

background. A three-dimensional sharpened Patterson function was computed; this was a beautifully detailed distribution, but many efforts to derive the structure by vector superposition methods have been unsuccessful.

Several attempts were made to prepare caesium, rubidium, silver, and thallium salts, but in no case could a crystalline sample be obtained.

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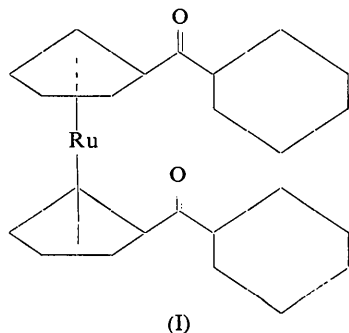
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The crystal structure of 1,1'-dibenzoylruthenocene. By JAMES TROTTER and SIMON H. WHITLOW, *Department of Chemistry, University of British Columbia, Vancouver 8, B.C., Canada*

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Crystals of 1,1'-dibenzoylruthenocene (I) are golden-yellow leaflets, m.p. 125–126 °C. The unit-cell parameters were determined from rotation, Weissenberg and precession films.



Crystal data (λ , Cu $K\alpha$ = 1.5418 Å; λ , Mo $K\alpha$ = 0.7107 Å).

Dibenzoylruthenocene, $C_{24}H_{18}O_2Ru$, M.W. 439.5.
 Monoclinic, $a = 7.34 \pm 0.02$, $b = 12.02 \pm 0.02$, $c = 20.02 \pm 0.04$
 Å, $\beta = 90^\circ$.
 $U = 1766 \text{ \AA}^3$.
 D_m (floatation in CCl_4-CHBr_3) = 1.65, $Z = 4$, $D_x = 1.65$
 g.cm^{-3} .

The diffraction pattern exhibits an interesting series of regions of diffuse scattering, the diffuseness being present only in the Hkl layers of the reciprocal lattice with $H = 2n + 1$. In addition the layers with $h = 2n$ show orthorhombic symmetry, but for those with $h = 2n + 1$ the symmetry is only monoclinic. This pattern is typical of an order-disorder structure (Dornberger-Schiff, 1956, Sedlacek & Dornberger-Schiff, 1965) built up from rows of molecules parallel to a . Within each row the molecular arrangement is perfectly ordered, but the sequence of rows is such that there is no true periodicity in the b and c directions. The disorder in the stacking must result from the fact that there are at least two energetically equivalent ways in which neighbouring rows can be placed with respect to one another. Since reflexions with $h = 2n$ show no diffuseness, ordered building blocks in the crystal are probably related to neighbouring blocks simply by translation $a/2$.

It seems virtually impossible to deduce the detailed structure since this would involve accounting quantitatively for

the diffuse scattering. In fact it is difficult to derive the space group unambiguously; the only definite systematic absence seems to be $0kl$ when k is odd, but in addition $h0l$ are absent for $h = 2n$ when $l = 2n + 1$, $0kl$ are weak when $(k/2 + l)$ is odd, $2kl$ are weak when k is even. Patterson projections were computed, but no outstanding Ru–Ru peaks were observed, a further indication of disordered ruthenium positions.

Although detailed analysis cannot be carried out, it is possible to indicate the type of molecular arrangement which probably exists in the crystal. Several crystal structures are known in which molecules with five-, six-, and even seven-membered rings show disorder in packing, suggesting that the spatial requirements of these rings are not very different, for example bisindenyl iron (Trotter, 1958) and azulene (Robertson, Shearer, Sim & Watson, 1962). By analogy with these structures it seems likely that in the dibenzoylruthenocene crystal two energetically equivalent molecular packings are possible. The most plausible structure is one with molecules all in a completely eclipsed orientation (I), the two molecular arrangements being related by 180° rotation about an axis parallel to, and midway between the C=O bonds. They may be represented schematically:



Any one row in the a direction contains molecules in only one of these orientations, but neighbouring rows are not related.

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