Donor–Stabilised Cations, Phosphinamide Anions, and Unusual Oxidative Cyclisation Products from Halogenated Phosphoranimines and Phosphinimines with a Bulky 2,4,6–Tri–*tert*–butylphenyl Substituent at Nitrogen

Keith Huynh,[†] Cindy P. Chun,[†] Alan J. Lough, [†] and Ian Manners[*†,‡]

Contribution from the [†]*Davenport Laboratories, Department of Chemistry, University of Toronto, 80 St. George Street, Toronto, Ontario, Canada, M5S 3H6*
and the
[‡]*School of Chemistry, University of Bristol, Bristol BS8 1TS, England*

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Table 1. Crystal data and structure refinement for **[9]OTf•CH_2Cl_2**.

Identification code	k0472	
Empirical formula	C27 H41 Cl4 F3 N3 O3 P S	
Formula weight	717.46	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*–1	
Unit cell dimensions	a = 8.7008(2) Å	α = 98.8000(16)°
	b = 10.6081(3) Å	β = 90.9900(18)°
	c = 20.7723(6) Å	γ = 112.4650(15)°
Volume	1744.88(8) $Å^3$	
Z	2	
Density (calculated)	1.366 Mg/m^3	
Absorption coefficient	0.493 mm^{-1}	
F(000)	748	
Crystal size	0.20 x 0.20 x 0.10 mm^3	
Theta range for data collection	2.60 to 27.53°	
Index ranges	$-11 \le h \le 11$, $-13 \le k \le 13$, $-26 \le l \le 26$	
Reflections collected	19547	
Independent reflections	7963 [R(int) = 0.0628]	
Completeness to theta = 27.53°	99.0%	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.970 and 0.703	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7963 / 0 / 391	
Goodness-of-fit on F^2	1.031	
Final R indices [$I>2\sigma(I)$]	R1 = 0.0492, wR2 = 0.1194	
R indices (all data)	R1 = 0.0808, wR2 = 0.1391	
Extinction coefficient	0.0044(11)	
Largest diff. peak and hole	0.490 and –0.458 e.$Å^{-3}$	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **[9]OTf•CH_2Cl_2**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	2582(1)	4830(1)	2225(1)	40(1)
Cl(2)	222(1)	6364(1)	2484(1)	39(1)
P(1)	2656(1)	6765(1)	2423(1)	26(1)
N(1)	3401(2)	7204(2)	3235(1)	26(1)
N(2)	5572(2)	8066(2)	5119(1)	29(1)
N(3)	3566(2)	7790(2)	2021(1)	27(1)
C(1)	2639(3)	6400(3)	3693(1)	31(1)
C(2)	3291(3)	6684(3)	4312(1)	31(1)
C(3)	4836(3)	7845(2)	4527(1)	26(1)
C(4)	5541(3)	8711(2)	4055(1)	28(1)
C(5)	4846(3)	8362(2)	3434(1)	26(1)
C(6)	4919(3)	7079(3)	5571(1)	36(1)
C(7)	7111(3)	9278(3)	5360(1)	34(1)
C(8)	4624(3)	8798(2)	1679(1)	24(1)
C(9)	4476(3)	10088(2)	1729(1)	26(1)
C(10)	5619(3)	11094(2)	1422(1)	29(1)
C(11)	6842(3)	10873(2)	1058(1)	28(1)
C(12)	6896(3)	9572(2)	998(1)	28(1)
C(13)	5819(3)	8508(2)	1293(1)	25(1)
C(14)	3080(3)	10429(3)	2071(1)	31(1)
C(15)	3083(3)	10327(3)	2802(1)	35(1)
C(16)	1374(3)	9459(3)	1714(1)	38(1)
C(17)	3245(4)	11919(3)	2043(2)	46(1)
C(18)	8041(3)	11976(3)	707(1)	33(1)
C(19)	8041(5)	13408(4)	939(2)	79(1)
C(20)	7543(5)	11567(4)	-22(2)	79(1)
C(21)	9825(4)	12082(4)	828(2)	68(1)
C(22)	5964(3)	7085(3)	1182(1)	30(1)
C(23)	7285(4)	7035(3)	705(2)	43(1)
C(24)	6539(3)	6800(3)	1828(1)	35(1)

C(25)	4303(3)	5947(3)	860(1)	36(1)
Cl(3)	9304(1)	8165(1)	3925(1)	56(1)
Cl(4)	6506(1)	5547(1)	3478(1)	54(1)
C(27)	8160(4)	6489(3)	4084(2)	47(1)
S(1)	-381(1)	2803(1)	4132(1)	39(1)
F(1)	-126(3)	2621(2)	2871(1)	86(1)
F(2)	-898(4)	674(2)	3206(1)	95(1)
F(3)	-2620(2)	1641(3)	3143(1)	78(1)
O(1)	1312(3)	2955(3)	4224(2)	76(1)
O(2)	-584(3)	4074(2)	4124(1)	51(1)
O(3)	-1553(3)	1884(2)	4502(1)	55(1)
C(26)	-1041(4)	1904(3)	3295(2)	51(1)

Table 3. Bond lengths [Å] and angles [°] for **[9]OTf•CH_2Cl_2**.

Cl(1)-P(1)	2.0065(9)
Cl(2)-P(1)	2.0044(9)
P(1)-N(3)	1.467(2)
P(1)-N(1)	1.725(2)
N(1)-C(5)	1.381(3)
N(1)-C(1)	1.383(3)
N(2)-C(3)	1.321(3)
N(2)-C(7)	1.470(3)
N(2)-C(6)	1.472(3)
N(3)-C(8)	1.414(3)
C(1)-C(2)	1.337(4)
C(2)-C(3)	1.439(3)
C(3)-C(4)	1.431(3)
C(4)-C(5)	1.350(3)
C(8)-C(9)	1.410(3)
C(8)-C(13)	1.420(3)
C(9)-C(10)	1.398(3)
C(9)-C(14)	1.548(3)
C(10)-C(11)	1.387(4)
C(11)-C(12)	1.385(3)
C(11)-C(18)	1.531(3)
C(12)-C(13)	1.396(3)
C(13)-C(22)	1.545(3)
C(14)-C(15)	1.538(4)
C(14)-C(16)	1.541(4)
C(14)-C(17)	1.541(3)
C(18)-C(20)	1.518(4)
C(18)-C(19)	1.521(4)
C(18)-C(21)	1.527(4)
C(22)-C(24)	1.536(4)
C(22)-C(23)	1.542(4)
C(22)-C(25)	1.543(3)
Cl(3)-C(27)	1.760(3)
Cl(4)-C(27)	1.764(3)

S(1)-O(1)	1.426(2)
S(1)-O(2)	1.428(2)
S(1)-O(3)	1.440(2)
S(1)-C(26)	1.818(4)
F(1)-C(26)	1.325(4)
F(2)-C(26)	1.342(4)
F(3)-C(26)	1.315(4)

N(3)-P(1)-N(1)	114.16(11)
N(3)-P(1)-Cl(2)	117.20(8)
N(1)-P(1)-Cl(2)	101.94(7)
N(3)-P(1)-Cl(1)	120.21(9)
N(1)-P(1)-Cl(1)	100.08(7)
Cl(2)-P(1)-Cl(1)	100.27(4)
C(5)-N(1)-C(1)	118.4(2)
C(5)-N(1)-P(1)	119.34(16)
C(1)-N(1)-P(1)	122.16(17)
C(3)-N(2)-C(7)	122.6(2)
C(3)-N(2)-C(6)	121.5(2)
C(7)-N(2)-C(6)	115.9(2)
C(8)-N(3)-P(1)	172.95(18)
C(2)-C(1)-N(1)	122.0(2)
C(1)-C(2)-C(3)	121.0(2)
N(2)-C(3)-C(4)	122.5(2)
N(2)-C(3)-C(2)	121.6(2)
C(4)-C(3)-C(2)	115.8(2)
C(5)-C(4)-C(3)	120.6(2)
C(4)-C(5)-N(1)	122.0(2)
C(9)-C(8)-N(3)	119.1(2)
C(9)-C(8)-C(13)	120.9(2)
N(3)-C(8)-C(13)	120.0(2)
C(10)-C(9)-C(8)	117.5(2)
C(10)-C(9)-C(14)	118.2(2)
C(8)-C(9)-C(14)	124.2(2)
C(11)-C(10)-C(9)	123.5(2)
C(12)-C(11)-C(10)	117.0(2)

C(12)-C(11)-C(18)	120.5(2)
C(10)-C(11)-C(18)	122.5(2)
C(11)-C(12)-C(13)	123.5(2)
C(12)-C(13)-C(8)	117.4(2)
C(12)-C(13)-C(22)	119.3(2)
C(8)-C(13)-C(22)	123.3(2)
C(15)-C(14)-C(16)	110.1(2)
C(15)-C(14)-C(17)	105.3(2)
C(16)-C(14)-C(17)	106.5(2)
C(15)-C(14)-C(9)	113.36(19)
C(16)-C(14)-C(9)	109.2(2)
C(17)-C(14)-C(9)	112.2(2)
C(20)-C(18)-C(19)	109.0(3)
C(20)-C(18)-C(21)	108.7(3)
C(19)-C(18)-C(21)	107.3(3)
C(20)-C(18)-C(11)	109.0(2)
C(19)-C(18)-C(11)	112.6(2)
C(21)-C(18)-C(11)	110.2(2)
C(24)-C(22)-C(23)	105.9(2)
C(24)-C(22)-C(25)	113.3(2)
C(23)-C(22)-C(25)	105.4(2)
C(24)-C(22)-C(13)	110.0(2)
C(23)-C(22)-C(13)	112.0(2)
C(25)-C(22)-C(13)	110.06(19)
Cl(3)-C(27)-Cl(4)	111.37(16)
O(1)-S(1)-O(2)	114.12(15)
O(1)-S(1)-O(3)	116.22(16)
O(2)-S(1)-O(3)	114.35(14)
O(1)-S(1)-C(26)	104.33(17)
O(2)-S(1)-C(26)	102.73(15)
O(3)-S(1)-C(26)	102.73(14)
F(3)-C(26)-F(1)	108.8(3)
F(3)-C(26)-F(2)	106.3(3)
F(1)-C(26)-F(2)	107.0(3)
F(3)-C(26)-S(1)	111.5(2)
F(1)-C(26)-S(1)	111.8(2)

F(2)-C(26)-S(1)	111.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **[9]OTf•CH_2Cl_2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	54(1)	21(1)	45(1)	8(1)	12(1)	12(1)
Cl(2)	26(1)	43(1)	43(1)	12(1)	3(1)	9(1)
P(1)	27(1)	22(1)	29(1)	6(1)	3(1)	8(1)
N(1)	26(1)	23(1)	29(1)	10(1)	3(1)	6(1)
N(2)	28(1)	30(1)	30(1)	12(1)	6(1)	9(1)
N(3)	31(1)	24(1)	29(1)	8(1)	6(1)	12(1)
C(1)	26(1)	26(1)	38(1)	13(1)	6(1)	5(1)
C(2)	28(1)	30(1)	35(1)	15(1)	8(1)	6(1)
C(3)	27(1)	24(1)	29(1)	8(1)	6(1)	12(1)
C(4)	28(1)	23(1)	33(1)	8(1)	4(1)	8(1)
C(5)	27(1)	21(1)	31(1)	11(1)	5(1)	7(1)
C(6)	37(1)	40(2)	33(1)	17(1)	6(1)	11(1)
C(7)	35(1)	30(1)	33(1)	7(1)	1(1)	8(1)
C(8)	26(1)	23(1)	21(1)	5(1)	-1(1)	8(1)
C(9)	30(1)	24(1)	24(1)	3(1)	0(1)	13(1)
C(10)	35(1)	22(1)	30(1)	6(1)	-1(1)	11(1)
C(11)	29(1)	28(1)	24(1)	6(1)	0(1)	8(1)
C(12)	30(1)	28(1)	23(1)	5(1)	4(1)	10(1)
C(13)	29(1)	26(1)	21(1)	3(1)	0(1)	12(1)
C(14)	34(1)	26(1)	37(1)	8(1)	7(1)	16(1)
C(15)	40(1)	35(1)	35(1)	2(1)	7(1)	21(1)
C(16)	35(1)	40(2)	44(2)	11(1)	3(1)	19(1)
C(17)	50(2)	36(2)	65(2)	17(1)	18(2)	28(1)
C(18)	37(1)	30(1)	30(1)	11(1)	2(1)	9(1)
C(19)	97(3)	33(2)	104(3)	25(2)	54(3)	15(2)
C(20)	87(3)	75(3)	38(2)	30(2)	-8(2)	-16(2)
C(21)	41(2)	60(2)	102(3)	45(2)	16(2)	8(2)
C(22)	34(1)	27(1)	32(1)	3(1)	5(1)	15(1)
C(23)	49(2)	33(2)	48(2)	3(1)	17(1)	20(1)
C(24)	35(1)	30(1)	43(2)	8(1)	2(1)	17(1)
C(25)	44(2)	26(1)	33(1)	0(1)	1(1)	10(1)

Cl(3)	49(1)	44(1)	71(1)	6(1)	6(1)	13(1)
Cl(4)	50(1)	42(1)	68(1)	17(1)	3(1)	12(1)
C(27)	55(2)	45(2)	52(2)	16(1)	7(1)	27(2)
S(1)	31(1)	29(1)	59(1)	19(1)	5(1)	10(1)
F(1)	90(2)	80(2)	70(2)	30(1)	44(1)	7(1)
F(2)	124(2)	48(1)	111(2)	3(1)	50(2)	33(1)
F(3)	49(1)	100(2)	60(1)	4(1)	-3(1)	8(1)
O(1)	41(1)	78(2)	120(2)	32(2)	-2(1)	30(1)
O(2)	46(1)	29(1)	78(2)	17(1)	9(1)	14(1)
O(3)	66(1)	36(1)	52(1)	20(1)	13(1)	3(1)
C(26)	49(2)	42(2)	59(2)	17(2)	24(2)	11(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for **[9]OTf•CH_2Cl_2**.

	x	y	z	U(eq)
H(1A)	1622	5621	3565	37
H(2A)	2724	6112	4614	37
H(4A)	6506	9538	4180	34
H(5A)	5367	8931	3124	32
H(6A)	4585	6132	5332	55
H(6B)	3949	7193	5763	55
H(6C)	5787	7256	5920	55
H(7A)	7004	10113	5256	50
H(7B)	8057	9159	5150	50
H(7C)	7296	9370	5834	50
H(7D)	7901	8982	5571	50
H(7E)	6848	9936	5677	50
H(7F)	7609	9725	4992	50
H(10A)	5554	11976	1465	35
H(12A)	7710	9394	743	33
H(15A)	4206	10870	3016	53
H(15B)	2752	9356	2850	53
H(15C)	2292	10689	3004	53
H(16A)	1232	8493	1703	57
H(16B)	1323	9633	1265	57
H(16C)	483	9632	1945	57
H(17A)	4303	12578	2277	69
H(17B)	2316	12079	2249	69
H(17C)	3221	12047	1587	69
H(19A)	8316	13664	1413	118
H(19B)	6933	13398	833	118
H(19C)	8873	14084	720	118
H(20A)	6431	11563	-110	119
H(20B)	7526	10640	-175	119
H(20C)	8352	12234	-253	119

H(21A)	10114	12234	1300	102
H(21B)	10595	12858	643	102
H(21C)	9911	11220	621	102
H(23A)	8378	7743	882	64
H(23B)	6980	7212	281	64
H(23C)	7338	6119	648	64
H(24A)	5769	6863	2158	52
H(24B)	7664	7486	1978	52
H(24C)	6552	5869	1760	52
H(25A)	3376	6034	1098	54
H(25B)	4326	5034	871	54
H(25C)	4151	6049	405	54
H(27A)	7704	6558	4516	57
H(27B)	8912	5990	4101	57

Table 6. Crystal data and structure refinement for **16**.

Identification code	k05127a	
Empirical formula	C18 H30 F2 N P	
Formula weight	329.40	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	*Pnma*	
Unit cell dimensions	a = 19.0219(7) Å	α = 90°
	b = 15.6776(8) Å	β = 90°
	c = 6.1422(3) Å	γ = 90°
Volume	1831.71(15) Å^3	
Z	4	
Density (calculated)	1.194 Mg/m^3	
Absorption coefficient	0.166 mm^{-1}	
F(000)	712	
Crystal size	0.40 x 0.26 x 0.14 mm^3	
Theta range for data collection	3.37 to 27.48°	
Index ranges	$-22 \le h \le 23$, $-20 \le k \le 20$, $-7 \le l \le 7$	
Reflections collected	8784	
Independent reflections	2133 [R(int) = 0.0431]	
Completeness to theta = 27.48°	99.5%	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.978 and 0.839	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2133 / 0 / 125	
Goodness-of-fit on F^2	1.040	
Final R indices [$I>2\sigma(I)$]	R1 = 0.0472, wR2 = 0.1141	
R indices (all data)	R1 = 0.0713, wR2 = 0.1288	
Extinction coefficient	none	
Largest diff. peak and hole	0.289 and –0.293 e.Å^{-3}	

Table 7. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **16**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
P(1)	2059(1)	2500	3841(1)	34(1)
F(1)	2629(1)	1768(1)	4252(2)	49(1)
N(1)	2030(1)	2500	1176(3)	25(1)
C(1)	1404(1)	2500	-174(4)	23(1)
C(2)	1112(1)	1713(1)	-845(3)	23(1)
C(3)	503(1)	1743(1)	-2123(3)	25(1)
C(4)	186(1)	2500	-2769(4)	23(1)
C(5)	1409(1)	822(1)	-213(3)	25(1)
C(6)	1224(1)	623(1)	2169(3)	31(1)
C(7)	2209(1)	749(1)	-579(3)	33(1)
C(8)	1078(1)	102(1)	-1586(3)	35(1)
C(9)	-485(1)	2500	-4139(4)	27(1)
C(10)	-755(3)	3371(3)	-4712(11)	64(2)
C(11)	-1055(2)	2037(4)	-2888(11)	79(2)
C(12)	-344(3)	2029(4)	-6270(9)	81(2)

Table 8. Bond lengths [Å] and angles [°] for **16**.

P(1)-F(1)#1	1.5990(12)
P(1)-F(1)	1.5990(12)
P(1)-N(1)	1.637(2)
N(1)-C(1)	1.452(3)
C(1)-C(2)	1.4143(19)
C(1)-C(2)#1	1.4143(19)
C(2)-C(3)	1.401(2)
C(2)-C(5)	1.556(2)
C(3)-C(4)	1.389(2)
C(4)-C(3)#1	1.389(2)
C(4)-C(9)	1.529(3)
C(5)-C(6)	1.537(2)
C(5)-C(7)	1.541(2)
C(5)-C(8)	1.543(2)
C(9)-C(10)#1	1.501(4)
C(9)-C(10)	1.501(4)
C(9)-C(11)	1.515(5)
C(9)-C(11)#1	1.515(5)
C(9)-C(12)	1.526(5)
C(9)-C(12)#1	1.526(5)
F(1)#1-P(1)-F(1)	91.72(9)
F(1)#1-P(1)-N(1)	100.40(7)
F(1)-P(1)-N(1)	100.40(7)
C(1)-N(1)-P(1)	126.72(16)
C(2)-C(1)-C(2)#1	121.5(2)
C(2)-C(1)-N(1)	119.22(10)
C(2)#1-C(1)-N(1)	119.22(10)
C(3)-C(2)-C(1)	117.28(15)
C(3)-C(2)-C(5)	118.07(14)
C(1)-C(2)-C(5)	124.63(15)
C(4)-C(3)-C(2)	123.20(16)
C(3)#1-C(4)-C(3)	117.5(2)
C(3)#1-C(4)-C(9)	121.27(10)

C(3)-C(4)-C(9)	121.27(10)
C(6)-C(5)-C(7)	110.44(14)
C(6)-C(5)-C(8)	106.17(14)
C(7)-C(5)-C(8)	105.65(14)
C(6)-C(5)-C(2)	109.66(14)
C(7)-C(5)-C(2)	112.87(14)
C(8)-C(5)-C(2)	111.80(14)
C(10)#1-C(9)-C(10)	131.0(4)
C(10)#1-C(9)-C(11)	55.8(4)
C(10)-C(9)-C(11)	108.0(4)
C(10)#1-C(9)-C(11)#1	108.0(4)
C(10)-C(9)-C(11)#1	55.8(4)
C(11)-C(9)-C(11)#1	57.3(6)
C(10)#1-C(9)-C(12)	54.5(3)
C(10)-C(9)-C(12)	107.4(4)
C(11)-C(9)-C(12)	109.2(4)
C(11)#1-C(9)-C(12)	142.4(3)
C(10)#1-C(9)-C(12)#1	107.4(4)
C(10)-C(9)-C(12)#1	54.5(3)
C(11)-C(9)-C(12)#1	142.4(3)
C(11)#1-C(9)-C(12)#1	109.2(4)
C(12)-C(9)-C(12)#1	57.9(6)
C(10)#1-C(9)-C(4)	114.50(19)
C(10)-C(9)-C(4)	114.50(19)
C(11)-C(9)-C(4)	108.6(2)
C(11)#1-C(9)-C(4)	108.6(2)
C(12)-C(9)-C(4)	109.0(2)
C(12)#1-C(9)-C(4)	109.0(2)

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z

Table 9. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **16**. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	40(1)	34(1)	28(1)	0	-8(1)	0
F(1)	58(1)	40(1)	48(1)	3(1)	-25(1)	8(1)
N(1)	22(1)	25(1)	27(1)	0	0(1)	0
C(1)	23(1)	24(1)	21(1)	0	-1(1)	0
C(2)	25(1)	21(1)	23(1)	1(1)	3(1)	1(1)
C(3)	26(1)	20(1)	28(1)	-1(1)	0(1)	-3(1)
C(4)	22(1)	23(1)	25(1)	0	0(1)	0
C(5)	29(1)	20(1)	27(1)	2(1)	-2(1)	3(1)
C(6)	35(1)	27(1)	32(1)	7(1)	1(1)	0(1)
C(7)	34(1)	25(1)	39(1)	4(1)	4(1)	6(1)
C(8)	44(1)	20(1)	41(1)	-1(1)	-7(1)	2(1)
C(9)	25(1)	23(1)	34(1)	0	-3(1)	0
C(10)	50(3)	34(2)	107(5)	12(3)	-47(3)	0(2)
C(11)	31(2)	107(4)	98(4)	58(4)	-21(3)	-23(3)
C(12)	61(3)	124(5)	57(3)	-45(3)	-33(3)	39(3)

Table 10. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for **16**.

	x	y	z	U(eq)
H(1A)	2437	2500	492	29
H(3A)	296	1220	-2569	30
H(6A)	1419	1067	3115	47
H(6B)	1424	69	2575	47
H(6C)	712	605	2337	47
H(7A)	2457	1058	576	49
H(7B)	2330	996	-1995	49
H(7C)	2347	147	-549	49
H(8A)	1298	-443	-1209	52
H(8B)	1152	221	-3136	52
H(8C)	572	72	-1286	52
H(10A)	-852	3690	-3374	96
H(10B)	-1188	3316	-5563	96
H(10C)	-401	3675	-5572	96
H(11A)	-1158	2347	-1540	118
H(11B)	-895	1459	-2535	118
H(11C)	-1481	2005	-3781	118
H(12A)	-772	2026	-7158	121
H(12B)	-203	1440	-5953	121
H(12C)	34	2317	-7066	121

Table 11. Crystal data and structure refinement for **18**.

Identification code	k0638	
Empirical formula	C18 H29 Cl N O2 P	
Formula weight	357.84	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.1257(3) Å	α = 90°
	b = 13.9396(7) Å	β = 95.608(3)°
	c = 15.6039(6) Å	γ = 90°
Volume	1975.45(14) Å^3	
Z	4	
Density (calculated)	1.203 Mg/m^3	
Absorption coefficient	0.283 mm^{-1}	
F(000)	768	
Crystal size	0.34 x 0.30 x 0.18 mm^3	
Theta range for data collection	2.68 to 27.48°	
Index ranges	$-11 \le h \le 11$, $-16 \le k \le 18$, $-20 \le l \le 20$	
Reflections collected	13533	
Independent reflections	4506 [R(int) = 0.0802]	
Completeness to theta = 27.48°	99.4%	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.958 and 0.808	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4506 / 0 / 217	
Goodness-of-fit on F^2	1.053	
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0575, wR2 = 0.1372	
R indices (all data)	R1 = 0.1084, wR2 = 0.1615	
Largest diff. peak and hole	0.433 and –0.613 e. Å^{-3}	

Table 12. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **18**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	4357(1)	3813(1)	2201(1)	51(1)
P(1)	3987(1)	2508(1)	2677(1)	36(1)
O(1)	4291(2)	1795(1)	1921(1)	33(1)
O(2)	2530(2)	2469(2)	2996(1)	41(1)
N(1)	5505(2)	2319(2)	3344(1)	35(1)
C(1)	5678(2)	1268(2)	2133(2)	29(1)
C(2)	6675(3)	1419(2)	1442(2)	30(1)
C(3)	8134(3)	1518(2)	1648(2)	28(1)
C(4)	8725(3)	1507(2)	2563(2)	29(1)
C(5)	7909(2)	1570(2)	3234(2)	28(1)
C(6)	6327(3)	1731(2)	2967(2)	28(1)
C(7)	5226(3)	183(2)	2234(2)	36(1)
C(8)	4396(3)	-147(2)	1385(2)	47(1)
C(9)	6589(3)	-457(2)	2433(2)	40(1)
C(10)	4216(3)	78(2)	2961(2)	46(1)
C(11)	9226(3)	1618(2)	970(2)	33(1)
C(12)	10018(3)	2581(2)	1109(2)	49(1)
C(13)	10363(3)	805(2)	1079(2)	47(1)
C(14)	8476(3)	1574(2)	57(2)	45(1)
C(15)	8500(3)	1593(2)	4179(2)	32(1)
C(16)	10095(3)	1221(2)	4303(2)	43(1)
C(17)	7554(3)	945(2)	4701(2)	40(1)
C(18)	8482(3)	2627(2)	4530(2)	41(1)

Table 13. Bond lengths [Å] and angles [°] for **18**.

Cl(1)-P(1)	2.0064(11)
P(1)-O(2)	1.4660(18)
P(1)-O(1)	1.5872(17)
P(1)-N(1)	1.670(2)
O(1)-C(1)	1.473(3)
N(1)-C(6)	1.291(3)
C(1)-C(2)	1.492(3)
C(1)-C(6)	1.520(3)
C(1)-C(7)	1.580(4)
C(2)-C(3)	1.346(3)
C(3)-C(4)	1.477(3)
C(3)-C(11)	1.528(3)
C(4)-C(5)	1.346(3)
C(5)-C(6)	1.480(3)
C(5)-C(15)	1.520(3)
C(7)-C(8)	1.531(4)
C(7)-C(9)	1.537(4)
C(7)-C(10)	1.538(4)
C(11)-C(14)	1.521(3)
C(11)-C(12)	1.530(4)
C(11)-C(13)	1.535(4)
C(15)-C(17)	1.536(4)
C(15)-C(16)	1.539(3)
C(15)-C(18)	1.543(4)
O(2)-P(1)-O(1)	117.32(11)
O(2)-P(1)-N(1)	120.44(11)
O(1)-P(1)-N(1)	99.78(10)
O(2)-P(1)-Cl(1)	110.54(9)
O(1)-P(1)-Cl(1)	104.18(8)
N(1)-P(1)-Cl(1)	102.41(9)
C(1)-O(1)-P(1)	110.74(14)
C(6)-N(1)-P(1)	107.43(17)
O(1)-C(1)-C(2)	109.79(19)

O(1)-C(1)-C(6)	103.59(18)
C(2)-C(1)-C(6)	110.16(19)
O(1)-C(1)-C(7)	105.86(18)
C(2)-C(1)-C(7)	113.0(2)
C(6)-C(1)-C(7)	113.9(2)
C(3)-C(2)-C(1)	120.1(2)
C(2)-C(3)-C(4)	119.3(2)
C(2)-C(3)-C(11)	122.8(2)
C(4)-C(3)-C(11)	117.9(2)
C(5)-C(4)-C(3)	125.0(2)
C(4)-C(5)-C(6)	112.9(2)
C(4)-C(5)-C(15)	125.8(2)
C(6)-C(5)-C(15)	120.9(2)
N(1)-C(6)-C(5)	124.2(2)
N(1)-C(6)-C(1)	117.7(2)
C(5)-C(6)-C(1)	117.7(2)
C(8)-C(7)-C(9)	108.8(2)
C(8)-C(7)-C(10)	109.1(2)
C(9)-C(7)-C(10)	109.1(2)
C(8)-C(7)-C(1)	108.3(2)
C(9)-C(7)-C(1)	111.2(2)
C(10)-C(7)-C(1)	110.2(2)
C(14)-C(11)-C(3)	112.4(2)
C(14)-C(11)-C(12)	109.3(2)
C(3)-C(11)-C(12)	108.3(2)
C(14)-C(11)-C(13)	108.2(2)
C(3)-C(11)-C(13)	109.6(2)
C(12)-C(11)-C(13)	109.1(2)
C(5)-C(15)-C(17)	109.8(2)
C(5)-C(15)-C(16)	110.9(2)
C(17)-C(15)-C(16)	108.0(2)
C(5)-C(15)-C(18)	110.4(2)
C(17)-C(15)-C(18)	109.5(2)
C(16)-C(15)-C(18)	108.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 14. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **18**. The anisotropic displacement factor exponent takes the form: $-2p^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	48(1)	42(1)	60(1)	-1(1)	-4(1)	9(1)
P(1)	27(1)	47(1)	33(1)	-7(1)	1(1)	7(1)
O(1)	25(1)	42(1)	29(1)	-6(1)	-2(1)	8(1)
O(2)	21(1)	66(1)	37(1)	-8(1)	7(1)	5(1)
N(1)	28(1)	47(1)	31(1)	-6(1)	5(1)	5(1)
C(1)	20(1)	37(2)	28(1)	-1(1)	0(1)	4(1)
C(2)	31(1)	36(1)	23(1)	0(1)	2(1)	5(1)
C(3)	28(1)	31(1)	26(1)	0(1)	3(1)	3(1)
C(4)	24(1)	35(1)	29(1)	0(1)	1(1)	2(1)
C(5)	25(1)	31(1)	28(1)	-1(1)	1(1)	-1(1)
C(6)	26(1)	33(1)	26(1)	3(1)	3(1)	0(1)
C(7)	30(1)	37(2)	41(2)	-5(1)	2(1)	-2(1)
C(8)	37(2)	44(2)	57(2)	-14(2)	-6(1)	0(1)
C(9)	36(1)	34(2)	50(2)	3(1)	2(1)	2(1)
C(10)	38(2)	45(2)	58(2)	1(2)	15(1)	-7(1)
C(11)	32(1)	39(2)	30(1)	2(1)	7(1)	3(1)
C(12)	51(2)	56(2)	41(2)	2(2)	17(1)	-8(2)
C(13)	38(2)	67(2)	37(2)	2(2)	10(1)	16(1)
C(14)	41(2)	67(2)	28(1)	3(1)	9(1)	7(1)
C(15)	26(1)	42(2)	27(1)	2(1)	2(1)	0(1)
C(16)	33(1)	69(2)	27(1)	4(1)	-2(1)	4(1)
C(17)	37(1)	52(2)	30(1)	7(1)	3(1)	1(1)
C(18)	46(2)	49(2)	29(1)	-3(1)	-1(1)	-7(1)

Table 15. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for **18**.

	x	y	z	U(eq)
H(2A)	6279	1446	856	36
H(4A)	9762	1452	2688	35
H(8A)	4018	-798	1454	70
H(8B)	5065	-145	931	70
H(8C)	3572	289	1228	70
H(9A)	6282	-1130	2451	60
H(9B)	7096	-276	2992	60
H(9C)	7257	-373	1984	60
H(10A)	3884	-589	2989	69
H(10B)	3360	500	2847	69
H(10C)	4760	255	3511	69
H(12A)	9310	3105	992	73
H(12B)	10792	2631	718	73
H(12C)	10459	2625	1706	73
H(13A)	9858	185	1011	70
H(13B)	10899	842	1654	70
H(13C)	11059	868	642	70
H(14A)	7748	2091	-27	67
H(14B)	7983	953	-37	67
H(14C)	9214	1651	-354	67
H(16A)	10139	577	4057	65
H(16B)	10428	1198	4919	65
H(16C)	10734	1654	4013	65
H(17A)	6531	1169	4632	59
H(17B)	7929	971	5311	59
H(17C)	7600	284	4494	59
H(18A)	7476	2880	4449	62
H(18B)	9131	3031	4219	62
H(18C)	8826	2626	5145	62