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The Crystal and Molecular Structure of *trans*-Bis(triphenylphosphine)-dicarbonyl(1,3-di-*p*-tolyltriazenido)rhenium(I)

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The structure of trans-*bis(triphenylphosphine)-dicarbonyl(1,3-di-*p*-tolyltriazenido)rhenium(I) has been determined from three-dimensional X-ray diffraction data collected by counter methods. The compound crystallizes in the triclinic space group* $P\bar{1}$ *with* a = *15.62(1),* b = *14.25(1),* c = *12.48(1) Å,* α = *66.34(4)°,* β = *108.84(4)°, and* γ = *115.48(4)°;* ρ_X = *1.46 g cm*$^{-3}$ *for* Z = *2. The structure was refined to the final R index of 0.056. The coordination about the rhenium atom is approximately octahedral. The triazenido ligand is coordinated to the metal atom in a bidentate mode with a N(1)–Re–N(3) angle of 57(1)° and a N(1)–N(2)–N(3) angle of 105(1)°. The Re–N(1) and Re–N(3) bond lengths are 2.21(1) and 2.18(1) Å and the N(1)–N(2) and N(2)–N(3) distances are 1.33(1) and 1.31(1) Å, respectively. The entire rhenium–triazenido system is essentially planar, indicative of* π *delocalization over the whole system.*

Introduction

There is much current interest in the chemistry of pseudo allyl ligands such as $[N(NR)_2]^-$, triazenido, and $[HC(NR)_2]^-$, formamidino, mainly because of their variety in mode of coordination which potentially may give rise to a rich chemistry. It has been proved that these ligands coordinate as monodentate [1–3], chelate [4–7], bridging [8–13], or as an orthometallated ligand [14]. In addition, the monodentate ligand may display a fluxional behaviour in solution [1, 3]; recently such dynamic processes have been proposed to occur also in the solid on the basis of X-ray diffraction studies [15]. Moreover a novel Rh–Hg complex has been reported containing two formamidino or triazenido groups, one chelating the rhodium atom and the other bridging a rhodium–mercury bond, for which a novel dynamic process has been established consisting of interchange of the bridging and the chelating organonitrogen ligands *via* a monodentate intermediate [16, 17].

Very recently we have synthesized new compounds of the type $[Re(N\text{–}N)(CO)_2(PPh_3)_2]$ (N–N = 1,3-diaryltriazenido or N,N'-diarylformamidino) which have been formulated as hexacoordinate complexes of Re(I) in which the organonitrogen ligand acts as a chelating ligand on the basis of i.r. and ^1H n.m.r.; this was confirmed by preliminary diffraction studies [18].

Here we present a full investigation on the structure of $[Re(dtt)(CO)_2(PPh_3)_2]$ (dtt = 1,3-di-*p*-tolyltriazenido).

Description of the Structure

The structure consists of discrete molecules of $[Re(dtt)(CO)_2(PPh_3)_2]$. Figures 1 and 2 show the molecular packing and the numbering system used throughout this paper. Figure 3 shows a detail of the molecule. The Re atom has a distorted octahedral coordination with *trans* PPh_3 groups being in the axial positions and the four equatorial sites being occupied by two *cis* carbonyl groups and by the chelate dtt ligand. Distortions from the idealized octahedral geometry arising from the constraints of the triazenido group are mainly localized in the base plane where this ligand subtends, at the metal atom, an angle of only 57°. Moreover (Table VI) mean planes calculations show that the equatorial atoms are only approximately coplanar, being alternatively displaced by ±0.01 to 0.02 Å from the base plane. A small deviation from linearity of the axial substituents (the P–Re–P angle is 176.1°) towards the carbonyls might be due

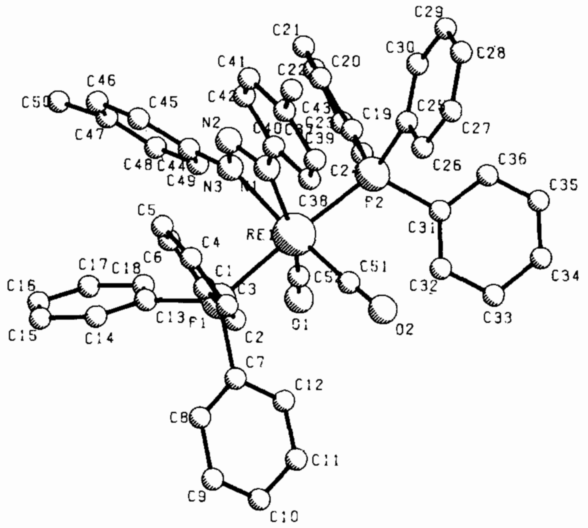

Fig. 1. Projection along the *a* axis.

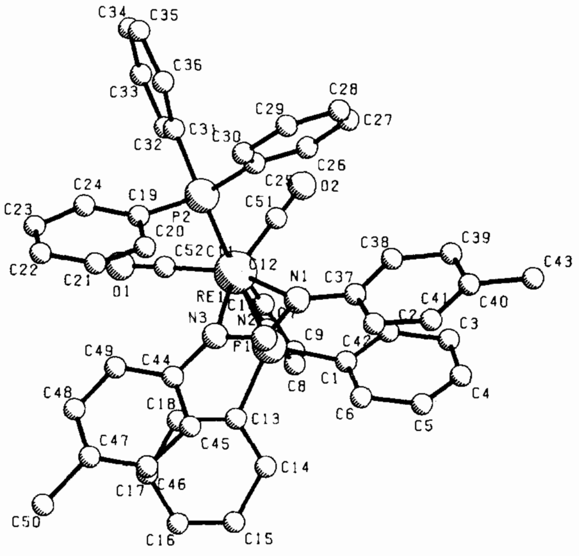

Fig. 2. Projection along the *c* axis.

TABLE I. Crystal Data for Re(dtt)$(CO)_2(PPh_3)_2$.

$C_{52}H_{44}N_3O_2P_2Re$	FW 991
Crystal description	Yellow-orange prisms
System	Triclinic
Space group	P$\bar{1}$
General positions	±(x, y, z)
Cell dimensions	a = 15.62(1)A
	b = 14.25(1)A
	c = 12.48(1) Å
	α = 66.33(4)°
	β = 108.84(4)°
	γ = 115.48(4)°
Volume	2258 A^3
Density	1.46 g cm^{-1}, for $Z = 2$
μ(Mo-Kα)	29.6 cm^{-1}
Total reflections	5223
Observed reflections with $I > 3\sigma(I)$	3939
Final R factor	0.056

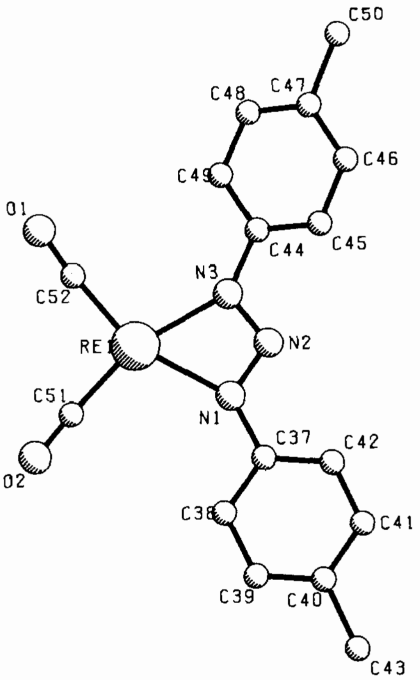

Fig. 3. Projection onto the equatorial plane. For clarity the axial substituents are omitted.

to steric hindrance between the bulky triphenylphosphine groups and the phenylene substituents. The most interesting feature of the present structure involves the rhenium–triazenido system. The Re–N(1) and Re–N(3) bond lengths as well as the N(1)–N(2) and N(2)–N(3) bond lengths are approximately the same, indicating that the ligand acts as chelating in a chemically equivalent environment. The latter distances are well in agreement with those found in $[Co(dpt)_3]$ [4], (dpt = 1,3-diphenyltriazenido) and in $[RuH(dtt)(CO)(PPh_3)_2]$ [7] in which the ligand acts also as chelating. In $[Ir(dtt)(CO)(PPh_3)_2]$, where there is a short Ir···N(3) contact (2.58(1) Å), the N(1)–N(2) and N(2)–N(3) distances are 1.31(1) and 1.28(2) respectively [15]; approximately the same values have been found in *trans*-$[PtH(dtt)(PPh_3)_2]$ [3] and in *cis*-$[Pt(dtt)_2(PPh_3)_2]$ [2], where there is a Pt···N contact of

TABLE II. Positional Parameters ($\times 10^4$) and Temperature Factors ($\times 10^3$).

Atom	X	Y	Z	U
Re	2753(0)	2098(0)	1119(0)	*
P(1)	3338(3)	1015(3)	619(3)	*
P(2)	2177(3)	3242(3)	1503(3)	*
N(1)	3799(8)	2028(10)	2813(10)	*
N(2)	3243(8)	1124(9)	3501(10)	*
N(3)	2415(8)	833(8)	2796(9)	*
O(1)	808(8)	1433(9)	–543(10)	*
O(2)	3866(9)	4104(10)	–712(10)	*
C(51)	3433(11)	3340(15)	–58(14)	*
C(52)	1556(14)	1714(12)	36(12)	*
C(1)	4630(16)	1263(18)	1263(19)	48
C(2)	5333(21)	2080(23)	594(25)	76
C(3)	6352(24)	2339(28)	1145(30)	99
C(4)	6564(22)	1780(25)	2349(27)	27
C(5)	5909(20)	1014(23)	3025(25)	77
C(6)	4883(17)	727(20)	2483(21)	58
C(7)	3295(15)	1297(17)	–962(19)	44
C(8)	3763(21)	866(23)	–1303(26)	78
C(9)	3750(21)	1094(24)	–2545(27)	84
C(10)	3235(20)	1713(23)	–3401(25)	76
C(11)	2792(20)	2186(24)	–3067(26)	79
C(12)	2798(19)	1960(21)	–1872(23)	67
C(13)	2778(14)	–500(16)	1098(18)	40
C(14)	3282(17)	–1172(19)	1299(20)	54
C(15)	2755(19)	–2358(21)	1588(22)	65
C(16)	1805(19)	–2760(22)	1678(23)	68
C(17)	1287(18)	–2083(21)	1459(22)	64
C(18)	1795(16)	–925(18)	1158(20)	51
C(19)	1112(14)	2450(16)	2240(18)	40
C(20)	1224(16)	1935(19)	3477(20)	53
C(21)	382(20)	1308(22)	3993(24)	72
C(22)	–541(21)	1128(24)	3288(26)	81
C(23)	–643(20)	1578(23)	2072(25)	76
C(24)	188(17)	2274(20)	1521(21)	56
C(25)	3064(15)	4182(17)	2392(18)	42
C(26)	3941(18)	4811(19)	1995(20)	55
C(27)	4641(19)	5585(22)	2601(24)	70
C(28)	4487(19)	5689(22)	3560(24)	70
C(29)	3598(20)	5064(22)	3930(24)	72
C(30)	7132(17)	5718(20)	6669(21)	58
C(31)	1697(15)	4246(17)	174(18)	43
C(32)	1546(18)	4212(20)	–945(22)	64
C(33)	1130(19)	4985(22)	–1948(24)	71
C(34)	927(18)	5731(20)	–1687(22)	66
C(35)	1097(18)	5749(21)	–561(23)	65
C(36)	1506(16)	5007(18)	418(20)	51
C(37)	4776(18)	2519(20)	3382(22)	52
C(38)	5385(18)	3387(20)	2635(22)	53
C(39)	6384(18)	3880(20)	3169(22)	60
C(40)	6692(18)	3457(20)	4382(22)	59
C(41)	6053(17)	2571(20)	5117(21)	62
C(42)	5093(18)	2120(20)	4613(22)	58
C(43)	7771(18)	4000(20)	4948(21)	83
C(44)	8298(18)	186(21)	6682(22)	38
C(45)	7999(17)	962(20)	5643(22)	69
C(46)	8617(18)	1971(20)	5195(21)	74
C(47)	9692(18)	2182(20)	5761(22)	63
C(48)	9956(17)	1422(20)	6760(22)	56
C(49)	9269(18)	398(20)	7254(21)	58
C(50)	471(21)	3344(23)	5259(25)	98

*Anisotropic thermal parameters in the form

$$T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + U_{12}hka^*b^* + U_{13}hla^*c^* + U_{23}klb^*c^*)]$$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Re	33	34	41	13	0	–11
P(1)	39	48	48	22	–1	–17
P(2)	45	39	40	19	4	–12
N(1)	44	50	49	2	–2	–21
N(2)	29	38	55	3	0	–11
N(3)	37	38	35	11	2	–1
O(1)	55	88	84	24	–16	–48
O(2)	102	60	70	18	44	8
C(51)	46	82	55	34	–8	–37
C(52)	44	65	45	27	–14	–27

TABLE III. Bond Distances (A).

Re–P(1)	2.44(1)	Re–P(2)	2.42(1)
Re–N(1)	2.21(1)	Re–N(3)	2.18(1)
Re–C(51)	1.92(2)	Re–C(52)	1.92(2)
C(51)–O(2)	1.15(2)	C(52)–O(1)	1.15(2)
N(1)–N(2)	1.33(1)	N(3)–N(2)	1.31(1)
N(1)–C(37)	1.44(2)	N(3)–C(44)	1.43(2)
C(37)–C(38)	1.39(3)	C(44)–C(45)	1.41(3)
C(38)–C(39)	1.46(3)	C(45)–C(46)	1.43(3)
C(39)–C(40)	1.39(3)	C(46)–C(47)	1.41(3)
C(40–C(41)	1.42(3)	C(47)–C(48)	1.36(3)
C(41)–C(42)	1.40(3)	C(48)–C(49)	1.42(3)
C(42)–C(37)	1.40(3)	C(49)–C(44)	1.41(3)
C(40)–C(43)	1.57(3)	C(47)–C(50)	1.58(3)
P(1)–C(1)	1.84(2)	P(2)–C(19)	1.85(3)
P(1)–C(7)	1.83(3)	P(2)–C(25)	1.88(3)
P(1)–C(13)	1.86(2)	P(2)–C(31)	1.89(2)
C(1)–C(2)	1.40(3)	C(19)–C(20)	1.41(3)
C(2)–C(3)	1.46(3)	C(20)–C(21)	1.42(4)
C(3)–C(4)	1.39(3)	C(21)–C(22)	1.40(3)
C(4)–C(5)	1.34(3)	C(22)–C(23)	1.37(3)
C(5)–C(6)	1.47(3)	C(23)–C(24)	1.44(4)
C(6)–C(1)	1.41(3)	C(24)–C(19)	1.41(3)
C(7)–C(8)	1.37(5)	C(25)–C(26)	1.40(4)
C(8)–C(9)	1.44(4)	C(26)–C(27)	1.44(4)
C(9)–C(10)	1.36(4)	C(27)–C(28)	1.37(5)
C(10)–C(11)	1.39(5)	C(28)–C(29)	1.40(4)
C(11)–C(12)	1.39(4)	C(29)–C(30)	1.46(4)
C(12)–C(7)	1.41(4)	C(30)–C(25)	1.37(5)
C(13)–C(14)	1.39(5)	C(31)–C(32)	1.36(4)
C(14)–C(15)	1.47(3)	C(32)–C(33)	1.48(3)
C(15)–C(16)	1.37(4)	C(33)–C(34)	1.40(5)
C(16)–C(17)	1.41(5)	C(35)–C(36)	1.43(3)
C(17)–C(18)	1.44(3)	C(34)–C(35)	1.35(4)
C(18)–C(13)	1.40(4)	C(36)–C(31)	1.40(5)

TABLE IV. Bond Angles (Degrees). The errors are referred to the last digit.

(a) Coordination			
P(1)–Re–P(2)	176.8(1)		
P(1)–Re–N(1)	91.3(5)	P(2)–Re–N(1)	90.8(5)
P(1)–Re–N(3)	92.4(4)	P(2)–Re–N(3)	90.8(4)
P(1)–Re–C(51)	87(1)	P(2)–Re–C(51)	90(1)
P(1)–Re–C(52)	90(1)	P(2)–Re–C(52)	88(1)
C(51)–Re–C(52)	96(1)	N(1)–Re–N(3)	57(1)
N(1)–Re–C(51)	105(1)	N(3)–Re–C(52)	102(1)
N(1)–Re–C(52)	159(1)	N(3)–Re–C(51)	162(1)
(b) dtt ligand			
N(1)–N(2)–N(3)	105(1)		
Re–N(1)–N(2)	98(1)	Re–N(3)–N(2)	100(1)
Re–N(1)–C(37)	146(1)	Re–N(3)–C(44)	144(1)
N(2)–N(1)–C(37)	116(1)	N(1)–N(3)–C(44)	115(1)
N(1)–C(37)–C(38)	116(2)	N(3)–C(44)–C(45)	120(2)
N(1)–C(37)–C(42)	122(2)	N(3)–C(44)–C(49)	118(2)
(c) CO and PPh_3			
Re–C(51)–O(2)	176(2)	Re–C(52)–O(1)	175(2)
Re–P(1)–C(1)	110(1)	Re–P(2)–C(19)	113(1)
Re–P(1)–C(7)	117(1)	Re–P(2)–C(25)	117(1)
Re–P(1)–C(13)	120(1)	Re–P(2)–C(31)	118(1)
C(1)–P(1)–C(7)	103(1)	C(19)–P(2)–C(25)	106(1)
C(1)–P(1)–C(13)	104(1)	C(19)–P(2)–C(31)	100(1)
C(7)–P(1)–C(13)	101(1)	C(25)–P(2)–C(31)	100(1)

ca. 3 Å. In $[Pd(1\text{-}3\text{-}\eta\text{-}C_3H_5)(dtt)]_2$, where the dtt ligand bridges two Pd atoms, the N–N distance is 1.30 Å. Thus it appears that the N–N bond distance is not much influenced by the coordination mode. Note, however, that in *cis*-$[PtCl(dtt)(PPh_3)_2]$, where the ligand acts as monodentate with a Pt···N contact of 3.01(2) Å, the N–N bond lengths are 1.26(3) and 1.26(3) respectively [3].

The N(1)–N(2), N(2)–N(3), N(1)–C(37), and N(3)–C(44) bond distances and the near planarity of the triazenido–rhenium system indicate that there is extensive delocalization over the entire ring. This feature is common to other related compounds, such as $[RuH(dtt)(CO)(PPh_3)_2]$ [7]. The N–N–N angle of 105° has the same value as the corresponding angle in $[Co(dpt)_3]$ (dpt = 1,3-diphenyltriazenido) and in $[RuH(dtt)(CO)(PPh_3)_2]$ in which both nitrogen atoms are coordinated to the metal to form a four membered chelate ring [4–7] and differs considerably from the values (mean 116°) found in complexes where the triazenido group is monodentate [2, 3] or where it bridges two metal centres [8–13]. This fact emphasizes the strained structure of the $\overline{\text{Re–N(1)–N(2)–N(3)}}$– four membered ring.

The values of the Re–C distances (1.92 Å) are about 0.1 to 0.2 Å less than the sum of the covalent

TABLE V. Contacts (Å).

Re···N(2)	2.74(1)		
N(1)···N(3)	2.10(1)		
Re···O(1)	3.08(1)	P(2)···O(1)	3.84(1)
Re···O(2)	3.08(1)	P(2)···O(2)	3.91(1)
P(1)···N(1)	3.33(1)	Re···C(1)	3.54(3)
P(1)···N(2)	3.71(1)	Re···C(7)	3.66(4)
P(1)···N(3)	3.34(1)	Re···C(13)	3.73(3)
P(2)···N(1)	3.30(1)	Re···C(19)	3.57(4)
P(2)···N(2)	3.70(1)	Re···C(25)	3.69(3)
P(2)···N(3)	3.28(1)	Re···C(31)	3.70(3)
P(1)···O(1)	3.96(1)	Re···C(37)	3.50(2)
P(1)···O(2)	3.83(1)	Re···C(44)	3.45(2)

Angles (Degrees)

N(1)–Re···N(2)	28.9(3)
N(3)–Re···N(2)	28.2(3)
N(1)–N(2)···Re	53.2(6)
N(3)–N(2)···Re	51.9(5)
P(1)–Re···N(2)	91.5(4)
P(2)–Re···N(2)	91.5(4)
C(51)–Re···N(2)	134.3(7)
C(52)–Re···N(2)	130.2(7)

TABLE VI. Least-squares Planes and Distances (A) of Atoms from the Planes. Equation of the plane is AX + BY + CZ = D referred to triclinic coordinates.

Plane I: N(1), N(3), C(51), C(52)
$-11.09X + 13.06Y + 7.50Z = 0.53$ A
[N(1) 0.02, N(3) −0.02, C(51) −0.01 C(52) 0.01, Re[a] 0.00, N(2)[a] −0.03, O(1)[a] 0.04, O(2)[a] 0.01]

Plane II: N(1), N(2), N(3)
$-11.20X + 12.87Y + 7.84Z = 0.56$ A

Plane III: C(37) to C(42)
$-10.34X + 13.46Y + 7.16Z = 0.88$ A
[C(37) 0.00, C(38) 0.00, C(39) 0.00, C(40) 0.00, C(41) 0.00, C(42) 0.01]

Plane IV: C(44) to C(49)
$-8.78X + 11.14Y + 10.85Z = 0.17$ A
[C(44) −0.01, C(45) 0.01, C(46) −0.01, C(47) 0.00, C(48) 0.00, C(49) 0.00]

Angles between the planes

Planes	Angle (Deg)	Planes	Angle (Deg)
I–II	2.3	II–III	6.4
I–III	4.5	II–IV	21.9
I–IV	23.8	III–IV	25.4

[a] These atoms were not used in the plane calculations.

radii for single bonds (2.05 Å), suggesting that the Re–C bond has some multiple bond character. Accordingly, the CO distances are 1.15 Å and should be compared with the value of 1.13 Å in carbon monoxide molecule: a further indication that in the carbonyl ligands the contribution of the structure with the triple bond is smaller than the sum in the CO molecule. The values of the *trans* Re–P distances (2.44 and 2.42 Å) are about 0.1 Å longer than the sum of the covalent radii. This feature is common to other mutually *trans* M–P bonds. The PPh_3 groups have their usual propeller configurations, and the P–C distances are all similar with observed values. It is noteworthy that, as already observed in complexes with coordinated triphenylphosphine groups, the Re–P–C angles (mean 116°) are larger and the C–P–C angles (mean 102°) are significantly smaller than the ideal value of 109° for sp^3 hybridized atoms. There are no abnormally short internuclear contacts in this structure.

Experimental

Yellow-orange crystals of $[Re(dtt)(CO)_2(PPh_3)_2]$ were obtained by recrystallization from CH_2Cl_2/EtOH [18]. A well formed crystal with approximate dimensions 0.10 × 0.15 × 0.10 mm was mounted on the end of a glass fiber with epoxy cement. Accurate cell dimensions were determined on a Philips PW 1100 four-circle computer-controlled automatic diffractometer using graphite-monochromatized MoKα radiation. Unit cell parameters were obtained by least-squares refinement of 30 medium angle reflections carefully determined. Crystal data are summarized in Table I. Intensity data were collected by the $\theta-2\theta$ scan technique out to a maximum of $2\theta = 50°$, with a scan rate of 2° min^{-1}. Two standard reflections measured at intervals of 100 were constant within counting statistics. The 5223 independent reflections measured were reduced to 3939 which were significantly above background ($F_o^2 \geqslant 3\sigma(F_o^2)$).

Solution of the structure was achieved by heavy-atom standard methods. Refinement of scale factor, positional and thermal parameters converged to the final agreement index R = 5.6% and Rw = 6.4% when the largest parameter shift in the last cycle was 0.2σ. The structure was refined with full-matrix least-squares, minimizing the function $\Sigma w(|F_o| - |F_c|)^2$ with w = 1. The agreement indices R and Rw are defined as $R = \Sigma \| F_o| - |F_c\| / \Sigma |F_o|$ and $Rw = (\Sigma w(|F_o| - |F_c|)^2 / \Sigma w F_o^2)^{1/2}$. A final difference Fourier synthesis showed no significant residuals. Scattering factors for Re were from Cromer and Waber [19], those for P, O, N, C from Cromer and Mann [20]. The rhenium scattering factor was corrected for anomalous dispersion with constant average values $\Delta f' = -1.34$ and $\Delta f'' = 7.34$ for the real and imaginary components. All calculations were done using the X-ray 70 program system [21]. Final atomic positional and thermal parameters are listed in Table II. Bond distances, bond angles, and relevant contact distances are reported in Tables III–V. Least-squares planes are given in Table VI.

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