The interactions between the sterically demanding trimesitylphosphine oxide and trimesityphosphine with scandium and selected lanthanide ions

Andrew W.G. Platt [a, *], Kuldip Singh [b]

[a] School of Sciences, Staffordshire University, Leek Road, Stoke on Trent, ST4 2DF, UK
[b] Chemistry Department, The University, Leicester, LE1 7RH, UK

ABSTRACT

The reactions between lanthanide nitrates, $Ln(NO_3)_3$ and scandium and lanthanide trifluoromethane sulfonates, $Ln(Tf)_3$ with trimesitylphosphine oxide, Mes_3PO show that coordination to the metal ions does not lead to crystalline complexes. Investigation of the reactions by 31-P NMR spectroscopy shows that weak complexes are formed in solution. The crystal structures of $Mes_3PO \cdot 0.5CH_3CN$ (**1**) and $[Mes_3PO]_3H_3O \cdot 2CH_3CN \cdot Tf$ (**2**), formed in the reaction between $ScTf_3$ and Mes_3PO, are reported. Trimesitylphosphine, Mes_3P, is protonated by scandium and lanthanide trifluoromethane sulfonates and lanthanide nitrates in CD_3CN and the structure of $[Mes_3PH]Cl \cdot HCl \cdot 2H_2O$ (**3**) is reported.

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1. Introduction

The coordination chemistry of phosphine oxides with lanthanide ions has attracted interest for a number of years because of the potential the complexes have in a variety of applications [1,2].

Complexes of lanthanide nitrates have been studied in some detail in the solvent extraction separation of lanthanide from actinides carried out in nitric acid solutions [3–5]. We have previously studied the effect on structures and solution properties of increasing the size of the R group in R_3PO where we found that solid state structures and solution properties depend on a balance between steric and electronic effects of the phosphine oxides and the size of the lanthanide ions [6–8]. Cone angles, are not available for phosphine oxides, and as a result we have used the cone angles of the parent phosphine [9] as a proxy for the steric demands of the oxides. Previous studies on trialkyl phosphine oxides R_3PO (R = Et [6], ^iBu [7], cyclohexyl [10] and ^tBu [8]) have shown that significant differences in the properties of the lanthanide nitrate complexes result with changes in the steric and electronic properties of the ligands.

We have extended our studies to include the more highly sterically demanding trimesitylphosphine oxide, for which the cone angle of the parent phosphine is 212° [9].

Lanthanide ions do not form complexes with soft donors such as phosphines in the presence of competition from hard donor ligands such as water. We have previously found that sterically crowded phosphine oxides are able to hydrogen bond to lanthanide-coordinated water molecules [11,12]. Theoretical studies of hydrogen bonding between phosphines and a variety of hydrogen donors have indicated that such bonding can give rise to stable complexes [13–15] and an $HF-PH_3$ dimer has been identified in the gas phase [16]. In view of this we have conducted a brief investigation into the interaction of the sterically demanding Mes_3P with hydrated lanthanide salts to investigate whether hydrogen bonding to coordinated water occurred.

2. Results and discussion

2.1. Synthesis and solid state structures

Attempted synthesis of complexes of Mes_3PO with representative lanthanide nitrates across the series (Ln = Pr, Nd, Gd, Ho, Er, Lu) was carried out in ethanol solution. The phosphine oxide is poorly soluble in warm ethanol but on addition of solutions of lanthanide nitrates clear solutions were obtained which implied that

* Corresponding author.
  E-mail address: a.platt@staffs.ac.uk (A.W.G. Platt).

interaction with the metal had taken place. Attempts to obtain crystalline materials from these reactions by slow evaporation, cooling to −30 °C or slow diffusion with diethylether or toluene followed by cooling were not successful. The reactions led to either recovery of unreacted Mes_3PO, no solid materials or to the formation of oils. The reason for the apparent lack of coordination with $Ln(NO_3)_3$ is probably a combination of the high affinity of lanthanide ions for nitrate and the increased steric demands of the ligand limiting access of the phosphoryl oxygen to the coordination sites on the metal.

Clearly there is some interaction between Mes_3PO and the lanthanide nitrates in solution, but this does not result in the formation of complexes which are sufficiently stable to permit isolation. The reactions of lanthanide and scandium trifluoromethane sulfonates (triflates) were examined as the triflate ion has a low coordinating ability and might thus lead to a less crowded coordination environment around the metal. In reactions with $LnTf_3$ Ln = (Ln = La, Nd, Eu, Er, Lu) in ethanol the initial suspensions of Mes_3PO cleared on addition of the lanthanide salt, again implying some complex formation. The reaction mixtures, however, failed to yield characterisable materials.

In order to assess the steric congestion around the oxygen in trimesitylphosphine oxide the crystal structure of Mes_3-$PO·0.5CH_3CN$ (**1**) was determined. The structure is shown in Fig. 1 details of the data collection and refinement are given in Table 1 and selected bond distances and angles in Table 2. The compound crystallises with two Mes_3PO molecules and one CH_3CN in the unit cell. There are no close contacts between the acetonitrile and the phosphine oxide. Comparison of this structure with Ph_3PO, which forms stable complexes with all simple lanthanide salts, indicates that the structure of Mes_3PO changes to reduce steric interactions between the methyl groups and in doing so increases the crowding of the environment around the O-atom, as described below. The P=O bond distance is 1.486 Å which is very similar to those reported for Ph_3PO, for example 1.483 [13] and 1.487 [14]. The C−P−C angles widen to reduce steric interactions between the methyl groups in Mes_3PO with an average of 110.2° compared to 106.4° in Ph_3PO. There is also a significant increase in the P−C bond distance compared with Ph_3PO from 1.800 Å to 1.828 Å which will further reduce steric crowding between the methyl groups. The structure

shows that, compared to Ph_3PO, reduction in steric strain by widening of the C−P−C angle has the effect of making the methyl groups effectively shield the phosphoryl oxygen and this probably limits the availability of the P=O group to coordinate to the lanthanide ion, where the environment around the metal will already contain coordinated nitrate ions and/or water molecules.

The reaction with scandium triflate in CH_3CN did produce crystalline material which was suitable for x-ray diffraction study. The solid does not, however, contain scandium but is $[Mes_3PO]_3H_3O^+$ $CF_3SO_3^-.2CH_3CN$ (**2**). The hydronium ions are formed are almost certainly by acid hydrolysis of hydrated Sc^{3+}. The structure is shown in Fig. 2 and details of the data collection and refinement are included in Table 1. Three Mes_3PO molecules are coordinated to H_3O^+ via hydrogen bonding and the CH_3CN and triflate ions are not involved in hydrogen bonding interactions. The P=O bonds are slightly longer (average 1.493 Å) than in Mes_3PO as expected on coordination to a positively charged centre. Larger increases in bond distances in similar structures such as $Ph_3PO·HBr$ (1.550 Å) [15] and $Ph_3PO·HNO_3$ (1.499 Å) [16] are probably due to the proton being coordinated to one phosphine oxide rather than three as in this study.

Trimesitylphosphine oxide is bulkier than trialkylphosphine oxides such as $^tBu_3PO$ (the cone angle $^tBu_3P$ is 182°) which forms only 1:2 complexes with lanthanide nitrates [8]. Trimesitylphosphine itself forms complexes with a number of metals and coordination compounds with Hg [17], Ag [18], Pt [19] and Au [20] have been characterised. The inability of Mes_3PO to form stable complexes might thus be considered to be surprising as the cone angle of the oxide will certainly be lower than that of the phosphine. A comparison of some of the parameters which might be relevant to their relative coordinating ability are given in Table 3.

The approach of Mes_3PO to the metal centre will be restricted by steric interaction with the methyl groups. The proximity of the methyls to the coordinating atoms indicates that the environments in Mes_3P and Mes_3PO are similar with P … O and C … O distances relatively short. Although the distances could be considered as indicative of hydrogen bonding (C … O average in Mes_3PO is 2.89 Å compared to a sum of Van der Waals radii for C and O of 3.2 Å and C … P average in Mes_3P is 2.72 Å compared to a sum of Van der Waals radii for P and C of 3.5 Å), it is more likely that these distances are imposed by the geometrical constraints of the molecules [21].

The differences in coordinating ability probably lies in the strong hydration of the lanthanide and scandium ions which combines with the steric congestion of the ligand to make coordination impossible under the conditions employed. In addition the metal ions coordinated to Mes_3P are likely to be less strongly solvated (for example using CH_2Cl_2/THF as the reaction medium) and hence the effective size of the metal ions will be lower than hydrated lanthanide or scandium ions.

2.2. Solution NMR investigation

Given that there is clearly some interaction between the lanthanide ions and Mes_3PO several lanthanide triflate/Mes_3PO systems were investigated by 31-P NMR spectroscopy.

The results are shown in Table 4. The absence of any large coordination shifts with the paramagnetic lanthanide ions indicates that there is no direct interaction between the phosphine oxide and the metal. However, the lines are broad and show a significant temperature dependence of the chemical shifts which whilst smaller than that observed for Ln−O=P systems, is an order of magnitude greater than that of free Mes_3PO. We recently reported the structures of $[Ln(H_2O)_5(Cy_3PO)_2]·2Cy_3PO·3X$ (X = Cl, Br) [11,12] where Cy = cyclohexyl, C_6H_{11} in which the Cy_3PO are either directly bonded to the metal ion or through hydrogen bonding to the
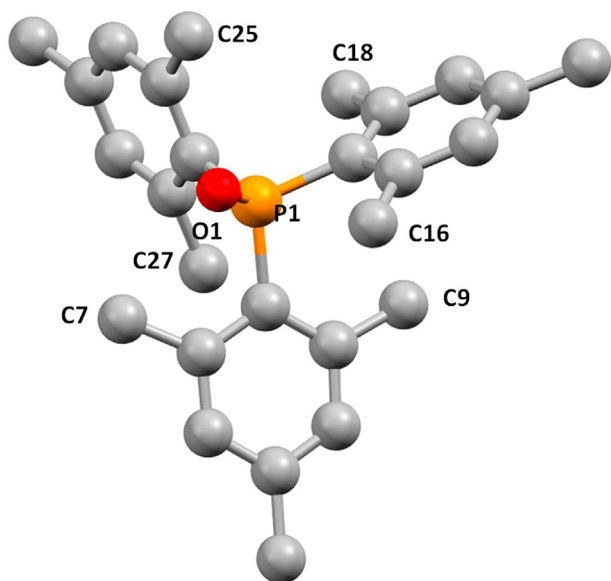

Fig. 1. The structure of $Mes_3PO·CH_3CN$ The acetonitrile molecule is omitted for clarity.

Table 1
Data collection and refinement for (1) and (2).

	Mes$_3$PO 0.5CH$_3$CN	[Mes$_3$PO]$_3$H$_3$O·CF$_3$SO$_3$
Empirical formula	C56 H69 N O2 P2	C86 H107 F3 N2 O7 P3 S
Formula weight	850.06	1462.71
Temperature	150(2) K	150(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	Triclinic	Triclinic
Space group	P-1	P-1
Unit cell dimensions	a = 8.101(5) Å α = 75.593(11)°	a = 16.176(4) Å α = 105.682(5)°
	b = 16.449(10) Å β = 83.298(12)°	b = 16.621(4) Å β = 106.410(6)°
	c = 19.055(12) Å γ = 79.329(13)°	c = 17.233(4) Å γ = 106.253(5)°
Volume	2410(3) Å^3	3948.1(17) Å^3
Z	2	2
Density (calculated)	1.171 Mg/m^3	1.230 Mg/m^3
Absorption coefficient	0.132 mm^{-1}	0.164 mm^{-1}
F(000)	916	1562
Crystal size	0.35 × 0.23 × 0.15 mm^3	0.25 × 0.20 × 0.18 mm^3
Theta range for data collection	1.89—26.00°	1.38—25.00°
Index ranges	−19 ≤ h ≤ 13, −19 ≤ k ≤ 19, −20 ≤ l ≤ 20	−19 ≤ h ≤ 13, −19 ≤ k ≤ 19, −20 ≤ l ≤ 20
Reflections collected	18,854	19,947
Independent reflections	9325 [R(int) = 0.0682]	13,161 [R(int) = 0.0960]
Completeness to theta = 26.00°	98.8%	94.6%
Absorption correction	Empirical	Empirical
Max. and min. transmission	0.981 and 0.762	0.862 and 0.548
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	9325/0/569	13161/0/948
Goodness-of-fit on F^2	0.932	0.839
Final R indices [I > 2sigma(I)]	R1 = 0.0606, wR2 = 0.1203	R1 = 0.0818, wR2 = 0.1372
R indices (all data)	R1 = 0.0978, wR2 = 0.1329	R1 = 0.1951, wR2 = 0.1730
Largest diff. peak and hole	0.367 and −0.441 e.Å^{-3}	0.403 and −0.401 e.Å^{-3}

Table 2
Selected bond distances and angles for (1) and (2).

	1	2
P(1)—O(1)	1.4858(18)	1.492(4)
P(1A)—O(1A)	1.4858(19)	1.500(4)
P(1B)—O(1B)		1.490(3)
P(1)—C(1)	1.831(3)	1.815(6)
P(1A)—C(1A)	1.826(3)	1.833(5)
P(1B)—C(1B)		1.843(6)
P(1)—C(10)	1.826(3)	1.808(6)
P(1A)—C(10A)	1.831(3)	1.831(6)
P(1B)—C(10B)		1.799(6)
P(1)—C(19)	1.825(3)	1.831(6)
P(1A)—C(19A)	1.828(3)	1.812(6)
P(1B)—C(19B)		1.821(6)
O(1)—P(1)—C(1)	107.65(12)	107.0(2)
O(1A)—P(1A)—C(1A)	108.94(11)	108.2(2)
O(1B)—P(1B)—C(1B)		108.4(2)
O(1)—P(1)—C(10)	109.86(11)	108.6(2)
O(1A)—P(1A)—C(10A)	108.02(12)	108.7(2)
O(1B)—P(1B)—C(10B)		107.9(2)
O(1)—P(1)—C(19)	108.60(11)	107.9(2)
O(1A)—P(1A)—C(19A)	108.94(11)	107.3(2)
O(1B)—P(1B)—C(19B)		109.2(2)
C(1)—P(1)—C(10)	108.92(12)	113.5(3)
C(1A)—P(1A)—C(10A)	111.10(11)	108.3(3)
C(1B)—P(1B)—C(10B)		113.0(3)
C(1)—P(1)—C(19)	111.07(12)	112.9(£0
C(1A)—P(1A)—C(19A)	108.94(11)	113.3(3)
C(1B)—P(1B)—C(19B)		106.9(3)
C(10)—P(1)—C(19)	110.69(12)	113.0(3)
C(10A)—P(1A)—C(19A)	110.06(11)	111.0(3)
C(10B)—P(1B)—C(19B)		111.5(3)

Fig. 2. The structure of [Mes$_3$PO]$_3$H$_3$O$^+$ CF$_3$SO$_3^-$ hydrogen atoms and acetonitrile are omitted for clarity. The dashed lines represent hydrogen bonds.

coordinated water molecules. In these systems the temperature dependence of the 31-P NMR shifts depends on whether the Cy$_3$PO is directly bonded to the metal for example +0.7 ppmK^{-1} for P═O—Eu and −0.1 ppmK^{-1} for P═O … H$_2$O—Eu. The same reversal in the sign of the temperature dependence is also found for Yb and seems to be indicative of th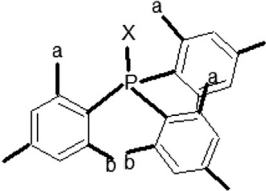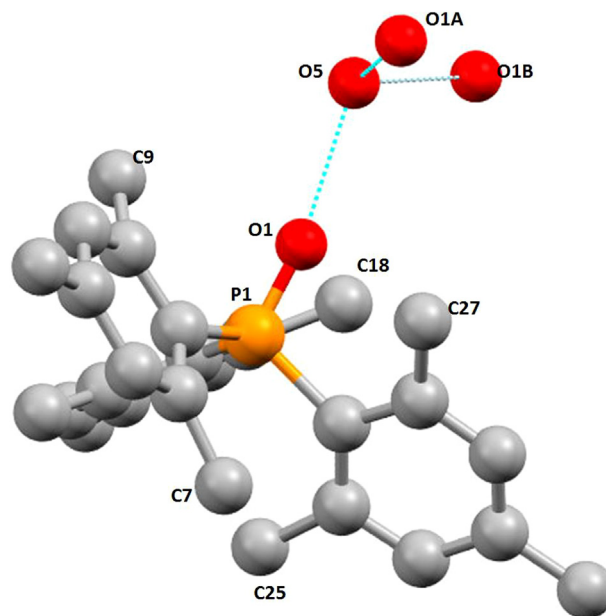e differing nature of the interaction. It is interesting that the temperature dependence in the Mes$_3$PO/Ln^{3+} systems is opposite of that found for 31-P shift for phosphine oxides directly bonded to lanthanides [7,8], but the same as for phosphine oxides hydrogen bonded to lanthanide coordinated water molecules. In the absence of crystallographic data from isolated compounds it is not possible to be certain of the nature of this interaction, but hydrogen bonding to the coordinated water molecules, as previously observed with other sterically crowded phosphine oxides [11,12] seems reasonable.

The reaction between scandium and lanthanide triflates

Table 3
Structural parameters for Mes_3P-X.

	Mes_3P	Mes_3PO	$[Mes_3PO]_3H_3O^+$
P—C	1.837(3)	1.828(3)	1.821(14)
Me(a) … Me(a)	4.76(12)	4.97(3)	4.96(14)
C(a) … P	2.72(5)		
C(a) … O		2.89(1)	2.89(3)
C—P—C	105.9(3.7)	110.2(1.1)	111.5(2.3)

Table 4
^{31}P NMR data for lanthanide triflate/Mes_3PO in CD_3CN.

|	δ/ppm	$W_{1/2}$/Hz[a]	Temperature dependence[b]/ppmK^{-1}
Ln			
Nd	31.9	71	0.04
Eu	29.9	135	−0.14
Ho	38.4	63	−0.15
Er	28.8	220	−0.17
Yb	29.1	13	−0.04
Lu	32.3	10	−0.001
Mes_3PO	26.9	5	0.003

[a] At 30 °C.
[b] Temperature range 30–70 °C.

(Ln = Eu, Lu) and lanthanide nitrates (Ln = La, Ce, Eu, Yb and Lu) and Mes_3P in CD_3CN was carried out to examine the possible formation of hydrogen bonded phosphine complexes. If such an interaction were to occur a larger paramagnetic shift might be expected than is observed for hydrogen bonded R_3PO as the effect would be transmitted through 3 bonds rather than 4. The NMR spectra show in addition to a signal from unreacted phosphine at −36.5 a peak at −26.9 ppm $^1J_{PH}$ 510 Hz which was independent of the metal ion used. This has very similar shift and coupling constant to that reported for Mes_3PH^+ [22]. The formation of the phosphonium salt undoubtedly proceeds via the protonation of the phosphine by the acidic hydrogens of the coordinated water molecules as indicated in Eq. (1).

$$LnX_3(H_2O)_n + Mes_3P \rightarrow LnX_2(OH)(H_2O)_{n-1} + Mes_3PH^+X^-$$
(1)
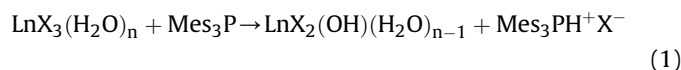
This was confirmed by carrying out the reaction with $ScTf(D_2O)_6$ which produced a characteristic 1:1:1 triplet ($^1J_{PD}$ = 76 Hz) expected from P–D coupling. Further confirmation was obtained by preparing a fully characterised Mes_3PH^+ salt by reaction of Mes_3P with aqueous HCl in CH_3CN. Single crystals of Mes_3PH^+ $Cl^-·HCl·2H_2O$ formed spontaneously from the reaction mixture. The 31-P NMR spectrum showed the expected single signal at −25.6 $^1J_{PH}$ = 511 Hz. Hydrogen bonding of the phosphine to the coordinated water is probably the first step in this reaction.

The structure of $Mes_3PH^+Cl^-·HCl·2H_2O$ is shown in Fig. 3, details of the data collection and refinement in Table 5 and selected bond distances and angles in Table 6. The structure is similar to those reported for the Mes_3PH^+ with other anions [23–26]. The P–C distances, at an average of 1.799 Å, are slightly below the lower end of the range reported for other structures where the P–C
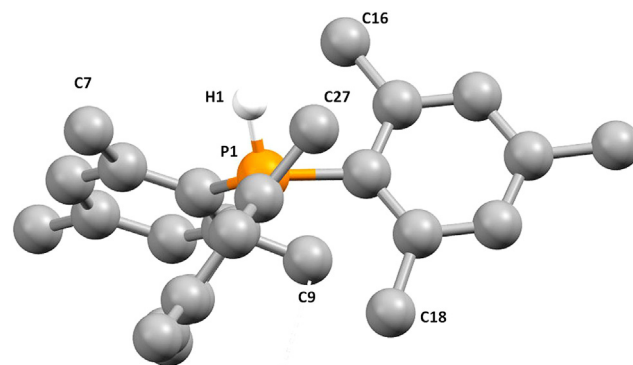
Fig. 3. The Structure of $[Mes_3PH]Cl·HCl·2H_2O$ Chloride ions, water, HCl and carbon and hydrogen atoms omitted for clarity.

distance ranges from 1.801 to 1.896 Å with an average of 1.833 Å. The C–P–C bond angles vary considerably in the previously reported structures from 106 to 118° (average 112.4°) and the average of 114.8° found here lies within that range. There are no interactions between the P–H and H_2O, HCl and Cl^- but hydrogen bonding is found between the lattice water, HCl and chloride ions. Details are given as supplementary information in Table S1. In the absence of short contacts with the phosphonium hydrogen the P–H distance of 1.311 Å lies within the range of 1.341−1.263 Å (average 1.301 Å) reported for the Mes_3PH^+ ion reported in other compounds [23–26].

3. Conclusion

Strong complexes between Mes_3PO and lanthanide ions do not form due to a combination of steric factors related to the congestion around the donor atom and probably the strong solvation of the lanthanide starting materials. The interaction which leads to increased solubility of Mes_3PO observed in reactions with the lanthanide ions is probably due to hydrogen bonding with coordinated water molecules in $Ln(NO_3)_3 6H_2O$ and $Ln(CF_3SO_3)_3 6H_2O$ used in the reactions. Trimesitylphosphine itself reacts with hydrated lanthanide ions to form the well-defined phosphonium salt rather than forming hydrogen bonded complexes.

4. Experimental

Data for x-ray structures were collected on a Bruker APEX 2000 CCD diffractometer using graphite-monochromated Mo-K_a radiation, λ = 0.71073 Å.

The data were corrected for Lorentz and polarisation effects and empirical absorption corrections applied. Structure solution by direct methods and structure refinement based on full-matrix least-squares on F^2 employed SHELXTL version 6.10 [27]. Hydrogen atoms were included in calculated positions (C–H = 0.95−0.98 Å) riding on the bonded atom with isotropic displacement parameters set to 1.5 $U_{eq}(C)$ for methyl H atoms and 1.2 $U_{eq}(C)$ for all other H atoms. All non-H atoms were refined with anisotropic displacement parameters.

Infrared spectra were recorded with a resolution of ±2 cm^{-1} on a Thermo Nicolet Avatar 370 FT-IR spectrometer operating in ATR mode. Samples were compressed onto the optical window and spectra recorded without further sample pre-treatment.

NMR spectra were obtained on a JEOL ECX −400

Trimesitlyphosphine oxide Trimesitylphosphine (5.08 g 13 mmol) was suspended in 25 mL acetone and 2.59 g 30% aqueous hydrogen peroxide was added over 10 min. The suspension was

Table 5
Crystal data and structure refinement for $Mes_3PH^+ \cdot Cl^- \cdot HCl \cdot 2H_2O$.

Empirical formula	C27 H39 Cl2 O2 P	
Formula weight	497.45	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 11.6750(19) Å	$\alpha = 90°$
	b = 14.603(2) Å	$\beta = 90°$
	c = 15.704(2) Å	$\gamma = 90°$
Volume	2677.4(7) Å^3	
Z	4	
Density (calculated)	1.234 Mg/m^3	
Absorption coefficient	0.324 mm^{-1}	
F(000)	1064	
Crystal size	0.16 × 0.09 × 0.04 mm^3	
Theta range for data collection	1.90—26.00°	
Index ranges	$-14 \le h \le 14, -18 \le k \le 17, -19 \le l \le 19$	
Reflections collected	21,143	
Independent reflections	5255 [R(int) = 0.1783]	
Completeness to theta = 26.00°	100.0%	
Absorption correction	Empirical	
Max. and min. transmission	0.862 and 0.488	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	5255/4/298	
Goodness-of-fit on F^2	0.748	
Final R indices [I > 2sigma(I)]	R1 = 0.0622, wR2 = 0.0854	
R indices (all data)	R1 = 0.1223, wR2 = 0.0980	
Absolute structure parameter	0.01(9)	
Largest diff. peak and hole	0.337 and −0.330 e.Å^{-3}	

Table 6
Bond lengths [Å] and angles [°] for (3).

P(1)—C(10)	1.785(4)
P(1)—C(1)	1.806(5)
P(1)—C(19)	1.810(5)
P(1)—H(1)	1.3117
C(10)—P(1)—C(1)	117.4(2)
C(10)—P(1)—C(19)	113.8(2)
C(1)—P(1)—C(19)	113.4(2)
C(10)—P(1)—H(1)	101.7
C(1)—P(1)—H(1)	105.0
C(19)—P(1)—H(1)	103.3

heated to 50 °C for 10 min and stirred at room temperature for 2 d. The mixture was filtered, washed with methanol and dried at the pump to give 5.06 g white solid. Recrystallisation from acetonitrile gave crystals of $[Mes_3PO]_2CH_3CN$ suitable for x-ray diffraction.

Synthesis $Mes_3PH^+Cl^- \cdot HCl \cdot 2H_2O$

Trimesitylphosphine 0.50 g (1.25 mmol) was suspended in CH_3CN (4.0 g) and 0.22 g 36% aqueous HCl, (2.17 mmol) was added. The mixture cleared on addition and crystals were deposited on standing overnight. The yellow crystals were filtered, washed with CH_3CN and dried at the pump to give 0.39 g (63%)

Analysis expected(found) C 65.19(65.00) H7.90(8.04)

NMR ^{1}H (400 MHz) 1.90(s) 9H CH_3, 2.33(s) 9H CH_3 2.52(s) 9H CH_3, 6.94(s) 3H CH, 7.07(s) 3H CH, 9.4(d) $^1J_{PH}$ 510 Hz 1H PH; ^{31}P (161 MHz) $CDCl_3$ d −25.6 $^1J_{PH}$ 510 Hz.

$ScTF_36D_2O$ was prepared by recrystallising the hydrated salt once from D_2O.

Appendix A. Supplementary data

CCDC 1419070—1419072 contain the supplementary crystallographic data for (1)—(3) respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving. html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ UK: fax (+44+1223-336-033) or e-mail: deposit@ccdc.cam.ac.uk.

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.molstruc.2016.01.073.

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