

sponding carboxylate group are 6.3 and 10.8° for fluorobenzoate (*A*) and (*B*), respectively.

Although not involved in Ca<sup>2+</sup> binding, the 4-fluorobenzoate ion (*B*) is extensively involved in the hydrogen-bonding scheme. All six H atoms from the three water molecules are hydrogen-bonded to the O(3*B*) and O(4*B*) carboxylate O atoms. In addition, there is a C(2*A*)—H(2*A*)...O(*w*3<sup>1</sup>) hydrogen bond with H(2*A*)...O(*w*3<sup>1</sup>) 2.37, C(2*A*)...O(*w*3<sup>1</sup>) 3.346 (3), C(2*A*)—H(2*B*) 1.08 Å and the C(2*A*)—H(2*A*)...O(*w*3<sup>1</sup>) angle 149°. These parameters are well within the range reported for C—H...O hydrogen bonds (Taylor & Kennard, 1982).

The two F atoms in this structure are not involved in short interactions with water molecules, which is in contrast to the situation in calcium bis(2-fluorobenzoate) dihydrate. The closest immediate neighbors about each F atom are aromatic H atoms with the

shortest distance being F(4*B*)...H(3*A*)(1−*x*, *y*, 2−*z*) at 2.40 Å with the C(4*B*)—F(4*B*)...H(3*A*) angle 139°.

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## **$\mu$ -{4-Methyl-1,2,6-triphosphatricyclo[2.2.1.0<sup>2,6</sup>]heptane-*P*<sup>1</sup>,*P*<sup>2</sup>,*P*<sup>3</sup>}-tris[pentacarbonylchromium(0)]**

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**Abstract.** [ $\{\text{Cr}(\text{CO})_5\}_3(\text{C}_5\text{H}_9\text{P}_3)$ ],  $M_r = 738.19$ , monoclinic,  $P2_1/c$ ,  $a = 9.632(1)$ ,  $b = 10.995(1)$ ,  $c = 27.807(8)$  Å,  $\beta = 92.84(2)^\circ$ ,  $V = 2941(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.67$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.70926$  Å,  $\mu = 12.88$  cm<sup>-1</sup>,  $F(000) = 1464$ ,  $T = 295(2)$  K; final  $R = 0.054$  for 3858 reflections (including 54 unobserveds). The complex consists of the  $P_3$ -nortricyclane  $\text{CH}_3\text{C}(\text{CH}_2\text{P})_3$  (4-methyl-1,2,6-triphosphatricyclo[2.2.1.0<sup>2,6</sup>]heptane), which coordinates *via* the P atoms as a tridentate ligand to three  $\text{Cr}(\text{CO})_5$  groups. The structure of the  $P_3$ -nortricyclane ligand has already been determined by X-ray analysis [Zimmermann, Gomm, Köck & Ellermann (1986). *Acta Cryst.* C42, 1757–1759]. In the complex  $\text{CH}_3\text{C}(\text{CH}_2\text{P})_3[\text{Cr}(\text{CO})_5]_3$  the P—P distances are slightly longer [P(1)—P(2) 2.218(1), P(2)—P(3) 2.217(1) and P(3)—P(1)

2.213(1) Å] than in the free ligand. The P—Cr distances range from 2.331(1) to 2.345(1) Å. Bond lengths and bond angles within the  $\text{Cr}(\text{CO})_5$  groups are normal.

**Introduction.** Organocyclotriphosphanes and heteronortriacyclanes with a  $P_3$  unit, *e.g.* (*t*-BuP)<sub>3</sub>, P[Si(CH<sub>3</sub>)<sub>2</sub>]<sub>3</sub>P<sub>3</sub>, P[PSi(CH<sub>3</sub>)<sub>3</sub>]<sub>3</sub>P<sub>3</sub>, and CH<sub>3</sub>C(CH<sub>2</sub>P)<sub>3</sub> are of interest due to their unusual coordination properties. P[PSi(CH<sub>3</sub>)<sub>3</sub>]<sub>3</sub>P<sub>3</sub> (Fritz, 1982; Fritz, Härer & Schneider, 1982) shows no coordination tendency, whereas the donor properties of (*t*-BuP)<sub>3</sub> (Baudler, Salzer & Hahn, 1982) and P[Si(CH<sub>3</sub>)<sub>2</sub>]<sub>3</sub>P<sub>3</sub> (Fritz, Uhlmann, Hoppe, Hönle & von Schnering, 1982) to one or two metal centres are well established by X-ray analysis (Tebbe & Feher, 1984; Hönle & von Schnering, 1980). The compound CH<sub>3</sub>C(CH<sub>2</sub>P)<sub>3</sub> (Ellermann & Demuth, 1984) coordinates to one, two and three

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metals (Ellermann & Demuth, 1987). The complex type with three metal centres is now confirmed by the crystal structure of the title compound  $\text{CH}_3\text{C}(\text{CH}_2\text{P})_3\text{[Cr}(\text{CO})_5\text{]}_3$ .

**Experimental.** Recrystallization from tetrahydrofuran gave yellow crystals; a spherical crystal with  $r = 0.11(2)$  mm was covered with a thin film of epoxy adhesive; Philips PW 1100 diffractometer (Mo  $K\alpha$  radiation, graphite monochromator);  $\omega$ - $2\theta$  scans, scan width  $(0.750 + 0.345\tan\theta)^\circ$ , profile analysis with a modified Lehmann & Larsen (1974) procedure; lattice parameters improved by least-squares refinement program *GIVER* (Krogmann, 1966) on basis of 48 reflections with  $15.9 \leq 2\theta \leq 29.9^\circ$ ; spherical absorption correction applied:  $\mu R = 0.14(2)$ , absorption correction factor  $A = 1.234(1)$ ;  $[(\sin\theta)/\lambda]_{\max} = 0.54 \text{ \AA}^{-1}$ , index range  $h \pm 10$ ,  $k 0/11$ ,  $l 0/29$ ; six standard reflections (intensity variation 0.004), 14 824 reflections ( $3 \leq \theta \leq 22.5^\circ$ ) were measured; after averaging symmetrical equivalents 3858 [54 unobserved included,  $I < 3\sigma(I)$ ] remained;  $R_{\text{int}} = 0.023$ . Structure solved by direct methods, subsequent difference Fourier map revealed all C and O atoms, after refinement all H atoms could be found in a difference Fourier map; blocked least-squares refinement based on  $F$  and weights  $w = 1/\sigma^2(|F_o|)$ ; final cycles refined scale factor, coordinates, anisotropic temperature factors for non-hydrogen atoms; the isotropic temperature factor  $B$  of some H atoms ranged to negative values and therefore the  $B$  value for all H atoms was fixed at  $2.5 \text{ \AA}^2/8\pi^2$ ;  $R = 0.054$ ,  $wR = 0.022$ ,  $S = 2.11$ ; max. shift-to-e.s.d. ratio 0.33,  $\Delta\rho = -0.31$  to  $0.32 \text{ e \AA}^{-3}$ ; atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974). Computations employed the *CRYSTAN* system (Burzlaff, Gomm & Zimmermann, 1986) and *ORTEPII* (Johnson, 1977) for plotting purposes.

**Discussion.** Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1. Selected bond lengths and bond angles are reported in Table 2.\*

The molecular structure deduced from spectroscopic results by Ellermann & Demuth (1987) is verified. The complex (see Fig. 1) consists of three  $\text{Cr}(\text{CO})_5$  groups, each attached to one P atom of the  $\text{P}_3$ -nortricyclane by a  $\sigma$  bond. The solid-state structure of the free  $\text{P}_3$ -nortricyclane ligand has already been determined by X-ray analysis (Zimmermann, Gomm, Köck & Ellermann, 1986) and provides direct comparative data. The P-P distances of  $2.200(1) \text{ \AA}$  in the free ligand are

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44357 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atom coordinates ( $\times 10^4$ ) with e.s.d.'s in parentheses and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^2$ )

$$U_{\text{eq}} = (1/6\pi^2) \sum_i \sum_j \beta_{ij} (\mathbf{a}_i \cdot \mathbf{a}_j).$$

	$x$	$y$	$z$	$U_{\text{eq}}$
Cr(1)	1143 (1)	2562 (1)	4588 (1)	4.1
Cr(2)	1805 (1)	2279 (1)	2650 (1)	4.0
Cr(3)	3927 (1)	-1227 (1)	3828 (1)	4.2
P(1)	3082 (1)	2408 (1)	4122 (1)	3.3
P(2)	3300 (1)	2338 (1)	3333 (1)	3.4
P(3)	4225 (1)	889 (1)	3801 (1)	3.4
O(11)	2984 (3)	3810 (3)	5348 (1)	11.3
O(12)	1789 (2)	198 (2)	5122 (1)	8.2
O(13)	-706 (2)	1280 (2)	3833 (1)	7.0
O(14)	448 (3)	4987 (2)	4117 (1)	9.4
O(15)	-1418 (2)	2921 (2)	5148 (1)	8.8
O(21)	2984 (3)	4690 (2)	2330 (1)	8.8
O(22)	-437 (3)	3765 (2)	3100 (1)	10.1
O(23)	467 (2)	-70 (2)	2957 (1)	8.0
O(24)	4067 (3)	1007 (2)	2123 (1)	10.0
O(25)	-40 (3)	2084 (2)	1747 (1)	10.7
O(31)	7012 (3)	-1568 (2)	3721 (1)	9.8
O(32)	3393 (3)	-1444 (2)	2746 (1)	9.5
O(33)	864 (2)	-1036 (2)	4029 (1)	7.0
O(34)	4613 (3)	-1305 (2)	4896 (1)	8.2
O(35)	3575 (3)	-3933 (2)	3839 (1)	11.5
C(1)	4656 (3)	3341 (2)	4231 (1)	4.0
C(2)	4895 (3)	3260 (2)	3356 (1)	4.2
C(3)	5926 (3)	1649 (3)	3876 (1)	4.2
C(4)	5656 (3)	3027 (2)	3841 (1)	3.8
C(5)	6995 (3)	3773 (3)	3898 (1)	5.8
C(11)	2283 (4)	3352 (3)	5060 (1)	6.7
C(12)	1548 (3)	1065 (3)	4914 (1)	5.4
C(13)	7 (3)	1750 (3)	4113 (1)	4.7
C(14)	730 (3)	4072 (3)	4286 (1)	5.5
C(15)	-420 (3)	2775 (3)	4948 (1)	5.8
C(21)	2549 (3)	3785 (3)	2448 (1)	5.2
C(22)	406 (3)	3189 (3)	2942 (1)	6.0
C(23)	996 (3)	801 (3)	2848 (1)	5.4
C(24)	3213 (4)	1464 (3)	2329 (1)	5.8
C(25)	680 (3)	2185 (3)	2086 (1)	6.1
C(31)	5848 (3)	-1421 (3)	3754 (1)	6.2
C(32)	3588 (3)	-1324 (3)	3151 (1)	6.0
C(33)	2000 (3)	-1079 (3)	3944 (1)	4.9
C(34)	4336 (3)	-1245 (3)	4494 (1)	5.4
C(35)	3713 (4)	-2905 (3)	3840 (1)	6.8

only slightly lengthened by coordination [P(1)-P(2) 2.218 (1), P(2)-P(3) 2.217 (1), P(3)-P(1) 2.213 (1)  $\text{\AA}$ ]. This fact was previously observed in similar systems (Tebbe & Feher, 1984). The P-C distances also show a small elongation; but C(4)-C(5) has the same length within the limits of error. As expected the P-P-P and P-C-C angles are essentially unchanged (see Table 2).

The bond lengths and bond angles within the  $\text{Cr}(\text{CO})_5$  groups are in good agreement with other results, e.g.  $\text{P}_4(\text{SiMe}_2)_3\text{Cr}(\text{CO})_5$  (Hönle & von Schnering, 1980),  $(\text{C}_6\text{H}_5)_3\text{PCr}(\text{CO})_5$  (Plastas, Stewart & Grim, 1973). The C-Cr-C angles range from  $86.9(1)$  to  $93.0(1)^\circ$  and the P-Cr-C angles lie between  $176.8(1)$  and  $179.0(1)^\circ$ ; deviations of the Cr-C-O angles from linearity are small. The Cr atoms have approximately octahedral coordination. The distances Cr-C<sub>trans</sub> 1.862, Cr-C<sub>cis</sub> 1.892, C<sub>trans</sub>-O 1.137 and C<sub>cis</sub>-O 1.144  $\text{\AA}$  agree with previous values, e.g.  $\text{P}_4(\text{SiMe}_2)_3\text{Cr}(\text{CO})_5$  (Hönle & von Schnering, 1980): Cr-C<sub>trans</sub> 1.849, Cr-C<sub>cis</sub> 1.890, C<sub>trans</sub>-O 1.149 and C<sub>cis</sub>-O 1.139  $\text{\AA}$  (mean values). The notations *trans* and *cis* are related to the position of the P atom.

Table 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses

P(1)—P(2)	2.218 (1)	C(11)—O(11)	1.140 (4)
P(2)—P(3)	2.217 (1)	C(12)—O(12)	1.134 (4)
P(3)—P(1)	2.213 (1)	C(13)—O(13)	1.138 (4)
P(1)—C(1)	1.842 (3)	C(14)—O(14)	1.137 (4)
P(2)—C(2)	1.839 (3)	C(15)—O(15)	1.145 (4)
P(3)—C(3)	1.841 (3)	C(21)—O(21)	1.135 (4)
C(1)—C(4)	1.526 (4)	C(22)—O(22)	1.135 (4)
C(2)—C(4)	1.524 (4)	C(23)—O(23)	1.133 (4)
C(3)—C(4)	1.540 (4)	C(24)—O(24)	1.143 (4)
C(4)—C(5)	1.530 (4)	C(25)—O(25)	1.147 (4)
Cr(1)—P(1)	2.331 (1)	C(31)—O(31)	1.141 (4)
Cr(1)—C(11)	1.882 (3)	C(32)—O(32)	1.140 (4)
Cr(1)—C(12)	1.909 (3)	C(33)—O(33)	1.132 (4)
Cr(1)—C(13)	1.894 (3)	C(34)—O(34)	1.137 (4)
Cr(1)—C(14)	1.895 (3)	C(35)—O(35)	1.139 (4)
Cr(1)—C(15)	1.864 (3)		
Cr(2)—P(2)	2.326 (1)	C—H	0.90 (2)—1.01 (3)
Cr(2)—C(21)	1.890 (3)	C—H	0.95 (mean)
Cr(2)—C(22)	1.893 (3)		
Cr(2)—C(23)	1.896 (3)	H(11)···O(34 <sup>b</sup> )	2.56 (2)
Cr(2)—C(24)	1.886 (3)	H(21)···O(24)	2.76 (2)
Cr(2)—C(25)	1.864 (3)	H(22)···O(32)	2.70 (3)
Cr(3)—P(3)	2.345 (1)	H(32)···O(34 <sup>b</sup> )	2.75 (2)
Cr(3)—C(31)	1.883 (3)	H(51)···O(11 <sup>b</sup> )	2.75 (2)
Cr(3)—C(32)	1.898 (3)		
Cr(3)—C(33)	1.907 (3)		
Cr(3)—C(34)	1.876 (3)		
Cr(3)—C(35)	1.857 (3)		
P(3)—P(1)—P(2)	60.05 (2)	C(11)—Cr(1)—C(12)	88.2 (1)
P(1)—P(2)—P(3)	59.88 (2)	C(12)—Cr(1)—C(13)	91.3 (1)
P(2)—P(3)—P(1)	60.07 (2)	C(13)—Cr(1)—C(14)	90.2 (1)
P(2)—P(1)—C(1)	93.70 (6)	C(14)—Cr(1)—C(11)	90.4 (1)
P(3)—P(1)—C(1)	93.72 (6)	C(15)—Cr(1)—P(1)	176.8 (1)
P(1)—P(2)—C(2)	93.75 (6)	C(21)—Cr(2)—C(22)	87.3 (1)
P(3)—P(2)—C(2)	93.69 (6)	C(22)—Cr(2)—C(23)	91.0 (1)
P(1)—P(3)—C(3)	93.96 (6)	C(23)—Cr(2)—C(24)	92.6 (1)
P(2)—P(3)—C(3)	94.01 (6)	C(24)—Cr(2)—C(21)	89.1 (1)
P(1)—C(1)—C(4)	107.5 (2)	C(25)—Cr(2)—P(2)	176.9 (1)
P(2)—C(2)—C(4)	107.7 (2)	C(31)—Cr(3)—C(32)	90.5 (1)
P(3)—C(3)—C(4)	107.1 (2)	C(32)—Cr(3)—C(33)	93.0 (1)
C(1)—C(4)—C(2)	107.3 (2)	C(33)—Cr(3)—C(34)	89.5 (1)
C(2)—C(4)—C(3)	107.1 (2)	C(34)—Cr(3)—C(31)	86.9 (1)
C(3)—C(4)—C(1)	106.7 (2)	C(35)—Cr(3)—P(3)	179.0 (1)
C(1)—C(4)—C(5)	111.3 (2)	O(11)—C(11)—Cr(1)	178.7 (1)
C(2)—C(4)—C(5)	111.6 (2)	O(12)—C(12)—Cr(1)	177.6 (1)
C(3)—C(4)—C(5)	112.5 (2)	O(13)—C(13)—Cr(1)	178.1 (1)
H—C—H(methylene)	104(2)—108(2)	O(14)—C(14)—Cr(1)	177.5 (1)
H—C—H(methyl)	104(2)—114(2)	O(15)—C(15)—Cr(1)	176.6 (1)
P—C—H(methylene)	107.2(7)—108.6(7)	O(21)—C(21)—Cr(2)	179.4 (1)
Cr(1)—P(1)—P(2)	132.05 (5)	O(22)—C(22)—Cr(2)	177.0 (1)
Cr(1)—P(1)—P(3)	135.01 (6)	O(23)—C(23)—Cr(2)	177.3 (1)
Cr(1)—P(1)—C(1)	122.96 (9)	O(24)—C(24)—Cr(2)	177.3 (1)
Cr(2)—P(2)—P(1)	136.40 (6)	O(25)—C(25)—Cr(2)	177.1 (1)
Cr(2)—P(2)—P(3)	132.37 (5)	O(31)—C(31)—Cr(3)	177.8 (1)
Cr(2)—P(2)—C(2)	121.78 (9)	O(32)—C(32)—Cr(3)	176.5 (1)
Cr(3)—P(3)—P(1)	132.20 (6)	O(33)—C(33)—Cr(3)	176.6 (1)
Cr(3)—P(3)—P(2)	133.25 (6)	O(34)—C(34)—Cr(3)	176.9 (1)
Cr(3)—P(3)—C(3)	123.79 (9)	O(35)—C(35)—Cr(3)	178.8 (1)

Symmetry codes: (i)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (ii)  $-x, -y, -z$ .

The P—Cr bonds [P(1)—Cr(1) 2.331 (1), P(2)—Cr(2) 2.326 (1), P(3)—Cr(3) 2.345 (1) Å] lie in the middle of the range [2.252 (1) Å (Preston, Stewart, Plastas & Grim, 1972) to 2.448 (2) Å (Tebbe & Feher, 1984)] which is observed for this type of compound. The Cr—P—P angles and the Cr—P—C angles (see Table 2) indicate that the triangle of Cr atoms is slightly twisted with respect to the P<sub>3</sub> unit. On the basis of these distances and angles the molecule has the symmetry C<sub>i</sub>≡1.

The shortest intermolecular distances are between H and O atoms (see Table 2). All P···P, P···Cr and Cr···Cr intermolecular distances are considerably longer than the sum of van der Waals radii. The central

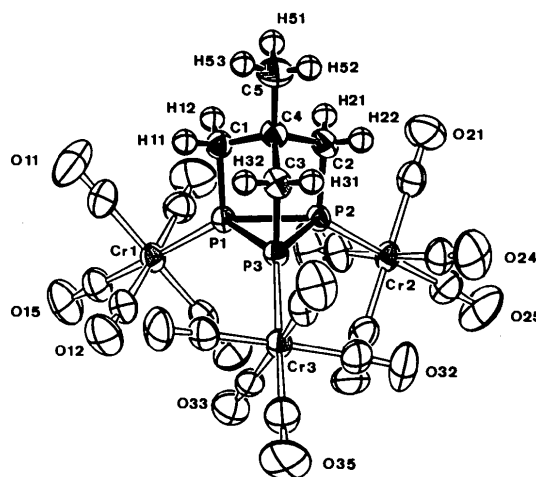


Fig. 1. Perspective view of the molecule with 50% probability thermal ellipsoids. The O atoms possess a two-digit symbol, whereby the first stands for the corresponding chromium atom and the second for the atom number. By looking along the Cr—P bond the atom number is laid down in a clockwise direction. The numbering of the carbonyl C atoms is the same as that of the neighbouring O atoms.

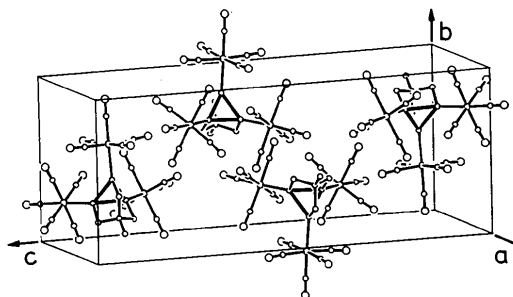


Fig. 2. Molecular packing in the crystal of CH<sub>3</sub>C[CH<sub>2</sub>PCr(CO)<sub>5</sub>]<sub>3</sub>.

points of the molecules form a distorted face-centred lattice. Fig. 2 gives a view of the crystal packing.

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## Structure of Tricarbonyl( $\eta^4$ -1,4-dibenzoyloxy-1,3-butadiene)iron(0)\*

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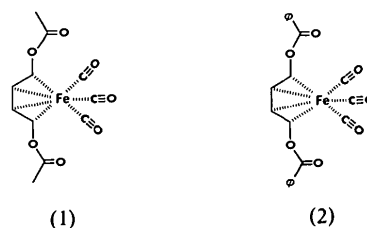
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**Abstract.**  $[\text{Fe}(\text{CO})_3(\text{C}_{18}\text{H}_{14}\text{O}_4)]$ ,  $M_r = 434.2$ , monoclinic,  $P2_1/c$ ,  $a = 6.152$  (2),  $b = 32.318$  (10),  $c = 10.210$  (4) Å,  $\beta = 104.26$  (3)°,  $V = 1967$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.47$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.802$  mm<sup>-1</sup>,  $F(000) = 888$ ,  $T = 293$  K,  $R = 0.048$  for 2681 observed reflections. The X-ray study confirms that the solid-state structure of tricarbonyl( $\eta^4$ -1,4-dibenzoyloxy-1,3-butadiene)iron(0) is as inferred from spectroscopic evidence. The molecule contains a diphenylester–vinyl unit bonded to the iron atom, to which are also bonded three carbonyl groups in an approximately trigonal arrangement. Both  $\varphi$ -COO–ester groups are planar and synclinal to each other about the central C(5)–C(6) bond. There is only one C–H...O intermolecular approach  $< 3.3$  Å. The crystal structure is stabilized by van der Waals forces.

**Introduction.** As part of our investigations concerning complexes between  $\text{Fe}(\text{CO})_3$  and vinyl ligands with various substituents, including phenyls, esters and acyl groups, the title compound (2) was synthesized. It was obtained from the reaction of tricarbonyl( $\eta^4$ -*syn*-1,4-diacetoxy-1,3-butadiene)iron(0) (1) (170 mg, 1 mmol) and MeLi (6.2 ml, 0.8 M) with 15 ml of anhydrous tetrahydrofuran (THF) at 195 K in an argon atmosphere, to give a red solution. After 10 min of stirring, benzoic anhydride (1.13 g, 5 mmol) in 5 ml of

THF was added, the temperature was raised to 298 K and stirring continued for 2 h. The brown product was purified by chromatographic methods and recrystallized from *n*-hexane to give pale yellow crystals, m.p. 455–456 K. Compound (2) was characterized by NMR, IR and mass spectrometry. The X-ray structure determination was undertaken to elucidate the stereochemistry of (2).



**Experimental.** Pale yellow crystal,  $0.28 \times 0.12 \times 0.40$  mm. Nicolet R3 four-circle diffractometer, graphite-monochromated Mo  $K\alpha$  radiation. Lattice parameters from 25 machine-centred reflections with  $5.0 < 2\theta < 20.2^\circ$ , 3482 reflections with  $3 < 2\theta < 50^\circ$  for two octants, 2681 independent with  $I > 2.5\sigma(I)$ , index range  $h -7 \rightarrow 7$ ,  $k 0 \rightarrow 38$ ,  $l 0 \rightarrow 12$ ,  $\omega$ -scan mode, variable scan speed, scan width  $1.0^\circ$  ( $\theta$ ), two standard reflections (041, 052) monitored every 50 measurements, Lp corrections and absorption ignored. Structure solved by combination of direct methods and partial structure expansion by iterative  $E$ -Fourier

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