bipyridine than for the phenanthroline complexes. The in-plane bending can be seen from the discrepancy between C(3)—C(2)—C(2′) and N—C(2)—C(2′) of 8–10°. The twisting of the ligands is in keeping with mechanisms for hydrolysis in which the ligands are twisted even more and the nucleophile attached to the central metal atom (Nord, 1975); *cf.* [Pt(phen)$_2$CN$^-$] (Wernberg & Hazell, 1980) where the angle between the ligands is 76° and CN$^-$ is bonded to Pt. Table 3 summarizes the geometries so far found for complexes with *trans* polypyridyl ligands. Although the tetrahedral, or twist, deformation is the commonest there are a surprising number with bow or tilt deformations in which the planar configuration at the metal atom is retained. So far no phenanthroline complexes have been reported with the bow deformation.

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The Structure of Bis(2,4,6-trimethoxyphenyl)mercury*

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Abstract. [Hg(C$_9$H$_{11}$O$_3$)$_2$], $M_r = 534{\cdot}96$, monoclinic, $P2_1/c$, $a = 14{\cdot}624\ (4)$, $b = 17{\cdot}731\ (5)$, $c = 7{\cdot}221\ (3)$ Å, $\beta = 93{\cdot}98\ (3)°$, $V = 1868\ (1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}93\ (2)$, $D_x = 1{\cdot}90$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 82{\cdot}4$ cm^{-1}, $F(000) = 1032$, $T = 293$ K. Final $R = 0{\cdot}045$ for 2292 observed reflections. Molecules of the title compound have near linear stereochemistry at Hg, C—Hg—C 176·7 (4)°, both Hg—C bonds 2·07 (1) Å, with an angle of 63·5 (4)° between the aromatic rings.

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* Organomercury Compounds. XXIX.

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The methyls of the *ortho*-methoxy groups are bent away from the mercury atom. The twisting of the aromatic rings from coplanarity is considered to arise from electrostatic repulsion between the oxygen atoms of the *ortho*-methoxy groups.

Introduction. Structures of diaryl- and diheteroarylmercury compounds range from those in which the two aromatic rings are coplanar, *e.g.* diphenylmercury (Grdenić, Kamenar & Nagl, 1977), di-*p*-tolylmercury (Mathew & Kunchur, 1970), bis(2,3,4,5-tetrafluorophenyl)mercury in which the two *ortho* fluorines are *transoid* (Brown, Massey & Wickens, 1980), di(2-furyl)mercury, which has the oxygen atoms in the *anti* conformation (Sikirica, Grdenić & Cimaš, 1982), and bis[(2-dimethylaminomethyl)phenyl]mercury in which *trans* dimethylamino groups are weakly bonded to mercury (Atwood, Berry, Stobart & Zaworotko, 1983), to those in which the two aryl rings are substantially inclined to one another, *e.g.* bis(pentafluorophenyl)mercury with a twist angle of 59·4° (Kunchur & Mathew, 1966), di(*o*-tolyl)mercury, twist angle 58·9° (Liptak, Ilsley, Glick & Oliver, 1980), and bis[(2,4,6-tri-*tert*-butyl)phenyl]mercury, twist angle 70·77° (Huffman, Nugent & Kochi, 1980). For the last two compounds, the twisting from the preferred coplanar arrangement can be attributed to steric repulsion between bulky *ortho* substituents. However, twisting for [$(C_6F_5)_2Hg$] has been attributed to both electrostatic repulsion between *ortho* fluorines (Kunchur & Mathew, 1966; Brown *et al.*, 1980) and steric repulsion (Liptak *et al.*, 1980). The latter explanation seems the less likely since the van der Waals radius of fluorine (1·35 Å) is not greatly larger than that (1·20 Å) of hydrogen (Cohen & Massey, 1970; Banks, 1970) [for a cautionary note, however, see Nyburg & Szymański (1968)] and in the sterically constrained bis(3,3′,4,4′,5,5′,-6,6′-octafluorobiphenylene)germanium, the 6,6′ fluorines approach within 2·45 Å (Cohen & Massey, 1970). The arrangement of aromatic rings in bis-(2,4,6-trimethoxyphenyl)mercury is of interest since space-filling models show that, despite the apparent steric congestion, a configuration with both rings coplanar is possible if all methyl groups are bent back away from mercury. We now report the structure of this compound.

Experimental. The title compound has been prepared by reaction (1), $R = 2,4,6\text{-}(MeO)_3C_6H_2$, in aqueous methanol at room temperature (Deacon, O'Donoghue, Stretton & Miller, 1982):

$$Hg(O_2CMe)_2 + 2RCO_2H \rightarrow R_2Hg + 2MeCO_2H + 2CO_2. \quad (1)$$

In the present work, the compound was obtained from an unsuccessful attempt to prepare phenyl(2,4,6-trimethoxyphenyl)mercury by decarboxylation of phenylmercuric 2,4,6-trimethoxybenzoate in refluxing toluene, reaction (2), $R = 2,4,6\text{-}(MeO)_3C_6H_2$:

$$2PhHgO_2CR \rightarrow Ph_2Hg + R_2Hg + 2CO_2. \quad (2)$$

After removal of the solvent under vacuum, the residue was shown to be a mixture of the two symmetrical mercurials by TLC and ^1H NMR (comparison with authentic samples) and mass spectroscopy (parent ions of symmetric mercurials but not PhHg*R* observed). Crystallization of the residue from ether/petroleum ether (b.p. ~340 K) gave single crystals of the title compound. D_m by flotation in $C_2H_2Br_4$ and CCl_4. Cell parameters determined using a Philips PW 1100 automatic four-circle diffractometer equipped with a graphite monochromator as described previously (Gatehouse & Miskin, 1974) and are the mean of three refinements of 24 medium- to high-angle reflections; three standard reflections monitored at 3 h intervals; no decomposition occurred. Data collected using the $\omega/2\theta$ scan technique with a symmetric scan width of ±0·60° in ω with allowance for dispersion, scan rate 0·05° s^{-1}; no attenuation filter required; data processed using the program of Hornstra & Stubbe (1972). Absorption correction applied based on indexed crystal faces, max. and min. transmission factors 0·4636 and 0·3985; 5442 reflections measured to $2\theta = 60°$ from a tabular crystal 0·11 × 0·11 × 0·21 mm, 2292 unique reflections [$I \geq 3\sigma(I)$] used in analysis; index range h −19/20, k 0/24, l 0/9. Hg-atom parameters found from Patterson synthesis and all non-H atoms located in subsequent difference Fourier synthesis. Function minimized in full-matrix least-squares refinement $\sum w(|F_o| - |F_c|)^2$, $w = [\sigma^2(F_o)]^{-1}$; Hg refined anisotropically, other non-H atoms refined isotropically; H-atoms inserted in calculated positions (C—H 1·08 Å, riding model), final $R = 0·045$ and $wR = 0·042$ (for observed reflections); max Δ/σ in final cycle 0·02. Final difference Fourier synthesis had $\Delta\rho$ within −1·10 and 1·00 e Å^{-3}. This was in the vicinity of the Hg atom. Scattering factors for neutral atoms and corrections for anomalous dispersion from *International Tables for X-ray Crystallography* (1974). All calculations performed on the Monash University VAX 11/780 computer system. Major programs used: *SHELX76* (Sheldrick, 1976), *ORFFE* (Busing, Martin & Levy, 1964) and *MEANPL* (Blow, 1960). Figure drawn using *ORTEP* (Johnson, 1965).

Discussion. Final fractional coordinates and thermal parameters are given in Table 1* and interatomic distances are in Table 2. The molecule (Fig. 1) is

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* Lists of structure factors, bond angles, H-atom parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43196 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

monomeric with near-linear C—Hg—C stereochemistry [angle C(1)—Hg—C(1′) = 176·7 (4)°] and a twist angle of 63·5 (4)° between the two aromatic rings. The four methyl groups of the *ortho*-methoxy substituents are all bent back away from Hg. A comparison of the Hg—C distance, the C—Hg—C angle, and the aromatic plane twist angle with those of other diarylmercurials and di(2-furyl)mercury is given in Table 3. Deviation from linear C—Hg—C stereochemistry is observed where the aromatic rings are not coplanar, and the greatest deviation is associated with the greatest twist angle. The title compound has the second largest twist angle. However, there is no relationship between the Hg—C distance and the deviation from coplanarity. The range of Hg—C for coplanar structures (2·06–2·10 Å) is similar to that (2·07–2·10 Å) for structures with twisted aromatic rings, and the average bond distance (2·08 Å) for the structure with the greatest twist is in the middle of the bond distance range. Whether twisting is associated with steric or electrostatic repulsion, these factors do not have a dominant effect on the Hg—C distance.

Mean-plane data* indicate that the C atoms of each aromatic ring are planar to within experimental error. One ring of bis[(2,4,6-tri-*tert*-butyl)phenyl]mercury has greater deviations (−0·041 → +0·028 Å, errors not given) (Huffman *et al.*, 1980). The bond distance range for the ring C(1′)—C(6′) is slightly larger than that for the ring C(1)—C(6) (Table 2), but even the anomalous 1·33 Å is within 3 e.s.d.'s of the mean (1·38 Å) for either or both rings. By contrast, for $(2,4,6\text{-Bu}^t_3\text{C}_6\text{H}_2)_2\text{Hg}$, ten distances cover a narrow range, but one C_{ipso}—C distance of each ring is extraordinarily long [1·450 (9) and 1·474 (9) Å] (Huffman *et al.*, 1980). As with bond distances, the bond angle range for the ring C(1′)—C(6′) [114·1 (11)—125·1 (11)°] is greater than that for the ring C(1)—C(6) [116·9 (10)–123·7 (10)°]. The overall range is similar to that of $(2,4,6\text{-Bu}^t_3\text{C}_6\text{H}_2)_2\text{Hg}$ and greater than that of diphenylmercury [117·5 (7)–122·4 (5)°] (Grdenić *et al.*, 1977). The

Table 1. *Fractional coordinates* (×10⁴) *and thermal parameters* (Å² ×10³) (Hg *coordinates* ×10⁵)

	x	y	z	U_{iso}
Hg	21525 (3)	3609 (2)	21304 (6)	*
C(1)	1104 (7)	1062 (6)	2865 (15)	44 (3)
C(2)	1244 (7)	1832 (6)	3224 (15)	43 (3)
C(3)	518 (6)	2309 (6)	3560 (14)	41 (3)
C(4)	−343 (7)	1999 (6)	3560 (17)	52 (3)
C(5)	−507 (7)	1240 (6)	3313 (15)	49 (3)
C(6)	235 (7)	794 (6)	2944 (16)	45 (3)
C(7)	2287 (7)	2883 (6)	3360 (17)	52 (3)
C(8)	−1057 (9)	3221 (7)	3710 (20)	70 (4)
C(9)	−758 (7)	−274 (7)	2413 (18)	64 (3)
O(1)	2135 (5)	2074 (4)	3222 (11)	50 (2)
O(2)	−1115 (5)	2418 (5)	3892 (12)	66 (2)
O(3)	152 (5)	31 (4)	2588 (11)	55 (2)
C(1′)	3158 (7)	−352 (6)	1255 (15)	47 (2)
C(2′)	3562 (7)	−246 (6)	−361 (15)	48 (3)
C(3′)	4227 (7)	−722 (6)	−1037 (18)	55 (3)
C(4′)	4455 (8)	−1336 (6)	103 (17)	54 (3)
C(5′)	4096 (8)	−1482 (7)	1713 (18)	56 (3)
C(6′)	3431 (7)	−992 (6)	2303 (16)	48 (3)
C(7′)	3600 (9)	517 (7)	−3155 (18)	74 (4)
C(8′)	5646 (9)	−1674 (8)	−1922 (21)	78 (4)
C(9′)	3129 (8)	−1778 (7)	4945 (19)	68 (4)
O(1′)	3263 (5)	385 (5)	−1368 (12)	65 (2)
O(2′)	5130 (6)	−1857 (5)	−361 (13)	76 (3)
O(3′)	3008 (5)	−1077 (5)	3946 (12)	63 (2)

* Anisotropic thermal parameters (Å² ×10⁴) of the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + \ldots + 2U_{12}hka^*b^* + \ldots)]$:

U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
418 (2)	294 (2)	530 (3)	46 (2)	55 (2)	−37 (3)

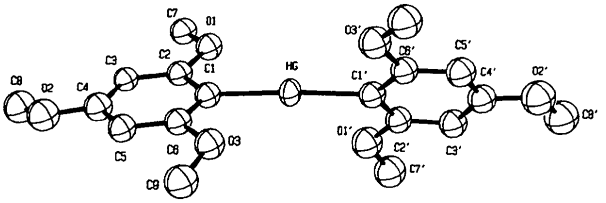

Fig. 1. Drawing of the title compound showing the stereochemistry of the molecule and the numbering scheme used.

Table 2. *Interatomic distances* (Å) *with e.s.d.'s in parentheses*

Hg—C(1)	2·07 (1)	Hg—C(1′)	2·07 (1)
C(1)—C(2)	1·40 (2)	C(1′)—C(2′)	1·36 (2)
C(2)—C(3)	1·39 (1)	C(2′)—C(3′)	1·40 (2)
C(3)—C(4)	1·37 (1)	C(3′)—C(4′)	1·39 (2)
C(4)—C(5)	1·38 (2)	C(4′)—C(5′)	1·33 (2)
C(5)—C(6)	1·38 (2)	C(5′)—C(6′)	1·39 (2)
C(6)—C(1)	1·36 (1)	C(6′)—C(1′)	1·41 (2)
C(2)—O(1)	1·37 (1)	C(2′)—O(1′)	1·39 (1)
C(4)—O(2)	1·39 (1)	C(4′)—O(2′)	1·41 (1)
C(6)—O(3)	1·38 (1)	C(6′)—O(3′)	1·38 (1)
C(7)—O(1)	1·45 (1)	C(7′)—O(1′)	1·43 (2)
C(8)—O(2)	1·43 (2)	C(8′)—O(2′)	1·44 (2)
C(9)—O(3)	1·43 (1)	C(9′)—O(3′)	1·44 (2)

Table 3. *Structural data for some diarylmercurials*

	Hg—C (Å)	C—Hg—C (°)	Twist angle (°)	
Ph_2Hg	2·085 (7)	180*	0	(Grdenić *et al.*, 1977)
$(2\text{-Furyl})_2\text{Hg}$	2·059 (5)	180*	0	(Sikirica *et al.*, 1982)
$(p\text{-MeC}_6\text{H}_4)_2\text{Hg}$	2·08 (2)	180*	0	(Mathew & Kunchur, 1970)
$(o\text{-HC}_6\text{F}_4)_2\text{Hg}$	2·096 (16)	180*	0	(Brown *et al.*, 1980)
$(o\text{-Me}_2\text{NCH}_2\text{C}_6\text{H}_4)_2\text{Hg}$	2·10 (2)	180*	0	(Atwood *et al.*, 1983)
$(o\text{-MeC}_6\text{H}_4)_2\text{Hg}$	2·09 (1)	178·0 (4)	58·9	(Liptak *et al.*, 1980)
$(\text{C}_6\text{F}_5)_2\text{Hg}$	2·09; 2·10	176·2 (12)	59·4 (12)	(Kunchur & Mathew, 1966)
$(2,4,6\text{-Bu}^t_3\text{C}_6\text{H}_2)_2\text{Hg}$	2·077 (6); 2·083 (6)	173·4 (2)	70·8	(Huffman *et al.*, 1980)
$[2,4,6\text{-(MeO)}_3\text{C}_6\text{H}_2]_2\text{Hg}$	2·07 (1)	176·7 (4)	63·5 (4)	(present work)

* Hg on centre of symmetry.

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* See deposition footnote.

planarity of the individual aromatic rings has not been discussed for the other two non-coplanar structures (Table 3). However, $(o\text{-MeC}_6H_4)_2Hg$ shows considerably less variation in C—C distances [1·38 (1)–1·40 (1) Å] and C—C—C angles [118·7 (10)–122·0 (9)°] than the present structure, while $(C_6F_5)_2$-Hg shows more variation (1·34–1·44 Å; 113·6–128·1°) but errors are not given and the *R* factor (0·081) is larger than for the other structures. The mercury atom of the title compound is significantly above the mean plane of both rings, the deviation being greatest from the C(1)—C(6) ring. Much greater displacements (0·279 and –0·778 Å) are observed for $(2,4,6\text{-Bu}^t_3C_6H_2)_2Hg$ (Huffman *et al.*, 1980) where the shifts are in opposite directions. Hg-atom deviations have not been discussed for the other non-coplanar structures. The *ortho* O atoms are staggered about the aromatic planes and the *para* oxygens deviate in opposite directions, but only one displacement is much greater than three e.s.d.'s.* For $(2,4,6\text{-Bu}^t_3C_6H_2)_2Hg$ (Huffman *et al.*, 1980), the two *ortho* α-carbon atoms of one ring are above the plane and the two of the other ring are below, and for one ring the displacements (0·183 and 0·256 Å) are quite large [*cf.* O-atom displacements in the title compound are 0·006–0·064 (8) Å].* Thus, for the non-coplanar diarylmercurials, distortion of individual rings from planarity falls in the sequence $(2,4,6\text{-Bu}^t_3C_6H_2)_2Hg > [2,4,6\text{-(MeO)}_3C_6H_2]_2Hg > (o\text{-MeC}_6H_4)_2Hg$ with the position of $(C_6F_5)_2Hg$ uncertain.

The methyls of the *ortho*-methoxy groups are all above the appropriate aromatic plane whereas the methyls of the *para*-methoxy groups are displaced more and in opposite directions. Moreover, the former methyls are displaced in the same sense as the mercury atom.

The aromatic C—O distances [*o*-MeO—C = 1·38 (ave.); *p*-MeO—C = 1·40 (ave.) Å] are less than the aliphatic C—O distances (ave. 1·44 Å) and the general C(paraffin)—O distance (1·43 Å) (Sutton, 1958). Although the errors are appreciable in relation to the difference, this behaviour is observed for all six aromatic carbon–oxygen distances and suggests partial double-bond character in these bonds owing to conjugation of oxygen lone pairs with the aromatic ring. However, conjugation is not as marked as in phenols (C—O = 1·36 Å) (Sutton, 1958).

The mercury–*ortho*-oxygen contacts [Hg···O(1), 3·139 (7); Hg···O(1'), 3·097 (8); Hg···O(3), 3·024 (8); Hg···O(3'), 3·091 (8) Å] are sufficiently close to the sum (3·13 Å) of the van der Waals radii of oxygen (1·4 Å) (Pauling, 1960) and mercury (*ca* 1·73 Å) (Canty & Deacon, 1980) for significant intramolecular mercury–oxygen bonding to be ruled out. In bis(pentafluorophenyl)mercury (Kunchur & Mathew, 1966), mercury–*ortho*-fluorine contacts are even longer (3·14–3·32 Å, ave. 3·23 Å, no errors given) compared with the sum of the van der Waals radii of mercury and fluorine of 3·08 Å.

For $(2,4,6\text{-Bu}^t_3C_6H_2)_2Hg$ the main source of molecular distortion is considered to be steric repulsion owing to short H···H and Hg···H contacts. Even with the rings twisted to minimize these interactions, there are five Hg···H and several H···H contacts significantly inside the sum of the appropriate van der Waals radii (Huffman *et al.*, 1980). In the present structure, only one Hg···H contact and three H···H contacts are within the sum of the van der Waals radii, even if the upper limit of 1·3 Å (Pauling, 1960) is used for the hydrogen radius. All are *inter*molecular contacts, and none is significant if the lower limit of the hydrogen van der Waals radius (1·1 Å) is used. With the non-coplanar aromatic rings, all O···O contacts are well outside twice the van der Waals radius of oxygen (2·8 Å) (Pauling, 1960), the shortest being O(3)···O(3') [4·66 (1) Å]. Intramolecular contacts between the methyl C atoms of *ortho*-methoxy groups are >6·0 Å but there is an intermolecular C(7)···C(7') approach of 3·64 Å, well within two van der Waals radii of methyl groups (4·0 Å) (Pauling, 1960).

With the methyl groups of the *ortho*-methoxy substituents orientated as in the present structure (Fig. 1), *viz* away from Hg, space-filling models show that a coplanar structure is sterically possible. Thus, it is likely that non-coplanarity arises from electrostatic repulsion between oxygen lone pairs. Although the bond length errors rule out a definite conclusion, it is interesting that the average $o\text{-MeO—C}_{Ar}$ distance is slightly shorter (0·02 Å) than the average $p\text{-MeO—C}_{Ar}$ distance, consistent with greater conjugation from the *o*-MeO groups owing to O···O electrostatic repulsion. The packing diagram showed no evidence for intermolecular effects which might favour non-coplanarity. Indeed, twisting occurs despite a resulting highly repulsive intermolecular C(7)···C(7') approach (see above).

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Structure of Dichlorobis(trimethylphosphine oxide)cobalt(II)

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Abstract. $[CoCl_2(C_3H_9OP)_2]$, $M_r = 314{\cdot}0$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}686\,(3)$, $b = 11{\cdot}271\,(3)$, $c = 11{\cdot}975\,(4)$ Å, $V = 1442\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}446\,(1)$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}70926$ Å, $\mu = 17{\cdot}4$ cm^{-1}, $F(000) = 644$, $T = 293$ K, $R = 0{\cdot}0482$ for 1351 observed reflections. The title compound is a mononuclear cobalt complex with approximate tetrahedral coordination of the two Cl and two O atoms around the central Co atom. Important bond lengths: Co—Cl 2·256 (2), Co—O 1·960 (3), P—O 1·508 (3), P—C 1·771 (4) Å.

Introduction. Adducts of Co^{II} halides with phosphine oxides have been the subject of detailed spectroscopic studies (Cotton, Barnes & Bannister, 1960; Brodie, Hunter, Rodley & Wilkins, 1968; Hunter, Langford, Rodley & Wilkins, 1968; Kato & Akimoto, 1974) and two complexes with aryl-substituted phosphine oxides have been structurally characterized [$(Ph_3PO)_2CoCl_2$: Mangion, Smith & Shore (1976); $(Bz_3PO)_2CoCl_2$: Santos & Mascarenhas (1979)]. We report here the first crystal structure of a simple alkyl derivative, $(Me_3PO)_2CoCl_2$.

Experimental. The title compound was prepared by mixing ethanol solutions of $CoCl_2$ and trimethylphosphine oxide as described by Pickard & Kenyon (1906). Dark blue, cube-shaped single crystals were obtained by recrystallization from acetone. Crystal 0·25 × 0·25 × 0·25 mm. Hilger & Watts diffractometer, $\theta/2\theta$ scan mode with $5 < 2\theta < 60°$, hkl range: $0 \le h \le 15$, $0 \le k \le 15$, $0 \le l \le 16$. Cell parameters obtained by least-squares refinement from setting angles of 20 automatically centred high-angle reflections ($20 \le 2\theta \le 55°$). 2061 independent, non-systematically absent (in $P2_12_12_1$) reflections measured, 710 considered as unobserved ($|F| \le 4\sigma|F|$). No absorption or secondary-extinction corrections. Structure solved by Patterson and Fourier methods (*SHELX*77; Sheldrick, 1977). No intensity drop of three standard reflections measured after each block of 97 reflections. H atoms calculated and refined with constrained C—H bond lengths of 0·96 Å, H—C—H angles of 104·7° (refined value), and a common temperature factor.

Least-squares refinement based on F. Scattering factors for all atoms except Co included in the *SHELX*77 program (Sheldrick, 1977); for Co from

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