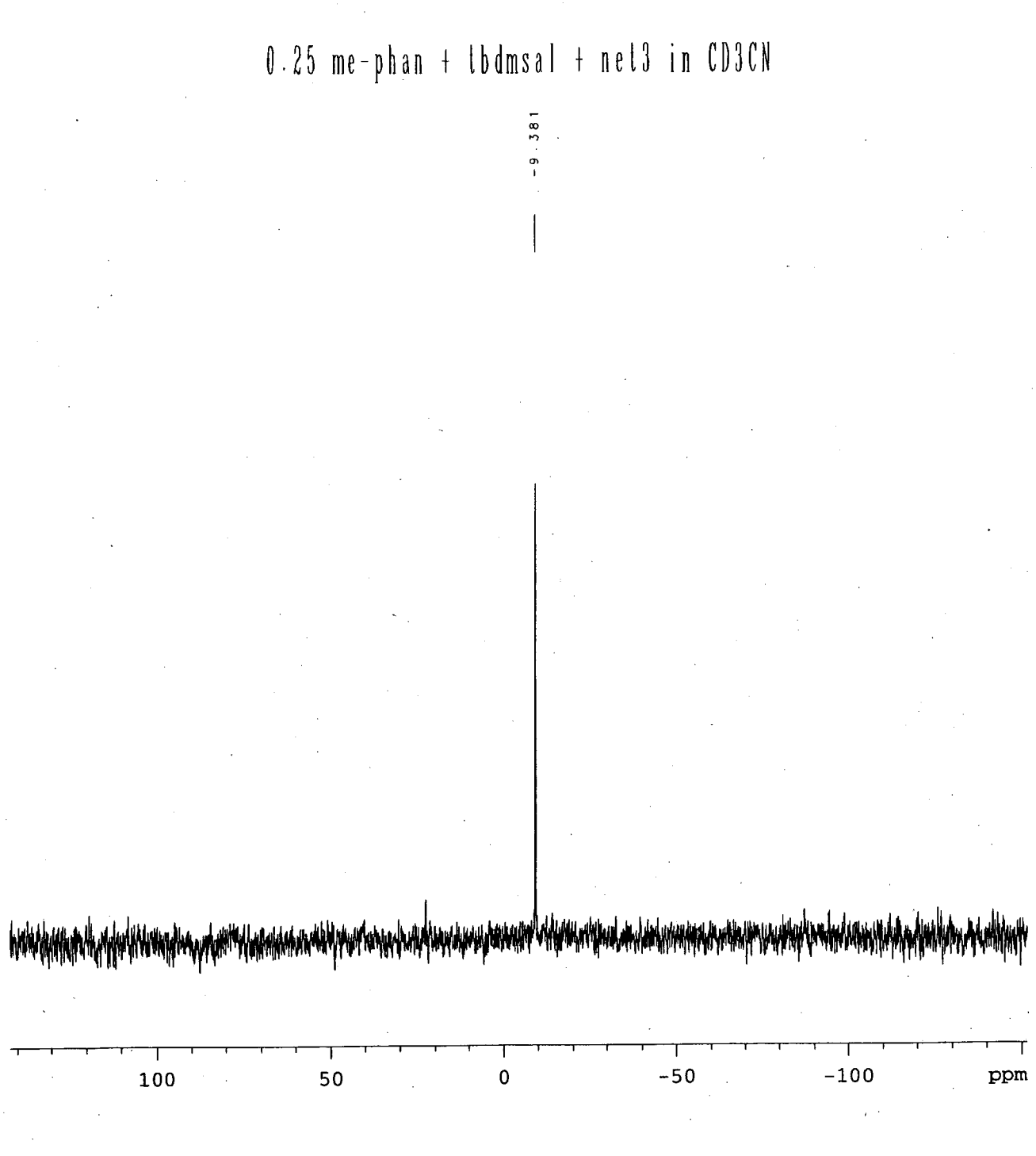
Inorganic Chemistry
including bioinorganic chemistry
Inorg. Chem., 1998, 37(20), 5189-5197, DOI:10.1021/ic9714652

H(9AB)	9148(18)	7129(8)	6475(8)	0.080
H(9AC)	7341(18)	7253(8)	6021(8)	0.080
H(1BA)	-2172(18)	7065(7)	-1607(8)	0.080
H(1BB)	-299(18)	6762(7)	-1836(8)	0.080
H(2BA)	175(17)	8163(7)	-1901(7)	0.080
H(2BB)	-1131(17)	8352(7)	-1190(7)	0.080
H(3BA)	576(14)	9278(6)	685(7)	0.080
H(3BB)	-798(14)	9170(6)	-35(7)	0.080
H(4BA)	1690(15)	9246(6)	-769(7)	0.080
H(4BB)	2789(15)	8801(6)	-96(7)	0.080
H(5BA)	4656(16)	6840(8)	-590(9)	0.080
H(5BB)	4629(16)	7749(8)	-201(9)	0.080
H(6BA)	3504(16)	8006(8)	-1432(8)	0.080
H(6BB)	2529(16)	7126(8)	-1502(8)	0.080
H(7BA)	-1948(17)	5686(7)	-1119(8)	0.080
H(7BB)	-192(17)	5487(7)	-652(8)	0.080
H(7BC)	-1886(17)	5699(7)	-155(8)	0.080
H(8BA)	-1995(17)	8702(8)	1220(8)	0.080
H(8BB)	-2864(17)	7912(8)	759(8)	0.080
H(8BC)	-1628(17)	7794(8)	1525(8)	0.080
H(9BA)	4708(18)	6597(8)	800(10)	0.080
H(9BB)	3325(18)	7014(8)	1378(10)	0.080

H(9BC)	2955(18)	6105(8)	1004(10)	0.080

Equivalent isotropic U defined as one third of the trace of the orthogonalized Uij tensor.



Supplementary Material

for **23**(Cl)

Unusual Phosphoryl Donor Properties of $O=P(MeNCH_2CH_2)_3N$

Xiao-Dong Liu and John G. Verkade*

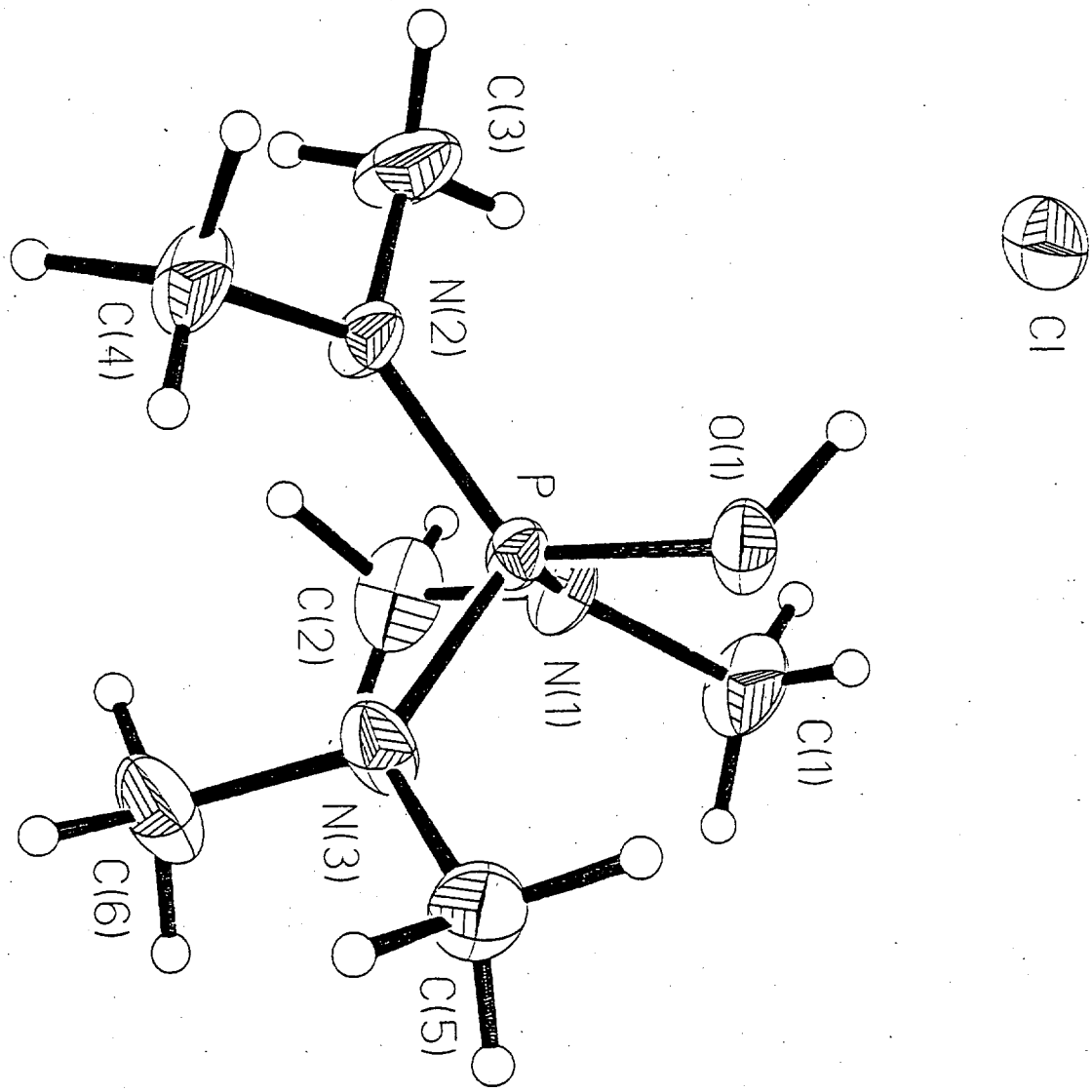


Fig 3

STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_6\ H_{19}\ N_3\ O\ P\ Cl$
Color, Habit	clear , square chip
Crystal Size (mm)	0.3x 0.2x 0.2
Crystal System	Monoclinic
Space Group	$P2_1/n$
Unit Cell Dimensions	a = 8.267(2) Å
	b = 11.700(2) Å
	c = 12.216(2) Å
	α = 90 °
	β = 107.63(3) °
	γ = 90 °
Volume	1126.1(4) $Å^3$
Z	4
Formula Weight	215.66
Density(calc.)	1.272 Mg/m^3
Absorption Coefficient	4.083 mm^{-1}
F(000)	464

STRUCTURE DETERMINATION SUMMARY

Data Collection

Diffractometer Used	Siemens P4RA
Radiation	CuKα (λ =1.54178Å)
Temperature (K)	203(2)
Monochromator	graphite
θ Range	5.36to 56.76°
Scan Type	ω
Standard Reflections	3 measured every 97 reflections
Index Ranges	-8$\leq h \leq$ 8 -1$\leq k \leq$ 12 -13$\leq l \leq$ 12
Reflections Collected	3191
Independent Reflections	1502(Rint = 0.0500)
Observed Reflections	1407(I $\geq 2\sigma$(I))
Max./Min. Transmission	0.808/ 0.488
Absorption Correction	Semi-empirical

STRUCTURE DETERMINATION SUMMARY

Solution and Refinement

System Used	SHELXL-93 (Sheldrick, 1993)
Solution	direct
Refinement Method	Full-matrixleast-squares on F^2
Extinction Correction	0.0122(9)
Extinction expression	$Fc^* = kFc[1 + 0.001xFc^2\lambda^3/sin(2\theta)]^{-1/4}$
Hydrogen Atoms	fixed isotropic
Weighting Scheme	w=calc w=$1/[\sigma^2(Fo^2) + (0.0478P)^2$ +1.0714P] where P=$(Fo^2 + 2Fc^2)/3$
Parameters Refined	114
Final R Indices [I$\geq 2\sigma(I)$]	R1 = 0.0416, wR2 =0.1082
R Indices (all data)	R1 = 0.0441, wR2 = 0.1104
GooF, Observed and All Data	1.127, 1.111
Largest and Mean Δ/σ	0.000, 0.000
Largest Difference Peak	0.341e/Å^{-3}
Largest Difference Hole	-0.393e/Å^{-3}

R1 = $\Sigma||Fo| - |Fc||/\Sigma|Fo|$
wR2 = $[\Sigma[w(Fo^2 - Fc^2)^2]/\Sigma[w(Fo^2)^2]]^{0.5}$
where w = $1/[\sigma^2(Fo^2) + (a * P)^2 + b * P + d + e * sin\,\theta]$
GooF = $[\Sigma[w(Fo^2 - Fc^2)^2]/(n - p)]^{0.5}$

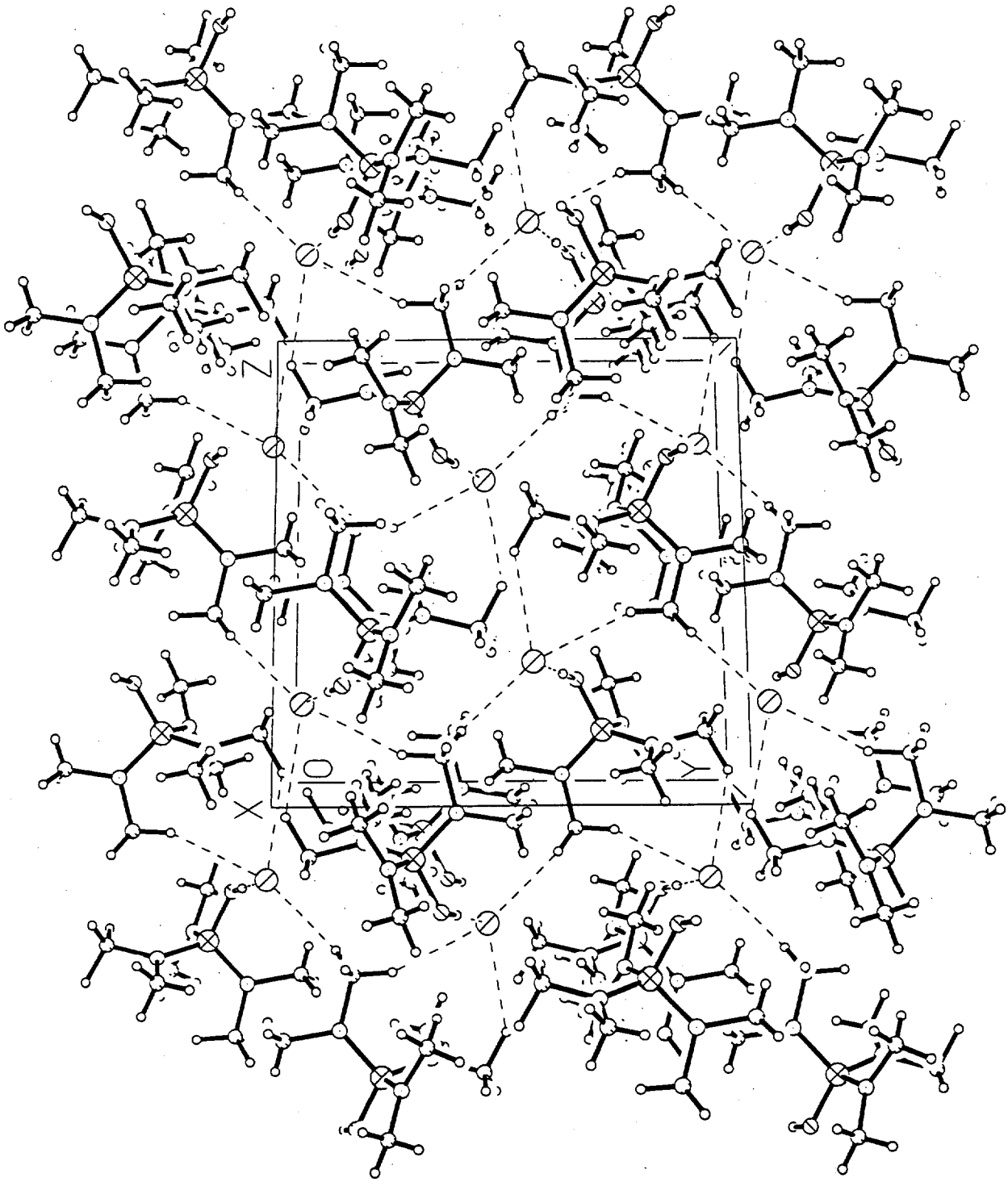

Z
O
X
Y

Table 1. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for Str1227.

Atom	x	y	z	U(eq)
P	2329(1)	2101(1)	3730(1)	18(1)
Cl	-1708(1)	533(1)	2070(1)	32(1)
O(1)	1559(3)	1512(2)	2559(2)	27(1)
N(1)	2366(3)	1163(2)	4718(2)	26(1)
N(2)	1322(3)	3181(2)	4025(2)	23(1)
N(3)	4133(3)	2579(2)	3659(2)	29(1)
C(1)	2383(4)	-75(3)	4512(3)	42(1)
C(3)	-158(4)	3046(3)	4442(3)	44(1)
C(2)	2956(5)	1459(3)	5945(3)	42(1)
C(4)	1579(4)	4368(2)	3738(3)	36(1)
C(5)	4859(4)	2316(3)	2742(3)	37(1)
C(6)	5303(4)	3155(3)	4658(3)	41(1)

Equivalent isotropic U defined as one third of the trace of the orthogonalized Uij tensor.

Table 2. Bond lengths (Å) Str1227.

P-O(1)	1.541(2)
P-N(2)	1.613(2)
P-N(3)	1.619(2)
P-N(1)	1.624(2)
O(1)-H(1D)	1.00(5)
N(1)-C(2)	1.470(4)
N(1)-C(1)	1.472(4)
N(2)-C(4)	1.464(4)
N(2)-C(3)	1.470(4)
N(3)-C(5)	1.456(4)
N(3)-C(6)	1.470(4)
C(1)-H(1A)	1.039(4)
C(1)-H(1B)	0.972(3)
C(1)-H(1C)	0.849(4)
C(3)-H(3A)	0.961(3)
C(3)-H(3B)	0.982(4)
C(3)-H(3C)	0.889(3)
C(2)-H(2A)	1.106(4)
C(2)-H(2B)	1.091(4)
C(2)-H(2C)	1.059(3)
C(4)-H(4A)	0.988(3)

C(4)-H(4B)	0.969(4)
C(4)-H(4C)	1.178(3)
C(5)-H(5A)	0.983(3)
C(5)-H(5B)	0.959(3)
C(5)-H(5C)	1.066(3)
C(6)-H(6A)	1.024(4)
C(6)-H(6B)	0.973(3)
C(6)-H(6C)	1.007(3)

Symmetry transformations used to generate equivalent atoms:

Table 3. Bond angles (°) for Str1227.

O(1)-P-N(2)	117.48(12)
O(1)-P-N(3)	103.52(12)
N(2)-P-N(3)	106.67(13)
O(1)-P-N(1)	107.29(12)
N(2)-P-N(1)	104.95(13)
N(3)-P-N(1)	117.55(13)
P-O(1)-H(1D)	117(3)
C(2)-N(1)-C(1)	112.8(3)
C(2)-N(1)-P	121.6(2)
C(1)-N(1)-P	122.5(2)
C(4)-N(2)-C(3)	112.4(3)
C(4)-N(2)-P	124.6(2)
C(3)-N(2)-P	122.3(2)
C(5)-N(3)-C(6)	114.5(2)
C(5)-N(3)-P	124.7(2)
C(6)-N(3)-P	120.0(2)
N(1)-C(1)-H(1A)	109.3(3)
N(1)-C(1)-H(1B)	108.4(3)
H(1A)-C(1)-H(1B)	119.0(3)
N(1)-C(1)-H(1C)	113.6(3)
H(1A)-C(1)-H(1C)	93.4(3)

H(1B)-C(1)-H(1C)	112.6(4)
N(2)-C(3)-H(3A)	114.1(3)
N(2)-C(3)-H(3B)	110.6(3)
H(3A)-C(3)-H(3B)	103.0(3)
N(2)-C(3)-H(3C)	109.0(3)
H(3A)-C(3)-H(3C)	116.4(4)
H(3B)-C(3)-H(3C)	102.9(4)
N(1)-C(2)-H(2A)	103.9(3)
N(1)-C(2)-H(2B)	112.3(3)
H(2A)-C(2)-H(2B)	122.4(3)
N(1)-C(2)-H(2C)	104.7(3)
H(2A)-C(2)-H(2C)	109.8(3)
H(2B)-C(2)-H(2C)	102.5(3)
N(2)-C(4)-H(4A)	105.6(3)
N(2)-C(4)-H(4B)	108.8(3)
H(4A)-C(4)-H(4B)	117.1(3)
N(2)-C(4)-H(4C)	105.0(3)
H(4A)-C(4)-H(4C)	111.6(3)
H(4B)-C(4)-H(4C)	108.0(3)
N(3)-C(5)-H(5A)	110.1(3)
N(3)-C(5)-H(5B)	108.1(3)
H(5A)-C(5)-H(5B)	98.0(3)

N(3)-C(5)-H(5C)	115.9(3)
H(5A)-C(5)-H(5C)	111.7(3)
H(5B)-C(5)-H(5C)	111.6(3)
N(3)-C(6)-H(6A)	110.4(3)
N(3)-C(6)-H(6B)	108.4(3)
H(6A)-C(6)-H(6B)	110.9(3)
N(3)-C(6)-H(6C)	111.8(3)
H(6A)-C(6)-H(6C)	105.7(3)
H(6B)-C(6)-H(6C)	109.7(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for Str1227.

Atom	U11	U22	U33	U23	U13	U12
P	21(1)	12(1)	23(1)	-1(1)	10(1)	-3(1)
Cl	30(1)	24(1)	41(1)	1(1)	10(1)	-9(1)
O(1)	31(1)	27(1)	26(1)	-10(1)	12(1)	-9(1)
N(1)	41(1)	10(1)	30(1)	3(1)	14(1)	-2(1)
N(2)	28(1)	13(1)	34(1)	1(1)	16(1)	0(1)
N(3)	25(1)	38(2)	27(1)	-9(1)	15(1)	-15(1)
C(1)	55(2)	11(2)	61(2)	3(2)	20(2)	0(2)
C(3)	37(2)	31(2)	75(3)	0(2)	36(2)	6(2)
C(2)	65(2)	32(2)	29(2)	6(2)	17(2)	-4(2)
C(4)	51(2)	12(2)	42(2)	3(1)	11(2)	-1(1)
C(5)	36(2)	39(2)	47(2)	-11(2)	29(2)	-10(2)
C(6)	31(2)	54(2)	38(2)	-11(2)	13(1)	-21(2)

The anisotropic displacement factor exponent takes the form: $-2\pi^2$ ($h^2a^{*2}U11$+ ... + 2hka*b*U12).

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for Str1227.

Atom	x	y	z	U(eq)
H(1D)	388(60)	1199(40)	2403(40)	0.09(2)
H(1A)	1478	-470	4817	0.050
H(1B)	3551	-341	4784	0.050
H(1C)	1890	-265	3820	0.050
H(2A)	4349	1353	6184	0.050
H(2B)	2191	1056	6425	0.050
H(2C)	2652	2335	5975	0.050
H(3A)	-1160	3433	3984	0.050
H(3B)	54	3404	5200	0.050
H(3C)	-275	2312	4588	0.050
H(4A)	477	4623	3214	0.050
H(4B)	2551	4403	3452	0.050
H(4C)	1929	4861	4620	0.050
H(5A)	5836	1801	3027	0.050
H(5B)	5464	2977	2615	0.050
H(5C)	3991	2011	1962	0.050
H(6A)	6307	2632	5046	0.050
H(6B)	4678	3358	5188	0.050
H(6C)	5815	3862	4428	0.050

Equivalent isotropic U defined as one third of the trace of the orthogonalized Uij tensor.