatic protons appear as two poorly-resolved doublets at δ 7.10 and 8.08 ppm. Integration of the peaks gave the expected ratios. The FI mass spectrum shows only the ion $[M - Cl]^+$.

Compound **7** was characterised from its IR and ^{1}H NMR spectra, its mass spectrum, and elemental analysis. The signal from the protons of the Me_3Si group shows two satellites due to the coupling with the ^{29}Si nucleus ($^2J(^{29}Si{-}^1H) = 7$ Hz). The mass spectrum contains $[M - Me]^+$ (m/z 425, 4%) as well as the ion $[M - Cl]^+$ (m/z 405, 58%).

The four-membered ring species **9** was obtained in 34% yield from the reaction of $RTeCl_3$ with **8** at 80 °C in toluene. In the IR spectrum the carbonyl bond is clearly observed at 1638 cm^{-1}. Bands from the CF_3 groups appear at 1365 and 1117 cm^{-1}. The ^{19}F NMR spectrum exhibits a singlet at δ −62.1 ppm. The FI mass spectrum of **9** confirms the proposed four-membered ring formulation, showing an $[M]^+$ ion at m/z 488.

Our work has shown Te–N bonds can be stabilised by electronic effects of appropriate substituents. The triphenylphosphorane iminato ligand in **2** and **3** and the sulfurdiimide ligand in **7** have electron-releasing properties, the *N*-ligands in **6** and **9** are more electron accepting.

In **6** and **9** the *p*-methoxyphenyl ligand has an important influence on the stability of the Te–N bond. (Reactions of **4** and **8** with tellurium(IV) chloride do not give stable products [11].) Attachment of the *p*-methoxyphenyl ligand to tellurium(IV) may thus Te–N compounds are to be prepared.

Experimental

All operations were carried out in carefully dried apparatus under dry nitrogen. Solvents were dried prior to use by standard methods. Commercially-available tellurium(IV) chloride was sublimed prior to use. *p*-Methoxyphenyltellurium trichloride, *N*-trimethylsilyl-*P*-triphenyliminophosphorane, **1**, *S*,*S*-dimethyl-*N*-trimethylsilylsulfoximine, **4**, *N*,*N*'-(bistrimethylsilyl)sulfurdiimide, **5**, and *N*-(3-trifluoromethylphenyl)-*N*'-methyl-*N*,*N*'-(bistrimethylsilyl)urea, **8**, were prepared by published methods.

The IR spectra were recorded on a Bio RAD FTS. 7 spectrometer as Nujol mulls between NaCl windows. Only strong (s) or very strong (ss) bands are reported. Mass spectra were obtained with Varian CH5 or Finnigan MAT 8230 instruments by electron impact (EI) or field ionisation (FI) methods. In the main we report only ions of highest intensity and high mass; simulations were used to deck the isotope patterns. ^1H NMR spectra were recorded on a 80 MHz Bruker WP 80 spectrometer with TMS as external standard. A Bruker AM 250 instrument was used to record ^{31}P NMR spectra, with 85% H_3PO_4 as an external standard. Microanalyses were performed by the analytic laboratory of the Institut für Anorganische Chemie der Universität Göttingen.

The X-ray structure analyses were performed in a profile-fitting scan mode [12] on a Siemens–Stoe AED2 Rev. 6.2 diffractometer using graphite monochromated Mo-K_α radiation. Absorption corrections were applied by azimuthal scans. The structure solution and the refinement were carried out with SHELXTL PLUS [13], using anisotropic displacement parameters for all non hydrogen atoms and a riding model for hydrogen atoms.

Crystallographic data for 2

Orthorhombic $Pna2_1$, $a = 2163.9(6)$, $b = 891.5(1)$, $c = 1718.5(4)$ pm, $V = 3.3153(13)$ nm^3, $D_x = 1.50$ g cm^{-3}, $Z = 4$, $\mu(\text{Mo-}K_\alpha) = 1.18$ mm^{-1}, $T = 20\,°$C, 4974 reflections collected up to $2\theta_{max} = 45°$, 4320 reflections independent, 4121 with $F \geqslant 3\sigma(F)$ used in the refinement, $R = 0.023$, $R_w = 0.026$, $w^{-1} = \sigma^2(F) + 0.0002 \cdot F^2$. The absolute structure was assigned by refinement of anomalous scattering [14].

Crystallographic data for 3 · toluene

$C_{32}H_{30}Cl_2NOPTe$ ($M = 674$), triclinic $P\bar{1}$, $a = 1029.6(2)$, $b = 1218.1(2)$, $c = 1331.3(3)$ pm, $\alpha = 78.37(3)$, $\beta = 68.75(3)$, $\gamma = 79.88(3)°$, $V = 1.5146(5)$ nm^3, $D_x = 1.48$ g cm^{-3}, $Z = 2$, $\mu(\text{Mo-}K_\alpha) = 1.24$ mm^{-1}, $T = 20\,°$C, 7896 reflections collected up to $2\theta_{max} = 45°$, 3948 reflections independent, 3729 with $F \geqslant 3\sigma(F)$ used in the refinement, $R = 0.020$, $R_w = 0.027$; $w^{-1} = \sigma^2(F) + 0.0002 \cdot F^2$.

Preparation of 2

A solution of **1** (2.80 g, 8.0 mmol) in 80 ml of toluene was added dropwise at 0 °C to tellurium(IV) chloride (1.07 g, 4.0 mmol) in 70 ml of toluene. The mixture was allowed to warm to room temperature and than stirred for 14 h. The white precipitate was filtered off, washed with cold acetonitrile, and dried *in vacuo* to give 1.70 g (2.3 mmol) of **2** (yield 56%). Recrystallisation from pyridine gave crystals suitable for an X-ray study.

IR spectrum: 1460ss, 1100s, 1050s, 720s, 680s cm^{-1}. Mass spectrum (EI, m/z): 717 ($M -$ Cl, 11%), 277 ($Ph_3P{=}NH$, 100%). ^1H NMR ($CDCl_3$): δ 7.40–8.0 ppm (m). ^{31}P NMR (CH_2Cl_2/C_6D_6): δ 21.8 ppm (s). Elemental analysis. Found: C, 57.2; H, 4.3; N, 3.8. $C_{36}H_{30}Cl_2N_2P_2Te$ ($M = 751.15$) calc.: C, 57.57; H, 4.03; N, 3.73%.

Preparation of 3

To a suspension of *p*-methoxyphenyltellurium trichloride (1.23 g, 3.5 mmol) in 100 ml of toluene was added dropwise a solution of **1** (1.54 g, 4.4 mmol) in 80 ml of toluene; during the addition a clear yellowish solution was formed. The mixture was stirred for 2 h at room temperature and then at 80 ° C for 3 h. As it cooled slowly to room temperature colorless crystals separated out and were filtered off and washed with n-hexane. The filtrate was cooled to -10 ° C to give additional **3** as a white powder. A total of 1.40 g (2.4 mmol) of **3** was obtained (yield 68%). Crystals suitable for an X-ray study were obtained by recrystallisation from toluene.

IR spectrum: 1583s, 1438ss, 1378s, 1288s, 1260ss, 1176s, 1064ss, 1025s, 998s, 810ss, 732ss, 690ss cm^{-1}. Mass spectrum (EI, *m/z*): 548 ($M-Cl$, 36%), 276 ($Ph_3P{=}N$, 100%). ^1H NMR ($CDCl_3$): δ 3.84 (s, 3H0; 7.05–8.23 (m, 19H) ppm. ^{31}P NMR ($CDCl_3$): δ 30.2 (s) ppm; $^1J(^{13}C-^{31}P) = 24$ Hz, $^2J(^{13}C-^{31}P) = 90$ Hz. Elemental analysis. Found: C, 50.7; H, 4.0; N, 2.2. $C_{25}H_{22}Cl_2NOPTe$ ($M = 581.94$) calc. C, 51.60; H, 3.81; N, 2.41%.

Preparation of 6

To a suspension of *p*-methoxyphenyltellurium trichloride (0.52 g, 1.5 mmol) in 100 ml toluene was added a solution of **4** (0.50 g, 3 mmol) in 40 ml of toluene. The mixture was stirred for 12 h, during which a white precipitate separated. It was filtered off and recrystallised from dichloromethane for give 0.24 g (0.6 mmol) of **6** (yield 40%).

IR spectrum: 1587s, 1494s, 1264ss, 1210ss, br, 1174s, 1080ss, 1058ss, 938s, 829s cm^{-1}. Mass spectrum (FI, *m/z*): 364 ($M-Cl$, 100%). ^1H NMR (CD_3CN): δ 3.30 (s, 3H); 3.86 (s, 3H); 7.10 (d, 2H, $^3J = 9$ Hz); 8.08 (d, 2H, $^3J = 9$ Hz) ppm. Elemental analysis. Found: C, 26.7; H, 3.3; N, 3.2. $C_9H_{13}Cl_2NO_2STe$, ($M = 397.77$) calc.: C, 27.18; H, 3.29; N, 3.52%.

Preparation of 7

A solution of **5** (0.73 g, 3.5 mmol) in 40 ml of dichloromethane was added dropwise to a suspension of *p*-methoxyphenyltellurium trichloride (1.0 g, 2.9 mmol) in 60 ml of dichloromethane at room temperature. The clear yellowish solution formed was heated under reflux for 8 h, concentrated *in vacuo* to a volume of 40 ml, and then kept at -80 ° C. The precipitate formed was filtered off and washed with n-hexane. 0.80 g (1.8 mmol) of **7** were obtained (yield 63%).

IR spectrum: 1589s, 1260ss, 1189s, 1170s, 1051s, 843s, 718s cm^{-1}. Mass spectrum (EI, *m/z*): 425 ($M-CH_3$, 4%), 405 ($M-Cl$, 58%), 142 (C_7H_7ClO, 100%). ^1H NMR ($CDCl_3$): δ 0.06 (s, 9H, $^2J(^{29}Si-^1H) = 7$ Hz); 3.84 (s, 3H); 7.0 (d, 2H, $^3J = 9$ Hz); 8.46 (d, 2H, $^3J = 9$ Hz) ppm. Elemental analysis. Found: C, 27.6; H, 4.0; N, 5.5. $C_{10}H_{16}Cl_2N_2OSSiTe$ ($M = 438.91$) calc.: C, 27.36; H, 3.67; N, 6.38%.

Preparation of 9

To a suspension of *p*-methoxyphenyltellurium trichloride (1.21 g, 3.5 mmol) in 150 ml of toluene was added dropwise a solution of **8** (1.28 g, 3.5 mmol) at room temperature. The bright yellow solution formed was stirred at room temperature for 2 h and then at 80 ° C for 2 h. The solvent was removed *in vacuo* and the residue recrystallised from dichloromethane/n-hexane. Filtration yielded 0.58 g (1.2 mmol) of **9** (yield 34%).

IR spectrum: 1668s, 1638ss, 1356ss, 1301s, 1260ss, 1173ss, 1117s, 1064s, 1027s, 818s, 794s cm^{-1}. Mass spectrum (FI, m/z): 488 (M). ^1H NMR (CD_3CN): δ 3.21 (s, 3H); 3.82 (s, 3H); 7.0–8.0 (m, 8H) ppm. ^{19}F NMR (CD_3CN): δ -62.1 ppm (s). Elemental analysis. Found: C, 38.5; H, 3.0; N, 5.2. $C_{16}H_{14}F_3N_2O_2Te$ ($M = 486.35$) calc.: C, 39.49; H, 2.90; N, 5.76%.

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