

Short Communications

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The crystal structure of diaquobisacetylacetonatomanganese(II). By H. MONTGOMERY, *Canadian Services College, Royal Roads, Victoria, B.C., Canada*, and E. C. LINGAFELTER, *Department of Chemistry, University of Washington, Seattle, Washington 98105, U.S.A.*

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Diaquobisacetylacetonatomanganese(II) is isostructural with the cobalt, nickel and magnesium compounds. The structure has been refined by full-matrix least-squares with three-dimensional photographic intensity data to a final value of $R=0.062$.

As a continuation of the work on hydrated acetylacetonate derivatives of transition metals (Montgomery & Lingafelter, 1963, 1964) the structure of the manganese derivative, $Mn(C_5H_7O_2)_2 \cdot 2H_2O$, has been determined.

The compound was prepared by the method of Charles (1960). It was recrystallized from acetone or alcohol by warming to 40°C only, to prevent decomposition, and cooling strongly in a refrigerator. In general, these crystals were twinned, and specimens for X-ray work were crystallized from dimethylformamide by the above technique.

The unit cell has dimensions (and standard deviations)

$$a_0 = 11.186 \pm 0.015, \quad b_0 = 5.426 \pm 0.008, \\ c_0 = 11.315 \pm 0.02 \text{ \AA}, \quad \beta = 106.16 \pm 0.2^\circ,$$

determined from zero-level Weissenberg and rotation photographs (calibrated with NaCl, $a_0 = 5.6387 \text{ \AA}$) using Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The space group is $P2_1/c$, $Z=2$. Intensities were measured photometrically from a set of integrated equi-inclination Weissenberg photographs, $h0l$ through $h3l$, taken with Fe $K\alpha$ radiation. 588 independent

reflections were examined, of which 498 were found to be above background (intensity range, 1.6 to 3650). Lorentz and polarization factors were applied, but no absorption correction was made. The structure factors were scaled by levels. The level scale factors were assigned initial values from the exposure times of the photographs and were later refined after anisotropic thermal parameters had been determined. The physical significance of the thermal parameters is therefore doubtful.

The structure was refined by full-matrix least squares with the X-ray 63 program system (Stewart, 1964), starting from the parameters of the nickel compound (Montgomery & Lingafelter, 1964). Scattering factors for manganese, oxygen, and carbon atoms were taken from *International Tables for X-ray Crystallography* (1962), that of Mn being corrected for the real part of the anomalous dispersion correction, $\Delta f' = -4.0$. The scattering factors from Table II of Stewart, Davidson & Simpson (1965) were used for hydrogen. A modified Hughes weighting scheme was used, and $\Sigma w(F_o - F_c)^2$ was minimized.

Table 1. Final parameters (with estimated standard deviations in parentheses)

	Fractional positional parameters $\times 10^4$			Anisotropic thermal parameters $\times 10^4$						
	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	
Mn	0	0	0	40 (1)	228 (12)	44 (1)	-1 (3)	6 (1)	-20 (3)	
O(1)	1531 (3)	2286 (8)	940 (3)	51 (4)	199 (22)	63 (4)	11 (6)	9 (3)	-26 (6)	
O(2)	1241 (3)	-1534 (8)	-920 (3)	54 (4)	320 (26)	48 (3)	16 (7)	6 (3)	-22 (7)	
O(3)	608 (4)	-2992 (9)	1428 (3)	104 (4)	299 (25)	47 (3)	8 (8)	14 (3)	21 (7)	
C(1)	3604 (5)	3320 (13)	2025 (5)	75 (6)	224 (40)	75 (6)	-16 (11)	-1 (5)	-15 (10)	
C(2)	2682 (5)	1752 (12)	1092 (4)	66 (6)	257 (38)	40 (5)	3 (11)	10 (4)	18 (10)	
C(3)	3127 (5)	-108 (13)	487 (5)	50 (5)	323 (41)	56 (5)	5 (10)	9 (5)	-11 (12)	
C(4)	2431 (5)	-1654 (13)	-452 (4)	75 (6)	340 (40)	32 (5)	2 (12)	23 (4)	12 (11)	
C(5)	3084 (6)	-3568 (14)	-989 (5)	102 (7)	295 (42)	75 (6)	68 (13)	39 (5)	28 (12)	
Hydrogen atom positions										
H(1)	C(1)	0.345	0.50	0.185						
H(2)		0.34	0.32	0.30						
H(3)		0.46	0.25	0.23						
H(4)	C(5)	0.295	-0.30	-0.17						
H(5)		0.275	-0.53	-0.09						
H(6)		0.39	-0.37	-0.05						
H(7)	C(3)	0.40	-0.02	0.08						
H(8)	O(3)	0.10	-0.45	0.13						
H(9)		0.06	-0.28	0.23						

After $R (R = \Sigma ||F_o| - |F_c| | / \Sigma |F_o|)$, observed reflections only) had been reduced to 0.085 by refinement of the parameters of the non-hydrogen atoms, all hydrogen atoms were located on a difference Fourier synthesis, and were assigned fixed values, $B = 3.3$, for their isotropic thermal parameters. Two final cycles of refinement of all parameters (except the thermal parameters of the hydrogen atoms) gave a final value of $R = 0.062$. The final list of observed and calculated structure factors may be obtained from the authors.

The final positions for all atoms and the final thermal parameters for the non-hydrogen atoms are given in Table 1 and the bond lengths and angles are in Table 2.

Table 2. Bond lengths and angles

Mn—O(1)	2.142 (8) Å	C(1)—C(2)	1.515 (16) Å
Mn—O(2)	2.123 (8)	C(2)—C(3)	1.387 (16)
Mn—O(3)	2.257 (8)	C(3)—C(4)	1.406 (16)
O(1)—C(2)	1.284 (11)	C(4)—C(5)	1.493 (16)
O(2)—C(4)	1.291 (11)	O(1)—O(2)	2.907 (10)
O(1)—Mn—O(2)	85.9 (3)°	O(1)—C(2)—C(3)	125.6 (7)°
O(1)—Mn—O(3)	91.2 (3)	O(2)—C(4)—C(3)	124.8 (7)
O(2)—Mn—O(3)	87.8 (3)	O(1)—C(2)—C(1)	115.4 (7)
Mn—O(1)—C(2)	124.7 (5) Å	O(2)—C(4)—C(5)	115.7 (7)
Mn—O(2)—C(4)	124.0 (5)	C(2)—C(3)—C(4)	127.4 (9)
C(5)—C(4)—C(3)	119.5 (9)°		
C(1)—C(2)—C(3)	119.0 (9)		

The structure is essentially identical with that previously reported for the cobalt (Bullen, 1959), nickel (Montgomery & Lingafelter, 1964) and magnesium (Morosin, 1967) com-

pounds. The average Mn—O(acetylacetonate) distance is 2.13 Å, while the Mn—O(water) distance is considerably longer, 2.26 Å. The water molecule is hydrogen-bonded to two oxygen atoms of an adjacent molecule with O...O distances 2.87 and 2.90 Å. The chelate ring O—C—C—O is planar with the manganese atom 0.58 Å out of the least-squares plane. One of the methyl carbon atoms is displaced significantly (0.13 Å) from this plane, while the displacement of the other methyl carbon atom (0.06 Å) is probably not significant.

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The crystal structure of α -glycylglycine: Correction. By EDWARD W. HUGHES, *Gates and Crellin Laboratories of Chemistry, California Institute of Technology, Pasadena, California 91109, U.S.A.*

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Corrections to lattice constants given in *Acta Cryst.* (1968), B24, 40.

The paper of the above title (Biswas, Hughes, Sharma & Wilson, 1968) unfortunately gives lattice constants which are in error by amounts considerably larger than the error estimates listed. The exploratory examination of the α crystals was made in 1939 but the investigation was soon abandoned in favor of that of the β crystals (Hughes & Moore, 1949) and both problems were delayed by World War II. When the research was reinstigated many years later the first rough estimates of the lattice constants were inadvertently substituted for the more accurate values. This error is entirely the fault of the present author and should

not reflect on his later colleagues in the structure determination and refinement.

The errors were called to my attention by Dr Hans C. Freeman, who has kindly supplied me with lattice constants determined in his laboratory by a least-squares adjustment based on numerous scattering angle measurements made on a goniometer with Cu $K\alpha$ radiation in the wide range ($\theta > 45^\circ$).

Table 1 gives the old (erroneous) and new (Freeman) values and also the corresponding values for the perdeuteroglycylglycine (Freeman, Paul & Sabine, 1967). Our better

Table 1. Values for unit-cell dimensions of α -glycylglycine

	Old	New	Perdeutero
<i>a</i>	7.70 ± 0.02 Å	7.812 ± 0.0024 Å	7.8271 ± 0.0003 Å
<i>b</i>	9.57 ± 0.03	9.566 ± 0.0030	9.5586 ± 0.0006
<i>c</i>	9.48 ± 0.03	9.410 ± 0.0029	9.4251 ± 0.0002
β	124°35' ± 20'	124°36' ± 1.0'	124°51' ± 0.1'
ρ calc.	1.516 g.cm ⁻³	1.512 g.cm ⁻³	