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## Structure of 9,9,9,9-Tetracarbonyl-2,3,4,5-tetrafluoro-7-pentacarbonylmanganio-9-mangano-8-oxabicyclo[4.3.0]nonane

BY WOLFGANG HILLER

*Institut für Anorganische Chemie der Universität Tübingen, Auf der Morgenstelle 18, D-7400 Tübingen, Federal Republic of Germany*

AND THOMAS KOENIG AND ROLF WINTER

*Department of Chemistry, University of Oregon, Eugene, Oregon 97403, USA*

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**Abstract.**  $C_{16}O_{10}F_4Mn_2$ ,  $M_r = 538.04$ , monoclinic,  $P2_1/c$ ,  $a = 10.325$  (2),  $b = 6.631$  (3),  $c = 27.734$  (3) Å,  $\beta = 90.673$  (3)°,  $V = 1898.8$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.882$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 13.667$  cm<sup>-1</sup>,  $F(000) = 1048$ ,  $T = 293$  K. The final

$R$  value is 0.063 for 1423 significant [ $I > 5\sigma(I)$ ] reflections. The molecule contains a planar aceto-tetrafluorophenyl group chelated through the acyl oxygen and the *ortho* carbon atom of the tetrafluorophenyl ring to a *cis* tetracarbonylmanganese

Table 1. Data collection and structure refinement parameters

Crystal shape	Prismatic
Diffractometer used	CAD-4, Enraf-Nonius
Method of intensity measurement	$\theta/2\theta$
No. and $\theta$ range of reflections for lattice parameters	25; 11–19°
Absorption coefficient $\mu$	13.667 cm <sup>-1</sup>
Method used for absorption correction	DIFABS (Walker & Stuart, 1983)
Minimum absorption correction	0.842
Maximum absorption correction	1.190
Average absorption correction	1.008
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurement	0.572 Å <sup>-1</sup>
Range of $h$ , $k$ and $l$	0–11, 0–7, –31–31
Standard reflections	228; 306; 400
Interval, standard reflections measured	2h, no intensity variation
Total number of reflections measured; $\theta$ range	2770; 24° (214 systematic absences included)
No. of unique reflections; $R_{int}$	2571; 0.023
No. of observed reflections	1423
Criterion for observed reflections	$I > 3\sigma(I)$
Methods used to solve structure	Patterson, subsequent difference Fourier
Use of $F$ or $F^2$ in LS refinement	$F$
Weighting scheme	$1/\sigma^2$
Parameters refined	140
Value of $R$	0.063
Value of $wR$	0.064
Ratio of max. LS shift to e.s.d. ( $\Delta/\sigma$ )	0.0002
Max. height in final $\Delta F$ map	0.547 e Å <sup>-3</sup>
Error in an observation of unit weight	2.794
Secondary-extinction coefficient	4.083 (1) $\times 10^{-7}$ (Zachariasen, 1963)
Sources of atomic scattering factors	<i>International Tables for X-ray Crystallography</i> (1974)
Computer used	DEC MicroVAXII
Programs used	VAXSDP, version 2.2 (1985) (Frenz, 1978)

Table 2. Positional parameters, isotropic and equivalent isotropic thermal parameters (Å<sup>2</sup>)

	$x$	$y$	$z$	$U_{eq}/U_{iso}$
	$U_{eq} = 1/3(\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$			
Mn1	0.8461 (2)	0.4835 (3)	0.33548 (6)	0.046 (1)
Mn2	0.4377 (2)	0.1939 (3)	0.37683 (6)	0.047 (1)
F2	1.0719 (7)	0.406 (1)	0.4232 (2)	0.072 (3)
F3	1.0427 (7)	0.243 (1)	0.5097 (2)	0.080 (3)
F4	0.8075 (7)	0.116 (1)	0.5389 (2)	0.075 (3)
F5	0.6044 (6)	0.140 (1)	0.4810 (2)	0.068 (3)
O8	0.6598 (7)	0.393 (1)	0.3436 (2)	0.053 (3)
O11	1.115 (1)	0.599 (2)	0.3250 (3)	0.103 (4)
O12	0.9128 (9)	0.084 (1)	0.2942 (3)	0.086 (3)
O13	0.7768 (9)	0.676 (2)	0.2426 (3)	0.091 (3)
O14	0.811 (1)	0.857 (2)	0.3914 (4)	0.105 (4)
O21	0.1654 (8)	0.052 (1)	0.3631 (3)	0.076 (3)
O22	0.3679 (9)	0.385 (1)	0.4706 (3)	0.085 (3)
O23	0.5110 (8)	–0.198 (1)	0.4216 (3)	0.077 (3)
O24	0.5218 (9)	0.016 (2)	0.2842 (3)	0.093 (3)
O25	0.3898 (9)	0.587 (1)	0.3264 (3)	0.084 (3)
C1	0.854 (1)	0.359 (1)	0.4022 (3)	0.038 (3)
C2	0.951 (1)	0.338 (2)	0.4348 (4)	0.052 (3)
C3	0.938 (1)	0.258 (2)	0.4796 (4)	0.051 (3)
C4	0.823 (1)	0.193 (2)	0.4943 (4)	0.055 (3)
C5	0.719 (1)	0.206 (2)	0.4639 (4)	0.053 (3)
C6	0.732 (1)	0.287 (2)	0.4179 (3)	0.038 (3)
C7	0.625 (1)	0.301 (2)	0.3811 (3)	0.043 (3)
C11	1.008 (1)	0.549 (2)	0.3305 (4)	0.068 (4)
C12	0.883 (1)	0.237 (2)	0.3096 (4)	0.056 (3)
C13	0.807 (1)	0.597 (2)	0.2789 (4)	0.063 (3)
C14	0.820 (1)	0.714 (2)	0.3683 (4)	0.060 (3)
C21	0.268 (1)	0.107 (2)	0.3686 (4)	0.058 (3)
C22	0.397 (1)	0.313 (2)	0.4349 (4)	0.060 (3)
C23	0.483 (1)	–0.046 (2)	0.4059 (4)	0.061 (3)
C24	0.486 (1)	0.084 (2)	0.3200 (4)	0.065 (3)
C25	0.405 (1)	0.435 (2)	0.3463 (4)	0.062 (3)

Table 3. Distances (Å) and angles (°) with e.s.d.'s

Mn1	O8	2.030 (7)	O11	C11	1.16 (2)		
Mn1	C1	2.03 (1)	O12	C12	1.15 (1)		
Mn1	C11	1.74 (1)	O13	C13	1.17 (1)		
Mn1	C12	1.83 (1)	O14	C14	1.15 (2)		
Mn1	C13	1.79 (1)	O21	C21	1.13 (1)		
Mn1	C14	1.80 (1)	O22	C22	1.14 (1)		
Mn2	C7	2.06 (2)	O23	C23	1.13 (1)		
Mn2	C21	1.85 (1)	O24	C24	1.15 (1)		
Mn2	C22	1.85 (1)	O25	C25	1.16 (1)		
Mn2	C23	1.84 (1)	C1	C2	1.35 (1)		
Mn2	C24	1.81 (1)	C1	C6	1.42 (1)		
Mn2	C25	1.85 (1)	C2	C3	1.36 (1)		
F2	C2	1.36 (1)	C3	C4	1.33 (2)		
F3	C3	1.37 (1)	C4	C5	1.36 (2)		
F4	C4	1.35 (1)	C5	C6	1.39 (1)		
F5	C5	1.35 (1)	C6	C7	1.50 (1)		
O8	C7	1.26 (1)					
O8	Mn1	C1	79.0 (4)	C22	Mn2	C24	176.9 (6)
O8	Mn1	C11	176.7 (5)	C22	Mn2	C25	89.2 (5)
O8	Mn1	C12	89.0 (4)	C23	Mn2	C24	87.9 (5)
O8	Mn1	C13	91.0 (4)	C23	Mn2	C25	176.0 (5)
O8	Mn1	C14	92.6 (4)	C24	Mn2	C25	90.1 (5)
C1	Mn1	C11	98.4 (5)	Mn1	O8	C7	120.9 (6)
C1	Mn1	C12	89.3 (4)	Mn1	C1	C2	132.4 (8)
C1	Mn1	C13	169.2 (6)	Mn1	C1	C6	112.7 (7)
C1	Mn1	C14	83.7 (4)	C2	C1	C6	114.8 (9)
C11	Mn1	C12	89.3 (6)	F2	C2	C1	118.8 (9)
C11	Mn1	C13	91.8 (6)	F2	C2	C3	116.6 (9)
C11	Mn1	C14	88.8 (6)	C1	C2	C3	125 (2)
C12	Mn1	C13	94.4 (5)	F3	C3	C2	120 (1)
C12	Mn1	C14	172.5 (6)	F3	C3	C4	119.5 (9)
C13	Mn1	C14	93.0 (5)	C2	C3	C4	121 (2)
C7	Mn2	C21	175.8 (4)	F4	C4	C3	121 (1)
C7	Mn2	C22	91.5 (5)	F4	C4	C5	119 (1)
C7	Mn2	C23	92.4 (5)	C3	C4	C5	119 (1)
C7	Mn2	C24	85.4 (5)	F5	C5	C4	117 (1)
C7	Mn2	C25	84.0 (5)	F5	C5	C6	122.9 (9)
C21	Mn2	C22	91.0 (5)	C4	C5	C6	120 (2)
C21	Mn2	C23	90.9 (5)	C1	C6	C5	120.2 (9)
C21	Mn2	C24	92.2 (5)	C1	C6	C7	115.0 (8)
C21	Mn2	C25	92.6 (5)	C5	C6	C7	124.7 (9)
C22	Mn2	C23	92.6 (5)	Mn2	C7	O8	113.3 (7)
Mn2	C7	C6	134.6 (8)	Mn2	C21	O21	179 (1)
O8	C7	C6	112.1 (9)	Mn2	C22	O22	179 (2)
Mn1	C11	O11	176 (1)	Mn2	C23	O23	177 (1)
Mn1	C12	O12	176 (2)	Mn2	C24	O24	177 (2)
Mn1	C13	O13	177 (2)	Mn2	C25	O25	177 (2)
Mn1	C14	O14	175 (2)				

group; furthermore, a pentacarbonylmanganese group is bound to the acyl carbon. In the five-membered manganese ring the Mn—O and Mn—C bond distances are both 2.03 (1) Å with the O—Mn—C bond angle 79.0 (4)°. The C—O distance in the acyl group is 1.26 (1) Å.

**Experimental.** The title compound is stable under normal atmospheric conditions and was recrystallized from CH<sub>2</sub>Cl<sub>2</sub>. The <sup>19</sup>F NMR spectrum (C<sub>6</sub>F<sub>6</sub>, 360 MHz) shows a chemical shift δ at 109.68 (quartet), 128.86 (sextet) and 145.73 (septet).

A yellow single crystal of approximate dimensions 0.03 × 0.15 × 0.15 mm was mounted on a glass fiber. All X-ray investigations were performed on a CAD-4 (Enraf-Nonius) diffractometer. The cell parameters are given in the *Abstract*; the parameters for intensity data collection, structure solution and refinement are listed in Table 1. Final atomic coordinates are given in Table 2, bond distances and angles in Table 3.\* Fig. 1 shows the molecular numbering scheme.

**Related literature.** A general method for the preparation of manganese rings has recently been reported by DeShong, Sidler, Rybczynski, Slough & Rheingold (1988). Related crystal structures of tetracarbonyl-2-acetylphenylmanganese and derivatives are reported by Haupt, Lohmann & Flörke (1985) and Huie, Knobler, Firestein, McKinney & Kaesz (1977).

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\* Lists of structure factors, anisotropic thermal parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51736 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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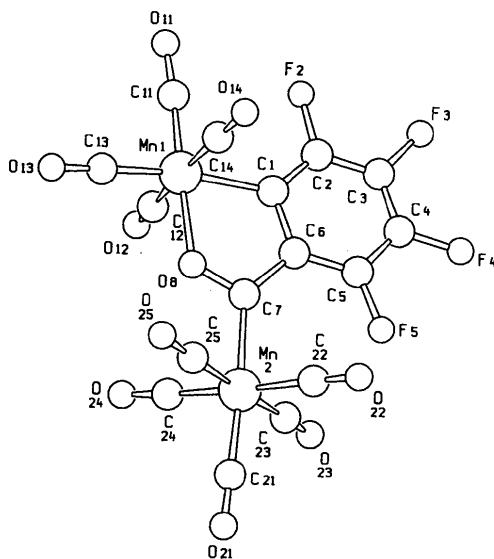


Fig. 1. Plot of the molecule produced using *SCHAKAL* (Keller, 1988).