

five-coordinate geometry of the complex is retained in chloroform solution, as shown by the magnitudes of the one- and two-bond NMR coupling constants (Holecek, Lycka, Handlir & Nadvornik, 1988).

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## Structure of ( $\eta^4$ -1,5-Cyclooctadiene)(1-ferrocenyl-1,3-butanedionato- $\kappa^2$ O,O')rhodium(I)

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**Abstract.** [Rh(C<sub>14</sub>H<sub>13</sub>FeO<sub>2</sub>)(C<sub>8</sub>H<sub>12</sub>)],  $M_r = 480.2$ , triclinic,  $P\bar{1}$ ,  $a = 11.658$  (1),  $b = 13.001$  (1),  $c = 15.251$  (2) Å,  $\alpha = 70.621$  (8),  $\beta = 68.538$  (8),  $\gamma = 66.049$  (7)°,  $V = 1919.8$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.63$ ,  $D_x = 1.66$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 16.15$  cm<sup>-1</sup>,  $T = 295$  K,  $F(000) = 976$ , final  $R = 0.0419$  for 8290 observed reflections. Two crystallographically independent molecules *A* and *B* are present in the unit cell. Both molecules have an approximately square-planar configuration about the Rh atom, but only the cyclopentadienyl (Cp) rings of *B* approach an eclipsed conformation. The O—Rh—O plane does not pass through the middle of the C=C bonds of the boat-like 1,5-cyclooctadiene (cod) ligand.

**Experimental.** The title compound was prepared by stirring 0.24 g (0.9 mmol) of solid 1,3-dioxo-1-butylferrocene (FcA) (Hauser & Lindsay, 1957) with a solution of 0.22 g (0.45 mmol) of [Rh<sub>2</sub>Cl<sub>2</sub>(cod)<sub>2</sub>] (Chatt & Venanzi, 1957) in a minimum amount of DMF for 5 min at room temperature. Excess water was added to the homogeneous reaction mixture and the precipitate was filtered, dissolved in ether then washed with water. Drying (MgSO<sub>4</sub>) of the ethereal solution and removal of the solvent, followed by recrystallization from an acetone–water mixture, gave 0.26 g (61%) of well formed red crystals of [Rh(FcA)(cod)] suitable for X-ray analysis. The den-

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sity was determined by flotation in sodium iodide solution. Data were collected on an Enraf–Nonius CAD-4F diffractometer with graphite-monochromated Mo  $K\alpha$  radiation, using a crystal of size  $0.19 \times 0.11 \times 0.08$  mm. The  $\omega/2\theta$ -scan technique was used with variable scan width  $\Delta\omega = (0.45 + 0.34\tan\theta)^\circ$ , a max. scan speed of  $5.49^\circ \text{ min}^{-1}$  in  $\omega$  and a max. scan time of 60 s per reflection. The unit-cell parameters were determined from a least-squares refinement of 25 reflections with  $18 < \theta < 20^\circ$ . Data were corrected for Lorentz and polarization effects, as well as for absorption (North, Phillips & Mathews, 1968) with min. and max. correction factors of 0.970 and 0.999, respectively. Three control reflections were measured every 3600 s of X-ray exposure time and varied from the initial value by less than 2%. Intensity data were corrected for this decay. The intensities of 11 384 reflections in the index range  $0 < h < 16$ ,  $-18 < k < 18$ ,  $-21 < l < 21$  with  $0.07 < \sin\theta/\lambda < 0.7 \text{ \AA}^{-1}$ , were measured. Of 10 106 unique reflections ( $R_{\text{int}} = 0.0204$ ), 8291 were considered observed with  $I > 3\sigma(I)$ . The structure was solved by the heavy-atom method using *SHELX86* (Sheldrick, 1990) and was subjected to least-squares refinement on *F* with 476 positional and anisotropic thermal parameters for all the non-H atoms using *SHELX76* (Sheldrick, 1976). The H-atom positions were calculated by assuming C—H = 1.08 Å, and refined with an overall temperature factor. Neutral-atom scattering factors (Cromer & Mann, 1968) and anomalous-dispersion corrections

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ ) for [Rh(*FcA*)(*cod*)] with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
Rh(1)	4971.1 (3)	4833.6 (3)	7919.2 (2)	31.2 (1)
Rh(2)	996.7 (3)	373.9 (3)	1209.3 (3)	34.9 (1)
Fe(1)	3187.0 (7)	2032.1 (6)	6660.8 (5)	37.0 (1)
Fe(2)	-726.2 (7)	2386.1 (6)	4226.4 (5)	40.9 (1)
O(12)	4094 (3)	4086 (3)	9266 (2)	38 (1)
O(22)	422 (3)	2037 (3)	450 (2)	41 (1)
O(11)	4180 (3)	4193 (3)	7293 (2)	39 (1)
O(21)	-326 (4)	819 (3)	2450 (2)	49 (1)
C(102)	2997 (4)	3264 (4)	8772 (3)	34 (1)
C(111)	2738 (4)	3459 (4)	7177 (3)	34 (1)
C(201)	-1012 (5)	1849 (4)	2538 (3)	38 (1)
C(103)	3369 (4)	3476 (3)	9443 (3)	33 (1)
C(211)	-1642 (5)	1988 (4)	3544 (3)	40 (1)
C(12)	5488 (5)	6000 (5)	6596 (3)	45 (1)
C(203)	-507 (5)	2848 (4)	810 (3)	39 (1)
C(101)	3367 (4)	3650 (3)	7766 (3)	33 (1)
C(11)	6507 (5)	4950 (5)	6655 (3)	43 (1)
C(112)	1637 (5)	3052 (4)	7517 (3)	39 (1)
C(113)	1318 (5)	3086 (4)	6686 (4)	45 (1)
C(15)	5000 (5)	6069 (4)	8518 (3)	44 (1)
C(16)	6151 (5)	5138 (4)	8522 (3)	40 (1)
C(22)	1017 (6)	-1349 (4)	1777 (4)	56 (1)
C(212)	-2453 (5)	3022 (5)	3884 (4)	49 (1)
C(202)	-1198 (5)	2815 (4)	1778 (3)	43 (1)
C(115)	3087 (5)	3731 (4)	6139 (3)	42 (1)
C(17)	7463 (5)	5171 (5)	7799 (4)	48 (1)
C(114)	2202 (5)	3493 (4)	5841 (4)	47 (1)
C(214)	-1996 (6)	1552 (6)	5192 (4)	57 (1)
C(215)	-1359 (5)	1076 (5)	4355 (4)	48 (1)
C(26)	2841 (5)	10 (5)	194 (4)	56 (1)
C(19)	2870 (5)	2985 (5)	10507 (3)	45 (1)
C(25)	1987 (5)	-197 (5)	-102 (4)	54 (1)
C(222)	921 (6)	2634 (6)	3223 (5)	65 (2)
C(213)	-2674 (6)	2726 (6)	4913 (4)	59 (1)
C(21)	1914 (6)	-1195 (5)	2071 (4)	60 (1)
C(18)	7731 (5)	4705 (5)	6927 (4)	49 (1)
C(14)	4844 (6)	7212 (5)	7804 (4)	58 (1)
C(29)	-882 (6)	3963 (4)	88 (4)	55 (1)
C(223)	1252 (6)	1762 (6)	4033 (5)	65 (2)
C(13)	5490 (6)	7085 (5)	6741 (4)	58 (1)
C(123)	4897 (7)	1060 (6)	5872 (5)	69 (2)
C(122)	4958 (7)	869 (6)	6801 (6)	74 (2)
C(221)	78 (6)	3658 (6)	3573 (6)	71 (2)
C(24)	1989 (8)	-1399 (7)	-5 (6)	90 (2)
C(121)	3968 (10)	454 (6)	7457 (5)	95 (2)
C(225)	-85 (7)	3406 (7)	4577 (6)	78 (2)
C(23)	1361 (8)	-1948 (5)	969 (5)	74 (2)
C(224)	628 (7)	2236 (7)	4863 (6)	74 (2)
C(124)	3881 (8)	778 (6)	5902 (6)	82 (2)
C(28)	3370 (7)	-1602 (7)	1636 (6)	88 (2)
C(125)	3260 (8)	383 (6)	6880 (10)	116 (3)
C(27)	3849 (7)	-883 (8)	699 (8)	109 (3)
<i>M</i> (11)*	2200 (10)	3367 (4)	6668 (9)	-
<i>M</i> (12)	4201 (5)	711 (2)	6582 (8)	-
<i>M</i> (21)	-2021 (7)	2067 (10)	4379 (8)	-
<i>M</i> (22)	556 (4)	2742 (5)	4056 (5)	-

\* Calculated centroids of Cp rings.

for rhodium and iron were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). A final *R* value of 0.0479 and *wR* = 0.0520 (unit weights) were obtained. Residual electron density was  $(\Delta\rho)_{\max} = 0.89$  and  $(\Delta\rho)_{\min} = -1.26 \text{ e \AA}^{-3}$ , with positional  $(\Delta/\sigma)_{\max}$  for displacement parameters = 0.20. Final atomic coordinates are given in Table 1\*

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55711 (51 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: L19126]

Table 2. Selected interatomic bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for [Rh(*FcA*)(*cod*)] with *e.s.d.*'s in parentheses

Molecule A		Molecule B	
Rh(1)—O(11)	2.063 (3)	Rh(2)—O(21)	2.039 (3)
Rh(1)—O(12)	2.037 (3)	Rh(2)—O(22)	2.048 (3)
Rh(1)—C(15)	2.111 (4)	Rh(2)—C(25)	2.118 (5)
Rh(1)—C(16)	2.120 (4)	Rh(2)—C(26)	2.114 (5)
Rh(1)—C(11)	2.109 (4)	Rh(2)—C(21)	2.123 (5)
Rh(1)—C(12)	2.135 (4)	Rh(2)—C(22)	2.110 (5)
C(111)—C(101)	1.477 (6)	C(211)—C(201)	1.475 (6)
C(101)—C(102)	1.405 (6)	C(201)—C(202)	1.401 (7)
C(102)—C(103)	1.385 (6)	C(202)—C(203)	1.395 (6)
C(103)—C(19)	1.518 (6)	C(203)—C(29)	1.511 (9)
C(18)—C(11)	1.513 (7)	C(28)—C(21)	1.511 (10)
C(11)—C(12)	1.400 (7)	C(21)—C(22)	1.381 (8)
C(15)—C(16)	1.396 (7)	C(25)—C(26)	1.377 (8)
C(16)—C(17)	1.531 (7)	C(26)—C(27)	1.510 (9)
C(11)—Rh(1)—C(16)	83.0 (2)	C(21)—Rh(2)—C(26)	83.4 (2)
C(16)—Rh(1)—C(15)	38.5 (2)	C(26)—Rh(2)—C(25)	38.0 (2)
C(15)—Rh(1)—C(12)	82.5 (2)	C(25)—Rh(2)—C(22)	83.3 (2)
C(12)—Rh(1)—C(11)	38.5 (2)	C(22)—Rh(2)—C(21)	38.1 (2)
Rh(1)—O(11)—C(101)	124.1 (3)	Rh(2)—O(21)—C(201)	124.6 (3)
Rh(1)—O(12)—C(103)	124.7 (3)	Rh(2)—O(22)—C(203)	123.8 (3)
C(101)—C(102)—C(103)	126.6 (4)	C(201)—C(202)—C(203)	125.7 (4)
C(101)—C(111)—C(112)	127.3 (4)	C(201)—C(211)—C(212)	128.4 (5)
O(12)—C(103)—C(19)	114.2 (4)	O(22)—C(203)—C(29)	114.6 (4)
O(12)—C(103)—C(102)	126.6 (4)	O(22)—C(203)—C(202)	127.4 (4)
O(11)—C(101)—C(102)	126.3 (4)	O(21)—C(201)—C(202)	125.7 (4)
O(11)—C(101)—C(111)	115.1 (4)	O(21)—C(201)—C(211)	114.6 (4)

Table 3. Distances ( $\text{\AA}$ ) of selected atoms above and below certain planes and selected twist angles ( $^\circ$ ) (*j* = 1,2,3,4 or 5, *M* = centroid)

Molecule A		Molecule B	
O(11)—Rh(1)—O(12)		O(21)—Rh(2)—O(22)	
C(11)	-0.865 (6)	C(21)	-0.543 (7)
C(12)	0.528 (6)	C(22)	0.831 (6)
C(15)	0.903 (6)	C(25)	0.530 (6)
C(16)	-0.484 (5)	C(26)	-0.541 (6)
C(111)—C(112)—C(113)—C(114)—C(115)		C(211)—C(212)—C(213)—C(214)—C(215)	
C(11) <sub>max</sub> = C(114)	-0.004 (6)	C(21) <sub>max</sub> = C(213)	0.008 (8)
Fe(1)	-1.648 (3)	Fe(2)	-1.641 (5)
C(121)	-3.347 (9)	C(221)	-3.217 (9)
C(122)	-3.350 (8)	C(222)	-3.249 (9)
C(123)	-3.287 (8)	C(223)	-3.331 (9)
C(124)	-3.241 (9)	C(224)	-3.349 (10)
C(125)	-3.284 (11)	C(225)	-3.290 (10)
<i>M</i> (11)	-0.001 (11)	<i>M</i> (21)	0.000 (12)
<i>M</i> (12)	-3.302 (8)	<i>M</i> (22)	-3.287 (8)
C(111)— <i>M</i> (11)— <i>M</i> (12)—C(122)	-20.1 (8)	C(211)— <i>M</i> (21)— <i>M</i> (22)—C(222)	-1.1 (8)
C(112)— <i>M</i> (11)— <i>M</i> (12)—C(121)	-20.5 (8)	C(212)— <i>M</i> (21)— <i>M</i> (22)—C(221)	-0.5 (8)
C(113)— <i>M</i> (11)— <i>M</i> (12)—C(125)	-18.7 (9)	C(213)— <i>M</i> (21)— <i>M</i> (22)—C(225)	-0.6 (8)
C(114)— <i>M</i> (11)— <i>M</i> (12)—C(124)	-18.0 (8)	C(214)— <i>M</i> (21)— <i>M</i> (22)—C(224)	0.2 (8)
C(115)— <i>M</i> (11)— <i>M</i> (12)—C(123)	-20.3 (8)	C(215)— <i>M</i> (21)— <i>M</i> (22)—C(223)	-0.4 (8)
Average	-19.5 (8)	Average	-0.5 (8)

with selected geometric parameters in Tables 2 and 3 according to the numbering scheme given in Figs. 1 and 2 (Johnson, 1976). A stereoview of the packing in the unit cell is given in Fig. 3.

**Related literature.** To examine substitution reactions (Leipoldt & Grobler, 1986) of  $\beta$ -diketone complexes of Rh<sup>I</sup> of the type [Rh( $\beta$ -diketone)(*L*)<sub>*n*</sub>] [where *L* are monodentate (*i.e.* *n* = 2) or bidentate (*i.e.* *n* = 1) electron-donating ligands, such as CO, ethylene or dienes], a knowledge of the relative *trans* influence of the different ligands is necessary. The *trans* influence of any ligand normally has an influence on the

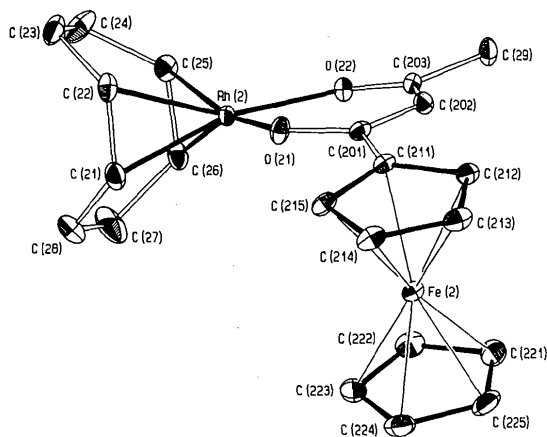


Fig. 1. Perspective view and atom labelling of molecule *B*. H atoms omitted for clarity. The numbering scheme in *A* and *B* is identical except for the first digit which is 1 for *A* and 2 for *B*. See also Fig. 2 and Tables 1–3.

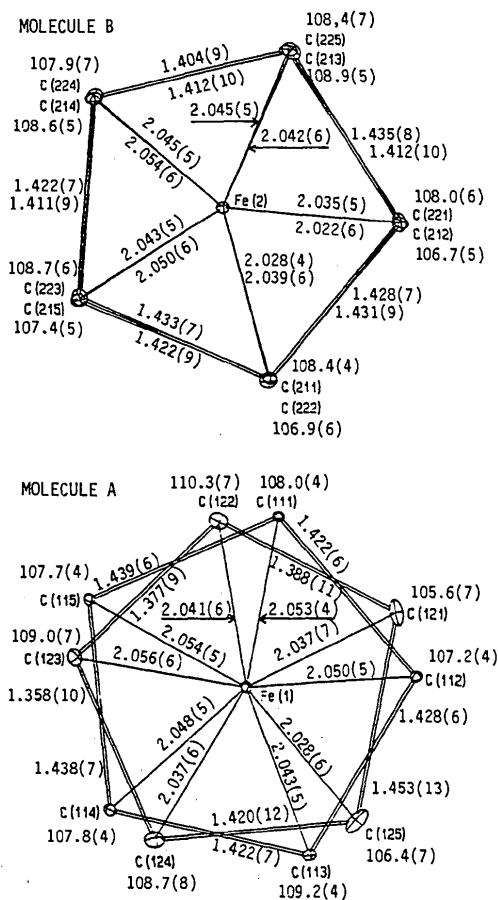


Fig. 2. A view from the top of the ferrocenyl groups. The bond angles ( $^{\circ}$ ) which are indicated above or below each atom number refer to the angle between two adjacent sides of the five-membered Cp rings. The upper printed bond lengths ( $\text{\AA}$ ) shown at each bond in molecule *B* are associated with atoms C(21 $_j$ ), while the lower printed bond lengths are associated with atoms C(22 $_j$ );  $j = 1, 2, \dots, 5$ .

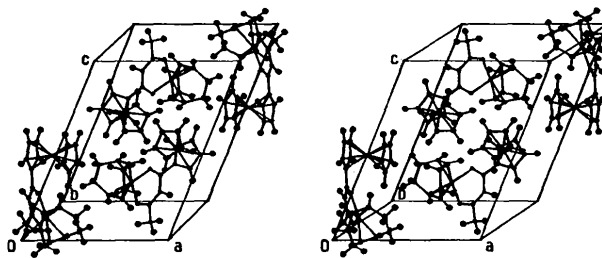


Fig. 3. Stereoview showing the packing in the unit cell.

metal–ligand bond strength and thus also on the bond lengths (Leipoldt, Basson, Bok & Gerber, 1978). Since it was shown that 1,5-cyclooctadiene (cod) does not influence the Rh–O bond lengths in  $\beta$ -diketone complexes of  $\text{Rh}^{\text{I}}$  (Leipoldt, Basson, Lamprecht, Bok & Schlebusch, 1980), the title compound was prepared in order to establish the effect the strong electron-donating ferrocenyl group has on the relative *trans* influence of the two  $\beta$ -diketone O atoms. The geometry of the approximately square-planar Rh centre, cod,  $\beta$ -diketone and ferrocenyl moieties of the title compound compare favourably with other structures (Graham, Lamprecht, Potgieter, Roodt & Leipoldt, 1990; Ibers & Snyder, 1962; Leipoldt, Basson, Lamprecht, Bok & Schlebusch, 1980; Roodt, Leipoldt, Swarts & Steyn, 1992; Takusagawa & Koetzle, 1979, and references therein; Luo, Barton & Robertson, 1990; Meetsma, Schoo, van de Grampel & Bosman, 1987, and references therein).

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## Structure of 5-Benzylamino-1-methyl-4-nitroimidazole

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**Abstract.** C<sub>11</sub>H<sub>12</sub>N<sub>4</sub>O<sub>2</sub>,  $M_r = 232.24$ , orthorhombic, *Pbca*,  $a = 6.962(4)$ ,  $b = 13.754(7)$ ,  $c = 22.974(12)$  Å,  $V = 2200(2)$  Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.40$ ,  $D_x = 1.402$  Mg m<sup>-3</sup>, graphite-monochromated Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å,  $\mu = 0.794$  mm<sup>-1</sup>,  $F(000) = 976$ ,  $T = 293$  K, final  $R = 0.035$  for 1143 unique observed [ $F \geq 4\sigma(F)$ ] reflections. The phenyl ring and the nitro group make angles of 50.8 and 3.6°, respectively, to the imidazole ring. An intermolecular hydrogen bond exists parallel to the  $b$  axis.

**Experimental.** Yellow crystals were obtained from 1,4-dioxane and were used for density measurements by flotation in *n*-heptane/CCl<sub>4</sub>. A prismatic crystal with approximate dimensions 0.1 × 0.2 × 0.4 mm was mounted for X-ray diffraction data collection on a Stoe Stadi-4 four-circle diffractometer with graphite-monochromated Cu  $K\alpha$  (reflection 200) radiation. Lattice and space group, *Pbca*, were determined from cell dimensions, observed symmetry and systematic absences. Unit-cell dimensions were obtained by least-squares refinement of setting angles of 30 reflections with  $30 \leq 2\theta \leq 50^\circ$ . X-ray intensities were collected in the  $\omega/2\theta$  scan mode up to a maximum  $\sin\theta/\lambda = 0.588$  Å<sup>-1</sup>, corresponding to  $2\theta_{\max} = 130^\circ$ , and for  $0 \leq h \leq 8$ ,  $0 \leq k \leq 16$ ,  $-27 \leq l \leq 27$ . Friedel pairs were also collected. Intensities of four standard reflections ( $\bar{2}20$ ,  $\bar{2}\bar{1}\bar{1}$ ,  $\bar{1}0\bar{4}$ ,  $00\bar{2}$ ), monitored every 60 min, showed an average decrease in inten-

sity of 1.92% in 96 h of radiation. The reflection intensities were rescaled using a linear regression. A total of 5911 reflections were measured. Symmetry-related reflections were averaged to give 1816 unique reflections ( $R_{\text{int, observed}, F} = 0.027$ ) of which 1143 were considered observed with  $F \geq 4\sigma(F)$ . Data reduction was with a locally adapted Stoe & Co. (1985) *REDU4* program. Lorentz and polarization corrections were applied, and absorption corrections were performed by the method of North, Phillips & Mathews (1968) based on the observed absorption of five reflections (302, 210, 200,  $\bar{2}\bar{1}\bar{1}$ , 111) as a function of  $\psi$ . Transmission factors varied between 0.81 and 1.00. Structure factors were calculated with scattering factors from Cromer & Waber (1974), and contracted H-atom form factors from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections were performed for all non-H atoms (Ibers & Hamilton, 1964). The phase problem was successfully solved by direct methods using *MULTAN82* (Main *et al.*, 1982) which revealed the positions of all non-H atoms. Full-matrix least-squares refinements were performed on  $F$ , first isotropically and then anisotropically. The 12 H atoms were located in a difference map. H-atom positions were refined and  $U_{\text{iso}}$  was fixed at  $0.05$  Å<sup>2</sup>.  $w = 4F^2/[\sigma^2(F^2) + (0.03F^2)^2]$ . Final  $R = 0.035$ ,  $wR = 0.035$ , with  $S = 2.05$ . Largest parameter shift/e.s.d. = 0.03. The ratio of reflections to refined parameters was 5.8. The residual electron density varied between  $-0.11$  and  $0.13$  e Å<sup>-3</sup>. All calculations were performed on a Digital PDP-11/73 and a Micro-VAX 2000 microcomputer using *SDP/VAX* (Enraf-Nonius, 1985) and *PARST* (Nardelli, 1983). An

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