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The Structure of Diacetylruthenocene

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Crystals of diacetylruthenocene are triclinic with two molecules in a unit cell of dimensions

$$a=5\cdot73,\ b=8\cdot15,\ c=13\cdot70\ \text{Å},\ \alpha=77\cdot8^\circ,\ \beta=85\cdot1^\circ,\ \gamma=69\cdot4^\circ,$$

space group $P\bar{1}$. The structure has been determined from Patterson and electron-density projections along the *a*- and *b*-axes. The molecular configuration is very close to *cis*.

In ferrocene (Dunitz, Orgel & Rich, 1956) and two of its derivatives, dibenzoylferrocene (Struchkov, 1956) and diindenyl iron (Trotter, 1958), X-ray analyses have shown that the five-membered rings are staggered, with substituents in *gauche* positions. In ruthenocene (Hardgrove & Templeton, 1959) and bisindenylruthenium (Webb & Marsh, 1962) on the other hand, the five-membered rings are eclipsed, with the six-membered rings in the latter compound in the *cis* configuration. The structure of diacetylruthenocene has now been examined to determine its molecular configuration.

The crystals are yellow needles elongated along *a*, with (010) developed and smaller (001) faces. The cell data were determined from various films and on the G. E. Spectrogoniometer, with single crystal orienter.

Crystal data (λ, Cu $K\alpha=1\cdot5418$, λ, Mo $K\alpha=0\cdot7107$ Å).
Diacetylruthenocene, $C_{14}H_{14}O_2Ru$, $M=316\cdot0$, m.p. = 149–150 °C.

Triclinic,

$$a=5\cdot73\pm0\cdot01,\ b=8\cdot15\pm0\cdot01,\ c=13\cdot70\pm0\cdot02\ \text{Å};$$
$$\alpha=77\cdot8\pm0\cdot1^\circ,\ \beta=85\cdot1\pm0\cdot1^\circ,\ \gamma=69\cdot4\pm0\cdot1^\circ.$$

$U=585\cdot5$ Å^3.
$D_m\sim1\cdot8$ g.cm^{-3}.
D_x (with $Z=2$) $=1\cdot78$ g.cm^{-3}.
$F(000)=316$.
Absorption coefficients for X-rays,

$$\lambda=1\cdot5418\ \text{Å},\ \mu=114\ \text{cm}^{-1};$$
$$\lambda=0\cdot7107\ \text{Å},\ \mu=14\cdot3\ \text{cm}^{-1}.$$

Space group $P1$ or $P\bar{1}$. $P\bar{1}$ from structure analysis.

Intensity data for $0kl$ and $h0l$ reflexions were measured with a scintillation counter using Mo $K\alpha$ radiation (Zr filter and pulse height analyser). The ruthenium position was determined from Patterson projections, and the parameters of the other atoms were determined, and all the parameters refined, by successive Fourier and difference syntheses. The final discrepancy factors were 0·14 for 242 $0kl$ reflexions and 0·15 for 164 $h0l$ reflexions (tables of structure factors are available from the author). The positional parameters are listed, as fractions of the unit cell edges, in Table 1, the final thermal parameters being $B_{Ru}=2\cdot5$, $B_{C,O}=4\cdot5$ Å^2. The oxygen atoms were distinguished by their distinctly greater peak heights in the Fourier and difference syntheses for both projections.

Table 1. *Final positions parameters*

Atom	*x*	*y*	*z*
Ru	0·1162	0·1500	0·2396
C1	0·298	0·026	0·123
2	0·392	0·146	0·162
3	0·194	0·324	0·133
4	0·007	0·293	0·093
5	0·056	0·128	0·087
6	0·142	−0·026	0·388
7	0·226	0·121	0·402
8	0·045	0·271	0·383
9	−0·157	0·239	0·349
10	−0·094	0·045	0·353
11	0·463	−0·165	0·135
12	0·329	−0·223	0·404
13	0·714	−0·262	0·178
14	0·239	−0·354	0·394
O1	0·384	−0·273	0·113
2	0·530	−0·244	0·441

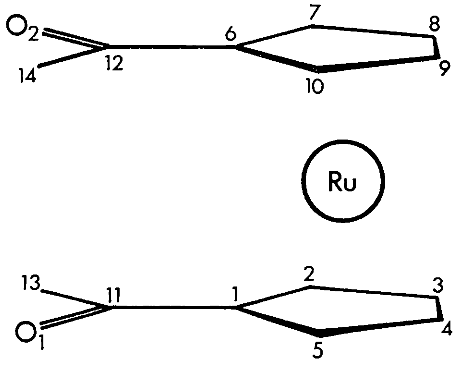

Fig. 1. Molecular configuration.

The molecular configuration is *cis* (Fig. 1), but with one acetyl group rotated 180° with respect to the other. The angles between corresponding bonds in the five-membered rings indicate a rotation of only a few degrees (which is however apparent on the *a*-axis electron-density projection) from the *cis* configuration.

Accurate molecular dimensions are not available from this two-dimensional study, and since the object was the determination of the molecular configuration, no three-dimensional analysis was carried out. The acetocyclopentadienyl residues are approximately planar and parallel, and the mean molecular dimensions are Ru–C = 2·18 Å (omitting one very short distance from the averaging), $C_{ar}-C_{ar}$ = 1·41 Å, other C–C = 1·48 Å, C=O = 1·23 Å, ∠ C–C–C (in five membered rings) = 108°.

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Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

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On the most probable differences occurring in the Lipson-Hesse difference method for indexing powder photographs.

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During our work we have found that the method described by Hesse (1948) and Lipson (1949) is one of the most powerful for indexing powder photographs with orthorhombic, hexagonal or tetragonal unit cells. But we have learned also that it is very important to know exactly what kind of differences may occur at all.

If we form the differences of the values

$$Q_{hkl}=4\sin^2\theta_{hkl}=h^2a^{*2}+k^2b^{*2}+l^2c^{*2}+2hka^*b^*\cos\gamma^*+2klb^*c^*\cos\alpha^*+2hla^*c^*\cos\beta^*,$$

we get in the monoclinic system (1st setting)

$$Q_{hkl}-Q_{hkl'}=N_3c^{*2}, \qquad \text{(I)}$$

in the orthorhombic system

$$Q_{hkl}-Q_{h'kl}=N_1a^{*2}, \text{ etc.} \qquad \text{(II)}$$

in the hexagonal system

$$Q_{hkl}-Q_{h'k'l}=Na^{*2}, \qquad \text{(IIIa)}$$

$$Q_{hkl}-Q_{hkl'}=N_3c^{*2}, \qquad \text{(IIIb)}$$

in the tetragonal system

$$Q_{hkl}-Q_{h'k'l}=Na^{*2}, \qquad \text{(IVa)}$$

$$Q_{hkl}-Q_{hkl'}=N_3c^{*2}, \qquad \text{(IVb)}$$

in the cubic system

$$Q_{hkl}-Q_{h'k'l'}=Na^{*2}, \qquad \text{(V)}$$

where N, N_1, N_2, N_3 are integers, the possible values of which (see Table 1) are determined by the Bravais lattice properties. (From these numbers all those which, in the form N_1a^{*2}, can also represent possible values of Q_{hkl} are printed in italics.) It is obvious that there are some forbidden values even for primitive unit cells; $N_3=l^2-l'^2$ cannot be equal to 2, 6, 10, 14, ... For

Table 1. *Possible values of* N, N_1, N_2, N_3

	Monoclinic	
P	N_3	= *1*, –, 3, *4*, 5, –, 7, 8, *9*, –, 11, 12, 13, –, . . .
B	N_3	= *4*, 8, 12, *16*, 20, 24, . . .
	Orthorhombic	
P	N_1, N_2, N_3	= *1*, –, 3, *4*, 5, –, 7, 8, *9*, –, 11, 12, 13, –, . . .
C	N_1, N_2	= *4*, 8, 12, *16*, 20, 24, . . .
	N_3	= *1*, –, 3, *4*, 5, –, 7, 8, *9*, –, 11, 12, 13, –, . . .
F, I	N_1, N_2, N_3	= *4*, 8, 12, *16*, 20, 24, . . .
	Hexagonal	
H	N	= *1*, 2, *3*, *4*, 5, 6, *7*, 8, *9*, 10, . . .
	N_3	= *1*, –, 3, *4*, 5, –, 7, 8, *9*, –, 11, 12, 13, –, . . .
R	$\frac{1}{3}$N	= *1*, 2, *3*, *4*, 5, 6, *7*, 8, *9*, 10, . . .
	$\frac{1}{3}N_3$	= 1, –, *3*, 4, 5, –, 7, 8, 9, –, 11, *12*, 13, –, . . .
	Tetragonal	
P	N	= *1*, *2*, 3, *4*, *5*, 6, 7, *8*, *9*, *10*, . . .
	N_3	= *1*, –, 3, *4*, 5, –, 7, 8, *9*, –, 11, 12, 13, –, . . .
I	N	= *2*, *4*, 6, *8*, *10*, 12, 14, *16*, *18*, . . .
	N_3	= *4*, 8, 12, *16*, 20, 24, 28, 32, . . .
	Cubic	
P	N	= *1*, *2*, *3*, *4*, *5*, *6*, 7, *8*, *9*, *10*, . . .
I	N	= *2*, *4*, *6*, *8*, *10*, *12*, *14*, *16*, . . .
F	N	= *1*, –, *3*, *4*, *5*, –, 7, *8*, *9*, –, *11*, . . .