X-ray Crystal Structure of [1,2-Bis(diphenylphosphino)ethane]bis(3,5-dimethylpyrazole-N)platinum(II) Tetrafluoroborate Dichloromethane Solvate, $[(Ph_2PCH_2CH_2PPh_2)Pt(3,5\text{-}Me_2pzH)_2][BF_4]_2\cdot CH_2Cl_2$

BRUNA BOVIO*

Dipartimento di Chimica Generale dell' Università, Viale Taramelli 12, 27100 Pavia, Italy

GUIDO BANDITELLI and ANNA LAURA BANDINI

Centro C.N.R., Dipartimento di Chimica Inorganica e Metallorganica dell' Università, Via Venezian 21, 20133 Milan, Italy

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Abstract

The crystal structure of the compound $[(Ph_2PCH_2CH_2PPh_2)Pt(3,5\text{-}Me_2pzH)_2][BF_4]_2\cdot CH_2Cl_2$ has shown that the ligands around the Pt atom are approximately in a square planar coordination, whereas the pyrazole rings point roughly in the same direction, away from the coordination plane. This unusual conformation is probably due to the hydrogen bonds with fluorine atoms of one BF_4^- anion. Bond distances and angles are compared with those in other pyrazole complexes.

Introduction

A series of $(LL)Pt(pz\text{-}N)_2$ compounds were prepared some years ago by Minghetti *et al.* [1], where LL indicates a bidentate ligand and pz is a pyrazole. Tetrafluoroboric acid often protonates both pyrazolato groups, affording $[(LL)Pt(pzH\text{-}N)_2]^{++}$ cations whose reactivity *e.g.* with BH_4^- [2] and spectroscopic properties [3] were investigated.

Owing to our interest towards these cations and in view also of the general interest towards platinum(II) chemistry [4], especially in the field of antitumoral agents, it was deemed useful to carry out an X-ray crystal structure determination of the title compound, an useful intermediate [2]. In this paper the structural features are reported and the relevance of hydrogen bonds in determining the unusual conformation of the complex is underlined.

Results and Discussion

The structure of the title compound with the numbering scheme, drawn with the program ORTEP [5], is given in Fig. 1.

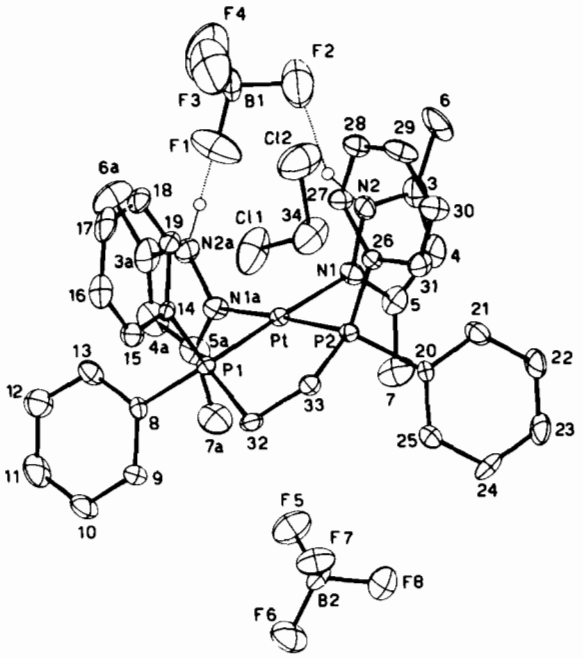

Fig. 1. ORTEP plot and numbering scheme of atoms, down *c* axis of cell. Thermal ellipsoids enclose 25% of the electron density. Carbon atoms are indicated only by numbers. Hydrogen atoms are omitted for clarity, except those involved in hydrogen-bonding.

The bond distances and angles are listed in Table I, with selected average values given in Table II. The complex exhibits no crystallographic symmetry, though a virtual *Cs* symmetry with the mirror plane passing through Pt atom and P(1)–P(2), C(32)–C(33), N(1)–N(1*a*), N(2)–N(2*a*) midpoints can be surmised.

The least-squares planes of the conjecturally planar regions in the complex, the displacements of atoms from them, the $\chi^2 = \Sigma_i(\Delta/\sigma_\perp)_i^2$ values with the probability *P* that the regions are non-planar and the angles between them, are all given in Table III.

*Author to whom correspondence should be addressed.

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TABLE I. Interatomic Distances (Å) and Bond Angles (°) with E.s.d. s in Parentheses.

In the coordination sphere:			
Pt–P(1)	2.245(2)	N(1)–Pt–N(1*a*)	87.8(2)
Pt–P(2)	2.253(2)	P(1)–Pt–N(1)	177.1(1)
Pt–N(1)	2.107(5)	P(2)–Pt–N(1*a*)	176.7(1)
Pt–N(1*a*)	2.102(5)	P(1)–Pt–N(1*a*)	95.0(1)
P(1)–Pt–P(2)	84.87(6)	P(2)–Pt–N(1)	92.3(1)
In the bidentate phosphine ligand:			
P(1)–C(32)	1.807(7)	C(32)–C(33)–H(33–1)	112(4)
P(2)–C(33)	1.830(7)	C(32)–C(33)–H(33–2)	112(3)
C(32)–C(33)	1.535(10)	H(33–1)–C(33)–H(33–2)	103(5)
C(32)–H(32–1)	0.96(5)	P(1)–C(8)–C(9)	122.3(5)
C(32)–H(32–2)	1.02(5)	P(1)–C(8)–C(13)	118.1(5)
C(33)–H(33–1)	0.90(6)	C(9)–C(8)–C(13)	119.6(6)
C(33)–H(33–2)	0.92(5)	C(8)–C(9)–C(10)	121.8(7)
P(1)–C(8)	1.817(6)	C(8)–C(9)–H(9)	125(3)
C(8)–C(9)	1.359(9)	C(10)–C(9)–H(9)	113(3)
C(9)–H(9)	0.97(5)	C(9)–C(10)–C(11)	118.5(8)
C(9)–C(10)	1.378(11)	C(9)–C(10)–H(10)	114(3)
C(10)–H(10)	1.10(6)	C(11)–C(10)–H(10)	127(3)
C(10)–C(11)	1.340(13)	C(10)–C(11)–C(12)	121.5(9)
C(11)–H(11)	0.96(7)	C(10)–C(11)–H(11)	119(4)
C(11)–C(12)	1.378(14)	C(12)–C(11)–H(11)	120(4)
C(12)–H(12)	1.03(6)	C(11)–C(12)–C(13)	118.7(8)
C(12)–C(13)	1.396(12)	C(11)–C(12)–H(12)	118(4)
C(13)–H(13)	0.86(6)	C(13)–C(12)–H(12)	123(4)
C(13)–C(8)	1.351(10)	C(12)–C(13)–C(8)	119.9(7)
P(1)–C(14)	1.794(6)	C(12)–C(13)–H(13)	118(4)
C(14)–C(15)	1.376(9)	C(8)–C(13)–H(13)	122(4)
C(15)–H(15)	0.90(5)	P(1)–C(14)–C(15)	121.3(5)
C(15)–C(16)	1.387(11)	P(1)–C(14)–C(19)	120.7(5)
C(16)–H(16)	0.95(6)	C(15)–C(14)–C(19)	118.0(6)
C(16)–C(17)	1.367(14)	C(14)–C(15)–C(16)	120.4(7)
C(17)–H(17)	0.86(6)	C(14)–C(15)–H(15)	116(4)
C(17)–C(18)	1.368(12)	C(16)–C(15)–H(15)	123(4)
C(18)–H(18)	0.96(6)	C(15)–C(16)–C(17)	120.3(7)
C(18)–C(19)	1.377(10)	C(15)–C(16)–H(16)	120(4)
C(19)–H(19)	0.88(6)	C(17)–C(16)–H(16)	120(4)
C(19)–C(14)	1.396(9)	C(16)–C(17)–C(18)	120.5(8)
P(2)–C(20)	1.836(6)	C(16)–C(17)–H(17)	120(4)
C(20)–C(21)	1.381(10)	C(18)–C(17)–H(17)	118(4)
C(21)–H(21)	0.87(5)	C(17)–C(18)–C(19)	119.2(7)
C(21)–C(22)	1.402(11)	C(17)–C(18)–H(18)	118(4)
C(22)–H(22)	0.99(6)	C(19)–C(18)–H(18)	121(4)
C(22)–C(23)	1.354(13)	C(18)–C(19)–C(14)	121.5(6)
C(23)–H(23)	0.98(6)	C(18)–C(19)–H(19)	117(4)
C(23)–C(24)	1.354(12)	C(14)–C(19)–H(19)	121(4)
C(24)–H(24)	0.99(6)	P(2)–C(20)–C(21)	119.7(5)
C(24)–C(25)	1.409(11)	P(2)–C(20)–C(25)	120.7(5)
C(25)–H(25)	0.94(5)	C(21)–C(20)–C(25)	119.5(6)
C(25)–C(20)	1.357(9)	C(20)–C(21)–C(22)	119.5(6)
P(2)–C(26)	1.805(6)	C(20)–C(21)–H(21)	124(4)
C(26)–C(27)	1.386(9)	C(22)–C(21)–H(21)	116(4)
C(27)–H(27)	1.06(5)	C(21)–C(22)–C(23)	120.7(7)
C(27)–C(28)	1.386(10)	C(21)–C(22)–H(22)	120(3)
C(28)–H(28)	0.98(6)	C(23)–C(22)–H(22)	117(3)
C(28)–C(29)	1.353(12)	C(22)–C(23)–C(24)	119.9(8)
C(29)–H(29)	1.04(6)	C(22)–C(23)–H(23)	115(3)
C(29)–C(30)	1.380(11)	C(24)–C(23)–H(23)	125(3)

(continued on facing page)

TABLE I *(continued)*

C(30)–H(30)	0.95(6)	C(23)–C(24)–C(25)	120.3(7)
C(30)–C(31)	1.391(10)	C(23)–C(24)–H(24)	118(4)
C(31)–H(31)	0.95(5)	C(25)–C(24)–H(24)	121(4)
C(31)–C(26)	1.413(9)	C(24)–C(25)–C(20)	120.1(6)
Pt–P(1)–C(32)	105.9(2)	C(24)–C(25)–H(25)	114(3)
Pt–P(1)–C(8)	117.6(2)	C(20)–C(25)–H(25)	126(3)
Pt–P(1)–C(14)	114.1(2)	P(2)–C(26)–C(27)	121.3(5)
C(32)–P(1)–C(8)	109.0(3)	P(2)–C(26)–C(31)	118.2(5)
C(32)–P(1)–C(14)	105.6(3)	C(27)–C(26)–C(31)	120.4(6)
C(8)–P(1)–C(14)	104.0(3)	C(26)–C(27)–C(28)	119.8(6)
Pt–P(2)–C(33)	108.3(2)	C(26)–C(27)–H(27)	120(3)
Pt–P(2)–C(20)	112.7(2)	C(28)–C(27)–H(27)	119(3)
Pt–P(2)–C(26)	115.4(2)	C(27)–C(28)–C(29)	120.0(7)
C(33)–P(2)–C(20)	108.0(3)	C(27)–C(28)–H(28)	119(3)
C(33)–P(2)–C(26)	105.7(3)	C(29)–C(28)–H(28)	120(3)
C(20)–P(2)–C(26)	106.3(3)	C(28)–C(29)–C(30)	121.5(7)
P(1)–C(32)–C(33)	107.2(4)	C(28)–C(29)–H(29)	119(3)
P(1)–C(32)–H(32–1)	115(3)	C(30)–C(29)–H(29)	120(3)
P(1)–C(32)–H(32–2)	103(3)	C(29)–C(30)–C(31)	120.3(7)
C(33)–C(32)–H(32–1)	110(3)	C(29)–C(30)–H(30)	122(3)
C(33)–C(32)–H(32–2)	117(3)	C(31)–C(30)–H(30)	118(3)
H(32–1)–C(32)–H(32–2)	105(4)	C(30)–C(31)–C(26)	118.0(6)
P(2)–C(33)–C(32)	112.2(5)	C(30)–C(31)–H(31)	116(3)
P(2)–C(33)–H(33–1)	113(4)	C(26)–C(31)–H(31)	126(3)
P(2)–C(33)–H(33–2)	104(3)		
In the dimethylpyrazole ligands:			
N(1)–N(2)	1.356(8)	N(1*a*)–N(2*a*)	1.347(8)
N(2)–H(2)	1.09(7)	N(2*a*)–H(2*a*)	1.08(7)
N(2)–C(3)	1.367(10)	N(2*a*)–C(3*a*)	1.364(11)
C(3)–C(4)	1.376(12)	C(3*a*)–C(4*a*)	1.360(14)
C(4)–H(4)	1.13(6)	C(4*a*)–H(4*a*)	1.10(7)
C(4)–C(5)	1.383(11)	C(4*a*)–C(5*a*)	1.373(12)
C(5)–N(1)	1.341(9)	C(5*a*)–N(1*a*)	1.323(9)
C(3)–C(6)	1.473(14)	C(3*a*)–C(6*a*)	1.446(17)
C(6)–H(6–1)	1.08(7)	C(6*a*)–H(6*a*–1)	1.05(5)
C(6)–H(6–2)	1.14(7)	C(6*a*)–H(6*a*–2)	1.00(7)
C(6)–H(6–3)	1.10(6)	C(6*a*)–H(6*a*–3)	1.15(7)
C(5)–C(7)	1.478(14)	C(5*a*)–C(7*a*)	1.474(14)
C(7)–H(7–1)	1.06(7)	C(7*a*)–H(7*a*–1)	0.94(7)
C(7)–H(7–2)	0.98(7)	C(7*a*)–H(7*a*–2)	1.01(7)
C(7)–H(7–3)	1.06(6)	C(7*a*)–H(7*a*–3)	1.08(8)
Pt–N(1)–N(2)	122.9(4)	Pt–N(1*a*)–N(2*a*)	120.7(4)
Pt–N(1)–C(5)	129.9(5)	Pt–N(1*a*)–C(5*a*)	132.9(5)
C(5)–N(1)–N(2)	107.2(5)	C(5*a*)–N(1*a*)–N(2*a*)	106.5(6)
N(1)–N(2)–C(3)	111.3(6)	N(1*a*)–N(2*a*)–C(3*a*)	111.3(6)
N(1)–N(2)–H(2)	119(3)	N(1*a*)–N(2*a*)–H(2*a*)	123(4)
C(3)–N(2)–H(2)	126(3)	C(3*a*)–N(2*a*)–H(2*a*)	125(4)
N(2)–C(3)–C(4)	104.4(7)	N(2*a*)–C(3*a*)–C(4*a*)	104.7(8)
N(2)–C(3)–C(6)	121.1(8)	N(2*a*)–C(3*a*)–C(6*a*)	122.2(9)
C(4)–C(3)–C(6)	134.5(8)	C(4*a*)–C(3*a*)–C(6*a*)	133.0(9)
C(3)–C(6)–H(6–1)	111(4)	C(3*a*)–C(6*a*)–H(6*a*–1)	113(3)
C(3)–C(6)–H(6–2)	109(3)	C(3*a*)–C(6*a*)–H(6*a*–2)	111(4)
C(3)–C(6)–H(6–3)	106(4)	C(3*a*)–C(6*a*)–H(6*a*–3)	117(4)
H(6–1)–C(6)–H(6–2)	116(5)	H(6*a*–1)–C(6*a*)–H(6*a*–2)	106(5)
H(6–1)–C(6)–H(6–3)	103(5)	H(6*a*–1)–C(6*a*)–H(6*a*–3)	106(5)
H(6–2)–C(6)–H(6–3)	111(5)	H(6*a*–2)–C(6*a*)–H(6*a*–3)	103(6)
C(3)–C(4)–C(5)	109.1(7)	C(3*a*)–C(4*a*)–C(5*a*)	108.4(8)
C(3)–C(4)–H(4)	121(3)	C(3*a*)–C(4*a*)–H(4*a*)	126(4)

(continued overleaf)

TABLE I *(continued)*

C(5)–C(4)–H(4)	129(3)	C(5*a*)–C(4*a*)–H(4*a*)	126(4)
C(4)–C(5)–N(1)	108.0(7)	C(4*a*)–C(5*a*)–N(1*a*)	109.2(7)
C(4)–C(5)–C(7)	130.2(8)	C(4*a*)–C(5*a*)–C(7*a*)	128.1(8)
N(1)–C(5)–C(7)	121.9(7)	N(1*a*)–C(5*a*)–C(7*a*)	122.8(7)
C(5)–C(7)–H(7–1)	117(4)	C(5*a*)–C(7*a*)–H(7*a*–1)	113(4)
C(5)–C(7)–H(7–2)	112(5)	C(5*a*)–C(7*a*)–H(7*a*–2)	109(4)
C(5)–C(7)–H(7–3)	106(3)	C(5*a*)–C(7*a*)–H(7*a*–3)	106(5)
H(7–1)–C(7)–H(7–2)	107(6)	H(7*a*–1)–C(7*a*)–H(7*a*–2)	111(6)
H(7–1)–C(7)–H(7–3)	106(5)	H(7*a*–1)–C(7*a*)–H(7*a*–3)	105(6)
H(7–2)–C(7)–H(7–3)	107(6)	H(7*a*–2)–C(7*a*)–H(7*a*–3)	113(6)
In the tetrafluoroborate anions:			
B(1)–F(1)	1.310(14)	B(2)–F(5)	1.381(10)
B(1)–F(2)	1.347(11)	B(2)–F(6)	1.359(10)
B(1)–F(3)	1.288(16)	B(2)–F(7)	1.381(10)
B(1)–F(4)	1.268(16)	B(2)–F(8)	1.357(10)
F(1)–B(1)–F(2)	105.0(9)	F(5)–B(2)–F(6)	112.6(7)
F(1)–B(1)–F(3)	108.6(1.0)	F(5)–B(2)–F(7)	108.5(6)
F(1)–B(1)–F(4)	107.1(1.0)	F(5)–B(2)–F(8)	108.8(7)
F(2)–B(1)–F(3)	112.5(1.0)	F(6)–B(2)–F(7)	111.2(7)
F(2)–B(1)–F(4)	109.9(1.0)	F(6)–B(2)–F(8)	108.6(7)
F(3)–B(1)–F(4)	113.1(1.0)	F(7)–B(2)–F(8)	107.0(6)
In the dichloromethane:			
C(34)–Cl(1)	1.700(13)	Cl(1)–C(34)–H(34–1)	111(4)
C(34)–Cl(2)	1.720(14)	Cl(1)–C(34)–H(34–2)	108(4)
C(34)–H(34–1)	0.94(5)	Cl(2)–C(34)–H(34–1)	111(3)
C(34)–H(34–2)	1.03(7)	Cl(2)–C(34)–H(34–2)	103(4)
Cl(1)–C(34)–Cl(2)	112.1(7)	H(34–1)–C(34)–H(34–2)	110(5)

TABLE II. Selected Average Bond Distances (Å) and Angles (°) with Their Standard Errors.

	N	x_m	σ_m	σ'_m
Pt–P	2	2.249	0.004	0.001
Pt–N	2	2.104	0.002	0.003
P–CH_2	2	1.818	0.011	0.005
P–C	4	1.813	0.009	0.003
N(1)–N(2)	2	1.351	0.005	0.006
N(2)–C(3)	2	1.366	0.001	0.007
C(3)–C(4)	2	1.369	0.008	0.009
C(4)–C(5)	2	1.378	0.005	0.008
C(5)–N(1)	2	1.332	0.009	0.006
C(3)–C(6)	2	1.462	0.013	0.011
C(5)–C(7)	2	1.476	0.002	0.010
P–Pt–N (*trans*)	2	176.9	0.2	0.1
P–Pt–N (*cis*)	2	93.6	1.3	0.1
Pt–P–CH_2	2	107.1	1.2	0.2
Pt–P–C	4	114.9	1.0	0.1
CH_2–P–C	4	107.1	0.8	0.1
C–P–C	2	105.1	1.2	0.2
Pt–N(1)–N(2)	2	121.9	1.1	0.3
Pt–N(1)–C(5)	2	131.3	1.5	0.3
N(2)–N(1)–C(5)	2	106.8	0.4	0.4
N(1)–N(2)–C(3)	2	111.26	0.01	0.44
N(2)–C(3)–C(4)	2	104.5	0.1	0.5
C(3)–C(4)–C(5)	2	108.8	0.4	0.5
C(4)–C(5)–N(1)	2	108.5	0.6	0.5
N(2)–C(3)–C(6)	2	121.6	0.6	0.6
C(4)–C(3)–C(6)	2	133.8	0.7	0.6
C(4)–C(5)–C(7)	2	129.1	1.1	0.6
N(1)–C(5)–C(7)	2	122.3	0.4	0.5

All values were calculated with the formulae taken from ref. [6].

$$x_m = \sum_{i=1}^{N} (x_i/\sigma_i^2) \Big/ \sum_{i=1}^{N} (1/\sigma_i^2)$$

$$\sigma_m = \left(\sum_{i=1}^{N} ((x_i - x_m)^2/\sigma_i^2) \Big/ (N-1) \sum_{i=1}^{N} (1/\sigma_i^2) \right)^{1/2}$$

$$\sigma'_m = \left(\sum_{i=1}^{N} 1/\sigma_i^2 \right)^{-1/2}$$

TABLE III. Planarity of Selected Regions.

Plane	Equation[a], χ^2, P	Atoms	Displacements (Å)
I	$-0.8989\,X' - 0.2340\,Y - 0.3704\,Z' + 4.4609 = 0$	Pt*	−0.0004(2)
		P(1)*	0.0025(16)
	$\chi^2 = 616.09$ (n = 2)	P(2)*	0.0112(16)
	$P > 99.0\%$	N(1)*	0.0242(47)
		N(1*a*)*	0.1088(47)
		C(32)	0.9115(61)
		C(33)	0.3142(68)
		N(2)	−1.1050(63)
		C(5)	1.0632(78)
		N(2*a*)	−0.9898(62)
		C(5*a*)	1.1008(78)
II	$0.1522\,X' + 0.6463\,Y - 0.7477\,Z' + 0.2876 = 0$	N(1)*	0.0067(48)
		N(2)*	−0.0085(63)
	$\chi^2 = 7.73$ (n = 2)	C(3)*	0.0035(77)
	$P = 97.8\%$	C(4)*	0.0067(76)
		C(5)*	0.0121(70)
		Pt	−0.0365(2)
		C(6)	0.0129(105)
		C(7)	−0.0408(107)
		H(2)	0.303(67)
		H(4)	0.060(56)
III	$0.2199\,X' - 0.7739\,Y - 0.5939\,Z' + 2.5576 = 0$	N(1*a*)*	0.0019(49)
		N(2*a*)*	−0.0019(79)
	$\chi^2 = 1.34$ (n = 2)	C(3*a*)*	0.0020(81)
	$P = 48.7\%$	C(4*a*)*	0.0063(89)
		C(5*a*)*	−0.0057(76)
		Pt	−0.0123(2)
		C(6*a*)	0.0686(143)
		C(7*a*)	−0.0022(106)
		H(2*a*)	0.129(86)
		H(4*a*)	0.070(67)

Angles (°) between the planes: I/II 90.6 I/III 78.3 II/III 91.3

[a]Transformation matrix from monoclinic X, Y, Z to orthogonal X', Y, Z' coordinates:

$$\begin{pmatrix} 1 & 0 & -\cos\beta^* \\ 0 & 1 & 0 \\ 0 & 0 & \sin\beta^* \end{pmatrix}$$

*These atoms were included in the calculation of the plane.

The packing of cations and anions in the crystal is represented in Fig. 2. The shortest approach distances are given in Table IV; there are no interanionic contacts below 3.6 Å. Although the arrangement of the ligands around the Pt atom is roughly in line with the expected square planar coordination, the best plane passing through Pt, P(1), P(2), N(1) and N(1*a*) (coordination plane) leaves the nitrogen atoms above it; both N–Pt–N and P–Pt–P angles are smaller than 90°, whereas the P–Pt–N ones are correspondingly larger. The angles (ϑ) between the coordination plane and the planes of the pyrazole rings are 90.6 and 78.3°; both the N(1)–N(2) and N(1*a*)–N(2*a*) vectors point roughly in the same direction, away from the coordination plane and towards the F(1) and F(2) atoms of one BF_4^- anion giving N(2)–F(2) and N(2*a*)–F(1) distances of 3.045 and 2.730 Å resp. Values for N(2)–H(2)···F(2) were 152° and for N(2*a*)–H(2*a*)···F(1) 165°, from which it can be inferred that a weaker and a stronger hydrogen bond are possible.

Comparison of the structural data pertaining to the title compound with those of other pyrazole complexes is made in Table V. In Table VI the pzH

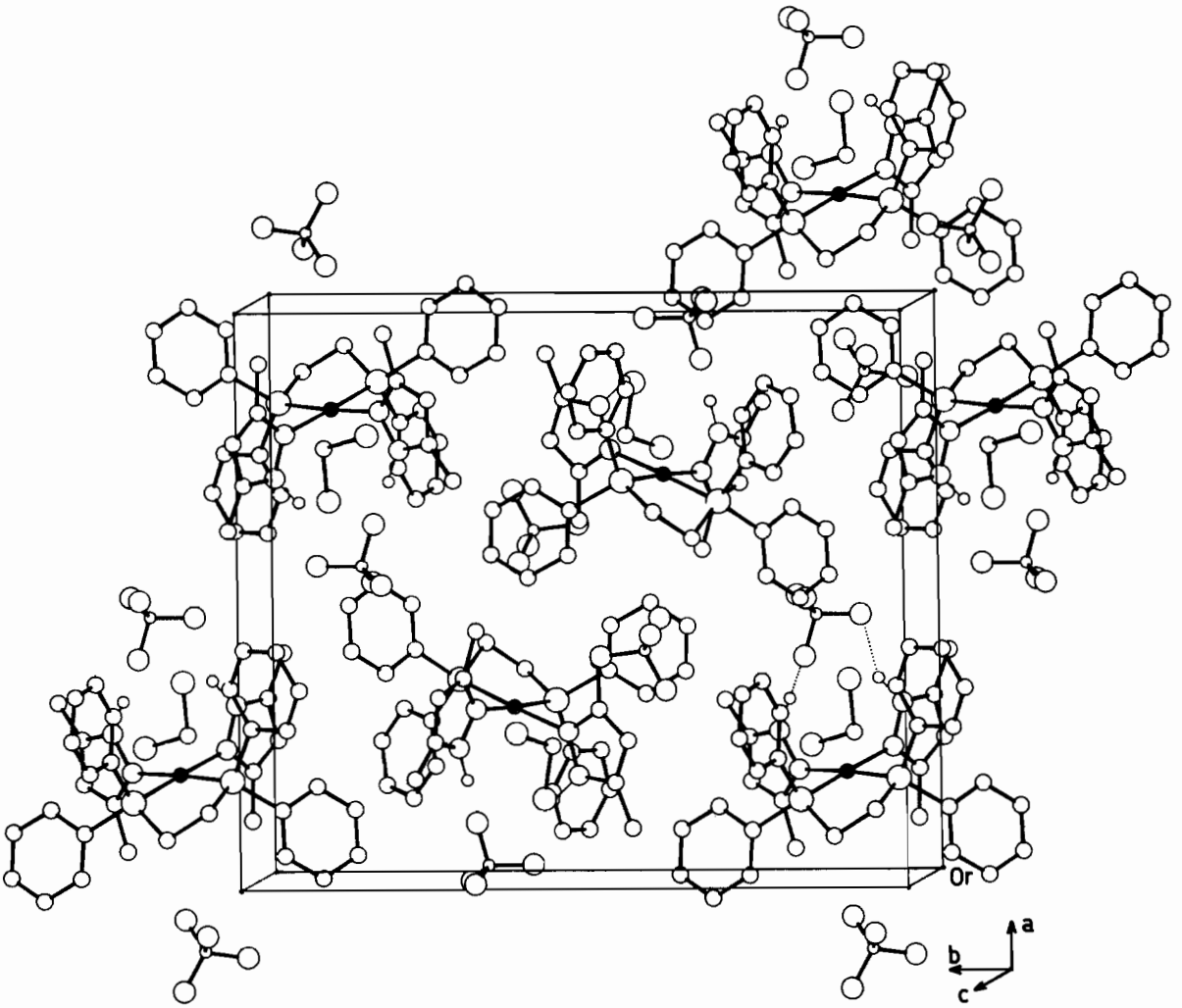

Fig. 2. Packing in the unit cell viewed along the *c* axis.

TABLE IV. Shortest Contact Distances (Å).

Between cations:			
C(16)(i)···C(4)(ii)	3.561	C(16)(i)···H(4)(ii)	2.53
Between cation and anions:			
N(2)(i)···F(1)(i)	3.404	C(17)(i)···F(6)(v)	3.210
N(2)(i)···F(2)(i)	3.045	C(19)(i)···F(1)(i)	3.407
H(2)(i)···F(1)(i)	2.40	C(24)(i)···F(8)(i)	3.284
H(2)(i)···F(2)(i)	2.04	C(25)(i)···F(7)(i)	3.507
C(6)(i)···F(2)(i)	3.564	C(25)(i)···F(8)(i)	3.322
C(7)(i)···F(8)(ii)	3.532	C(30)(i)···F(6)(vi)	3.344
N(1*a*)(i)···F(1)(i)	3.553	C(31)(i)···F(6)(vi)	3.303
N(2*a*)(i)···F(1)(i)	2.730	C(31)(i)···F(7)(vi)	3.238
H(2*a*)(i)···F(1)(i)	1.67	H(31)(i)···F(7)(vi)	2.43
C(6*a*)(i)···F(6)(iv)	3.581	C(32)(i)···F(5)(i)	3.553
H(6*a*−3)(i)···F(6)(iv)	2.49	C(32)(i)···F(7)(i)	3.189
C(7*a*)(i)···F(5)(i)	3.508	H(32−1)(i)···F(7)(i)	2.29
C(9)(i)···F(5)(i)	3.490	C(33)(i)···F(7)(vi)	3.475
C(16)(i)···F(6)(v)	3.268		
Between cation and solvent molecules:			
C(3)(i)···Cl(2)(i)	3.415	C(17)(i)···Cl(1)(vii)	3.588
C(16)(i)···Cl(1)(vii)	3.462		

TABLE IV *(continued)*

Between anion and solvent molecule:			
F(8)(i)···C(34)(iii)	3.063	F(8)(i)···Cl(1)(iii)	3.565

Symmetry code:

(i)	x	y	z
(ii)	$1/2-x$	$1/2+y$	$1/2-z$
(iii)	$-x$	$-y$	$-z$
(iv)	$1/2+x$	$1/2-y$	$-1/2+z$
(v)	$-1/2+x$	$1/2-y$	$1/2+z$
(vi)	$-x$	$-y$	$1-z$
(vii)	x	y	$1+z$

bond distances are reported for pyrazole and for Pt or Pd derivatives of various pyrazoles, as well as for representative examples of complexes of other metals: the values found in the title compound compare well with the literature data, since the Pt–N distances of 1.95 and 1.96 Å reported for **2** and **3** have been considered rather short [7].

TABLE V. Comparison of Distances and Angles in the Coordination Sphere.

Compound	No.	M–P Å	M–N Å	P–M–P deg.	P–M–N deg.	ϑ deg.	Reference
$[(Ph_2PCH_2)_2Pt(3,5\text{-}Me_2pzH)_2][BF_4]_2\cdot CH_2Cl_2$	1	2.245(2) 2.253(2)	2.107(5) 2.102(5)	84.87(6)	95.0(1) 92.3(1)	90.6 78.3	This work
$[cis\text{-}Cl(Et_3P)_2Pt(3\text{-}CF_3\text{-}5\text{-}MepzH)][BF_4]$	2	2.266(5)[a] 2.265(5)[b]	1.95(3)	98.5(2)	92.7(5)	93.0	[7]
$[cis\text{-}Cl(Et_3P)_2Pt(indazole)][BF_4]\cdot(CH_3)_2CO$	3	2.26(1)[a] 2.28(1)[b]	1.96(3)	98.0(4)	92.3(8)	83.9	[7]
$[cis\text{-}Cl(Et_3P)_2Pd(3,5\text{-}Me_2pzH)][BF_4]$	4	2.274(4)[a] 2.290(4)[b]	2.102(2)	97.9(1)	91.9(1)	89.3	[8]
$trans\text{-}Cl_2Pt(C_2H_4)$(pyrazole)	5	–	2.06(1)	–	–	3.5	[9]

[a]*trans* to chlorine. [b]*trans* to nitrogen.

TABLE VI. Comparison of the Bond Lengths inside the Pyrazole Rings in Selected Compounds and N···F Distances (Å) in the Case of Hydrogen Bonds.

Compound[a]	M	R′	R″	N(1)–N(2)	N(2)–C(3)	C(3)–C(4)	C(4)–C(5)	C(5)–N(1)	N···F separations	Reference
1	Pt	Me	Me	1.356(8) 1.347(8)	1.367(10) 1.364(11)	1.376(12) 1.360(14)	1.383(12) 1.373(12)	1.341(9) 1.323(9)	3.045(9) 2.730(9)	This work
2	Pt	CF_3	Me	1.49(3)	–[b]	1.39(3)	1.37(2)	1.38(3)	2.69(3)	[7]
4	Pd	Me	Me	1.355(9)	1.349(6)	1.35(1)	1.393(9)	1.319(7)	2.79(1)	[8]
5	Pt	H	H	1.33(2)	1.35(3)	1.33(3)	1.37(3)	1.28(3)	3.08(2)[c]	[9]
[e]	Au	Me	Me	1.40(2)	1.33(3)	1.41(3)	1.42(3)	1.36(2)	2.84 3.21	[10]
[f]	Sn	Me	Me	1.36(1)	1.35(1)	1.37(1)	1.39(1)	1.34(1)	2.76[d] 2.56[d]	[11]
[g]	H	H	H	1.344(2)	1.323(8)	1.369(10)	1.361(11)	1.353(3)	–	[12]

[a]For numbers see Table V. [b]Stated in the range 1.36–1.39. [c]N···Cl separation. [d]H···Cl separations. [e]$[Ph_3PAu(3,5\text{-}Me_2pzH)][BF_4]$. [f]$Me_2SnCl_2(3,5\text{-}Me_2pzH)_2$. [g]Pyrazole.

As can be seen from Table V, in the complexes with one pyrazole ring, *i.e.* **2**, **3** and **4**, the ring is normal (or nearly normal) to the coordination plane ($\vartheta \sim 90°$) except in the case of *trans*-$Cl_2Pt(C_2H_4)$-(pyrazole), **5**. Here, owing to the interaction between one chlorine atom and the NH group of the heterocycle, the angle ϑ between the coordination plane and the pyrazole ring is only 3.5°. In the title compound, **1**, ϑ is 90.6° for one pz ring and 78.3° for the other, and both rings lie on the same side with respect to the coordination plane. This orientation was not expected on the ground of steric considerations, and because in other platinum (II) derivatives containing two diazoles the rings lie in opposite sides, as in *cis*-Cl_2Pt(N-methylimidazole)$_2$, **6** [13] and in *cis*- or in *trans*-Cl_2PtL_2, where L is 1-(2-hydroxyethyl)-2-methyl-5-nitroimidazole (metronidazole) [14].

Therefore, the unusual reciprocal orientation of the two pzH rings in **1** is likely to be due to the hydrogen bonds with fluorine atoms of one BF_4^- anion. Such an interaction stabilizes the molecule into a well defined arrangement, while its absence may be the reason for the existence of multiple crystalline forms observed in **6**, probably due to different orientations of the diazole ligands [14]. The second BF_4^- anion does not take part in hydrogen-bonding since the two pzH rings are already involved in the hydrogen bonds with the other BF_4^- anion; bond lengths and angles are those expected.

The results reported here suggest that in the presence of hydrogen-bonding the angle ϑ is not related to any metal–pyrazole π–bonding. In addition, the two hydrogen-bonding interaction observed in **1** may be responsible for other features, such as deviation

from the planarity of the pyrazole rings both in the title compound **1** and in the palladium derivative **4**. In this respect it should be noted that the five-membered ring in crystalline indazole is also not planar owing to intermolecular hydrogen bonding [15].

Bond distances and angles within the solvent molecule CH_2Cl_2 are also as expected.

Experimental

The title compound non-solvated was prepared according to ref. [1]; suitable crystals were obtained from Et_2O/CH_2Cl_2 in solvated form, as colourless prisms.

Data Collection

A crystal of approximate dimensions 0.41 × 0.51 × 0.32 mm was chosen for data collection. Accurate unit-cell parameters were obtained by a least-squares fit of 2 ϑ values for 25 reflections measured on a Philips PW 1100 computer-controlled single-crystal diffractometer with graphite-monochromated Mo-Kα radiation at the Centro di Studio per la Cristallografia Strutturale del C.N.R., Pavia, Italy. The intensities of 7549 independent reflections were collected at room temperature within the angular range $2 \leqslant \vartheta \leqslant 25°$, using the $\omega/2\vartheta$ scan technique and a constant speed of 0.20° s^{-1} in ω: three standard reflections, monitored every 240 min, showed only small random deviations about their mean intensities. The intensities were corrected for Lorentz and polarization effects and for absorption (North *et al.*, 1968) [16]. An approximate absolute scale and a mean thermal factor were determined by Wilson's method [17]. A total of 4372 reflections having $I \geqslant 3\sigma(I)$ were considered to have observable intensity and were employed in the structure analysis.

Crystal Data

$C_{37}H_{42}B_2Cl_2F_8N_4P_2Pt$, F.W. 1044.37, monoclinic, *a* 17.418(5), *b* 20114(2), *c* 12252(2) Å, β 92.26(2)°; *V* 4289.1 Å^3; $Z = 4$, D_c 1.62 g cm^{-3}; *F*(000) 2064; λ (Mo-Kα) 0.7107 Å; μ (Mo-Kα) 37.11 cm^{-1}. Absent spectra: (*h*00), *h* odd; (0*k*0) *k* odd; (00*l*) *l* odd; (*h*0*l*) *h* + *l* odd. Space group $P2_1/n$.

Structure Determination and Refinement

The structure was solved by Patterson and Fourier methods. The positional and isotropic thermal parameters of the platinum, two phosphorus and two nitrogen atoms of the coordination sphere, derived from the three-dimensional Patterson map, were refined to *R* 0.236 in three cycles of least-squares refinement. A three-dimensional difference Fourier synthesis phased on these five atoms revealed all the remaining non-hydrogen atoms. Three cycles of full-matrix least-squares refinement, including scale factor and individual isotropic parameters, reduced the *R* index to 0.135. Individual anisotropic thermal parameters were then introduced and the *R* index dropped to 0.081 after three cycles. At this stage the positions of hydrogen atoms were calculated from the geometry of the compound and checked on a three-dimensional difference Fourier map. The subsequent least-squares refinement, including the hydrogen atoms with the same isotropic thermal parameters as the atom to which they were attached, reduced *R* to 0.041 ($Rw = 0.033$) after five cycles. In the early stages of the structure analysis the observed reflections were given unit weights; in the last two cycles a weight $w = \sigma^{-2}(|F_o|)$ was used.

Scattering factors for neutral atoms were taken from 'International Tables for X-ray Crystallography' (1974) [18] and those for hydrogen atoms from Stewart *et al.* (1965) [19]. Anomalous dispersion effects were included in the scattering factors.

The final positional and thermal parameters are listed in Tables VII and VIII. A list of structure factors can be obtained from one of the authors (B. Bovio) on request.

TABLE VII. Final Coordinates (with E.s.d.'s in Parentheses).

Atom	*x*	*y*	*z*
Pt	0.19311(1)	0.10172(1)	0.23394(2)
N(1)	0.2396(3)	0.0266(2)	0.1367(4)
N(2)	0.3156(4)	0.0116(3)	0.1391(5)
C(3)	0.3305(5)	−0.0396(4)	0.0698(6)
C(4)	0.2596(5)	−0.0569(4)	0.0248(6)
C(5)	0.2042(5)	−0.0165(4)	0.0679(5)
C(6)	0.4094(6)	−0.0639(5)	0.0573(9)
C(7)	0.1199(7)	−0.0158(7)	0.0478(7)
H(2)	0.3571(36)	0.0486(32)	0.1693(57)
H(4)	0.2532(30)	−0.0970(29)	−0.0392(44)
H(6−1)	0.4334(40)	−0.0443(35)	−0.0160(56)
H(6−2)	0.4442(38)	−0.0554(34)	0.1370(53)
H(6−3)	0.4037(39)	−0.1173(30)	0.0394(55)
H(7−1)	0.0873(39)	0.0142(34)	0.0994(54)
H(7−2)	0.1053(50)	−0.0034(44)	−0.0278(55)
H(7−3)	0.1015(32)	−0.0652(28)	0.0597(45)
N(1*a*)	0.2017(3)	0.1663(2)	0.1002(4)
N(2*a*)	0.2695(3)	0.1941(4)	0.0772(6)
C(3*a*)	0.2625(6)	0.2346(4)	−0.0119(7)
C(4*a*)	0.1870(6)	0.2308(5)	−0.0440(6)
C(5*a*)	0.1508(5)	0.1892(4)	0.0265(5)
C(6*a*)	0.3278(8)	0.2676(8)	−0.0571(8)
C(7*a*)	0.0691(7)	0.1699(5)	0.0244(9)
H(2*a*)	0.3235(39)	0.1799(44)	0.1177(68)
H(4*a*)	0.1600(38)	0.2545(34)	−0.1168(52)
H(6*a*−1)	0.3566(32)	0.2994(26)	−0.0007(36)
H(6*a*−2)	0.3669(38)	0.2349(38)	−0.0802(64)
H(6*a*−3)	0.3159(41)	0.2991(36)	−0.1347(55)

(continued on facing page)

TABLE VII *(continued)*

Atom	*x*	*y*	*z*
H(7*a*-1)	0.0356(38)	0.2067(32)	0.0249(59)
H(7*a*-2)	0.0576(39)	0.1409(34)	−0.0418(52)
H(7*a*-3)	0.0609(49)	0.1442(36)	0.1005(66)
P(1)	0.14144(9)	0.17774(8)	0.34329(13)
C(32)	0.0752(3)	0.1328(3)	0.4260(6)
H(32−1)	0.0286(28)	0.1183(25)	0.3883(42)
H(32−2)	0.0578(26)	0.1683(24)	0.4797(39)
P(2)	0.17698(9)	0.03106(8)	0.37386(13)
C(33)	0.1188(4)	0.0726(3)	0.4746(5)
H(33−1)	0.0875(31)	0.0446(27)	0.5091(46)
H(33−2)	0.1536(28)	0.0848(25)	0.5289(40)
C(8)	0.0915(4)	0.2476(3)	0.2787(5)
C(9)	0.0136(4)	0.2526(4)	0.2742(6)
C(10)	−0.0236(5)	0.3055(4)	0.2242(7)
C(11)	0.0187(6)	0.3516(5)	0.1752(7)
C(12)	0.0977(5)	0.3481(4)	0.1772(7)
C(13)	0.1340(4)	0.2947(4)	0.2308(6)
H(9)	−0.0213(29)	0.2223(27)	0.3091(43)
H(10)	−0.0864(33)	0.3002(28)	0.2189(45)
H(11)	−0.0070(42)	0.3882(34)	0.1384(56)
H(12)	0.1273(36)	0.3883(31)	0.1488(52)
H(13)	0.1828(32)	0.2899(28)	0.2236(49)
C(14)	0.2091(3)	0.2160(3)	0.4379(5)
C(15)	0.1851(4)	0.2521(4)	0.5256(6)
C(16)	0.2383(6)	0.2823(4)	0.5968(6)
C(17)	0.3153(6)	0.2759(4)	0.5813(7)
C(18)	0.3407(4)	0.2411(4)	0.4939(7)
C(19)	0.2880(4)	0.2110(3)	0.4232(5)
H(15)	0.1347(30)	0.2501(30)	0.5378(47)
H(16)	0.2211(34)	0.3071(30)	0.6570(46)
H(17)	0.3481(35)	0.2994(32)	0.6191(52)
H(18)	0.3942(31)	0.2301(29)	0.4933(47)
H(19)	0.3055(34)	0.1927(31)	0.3637(44)
C(20)	0.1274(3)	−0.0458(3)	0.3312(5)
C(21)	0.1696(4)	−0.0996(4)	0.2975(6)
C(22)	0.1314(5)	−0.1577(4)	0.2632(7)
C(23)	0.0537(5)	−0.1609(4)	0.2601(6)
C(24)	0.0122(4)	−0.1075(5)	0.2898(6)
C(25)	0.0495(4)	−0.0491(3)	0.3267(5)
H(21)	0.2193(29)	−0.1025(31)	0.3042(44)
H(22)	0.1589(32)	−0.1922(30)	0.2224(50)
H(23)	0.0321(32)	−0.2011(28)	0.2258(47)
H(24)	−0.0443(32)	−0.1124(31)	0.2934(51)
H(25)	0.0160(30)	−0.0163(25)	0.3508(43)
C(26)	0.2640(3)	0.0058(3)	0.4471(5)
C(27)	0.3338(4)	0.0361(3)	0.4287(5)
C(28)	0.3985(4)	0.0187(4)	0.4919(7)
C(29)	0.3929(4)	−0.0262(4)	0.5733(6)
C(30)	0.3246(4)	−0.0583(4)	0.5919(6)
C(31)	0.2586(4)	−0.0429(3)	0.5296(6)
H(27)	0.3364(27)	0.0778(23)	0.3766(40)
H(28)	0.4473(32)	0.0415(28)	0.4803(48)
H(29)	0.4400(32)	−0.0329(28)	0.6276(46)
H(30)	0.3205(30)	−0.0898(28)	0.6498(45)
H(31)	0.2153(30)	−0.0698(26)	0.5417(44)
B(1)	0.4657(7)	0.1440(5)	0.2244(11)
F(1)	0.3936(3)	0.1615(3)	0.2096(5)
F(2)	0.4654(4)	0.0771(3)	0.2174(5)
F(3)	0.4902(4)	0.1648(3)	0.3189(7)
F(4)	0.5021(5)	0.1685(4)	0.1464(8)
B(2)	−0.1138(5)	0.0686(4)	0.2657(7)
F(5)	−0.0557(2)	0.0946(3)	0.2051(3)
F(6)	−0.1819(2)	0.1003(2)	0.2469(3)
F(7)	−0.0910(2)	0.0712(2)	0.3748(3)
F(8)	−0.1230(3)	0.0034(2)	0.2395(4)
C(34)	0.2754(7)	0.0678(5)	−0.1870(11)
Cl(1)	0.25704(17)	0.12681(12)	−0.28401(20)
Cl(2)	0.36479(15)	0.07745(15)	−0.12414(25)
H(34−1)	0.2691(31)	0.0247(23)	−0.2162(46)
H(34−2)	0.2387(37)	0.0753(35)	−0.1248(54)

TABLE VIII. Thermal Parameters, with E.s.d.'s in Parentheses[a].

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Pt	0.00195(1)	0.00155(1)	0.00350(1)	−0.00008(1)	0.00023(1)	−0.00014(1)
N(1)	0.0028(2)	0.0025(2)	0.0050(4)	0.0004(2)	0.0008(2)	−0.0007(2)
N(2)	0.0033(3)	0.0019(2)	0.0079(6)	0.0002(2)	0.0018(3)	−0.0008(3)
C(3)	0.0055(4)	0.0024(3)	0.0079(7)	0.0003(3)	0.0035(5)	−0.0002(3)
C(4)	0.0063(5)	0.0026(3)	0.0065(7)	−0.0002(3)	0.0026(5)	−0.0023(3)
C(5)	0.0047(4)	0.0028(3)	0.0043(6)	−0.0007(3)	0.0010(4)	−0.0013(3)
C(6)	0.0068(6)	0.0039(3)	0.0154(13)	0.0029(4)	0.0060(6)	0.0004(5)
C(7)	0.0051(5)	0.0057(5)	0.0073(8)	−0.0011(4)	−0.0004(6)	−0.0024(5)
N(1*a*)	0.0030(2)	0.0027(2)	0.0044(4)	0.0001(2)	0.0011(2)	0.0005(2)
N(2*a*)	0.0033(3)	0.0026(2)	0.0079(7)	−0.0005(2)	0.0019(4)	0.0005(3)
C(3*a*)	0.0068(5)	0.0028(3)	0.0073(8)	−0.0004(3)	0.0032(5)	0.0015(4)
C(4*a*)	0.0069(5)	0.0041(4)	0.0051(8)	0.0008(3)	0.0012(5)	0.0014(4)
C(5*a*)	0.0043(4)	0.0039(3)	0.0028(5)	0.0005(3)	0.0006(4)	0.0004(3)
C(6*a*)	0.0073(8)	0.0068(6)	0.0114(12)	−0.0025(5)	0.0022(7)	0.0030(7)
C(7*a*)	0.0053(5)	0.0050(5)	0.0085(9)	0.0006(4)	−0.0022(6)	−0.0004(5)
P(1)	0.00194(6)	0.00150(5)	0.00432(13)	−0.00003(4)	0.00031(7)	0.00010(7)

(continued overleaf)

TABLE VIII *(continued)*

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(32)	0.0018(3)	0.0023(2)	0.0068(6)	−0.0003(2)	0.0006(3)	−0.0006(3)
P(2)	0.00190(6)	0.00163(5)	0.00424(13)	−0.00005(5)	0.00006(7)	−0.00009(7)
C(33)	0.0025(3)	0.0020(2)	0.0048(6)	0.0000(2)	0.0006(3)	0.0002(3)
C(8)	0.0029(3)	0.0013(2)	0.0049(5)	0.0002(2)	−0.0002(3)	−0.0003(3)
C(9)	0.0022(3)	0.0023(2)	0.0083(7)	0.0003(2)	0.0002(3)	−0.0003(3)
C(10)	0.0033(3)	0.0031(3)	0.0114(9)	0.0012(3)	−0.0020(4)	0.0001(4)
C(11)	0.0060(5)	0.0030(4)	0.0103(10)	0.0011(3)	−0.0019(6)	0.0019(4)
C(12)	0.0046(4)	0.0034(3)	0.0099(9)	0.0002(3)	0.0008(5)	0.0010(4)
C(13)	0.0036(3)	0.0026(3)	0.0076(7)	0.0009(3)	0.0000(4)	0.0008(3)
C(14)	0.0022(2)	0.0011(2)	0.0045(5)	−0.0003(2)	−0.0002(3)	0.0003(2)
C(15)	0.0031(3)	0.0023(2)	0.0064(6)	0.0000(2)	0.0001(4)	−0.0004(3)
C(16)	0.0063(5)	0.0022(2)	0.0049(6)	0.0000(3)	−0.0020(5)	−0.0002(3)
C(17)	0.0065(5)	0.0021(3)	0.0093(9)	−0.0011(3)	−0.0043(5)	−0.0002(4)
C(18)	0.0034(3)	0.0022(2)	0.0098(8)	−0.0007(2)	−0.0027(5)	0.0001(4)
C(19)	0.0028(3)	0.0020(2)	0.0062(6)	−0.0003(2)	−0.0008(4)	0.0006(3)
C(20)	0.0025(3)	0.0011(2)	0.0045(5)	0.0002(2)	0.0000(3)	0.0000(2)
C(21)	0.0031(3)	0.0027(2)	0.0090(6)	0.0008(3)	−0.0008(3)	−0.0017(4)
C(22)	0.0049(4)	0.0019(3)	0.0127(9)	0.0004(3)	−0.0016(5)	−0.0028(4)
C(23)	0.0056(4)	0.0018(2)	0.0096(8)	−0.0004(3)	−0.0022(5)	−0.0013(4)
C(24)	0.0036(3)	0.0030(3)	0.0095(7)	−0.0020(3)	−0.0003(4)	−0.0002(4)
C(25)	0.0029(3)	0.0022(2)	0.0059(6)	0.0005(2)	0.0003(3)	−0.0003(3)
C(26)	0.0022(2)	0.0017(2)	0.0046(5)	0.0002(2)	0.0002(3)	0.0002(3)
C(27)	0.0023(3)	0.0025(2)	0.0069(6)	0.0000(2)	−0.0002(3)	−0.0001(3)
C(28)	0.0027(3)	0.0030(3)	0.0102(8)	0.0004(2)	−0.0007(4)	−0.0001(4)
C(29)	0.0028(3)	0.0048(3)	0.0083(8)	0.0015(3)	−0.0009(4)	0.0008(4)
C(30)	0.0034(3)	0.0038(3)	0.0068(7)	0.0004(3)	0.0006(4)	0.0019(4)
C(31)	0.0034(3)	0.0025(2)	0.0062(6)	−0.0003(2)	0.0001(4)	0.0014(3)
B(1)	0.0060(6)	0.0015(3)	0.0146(14)	0.0001(3)	−0.0006(7)	0.0002(5)
F(1)	0.0066(3)	0.0105(3)	0.0233(8)	0.0044(3)	−0.0027(4)	0.0003(4)
F(2)	0.0127(4)	0.0042(2)	0.0248(8)	0.0006(2)	−0.0034(5)	−0.0003(3)
F(3)	0.0163(6)	0.0070(3)	0.0291(10)	0.0011(3)	−0.0126(7)	−0.0047(5)
F(4)	0.0204(7)	0.0080(4)	0.0445(16)	−0.0034(4)	0.0231(9)	−0.0019(6)
B(2)	0.0032(4)	0.0024(3)	0.0067(8)	−0.0012(3)	−0.0002(4)	0.0003(4)
F(5)	0.0063(2)	0.0063(2)	0.0106(4)	−0.0020(2)	0.0022(2)	0.0011(3)
F(6)	0.0053(2)	0.0055(2)	0.0132(5)	0.0015(2)	−0.0005(2)	0.0033(3)
F(7)	0.0040(2)	0.0064(2)	0.0067(3)	−0.0008(1)	−0.0006(2)	−0.0002(2)
F(8)	0.0061(2)	0.0035(2)	0.0197(6)	−0.0008(2)	−0.0013(3)	−0.0011(3)
C(34)	0.0072(6)	0.0053(5)	0.0129(10)	−0.0015(4)	−0.0030(6)	0.0036(6)
Cl(1)	0.01185(17)	0.00483(11)	0.01252(26)	−0.00254(11)	−0.00447(18)	0.00248(12)
Cl(2)	0.00720(14)	0.00829(15)	0.02251(37)	−0.00222(12)	−0.00224(19)	0.00741(19)

Atom	B (Å^2)	Atom	B (Å^2)	Atom	B (Å^2)
H(2)	3.92	H(7*a*−2)	6.60	H(18)	4.59
H(4)	5.20	H(7*a*−3)	6.60	H(19)	3.47
H(6−1)	7.81	H(32−1)	3.33	H(21)	4.51
H(6−2)	7.81	H(32−2)	3.33	H(22)	5.62
H(6−3)	7.81	H(33−1)	3.04	H(23)	5.18
H(7−1)	6.60	H(33−2)	3.04	H(24)	5.00
H(7−2)	6.60	H(9)	3.79	H(25)	3.54
H(7−3)	6.60	H(10)	5.36	H(27)	3.69
H(2*a*)	4.31	H(11)	6.16	H(28)	4.77
H(4*a*)	6.01	H(12)	5.66	H(29)	5.44
H(6*a*−1)	8.84	H(13)	4.38	H(30)	4.79
H(6*a*−2)	8.84	H(15)	3.80	H(31)	3.93
H(6*a*−3)	8.84	H(16)	4.77	H(34−1)	8.42
H(7*a*−1)	6.60	H(17)	5.71	H(34−2)	8.42

[a]The anisotropic parameters are in the form: $\exp -(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)$.

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