



Dedicated to the memory of
Professor Candin Liteanu on his 100th anniversary

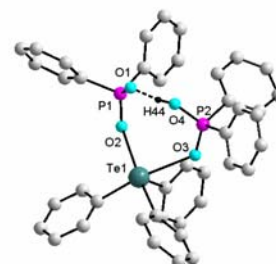
NEW TRIPHENYLTELLURONIUM SALTS OF ORGANOPHOSPHORUS LIGANDS

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New triorganotellurium(IV) compounds of type $[\text{Ph}_3\text{Te}][(\text{R}_2\text{PS})(\text{R}'\text{SO}_2)\text{N}]$, $[\text{R} = \text{Ph}$, $\text{R}' = \text{Me}$ (**1**), $\text{R} = \text{OEt}$; $\text{R}' = 4\text{-ClC}_6\text{H}_4$ (**2**)], as well as $[\text{Ph}_3\text{Te}][\text{O}_2\text{PPh}_2]$ (**3**) were obtained by reacting Ph_3TeCl with the potassium salt of the appropriate organophosphorus acid in an 1 : 1 molar ratio. The new species were characterized by multinuclear NMR (^1H , ^{13}C , ^{31}P and ^{125}Te) and mass spectrometry. The molar conductivity in DMSO solution suggested their behaviour as 1 : 1 electrolytes. The single-crystal X-ray diffraction structure of $\mathbf{3} \cdot \text{Ph}_2\text{PO}_2\text{H}$ revealed strong $\text{Te} \cdots \text{O}$ interactions between the triphenyltelluronium cation and the Ph_2PO_2^- anion and the free acid, respectively.



INTRODUCTION

Organotellurium compounds attracted a considerable increased interest in recent years due to their potential applications in catalysis, biology and nanomaterials.¹⁻⁶ Their structural features in solid state, *i.e.* dimeric or polymeric associations by secondary bonding was also systematically investigated for several classes of compounds, mainly organotellurium halides, which usually are associated by $\text{Te} \cdots \text{X}$ ($\text{X} = \text{halogen}$) intermolecular interactions.^{7,8} Triorganotellurium(IV) derivatives, R_3TeL ($\text{R} = \text{Me}$, Ph), with simple anionic ligands ($\text{L} = \text{Cl}^-$, N_3^- , NCO^- , NCS^- , NCSe^-) have shown an extended coordination sphere around tellurium and complex 3D networks realized through secondary interactions.⁹⁻¹² For several species containing 1,1-dithioato ligands, capable to act as chelating moieties,

either dimeric ($[\text{Ph}_3\text{TeS}_2\text{CNPr}_2]_2$,¹³ $[\text{Ph}_3\text{TeS}_2\text{COMe}]_2$,¹⁴ $[\text{Ph}_3\text{TeS}_2\text{COEt}]_2$,¹⁵) or monomeric structures ($\text{Ph}_3\text{TeS}_2\text{COBu}$,¹⁴ $\text{Ph}_3\text{TeS}_2\text{PPh}_2$, $\text{Ph}_3\text{Te}(\text{SPPPh}_2)_2\text{N}$,¹⁶ $\text{Ph}_3\text{Te}(\text{OPPh}_2)_2\text{N}$, $\text{Ph}_3\text{Te}(\text{SPPPh}_2)(\text{OPPh}_2)\text{N}$,¹⁷) were evidenced. The triorganotellurium(IV) compounds, either halides and pseudohalides or species containing chelating ligands, are described as triorganotelluronium salts which behave as 1 : 1 electrolytes, at least in polar solvents.¹⁷ The single-crystal X-ray diffraction studies brought evidences for secondary interactions between tellurium and the chalcogen donor atoms in compounds with such ligands. For the investigated species either a monodentate or a bidentate behaviour of the ligands was evidenced. However, the secondary interactions¹⁸ cannot be ignored in such species and this concept applied to triorganotelluronium species was subject of several detailed studies.¹⁹⁻²¹

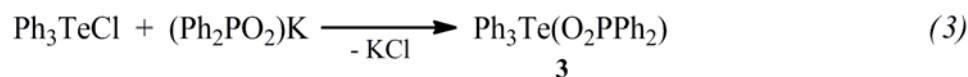
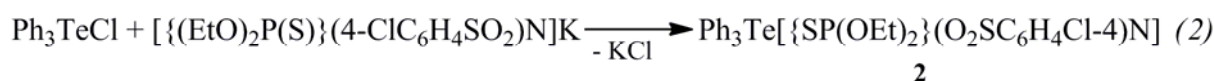
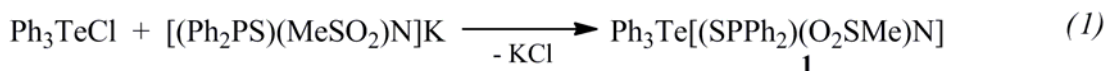
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Previously we have reported several triorganotelluronium salts of tetraorganodichalcogenoimidodiphosphinato and dithiophosphinato ligands, respectively.¹⁶⁻¹⁷ We developed during last years a new type of organophosphorus ligands, namely $[(R_2PX)(R'SO_2)N]^-$ ($R = Ph$ or OEt ; $R' =$ alkyl, aryl; $X = S, O$)^{22,23} and we report here the synthesis and solution behavior of two triorganotellurium(IV) compounds with such ligands, as well as the diphenyldioxophosphinato derivative $[Ph_3Te][O_2PPh_2]$.

RESULTS

Synthesis

Triorganotelluronium salts of the organophosphorus ligands $[(Ph_2PS)(MeSO_2)N]^-$, $[\{(EtO)_2P(S)\}(4-ClC_6H_4SO_2)N]^-$ and $(Ph_2PO_2)^-$, respectively, were prepared by salt metathesis reactions between Ph_3TeCl and the potassium salt of the appropriate organophosphorus acid, in an 1:1 molar ratio, using dichloromethane as solvent (*eq. 1 - 3*).



The described compounds are ionic species containing $[Ph_3Te]^+$ cations and organophosphorus anions, soluble in polar solvents. They were investigated by multinuclear NMR (1H , ^{13}C , ^{31}P and ^{125}Te), mass spectrometry and molar conductivity.

DISCUSSION

Spectroscopy

In the aromatic region the 1H NMR spectra of all three species present multiplet resonances characteristic for the *ortho*, *meta* and *para* protons of the phenyl groups attached to tellurium and also of the phenyl groups attached to phosphorus in compounds **1** and **3** and the 4- ClC_6H_4 group attached to sulfur in **2**. In the aliphatic region the 1H NMR spectrum of compound **1** presents a singlet assigned to the $MeSO_2$ group. The 1H NMR spectrum of **2** is more complex in the aliphatic region, due to diastereotopicity of the CH_2 protons in the ethoxy groups and the $^1H - ^1H$ and $^1H - ^{31}P$ couplings, respectively. While the OCH_2CH_3 protons appear as a triplet, the $OCH_AH_BCH_3$ resonance has a second order $ABXY_3$ pattern, determined by the combined $CH_3 - CH_A$, $CH_3 - CH_B$, $CH_A - CH_B$ and phosphorus-proton couplings, respectively. As a consequence, the complex appearance of the CH_2 resonance is determined both by the non-equivalent

diastereotopic CH_2 protons and the different values of the $^2J_{HH}$, $^3J_{HH}$ and $^3J_{PH}$ coupling constants. To better understand the splitting pattern of the CH_2 resonance in **2** and to unambiguously determine the coupling constants, we simulated the 1H NMR spectrum of **2** in the respective region. The ^{13}C NMR spectra are characteristic for the organic groups in the investigated compounds. The resonances of the carbons in the organic groups attached to phosphorus appear as doublets, due to the $^{31}P - ^{13}C$ couplings. The ^{13}C NMR spectrum of compound **2** displays two doublet resonances in the aliphatic region, characteristic for the OCH_2CH_3 and OCH_2CH_3 carbons, respectively, thus confirming the equivalence of the two ethoxy groups in solution.

The ^{31}P and ^{125}Te NMR spectra display singlet resonances in the regions characteristic for $P(V)=O$ and $P(V)=S$ and for the $[Ph_3Te]^+$ cation, respectively.

The mass spectra are in accordance with the ionic structure of the investigated species. For all three compounds the ESI+ MS spectra display the base peak corresponding to the $[Ph_3Te]^+$ cation, at m/z 361.01, while in the ESI- MS spectra peaks corresponding to the respective organophosphorus anions are present.

The molar conductivity of compounds **1 - 3** in 10^{-4} DMSO solutions are of the same order of magnitude as that one of the starting Ph_3TeCl ($\Lambda_M = 79.9 \text{ S}\cdot\text{cm}^{-1}\cdot\text{mol}^{-1}$) and it is in accordance with their behavior as 1 : 1 electrolytes.

Crystal and molecular structure of
 $[Ph_3Te][O_2PPh_2] \cdot Ph_2P(O)OH$ ($3 \cdot Ph_2P(O)OH$)

The Ortep-like diagram and the numbering scheme for compound $3 \cdot Ph_2P(O)OH$ is presented in Fig. 1, while interatomic distances and bond angles are given in Table 1.

The compound crystallizes in the $P-1$ space group. In the absence of any $Te \cdots O$ interaction, the structure might be regarded as a pure ionic species comprising pyramidal Ph_3Te^+ cations with $C-Te-C$ angles in the range $93.95(13) - 96.96(13)^\circ$ and $Ph_2PO_2^-$ anions, cocrystallized with free Ph_2PO_2H acid, probably formed by hydrolysis during the process of growing crystals. Anyway, the strong secondary $Te \cdots O$ interactions $Te1 \cdots O2$ [$2.628(3)$ Å] and $Te1 \cdots O3$ [$2.742(4)$ Å] can not be ignored ($\Sigma r_{vdw}(Te, O) = 3.60$ Å [24]) and they should be considered in the coordination sphere of tellurium in the discussed species. Both the $Ph_2PO_2^-$ anion and the free acid interact with tellurium through only one oxygen, thus resulting in a distorted square pyramidal coordination geometry about

tellurium, with a phenyl group in apices and the base formed by two carbon atoms belonging to the other phenyl groups attached to tellurium and the two oxygen atoms. The monometallic monoconnective behavior of the dioxodiphosphinato ligand in this species contrasts with the bidentate behavior previously observed in other triorganotelluronium compounds with dithio, monothio or dioxo organophosphorus(V) ligands.¹⁷ This behavior should be a consequence of the combined steric effects determined by the bulky cation and the presence of both $Ph_2PO_2^-$ and $Ph_2P(O)OH$ in the coordination sphere of tellurium. Moreover, the OH proton in the neutral ligand is involved in hydrogen bonding with the oxygen non-interacting with tellurium from the anionic ligand ($O1 \cdots H44$ 1.478 Å, vs. $\Sigma r_{vdw}(H, O) = 2.60$ Å)²⁴). The phosphorus – oxygen interatomic distances have typical values for single P–O and double P=O bonds, cf. P=O $1.486(6)$ and P–O $1.526(6)$ Å in $Ph_2P(O)OH$ ²⁵, in accordance also with their monodentate coordination behavior.

Table 1

Selected interatomic distances (Å) and angles (deg) in compound $3 \cdot Ph_2P(O)OH$

Te1–C1	2.110(3)	C1–Te1–C7	93.95(13)
Te1–C7	2.140(3)	C1–Te1–C13	96.96(13)
Te1–C13	2.118(3)	C13–Te1–C7	95.26(13)
Te1–O2	2.742(4)	O2–Te1–O3	96.60(9)
Te1–O3	2.628(3)	C1–Te1–O2	80.16(12)
		C1–Te1–O3	80.63(11)
		C7–Te1–O2	88.39(11)
P1–O1	1.516(3)	C7–Te1–O3	171.85(10)
P1–O2	1.489(3)	C13–Te1–O2	175.50(13)
P2–O3	1.491(3)	C13–Te1–O3	79.46(11)
P2–O4	1.530(3)	O1–P1–O2	118.52(16)
		O3–P2–O4	116.49(15)
O1 \cdots H44	1.478	O1 \cdots H44–O4	161.63(6)

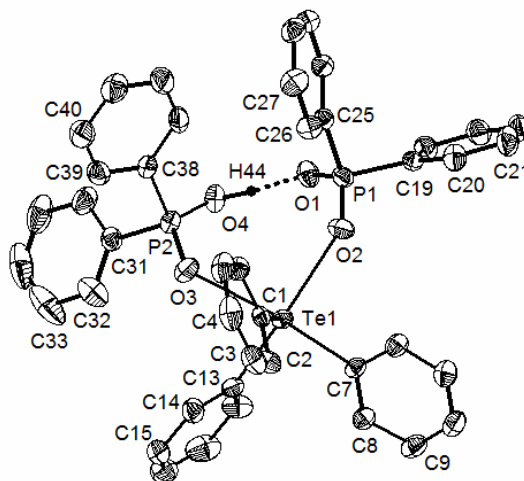


Fig. 1 – ORTEP representation at 30% probability and atom numbering scheme for the compound $3 \cdot Ph_2P(O)OH$.

EXPERIMENTAL

The starting materials were obtained following literature procedures: Ph_3TeCl ,²⁶ $[(\text{Ph}_2\text{PS})(\text{MeSO}_2)\text{N}]\text{K}$, $[(\text{EtO})_2\text{P}(\text{S})](4\text{-ClC}_6\text{H}_4\text{SO}_2\text{N})\text{K}$ ^{22,23} and $(\text{Ph}_2\text{PO}_2)\text{K}$.²⁵ Elemental analysis were performed on a Flash EA 1112 analyzer. Melting points were measured on an Electrothermal 9200 apparatus and are not corrected. ^1H , ^{13}C , ^{31}P and ^{125}Te NMR spectra were recorded on a BRUKER Avance instrument operating at 600.13, 100.61, 242.94 and 126.24 MHz, respectively. The chemical shifts are reported in δ units (ppm) relative to TMS (ref. CHCl_3 : ^1H 7.26 ppm, ^{13}C 77.0 ppm), H_3PO_4 85% and Ph_2Te_2 , respectively. The NMR spectra were processed using the MestReC and MestReNova software.²⁷ Mass spectra were recorded on a LTQ Orbitrap - XL instrument. Conductivity of 10^{-4} M solutions in DMSO was determined at 25°C with a Bench Conductivity/TDS Meter CON 510 instrument containing 2-ring stainless steel Ultem-body Conductivity/TDS electrode (cell constant $K = 1.0$) with build-in temperature sensor (EC-CONSEN 91W/35608-50).

Synthesis of $[\text{Ph}_3\text{Te}][(\text{SPPH}_2)(\text{O}_2\text{SMe})\text{N}]$ (1)

A reaction mixture of $[(\text{Ph}_2\text{PS})(\text{MeSO}_2)\text{N}]\text{K}$ (0.1013 g, 0.29 mmol) and Ph_3TeCl (0.1114 g, 0.29 mmol) in 20 mL of anhydrous methylene chloride was stirred at room temperature for 24 h. The resulting solid was removed by filtration. The solvent was evaporated in vacuum and the title compound was obtained as a colorless powder. Yield: 0.173 g (89%). M.p. 75 – 77°C. Anal. Calcd. for $\text{C}_{31}\text{H}_{28}\text{NO}_2\text{PS}_2\text{Te}$ (MW 669.26): C, 55.63; H, 4.22; N, 2.09%. Found: C, 55.28; H, 4.32; N, 2.33%. ^1H NMR: δ 2.80s (3H, SO_2CH_3), 7.35m (6H, P-C₆H₅-meta + para), 7.46t (6H, Te-C₆H₅-meta, $^3J_{\text{HH}}$ 7.5 Hz), 7.56t (3H, P-C₆H₅-para, $^3J_{\text{HH}}$ 7.1 Hz), 7.59d (6H, Te-C₆H₅-ortho, $^3J_{\text{HH}}$ 7.71 Hz), 8.07ddd (4H, P-C₆H₅-ortho, $^3J_{\text{HH}}$ 5.80, $^4J_{\text{HH}}$ 2.36, $^3J_{\text{PH}}$ 13.47 Hz). ^{13}C NMR: δ 43.92s (S-CH₃), 126.08s (Te-C₆H₅-ipso), 127.87d (P-C₆H₅-meta, $^3J_{\text{PH}}$ 13.10 Hz), 130.35d (P-C₆H₅-para, $^4J_{\text{PH}}$ 2.76 Hz), 130.54s (Te-C₆H₅-meta), 131.46d (P-C₆H₅-ortho, $^2J_{\text{PH}}$ 11.10 Hz), 131.86s (Te-C₆H₅-para), 134.79s (Te-C₆H₅-ortho, $^2J_{\text{TeC}}$ 33.10 Hz), 138.41d (P-C₆H₅-ipso, $^1J_{\text{PH}}$ 107.02 Hz) ^{31}P NMR: δ 37.15s. ^{125}Te NMR: δ 752.84s. ESI+ MS (m/z, %): 361.02 (100) $[\text{Ph}_3\text{Te}^+]$. ESI- MS (m/z, %): 310.01 (100) $[(\text{SPPH}_2)(\text{O}_2\text{SMe})\text{N}]^-$. Molar conductivity: $\Lambda_{\text{M}} = 76.9 \text{ S}\cdot\text{cm}^{-1}\cdot\text{mol}^{-1}$.

Synthesis of $[\text{Ph}_3\text{Te}][\{(\text{SP}(\text{OEt})_2)(\text{O}_2\text{SC}_6\text{H}_4\text{Cl-4})\text{N}\}]$ (2)

A reaction mixture of $[(\text{EtO})_2\text{P}(\text{S})](4\text{-ClC}_6\text{H}_4\text{SO}_2\text{N})\text{K}$ (0.107 g, 0.28 mmol) and Ph_3TeCl (0.1104 g, 0.28 mmol) in 20 mL of anhydrous methylene chloride was stirred at room temperature for 48 h. KCl was removed by filtration and from the clear solution the solvent was evaporated under vacuum. The obtained colorless oil was washed several times with n-hexane. The title compound was obtained as a yellowish oil.

Yield: 0.18 g (91.5%). Anal. Calcd. for $\text{C}_{28}\text{H}_{29}\text{ClNO}_4\text{PS}_2\text{Te}$ (MW 701.69): C, 47.93; H, 4.17; N, 2.00%. Found: C, 48.22; H, 4.38; N, 2.15%. ^1H NMR: δ 1.17t (6H, $-\text{OCH}_2\text{CH}_3$, $^3J_{\text{HH}}$ 7.08 Hz), 3.97m (ABXY₃ system with δ_{A} 3.951 and δ_{B} 3.936 ppm, 4H, $-\text{OCH}_2\text{CH}_3$, $^2J_{\text{HH}}$ 9.9, $^3J_{\text{HH}}$ 7.2 and 7.0, $^3J_{\text{PH}}$ 8.4 and 8.6 Hz), 7.28d (2H C₆H₄-meta, $^3J_{\text{HH}}$ 8.61 Hz), 7.51t (6H, C₆H₅-meta, $^3J_{\text{HH}}$ 7.60 Hz), 7.58t (3H, C₆H₅-para, $^3J_{\text{HH}}$ 7.32 Hz), 7.77m (8H, C₆H₅-ortho + C₆H₄-ortho). ^{13}C NMR: δ 15.91d (OCH_2CH_3 , $^3J_{\text{PC}}$ 8.61 Hz), 62.51d (OCH_2CH_3 , $^2J_{\text{PC}}$ 6.51 Hz), 126.17 (Te-C₆H₅-ipso), 127.85 (C₆H₄-meta), 128.08 (C₆H₄-ortho), 130.66 (Te-C₆H₅-meta), 131.95 (Te-C₆H₅-para), 134.86 (Te-C₆H₅-ortho, $^2J_{\text{TeC}}$ 33.0 Hz), 134.86 (C₆H₄-ipso), 136.23 (C₆H₄-para). ^{31}P NMR: δ 51.76s. ^{125}Te NMR: δ 746.67s. ESI+ MS (m/z, %): 361.02 (100) $[\text{Ph}_3\text{Te}^+]$. ESI- MS (m/z, %): 341.98 (100) $[\{(\text{SP}(\text{OEt})_2)(\text{O}_2\text{SC}_6\text{H}_4\text{Cl-4})\text{N}\}]^-$. Molar conductivity: $\Lambda_{\text{M}} = 43.4 \text{ S}\cdot\text{cm}^{-1}\cdot\text{mol}^{-1}$.

Synthesis of $[\text{Ph}_3\text{Te}][\text{O}_2\text{PPh}_2]$ (3)

A reaction mixture of $(\text{Ph}_2\text{PO}_2)\text{K}$ (0.359 g, 1.40 mmol) and Ph_3TeCl (0.551 g, 1.40 mmol) in 30 mL of anhydrous methylene chloride was stirred at room temperature for 24 h. The resulting KCl was removed by filtration. The solvent was evaporated in vacuum and the title compound was obtained as a colorless powder. Yield: 0.742g (92%). M.p. 130 – 132°C. Anal. Calcd. for $\text{C}_{30}\text{H}_{25}\text{O}_2\text{P}_2\text{Te}$ (MW 576.10): C 62.55, H 4.37%. Found: C 62.89; H 4.64%. ^1H NMR: δ 7.19dt (6H, P-C₆H₅-meta, $^3J_{\text{HH}}$ 7.5, $^4J_{\text{HH}}$ 2.76 Hz), 7.26t (2H, P-C₆H₅-para, $^3J_{\text{HH}}$ 7.21 Hz), 7.36t (6H, Te-C₆H₅-meta, $^3J_{\text{HH}}$ 7.66 Hz), 7.45t (3H, Te-C₆H₅-para, $^3J_{\text{HH}}$ 7.38 Hz), 7.61ddd (4H, P-C₆H₅-ortho, $^3J_{\text{HH}}$ 6.80, $^4J_{\text{HH}}$ 1.51, $^3J_{\text{PH}}$ 11.57 Hz), 7.70d (6H, Te-C₆H₅-ortho, $^3J_{\text{HH}}$ 8.39 Hz). ^{13}C NMR: δ 127.64d (P-C₆H₅-meta, $^3J_{\text{PH}}$ 12.41 Hz), 128.85s (Te-C₆H₅-ipso), 129.88d (P-C₆H₅-para, $^4J_{\text{PC}}$ 1.78 Hz), 129.97s (Te-C₆H₅-meta), 131.00s (Te-C₆H₅-para), 131.38d (P-C₆H₅-ortho, $^2J_{\text{PC}}$ 9.69 Hz), 134.90s (Te-C₆H₅-ortho, $^2J_{\text{TeC}}$ 32.43 Hz), 137.95d (P-C₆H₅-ipso, $^1J_{\text{PH}}$ 104.3 Hz), ^{31}P NMR: δ 20.95s. ^{125}Te NMR: δ 761.86s. ESI+ MS (m/z, %): 361.02 (100) $[\text{Ph}_3\text{Te}^+]$. ESI- MS (m/z, %): 217.04 (100) $[\text{Ph}_2\text{PO}_2]^-$. Molar conductivity: $\Lambda_{\text{M}} = 56.9 \text{ S}\cdot\text{cm}^{-1}\cdot\text{mol}^{-1}$.

X-ray structure determination

The details of the crystal structure determination and refinement for compound $3\cdot\text{Ph}_2\text{PO}_2\text{H}$ are given in Table 2. Data were collected on a Bruker SMART APEX diffractometer by using graphite-monochromated Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$). The crystals were attached with paratone/N oil on cryoloops and the data were collected at room temperature (297 K). The structure was refined with anisotropic thermal parameters. The hydrogen atoms were refined with a riding model and a mutual isotropic thermal parameter. For structure solving and refinement the software package SHELX-97 was used [28]. The drawings were created with the Diamond program [29].

Table 2

X-ray crystal data and structure refinement for compound $3\cdot\text{Ph}_2\text{P}(\text{O})\text{OH}$

Molecular formula	$\text{C}_{42}\text{H}_{36}\text{O}_4\text{P}_2\text{Te}$
<i>M</i>	296.38
Crystal system	Triclinic
Space group	<i>P</i> -1
Temperature (K)	293(2)
<i>a</i> /Å	11.257(3)
<i>b</i> /Å	11.616(3)
<i>c</i> /Å	16.160(4)

Table 2 (continued)

α°	97.993(5)
β°	103.336(4)
γ°	112.368(4)
$V/\text{\AA}^3$	1839.9(8)
Z	2
$D_{\text{calc}}/\text{gcm}^{-3}$	1.434
$F(000)$	804
$\mu(\text{Mo-K}\alpha)/\text{mm}^{-1}$	0.937
Crystal size (mm^3)	0.40 x 0.30 x 0.20
θ range for data collection ($^\circ$)	1.96 to 25.00
Reflections collected	17580
Independent reflections	6449 [$R_{\text{int}} = 0.0364$]
Absorption correction	Semi-empirical from equivalents ²⁸
Data / restraints / parameters	6449 / 0 / 446
Goodness-of-fit on F^2	1.127
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0397$, $wR_2 = 0.0890$
R indices (all data) ^a	$R_1 = 0.0450$, $wR_2 = 0.0914$
Largest difference peak and hole (e \AA^{-3})	0.825 and -0.560

CONCLUSIONS

New triorganotellurium(IV) compounds of type $[\text{Ph}_3\text{Te}][(\text{R}_2\text{PS})(\text{R}'\text{SO}_2)\text{N}]$, ($\text{R} = \text{Ph}$, OEt ; $\text{R}' = \text{Me}$, $4\text{-ClC}_6\text{H}_4$), as well as the species $[\text{Ph}_3\text{Te}][\text{O}_2\text{PPh}_2]$, were obtained by salt metathesis reactions between Ph_3TeCl and the potassium salt of the appropriate organophosphorus acid. The new compounds are ionic species, which behave as 1 : 1 electrolytes in DMSO solution. For the adduct $[\text{Ph}_3\text{Te}][\text{O}_2\text{PPh}_2] \cdot \text{Ph}_2\text{P}(\text{O})\text{OH}$ the single-crystal X-ray diffraction structure revealed strong $\text{Te} \cdots \text{O}$ interactions between the triphenyltelluronium cation and the Ph_2PO_2^- anion and the free $\text{Ph}_2\text{PO}_2\text{H}$ acid, respectively. The two ligands have a monodentate behaviour towards tellurium, thus determining a square pyramidal coordination geometry about the heavy chalcogen.

Supplementary material

CCDC 1027018 contains the supplementary crystallographic data for compound $3 \cdot \text{Ph}_2\text{P}(\text{O})\text{OH}$. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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