

Table 6. Powder diffraction data of quinhydrone

Triclinic		Monoclinic	
<i>d</i>	Intensity	<i>d</i>	Intensity
5.44 Å	<i>s</i>	5.34 Å	<i>s</i>
3.77	<i>w</i>	3.98	<i>w</i>
3.12	<i>m</i>	3.58	<i>m</i>
3.06	<i>s</i>	3.22	<i>m</i>
2.78	<i>m</i>	3.08	<i>s</i>
2.66	<i>m</i>	2.87	<i>w</i>
2.54	<i>m</i>	2.72	<i>w</i>
2.32	<i>w</i>	2.59	<i>m</i>
2.16	<i>m</i>	2.42	<i>m</i>
2.01	<i>w</i>	2.30	<i>m</i>
1.90	<i>w</i>	2.15	<i>w</i>
1.82	<i>w</i>	2.09	<i>w</i>
1.76	<i>w</i>	1.80	<i>w</i>
1.75	<i>w</i>	1.69	<i>w</i>
1.66	<i>w</i>	1.59	<i>m</i>
1.60	<i>m</i>		
1.51	<i>w</i>		
1.16	<i>w</i>		

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The Crystal Structure of α -Keto-1,1'-trimethyleneferrocene*

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The crystal structure of α -keto-1,1'-trimethyleneferrocene has been determined by three-dimensional X-ray diffraction analysis. This compound, $C_{13}H_{12}OFe$, crystallizes in the centrosymmetric monoclinic space group $P2_1/a$. The unit cell contains four molecules and has the dimensions

$$a = 22.981 \pm 0.002, b = 7.381 \pm 0.001, c = 5.833 \pm 0.001 \text{ \AA}; \beta = 93.38 \pm 0.02^\circ.$$

A trial structure was obtained from the locations of the iron-iron vectors in the (001) Patterson projection and was refined in both two and three dimensions by the method of least squares. The final reliability factor, R , was 0.067.

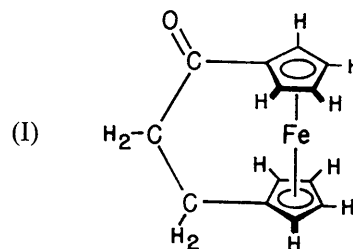
The dihedral angle between the best planes through the two nearly planar cyclopentadienyl rings is about 8.8° . The average ring carbon-carbon bond distance is $1.424 \pm 0.010 \text{ \AA}$ and the average iron-carbon bond distance is $2.039 \pm 0.006 \text{ \AA}$. The principal thermal motion is a vibration of the entire molecule in a direction roughly perpendicular to the ab plane.

Introduction

The stable bridged 'sandwich' compound α -keto-1,1'-trimethyleneferrocene, $C_{13}H_{12}OFe$ (I), may be prepared by the procedure of Rinehart & Curby (1957).

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The determination of the crystal structure of this compound was prompted by its unusual geometry and more specifically by recent studies (Hill & Richards, 1961; Richards & Hill, 1959) on the reactivities of α -metallocenyl carbonium ions.

These X-ray results are of significance in the interpretation of the solvolysis rate of α -acetoxy-1,1'-trimethyleneferrocene which is depressed by a surprisingly small degree (*i.e.* by a factor of 291) when compared with an analogue in which the α -carbon atom can be coplanar with the cyclopentadienyl ring to which it is attached, *e.g.* α -acetoxyethylferrocene. The reasons for this small rate difference will be discussed in detail in a paper on the mechanistic interpretation of these solvolytic processes.

Experimental

α -Keto-1,1'-trimethyleneferrocene crystallizes from *n*-heptane in the form of red-orange monoclinic needles melting at 144.0–144.5°. From a crystal mounted to rotate about the needle axis, *c*, rotation and equi-inclination Weissenberg photographs were obtained for layers 0 through 3. The absence on these photographs of reflections $0k0$ with *k* odd and $h0l$ with *h* odd is indicative of the centrosymmetric space group $P2_1/a$.

The unit-cell dimensions were obtained from Straumanis-type, zero-layer Weissenberg photographs taken at room temperature with nickel-filtered copper radiation using crystals mounted about the *b* and *c* axes. The measured Bragg angles for 189 $h0l$ and $hk0$ reflections were used in a least-squares calculation of the unit-cell dimensions; the resulting values are

$$\begin{aligned} a &= 22.981 \pm 0.002, \quad b = 7.381 \pm 0.001, \\ c &= 5.833 \pm 0.001 \text{ \AA}; \quad \beta = 93.38 \pm 0.02^\circ, \\ [\lambda(\text{Cu } K\alpha_1) &= 1.54051; \quad \lambda(\text{Cu } K\alpha_2) = 1.54433 \text{ \AA}] \end{aligned}$$

where the uncertainties listed are about ten times the calculated standard deviations. Absorption and eccentricity parameters were included in the least-squares calculation but were found to be negligible.

The density measured by flotation in mixed solvents was found to be 1.60 g.cm⁻³; the density calculated on the basis of four molecules of C₁₃H₁₂OFe (M.W. 240.1) in the unit cell is 1.61 g.cm⁻³.

In order to minimize absorption errors a small acicular crystal, approximately 0.1 mm. in diameter and with roughly hexagonal cross section, was chosen for the *c*-axis intensity photographs. Reflections $hk0$ to $hk5$, inclusive, were recorded using the multiple-film, equi-inclination Weissenberg technique. (α -Keto-1,1'-trimethyleneferrocene is quite stable to X-rays, there being no apparent change in the crystal after exposure to radiation for over 250 hours. However, all X-ray photographs taken from crystals of this compound exhibit a high background due to fluorescent scattering by the iron atoms.) Some difficulty was experienced in cutting a crystal in order to obtain satis-

factory diffraction photographs about the *b* axis. The long, slender needles fractured unevenly; after several dozen unsuccessful attempts, a rather unsatisfactory fragment was mounted. From this crystal the reflections $h0l$ to $h2l$, inclusive, were recorded as before. Intensities were estimated by visual comparison with a graduated scale prepared from the crystal mounted to rotate about the *c* axis. After correction for Lorentz and polarization effects, the F^2 values from the several photographs were put on the same arbitrary scale using a weighted least-squares procedure. Because of a number of serious discrepancies in intensities of high-angle reflections measured about the two axes (and, in retrospect, apparently caused by misorientation of the crystal mounted along *b*), the *b*-axis data were used only to scale the *c*-axis data, the latter being used in all subsequent calculations.

Determination and refinement of the structure

The *x* and *y* coordinates of the iron atom were readily determined from iron-iron vector peaks in the (001) Patterson projection. Using these coordinates, the contributions of the iron atom to about 100 of the largest $hk0$ structure factors were calculated. For about 25 of these reflections the iron atom contributions were sufficiently large to determine with certainty the signs of the structure factors. A (001) electron density map prepared from these phased structure factors clearly showed the positions of the iron atom, the non-carbonyl carbon atoms of the bridge and one carbon atom in each ring. The atoms of the carbonyl group and the rest of the ring atoms were poorly resolved owing to overlaps in projection. However, from consideration of the geometry of the molecule it was possible to assign trial coordinates to all atoms.

The two-dimensional structure was refined rapidly using a block-diagonal least-squares program written for the Burroughs 220 computer. This program minimizes the quantity $\sum w(F_o^2 - F_c^2)^2$. Atomic form factors for carbon and oxygen were an average of the values of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) and Hoerni & Ibers (1954) and for iron were taken from Thomas & Umeda (1957). Each observation was weighted according to the function $1/w = 1/f_c$, where f_c is the form factor of carbon at the calculated value of $\sin \theta/\lambda$. (We have found this weighting function frequently useful for rapid convergence in the initial stages of least-squares refinement.) After several least-squares cycles using individual isotropic temperature factors, the *R* index, based on observed reflections only, was 0.085.

From packing considerations, approximate *z* coordinates were calculated for all atoms. The first cycle of three-dimensional structure-factor calculations and least-squares refinement was run using isotropic temperature factors with $B = 3.0 \text{ \AA}^2$ for all atoms; the observations were weighted as before. The *R* index was 0.50 and there were large negative *z* shifts indicated for

Table 1. Final heavy atom parameters and their calculated standard deviations
All values have been multiplied by 10^4 . The temperature factors are expressed in the form
 $T = \exp(-B_{11}h^2 - B_{22}k^2 - B_{33}l^2 - B_{12}hk - B_{13}hl - B_{23}kl)$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> ₁₁	<i>B</i> ₂₂	<i>B</i> ₃₃	<i>B</i> ₁₂	<i>B</i> ₁₃	<i>B</i> ₂₃
Fe	1515(-4)	1259(1)	1913(1)	12(-2)	75(2)	211(4)	-4(1)	-3(1)	4(3)
O	955(3)	4880(7)	6090(9)	26(2)	188(13)	274(21)	34(7)	26(9)	-83(22)
C(1)	1584(3)	3702(8)	3418(12)	12(1)	88(11)	264(25)	-13(6)	-12(9)	-52(21)
C(2)	1925(3)	2429(9)	4727(11)	14(2)	115(12)	262(26)	-9(6)	-32(10)	-13(24)
C(3)	2350(3)	1701(10)	3344(12)	12(2)	157(15)	293(28)	8(6)	-40(10)	-8(26)
C(4)	2278(3)	2544(9)	1141(14)	12(2)	115(13)	404(33)	-15(7)	10(11)	9(29)
C(5)	1794(3)	3767(8)	1161(12)	15(2)	85(11)	264(25)	-23(6)	-11(10)	55(22)
C(6)	634(3)	985(8)	1599(13)	12(2)	101(13)	344(29)	-22(6)	-4(11)	-37(24)
C(7)	892(3)	-438(9)	3029(12)	17(2)	120(13)	243(25)	-28(7)	10(10)	15(24)
C(8)	1285(3)	-1426(8)	1736(14)	14(2)	56(11)	446(33)	-9(6)	23(12)	3(24)
C(9)	1275(4)	-637(9)	-508(13)	22(2)	116(13)	297(29)	-15(8)	-26(12)	-125(27)
C(10)	873(3)	815(8)	-580(12)	14(2)	102(12)	244(26)	-6(6)	-28(10)	-52(22)
C(11)	1014(3)	4421(8)	4123(12)	15(2)	78(11)	256(25)	-3(6)	18(10)	-49(22)
C(12)	501(3)	4343(9)	2391(12)	14(2)	108(12)	275(26)	22(6)	1(10)	-47(24)
C(13)	224(3)	2460(9)	2396(13)	11(2)	148(14)	351(31)	-6(7)	8(11)	-68(29)

many atoms; accordingly, the molecule was moved as a unit by -0.05 in z and another structure-factor least-squares calculation was performed. R for this calculation dropped to 0.37 and the shifts in the z coordinates, although still mostly negative, were considerably smaller. Several more least-squares cycles, in which the individual isotropic temperature factors were permitted to shift, saw R decrease rapidly to 0.12 . At this point anisotropic temperature parameters were introduced for the iron atom and the weighting function was changed to that suggested by Hughes (1941). After two more least-squares cycles R had dropped to 0.092 . Contributions of the hydrogen atoms were then included in the structure factors, based on assumed C-H bond distances of 1.0 Å and isotropic temperature factors equal to those of the heavy atoms to which they are attached. The parameters of the hydrogen atoms were not refined. Anisotropic temperature factors for the oxygen atom and carbon atoms were introduced, and R dropped to 0.080 after four more least-squares cycles.

The weighting scheme was then changed to

$$\sigma(F^2) = 1/\sqrt{w} = KF_o^2 \text{ for } F_o \geq 3.0 \\ = 3KF_o \text{ for } F_o < 3.0,$$

which we believe adequately represents the uncertainties in F_o^2 . After five more cycles of least-squares, R was 0.067 and no parameter shift was larger than one-third of its standard deviation; the refinement was considered complete.

A total of 1466 reflections were included in the final structure-factor calculation, of which 1196 contributed to the least-squares sums (and to the R index). Of the latter, 14 are 'less than' reflections with calculated structure factors larger than their estimated threshold values. Table 1 lists the final heavy-atom parameters and their standard deviations; Table 2 gives the assumed parameters of hydrogen atoms. The standard deviations in the heavy-atom parameters were calculated from the diagonal terms of the inverse block-diagonal matrices. The final observed and calculated structure factors are given in Table 3.

The observed structure factors and their calculated signs were used in the calculation of a three-dimensional electron density map, shown in Fig. 1. A three-dimensional difference map, shown in Fig. 2, was also prepared using ΔF 's derived from structure factors calculated from only the heavy atoms, but with the signs of the F_o 's being those of the final F_c 's. Electron

Table 2. Assumed hydrogen atom parameters

The temperature factors are expressed in the form
 $T = \exp(-B \sin^2 \theta/\lambda^2)$.

	Bonded to	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(14)	C(2)	0.187	0.211	0.641	3.5
H(15)	C(3)	0.267	0.075	0.374	4.4
H(16)	C(4)	0.252	0.228	-0.020	4.1
H(17)	C(5)	0.164	0.453	0.019	3.2
H(18)	C(7)	0.080	-0.063	0.465	3.9
H(19)	C(8)	0.153	-0.246	0.228	3.7
H(20)	C(9)	0.153	-0.109	-0.183	3.7
H(21)	C(10)	0.073	0.162	-0.192	2.7
H(22)	C(12)	0.066	0.455	0.076	3.8
H(23)	C(12)	0.023	0.531	0.264	3.8
H(24)	C(13)	-0.015	0.245	0.140	4.4
H(25)	C(13)	0.013	0.219	0.403	4.4

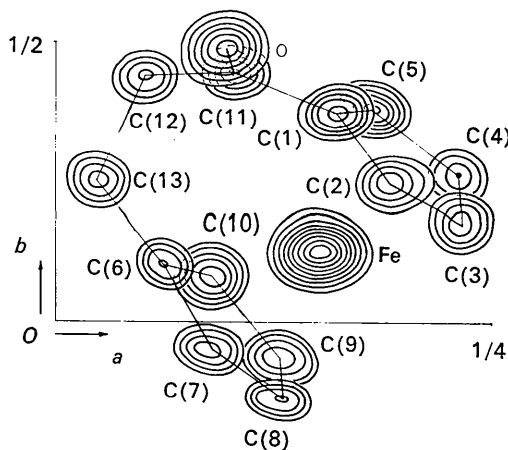


Fig. 1. The final composite three-dimensional electron density map projected onto (001). The contours are drawn at 4, 5, 6, ... e.Å⁻³, except for the iron atom, where the contours are at 5, 10, 15, ... e.Å⁻³.

Table 3. Observed and calculated structure factors

The three columns within each group contain the values of h, 10F_o and 10F_c. Observed values indicated by an asterisk (*) were of doubtful validity and were omitted from the least-squares calculations and from the R index.

Table with 30 columns and 1000+ rows of numerical data. Each row represents a data point with columns for h, 10F_o, and 10F_c values, often accompanied by a group label (e.g., h01, h02) and a sub-index (e.g., 1, 2, 3).

Table 4. *Magnitudes and direction cosines of the principal axes of the thermal ellipsoids*

The direction cosines, q , are taken relative to a , b and c^* . The root mean square deviation, U , is given by $(B/8\pi^2)^{\frac{1}{2}}$.

	Axis						Axis	Axis					
	i	B_i	U_i	q_{ia}	q_{ib}	q_{ic}^*		i	B_i	U_i	q_{ia}	q_{ib}	q_{ic}^*
Fe	1	2.93	0.192	-0.346	0.061	0.926	C(7)	1	4.19	0.231	0.854	-0.509	0.122
	2	2.39	0.174	0.925	-0.136	0.379		2	3.32	0.205	-0.003	0.182	0.983
	3	1.61	0.143	0.148	0.989	-0.005		3	1.99	0.159	0.517	0.841	-0.139
O	1	6.20	0.279	0.879	0.468	0.099	C(8)	1	6.11	0.278	0.138	-0.003	0.994
	2	4.49	0.239	0.257	-0.602	0.763		2	2.99	0.194	0.975	-0.169	-0.108
	3	2.64	0.183	-0.397	0.647	0.639		3	1.16	0.121	0.168	0.986	-0.016
C(1)	1	3.78	0.219	-0.271	-0.165	0.940	C(9)	1	5.32	0.260	0.801	0.090	-0.567
	2	2.78	0.188	0.846	-0.509	0.179		2	4.26	0.232	0.535	-0.546	0.660
C(2)	3	1.53	0.139	0.457	0.844	0.292	3	1.74	0.148	0.264	0.832	0.494	
	1	4.30	0.232	-0.610	0.055	0.772	C(10)	1	4.05	0.226	-0.618	-0.122	0.758
	2	2.72	0.186	-0.437	0.808	-0.407		2	2.70	0.185	-0.637	0.655	-0.424
3	2.08	0.162	0.659	0.587	0.489	3		1.81	0.152	0.458	0.746	0.497	
C(3)	1	4.70	0.243	-0.502	-0.152	0.836	C(11)	1	3.80	0.219	0.483	-0.193	0.868
	2	3.41	0.208	0.035	0.980	0.197		2	2.86	0.190	0.874	0.107	-0.446
	3	1.87	0.154	0.863	-0.128	0.512		3	1.61	0.143	-0.001	0.975	0.220
C(4)	1	5.48	0.264	0.034	0.021	0.999	C(12)	1	3.95	0.224	-0.339	-0.383	0.849
	2	2.98	0.194	-0.684	0.728	-0.012		2	3.26	0.203	0.756	0.431	0.514
	3	1.97	0.158	0.727	0.685	-0.018		3	1.75	0.149	-0.558	0.817	0.127
C(5)	1	4.14	0.230	-0.595	0.358	0.701	C(13)	1	4.96	0.251	0.053	-0.321	0.947
	2	3.02	0.196	0.700	-0.205	0.704		2	3.05	0.197	-0.211	0.922	0.319
	3	1.45	0.135	0.392	0.911	-0.118		3	2.31	0.171	0.974	0.217	0.047
C(6)	1	4.72	0.244	-0.074	-0.105	0.990							
	2	3.09	0.198	0.767	-0.640	0.012							
	3	1.51	0.138	0.635	0.761	0.146							

and are shown in Fig. 4. The standard deviations in the positional parameters (Table 1) are approximately 0.007 Å for the oxygen and carbon atoms and 0.0009 Å for the iron atom. These values correspond to uncertainties of approximately 0.010 Å for the C-C and C-O bonds and 0.007 Å for the Fe-C bonds. The excellent agreement among the ten C-C bond lengths in the rings indicates that these standard deviations are realistic.

Temperature parameters

The magnitudes and orientations of the thermal ellipsoids for each of the heavy atoms were calculated by the method of Rollett & Davies (1955) and are listed in Table 4. The anisotropies in the thermal motions are sizable. The major and minor axes of the ellipsoids for all atoms except the oxygen atom and C(7) are oriented approximately along the c and a axes, respectively, suggesting a relatively large lattice vibration. (Since only c -axis data were used in the refinement of the structure, the thermal anisotropy may be, in part at least, an artifact arising from systematic errors in the data.) Superimposed on this vibration are the motions of the individual atoms; the libration of the ring carbon atoms may be clearly seen in Fig. 3.

Molecular geometry

The least-squares planes (Schomaker, Waser, Marsh & Bergman, 1959) through each of the cyclopentadienyl rings and through the carbonyl group are given in Table 5. The ring atoms do not deviate significantly from coplanarity. The three-carbon bridge causes tilting of the two cyclopentadienyl rings so they are no longer parallel as in the parent compound, ferrocene,

but without causing noticeable ring bond distortions. It also appears that there is little strain in the α -keto-trimethylene bridge, since the bond lengths and angles are quite close to the expected values. The dihedral angle between the cyclopentadienyl rings, defined here as the acute angle formed by the normals to the best planes, is 8.8°.

Table 5. *Best planes through the rings and the carbonyl group*

The values q_i are the direction cosines of the normal to the best plane with respect to the orthogonal axes a , b , and c^* . Atoms indicated by an asterisk (*) were not included in the calculation of the plane.

Direction cosines	Atom	Deviation
$q_a = 0.613$	C(1)	-0.005 Å
$q_b = 0.731$	C(2)	-0.001
$q_c^* = 0.300$	C(3)	0.006
	C(4)	-0.010
	C(5)	0.009
	*C(11)	0.301
	*Fe	1.640
	*Origin	4.748
$q_a = 0.722$	C(6)	-0.006
$q_b = 0.622$	C(7)	0.003
$q_c^* = 0.303$	C(8)	0.001
	C(9)	-0.004
	C(10)	0.006
	*C(13)	-0.123
	*Fe	-1.640
	*Origin	1.741
$q_a = 0.246$	C(1)	-0.012
$q_b = 0.929$	C(11)	0.039
$q_c^* = -0.276$	C(12)	-0.012
	O	-0.012
	*C(13)	-1.462
	*Origin	-2.870

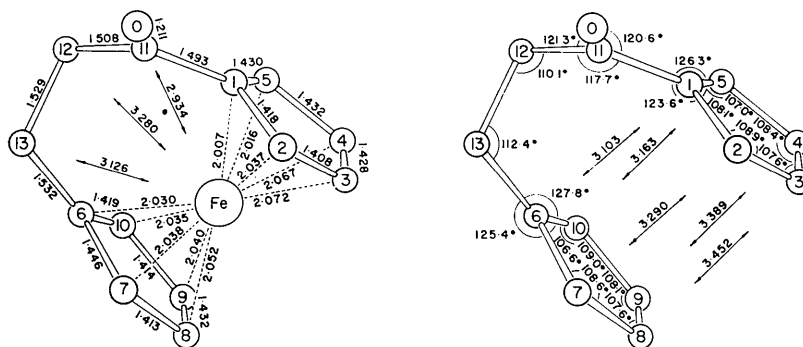
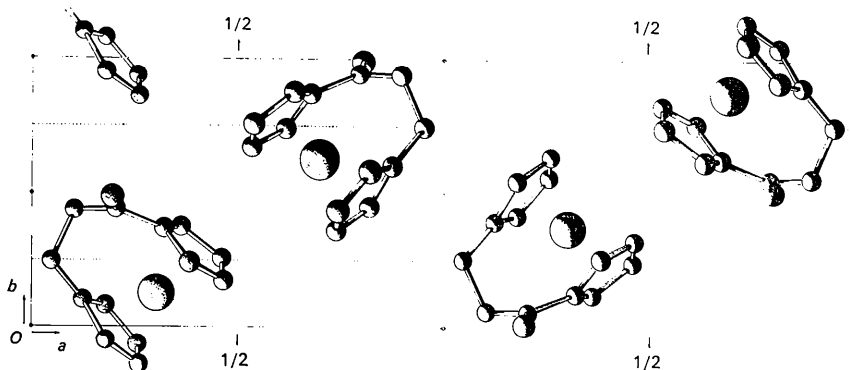


Fig. 4. Bond angles and interatomic distances.

Fig. 5. The structure viewed along the c axis.

In the crystal structure of ferrocene as determined by Dunitz, Orgel & Rich (1956) the iron atoms lie at centers of symmetry and the rings are therefore staggered by exactly 36° . In our bridged ketone the rings are more nearly eclipsed, being staggered by only 11.8° . (The degree of staggering is defined here as the angle between the lines from the centroid of each ring to the carbon atom attached to the bridge after the lines have been projected onto an 'average' plane. The 'average' plane is taken so that its normal bisects the acute angle formed by the normals to the best planes through the two rings.) The average ring C-C bond length in ferrocene is $1.403 \pm 0.020 \text{ \AA}$ and the average Fe-C bond is $2.045 \pm 0.010 \text{ \AA}$; for the bridged ketone these bonds are $1.424 \pm 0.010 \text{ \AA}$ and $2.039 \pm 0.006 \text{ \AA}$, respectively. Thus the two determinations agree well within the estimated standard deviations.

Table 6. Intermolecular distances less than 3.7 \AA

The atoms in the first column belong to the reference molecule at x, y, z .

From atom	To atom	In molecule at	Distance
C(11)	C(8)	$x, 1+y, z$	3.439
O	C(13)	$\bar{x}, 1-y, 1-z$	3.501
O	C(5)	$x, y, 1+z$	3.532
O	C(12)	$\bar{x}, 1-y, 1-z$	3.559
O	C(10)	$x, y, 1+z$	3.585
C(10)	C(13)	$\bar{x}, \bar{y}, \bar{z}$	3.606
C(4)	C(9)	$\frac{1}{2}-x, \frac{1}{2}+y, \bar{z}$	3.624
C(12)	C(8)	$x, 1+y, z$	3.637
C(12)	C(12)	$\bar{x}, 1-y, \bar{z}$	3.642
C(6)	C(6)	$\bar{x}, \bar{y}, \bar{z}$	3.668

Molecular packing

The molecular packing as viewed along the c axis is shown in Fig. 5 and the shortest intermolecular distances are listed in Table 6. The H-H and H-heavy atom distances are not included since the coordinates of the hydrogen atoms are only assumed.

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