# SHORT STRUCTURAL PAPERS

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## Tris-(2,4-pentanedionato)rhodium(III)

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**Abstract.** Monoclinic, $P2_1/c$, $a=13{\cdot}925$ (4), $b=7{\cdot}483$ (4), $c=16{\cdot}392$ (7) Å, $\beta=98{\cdot}63$ (2)°, 25°C, $(C_5H_7O_2)_3Rh$, $M=400{\cdot}23$, $Z=4$, $D_m=1{\cdot}56$, $D_x=1{\cdot}574$ g cm$^{-3}$ crystallized from aqueous ethanol. The compound is monomeric; the central Rh atom is octahedrally coordinated to six oxygen atoms.

**Introduction.** Cell dimensions were obtained from 12 reflections measured with Mo $K\alpha$ radiation ($\lambda=0{\cdot}7093$ Å) on a Picker four-circle diffractometer. The crystal was a prism $0{\cdot}1\times0{\cdot}2\times0{\cdot}5$ mm. Systematic absences: $h0l$ for $l$ odd, $0k0$ for $k$ odd. The intensities measured with Mo $K\alpha$ radiation were those for reflections with $\pm h, k\geq0, l\geq0$ in the range $2°\leq2\theta\leq62°$. Of the 5779 unique data in this range, 2881 with an intensity greater than three times the e.s.d. (counting statistics) of the intensity were used to determine the structure. Absorption, Lorentz, and polarization corrections were applied. Rh atom coordinates were determined from Patterson projections. The positions of the other atoms, except for a single H atom, were found from Fourier syntheses. The structure was refined by full-matrix least-squares calculations with weights derived from counting statistics. Coordinates and anisotropic thermal parameters for non-H atoms were refined, but, for 20 H atoms, only coordinates were treated as variables. Each H atom was assigned an isotropic thermal parameter derived from the thermal parameters of the carbon atom to which it is bonded. The final conventional residual was 3·5%. The weighted residual dropped from 5·1% to the final value of 3·6% with inclusion of the coordinates of 20 H atoms as variables, and Hamilton's (1965) significance test showed the inclusion of these 60 variables to be justified with an $\alpha$ below the 0·5% level.

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Table 1. *Final fractional coordinates* ($\times10^4$) *and thermal parameters* ($\times10^3$) *for non-hydrogen atoms*

Thermal factors are:

$$\exp\left[-2\pi^2(U_{11}a^{*2}h^2+U_{22}b^{*2}k^2+U_{33}c^{*2}l^2+2U_{12}a^*b^*hk+2U_{13}a^*c^*hl+2U_{23}b^*c^*kl)\right]$$

Standard deviations, given in parentheses, refer to the least significant digits.

| | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Rh | 2390·8 (2) | 2666·1 (4) | 4693·5 (2) | 44·9 (2) | 43·0 (2) | 34·1 (1) | 6·6 (2) | 5·2 (1) | −2·7 (1) |
| O(11) | 1120 (2) | 3404 (4) | 4073 (2) | 60 (2) | 74 (2) | 52 (2) | 19 (2) | −8 (1) | −11 (1) |
| O(12) | 1862 (2) | 1857 (4) | 5695 (2) | 57 (2) | 64 (2) | 45 (2) | −2 (1) | 13 (1) | −3 (1) |
| C(11) | 327 (4) | 3120 (7) | 4350 (3) | 54 (3) | 96 (4) | 71 (3) | 15 (3) | −9 (2) | −32 (3) |
| C(*M*11) | −541 (4) | 3707 (12) | 3722 (5) | 64 (4) | 183 (8) | 122 (5) | 43 (4) | −30 (4) | −66 (6) |
| C(12) | 951 (4) | 1865 (7) | 5727 (3) | 64 (3) | 90 (4) | 60 (3) | −15 (3) | 23 (2) | −16 (2) |
| C(*M*12) | 729 (6) | 1193 (13) | 6547 (5) | 91 (5) | 167 (9) | 97 (5) | −27 (5) | 48 (4) | 0 (5) |
| C(13) | 226 (3) | 2389 (9) | 5101 (4) | 48 (3) | 148 (6) | 80 (3) | −8 (4) | 13 (2) | −19 (4) |
| O(21) | 2532 (2) | 5124 (3) | 5174 (2) | 61 (2) | 48 (2) | 42 (2) | 10 (1) | 7 (1) | −6 (1) |
| O(22) | 3704 (2) | 1833 (3) | 5214 (2) | 46 (2) | 52 (2) | 50 (2) | 8 (1) | 6 (1) | 1 (1) |
| C(21) | 3316 (3) | 5654 (5) | 5593 (2) | 73 (3) | 47 (2) | 32 (2) | −6 (2) | 14 (2) | 0 (2) |
| C(*M*21) | 3279 (5) | 7524 (7) | 5907 (3) | 102 (4) | 55 (3) | 62 (3) | −6 (3) | 13 (3) | −10 (2) |
| C(22) | 4322 (3) | 2910 (5) | 5606 (2) | 44 (2) | 64 (3) | 38 (2) | 1 (2) | 11 (2) | 6 (2) |
| C(*M*22) | 5289 (3) | 2065 (9) | 5897 (3) | 49 (3) | 109 (5) | 64 (3) | 12 (3) | 4 (2) | 2 (3) |
| C(23) | 4169 (3) | 4666 (6) | 5782 (3) | 54 (3) | 60 (3) | 55 (2) | −9 (2) | 5 (2) | −3 (2) |
| O(31) | 3000 (2) | 3553 (3) | 3747 (1) | 70 (2) | 48 (2) | 40 (2) | 1 (1) | 14 (1) | −3 (1) |
| O(32) | 2139 (2) | 207 (3) | 4248 (1) | 57 (2) | 50 (2) | 45 (2) | 1 (1) | 6 (1) | −5 (1) |
| C(31) | 3067 (3) | 2576 (6) | 3123 (2) | 52 (2) | 65 (3) | 36 (3) | 7 (2) | 5 (1) | 1 (2) |
| C(*M*31) | 3533 (4) | 3478 (8) | 2471 (3) | 85 (4) | 87 (4) | 47 (3) | −13 (3) | 14 (2) | −2 (3) |
| C(32) | 2339 (3) | −258 (5) | 3547 (2) | 46 (2) | 51 (2) | 46 (2) | 12 (1) | −7 (2) | −7 (2) |
| C(*M*32) | 2091 (4) | −2151 (7) | 3331 (3) | 69 (3) | 57 (3) | 59 (3) | 3 (2) | −9 (2) | −12 (2) |
| C(33) | 2759 (3) | 819 (6) | 3008 (2) | 67 (3) | 55 (3) | 40 (2) | 7 (2) | 8 (2) | −13 (2) |

Coordinates and thermal parameters are listed in Tables 1 and 2. A table of structure factors is obtainable.* Average bond lengths and angles are in Fig. 1.

**Discussion.** This structure was studied to determine the degree of regularity in the environment of the metal atom. Recent work by Bursey & Rogerson (1971) has focused attention on the energy relationship between Rh and the ring atoms. The structure is essentially that of $(C_5H_7O_2)_3Cr$ (Morosin, 1965). The thermal motion of C(*M*11) and C(*M*12) is very much less than that of corresponding atoms in the Cr analog. The mean of Rh–O intraring distances is significantly shorter than the mean Rh–O distance 2·02 A observed in potassium tris(oxalato)rhodate(III) by Dalzell & Eriks (1971).

* This table has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30057. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

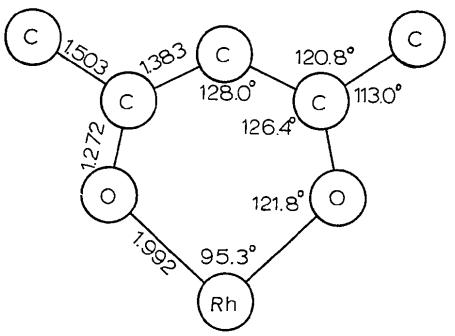


Fig. 1. Mean values of bond lengths and angles for the three rings in $(C_5H_7O_2)_3Rh$. E.s.d.'s are as follows: Rh–O, 0·003; C–O, 0·005; C–C, 0·008 Å; O–Rh–O, 0·1°; other angles, 0·5°.

Table 2. *Final fractional coordinates* ($\times 10^3$) *and assigned thermal parameters for the hydrogen atoms*

The parameter $B$ in the expression $\exp[-B\sin^2\theta/\lambda^2]$ was not varied. Atom H(111) was not resolved in the difference maps.

| | *x* | *y* | *z* | *B* | Bonded to |
|---|---|---|---|---|---|
| H(112) | −100 (4) | 411 (6) | 402 (3) | 6·7 | C(*M*11) |
| H(113) | −87 (4) | 291 (7) | 376 (4) | 6·7 | C(*M*11) |
| H(121) | 14 (4) | 72 (7) | 648 (3) | 7·4 | C(*M*12) |
| H(122) | 116 (4) | 56 (7) | 694 (3) | 7·4 | C(*M*12) |
| H(123) | 50 (5) | 206 (8) | 670 (4) | 7·4 | C(*M*12) |
| H(13) | −38 (4) | 223 (6) | 524 (3) | 7·3 | C(13) |
| H(211) | 380 (3) | 797 (6) | 614 (3) | 5·8 | C(*M*21) |
| H(212) | 275 (3) | 764 (6) | 621 (3) | 5·8 | C(*M*21) |
| H(213) | 291 (3) | 832 (6) | 551 (3) | 5·8 | C(*M*21) |
| H(221) | 555 (3) | 150 (6) | 551 (3) | 5·9 | C(*M*22) |
| H(222) | 589 (3) | 311 (6) | 590 (3) | 5·9 | C(*M*22) |
| H(223) | 533 (3) | 178 (6) | 644 (3) | 5·9 | C(*M*22) |
| H(23) | 468 (3) | 522 (5) | 605 (2) | 4·4 | C(23) |
| H(311) | 374 (3) | 466 (6) | 253 (3) | 5·8 | C(*M*31) |
| H(312) | 314 (3) | 342 (6) | 200 (3) | 5·8 | C(*M*31) |
| H(313) | 416 (4) | 322 (7) | 257 (3) | 5·8 | C(*M*31) |
| H(321) | 138 (3) | −245 (5) | 332 (3) | 4·9 | C(*M*32) |
| H(322) | 207 (4) | −231 (6) | 291 (3) | 4·9 | C(*M*32) |
| H(323) | 234 (3) | −298 (5) | 371 (3) | 4·9 | C(*M*32) |
| H(33) | 287 (3) | 28 (5) | 252 (2) | 4·2 | C(33) |

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## 1-Morpholinomethyl-3-methyl-3-phenylpyrrolidin-2,5-dione

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**Abstract.** The structure determination is reported of an efficacious antiepileptic drug [registered under the names 'Perlepsyn' (in Hungary) and 'Morpholep' (Seres, Tardos & Leszkovszki, 1962, Hungarian patent No. 151425)] which crystallizes from ethanol in prismatic needles. Its space group is monoclinic $P2_1/c$ (No. 14), $a=11{\cdot}659$ (10), $b=5{\cdot}795$ (10), $c=22{\cdot}861$ (10) Å, $\beta=100{\cdot}75$ (5)°, (from oscillation and Weissenberg photographs and refined on a diffractometer), $Z=4$, $D_c=1{\cdot}269$, $D_x=1{\cdot}258$ g cm$^{-3}$ (by flotation). The

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