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Bis-(2-hydroxy-5-methylacetophenato)nickel (II)

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Abstract. Monoclinic, $C2/c$, $a=11{\cdot}432\,(2)$, $b=8{\cdot}579\,(2)$, $c=16{\cdot}693\,(4)$ Å, $\beta=101{\cdot}3\,(1)°$, 22°C, $(C_9H_9O_2)_2Ni$, $M=357{\cdot}05$, $Z=4$, $D_m=1{\cdot}45$, $D_x=1{\cdot}48$ g cm^{-3}, crystallized from aqueous ethanol. The compound is diamagnetic and monomeric. The Ni atom is at a center of symmetry with square planar coordination to four oxygen atoms; the entire complex is planar.

Introduction. Cell dimensions were obtained from 10 high-angle reflections measured with Mo $K\alpha_1$ radiation ($\lambda=0{\cdot}70926$) on a Picker FACS-1 four-circle diffractometer. The red parallelepiped crystal measured 0·31 × 0·16 × 0·36 mm. Systematic absences: hkl for $h+k$ odd, $h0l$ for l odd. One quadrant of data was collected in the range $3°<2\theta<52°$. Of the 1678 unique data in this range, 1134 with an intensity greater than $3\sigma(F_0^2)$ based on counting statistics were used for the structure determination. Lorentz, polarization and absorption (Ni only) corrections were applied. The structure was solved by analysis of a series of Fourier syntheses. Refinement was by full-matrix least-squares techniques with weights derived from counting statistics. Three positional parameters for all atoms except Ni, anisotropic temperature factors for non-hydrogen atoms, isotropic temperature factors for hydrogen atoms and one scale factor (total of 142) were refined. The final conventional residual index was 0·049. Inclusion of 544 unobserved data without further refinement raised the residual to 0·080. Atomic coordinates and thermal parameters are listed in Table 1. Bond lengths and angles are included in Fig. 1.*

Discussion. Graddon & Mockler (1968) report that this compound forms as a deep green monomeric high-

* The table of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30259 (8 pp.). Copies may be obtained from the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final structure parameters with standard deviations in parentheses*

(*a*) Heavy atoms (anisotropic thermal parameters)

The anisotropic thermal parameters are in the form $\times 10^{-4}$ exp $[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})]$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ni	0·2500	0·2500	0·5000	80 (1)	65 (1)	25 (1)	−2 (1)	14 (1)	−2 (1)
O(1)	0·1831 (3)	0·4097 (4)	0·4316 (2)	121 (4)	121 (6)	43 (2)	−8 (4)	20 (2)	−2 (2)
O(2)	0·2692 (3)	0·1476 (3)	0·4069 (2)	105 (3)	74 (4)	26 (1)	10 (3)	16 (2)	1 (2)
C(1)	0·1431 (4)	0·4080 (5)	0·0315 (2)	53 (4)	77 (5)	22 (2)	6 (4)	11 (2)	4 (2)
C(2)	0·1880 (4)	0·5060 (5)	0·0986 (3)	64 (4)	70 (6)	27 (2)	7 (4)	14 (2)	−1 (2)
C(3)	0·1855 (5)	0·4487 (5)	0·1773 (3)	93 (5)	90 (6)	24 (2)	−5 (5)	11 (3)	−4 (3)
C(4)	0·1410 (4)	0·3041 (6)	0·1894 (3)	85 (5)	102 (6)	23 (2)	−3 (5)	14 (3)	8 (3)
C(5)	0·0957 (4)	0·2054 (5)	0·1244 (3)	62 (4)	92 (6)	31 (2)	−2 (4)	13 (2)	9 (3)
C(6)	0·0987 (4)	0·2599 (6)	0·0469 (3)	58 (4)	86 (6)	25 (2)	5 (5)	7 (2)	−3 (3)
C(7)	0·3591 (4)	0·0452 (5)	0·0529 (3)	52 (4)	75 (6)	29 (2)	15 (4)	8 (2)	4 (3)
C(8)	0·4095 (5)	0·1506 (6)	0·1222 (3)	73 (5)	100 (7)	24 (2)	0 (5)	7 (3)	−6 (3)
C(9)	0·0458 (7)	0·0468 (8)	0·1369 (4)	112 (7)	121 (9)	36 (2)	−41 (6)	15 (4)	9 (4)

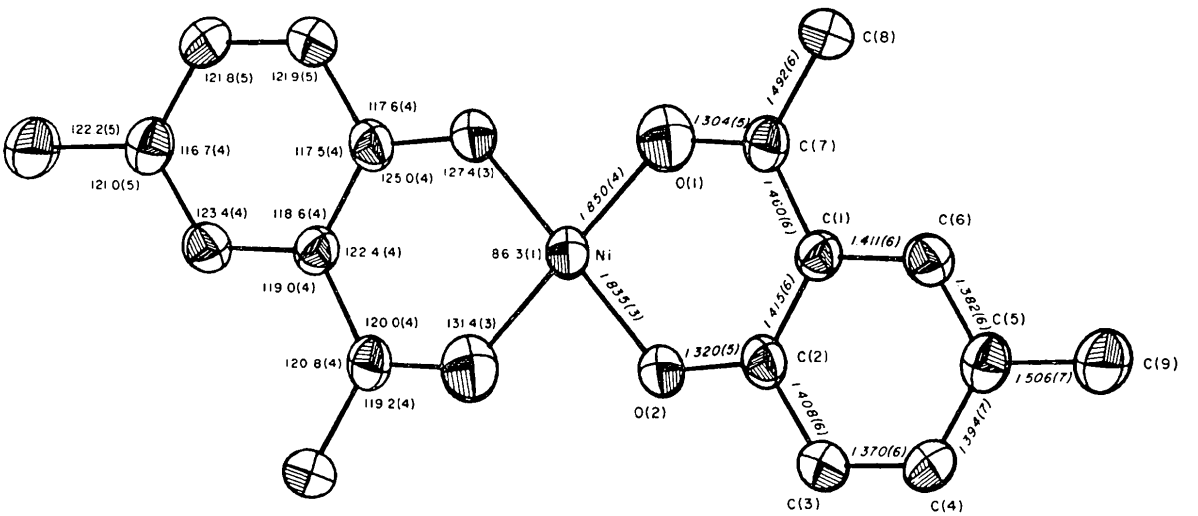

Fig. 1. Bond distances and angles for bis-(2-hydroxy-5-methylacetophenato)nickel(II). Standard errors were computed from the least-squares variance-covariance matrix.

Table 1 (*cont.*)

(*b*) Hydrogen atoms (isotropic thermal parameters)

	x	*y*	*z*	*B*
H(C3)	0·285 (5)	0·007 (6)	0·280 (3)	2·5 (1·5)
H(C4)	0·146 (4)	0·276 (4)	0·237 (3)	2·9 (0·9)
H(C6)	0·071 (4)	0·196 (6)	0·000 (3)	2·9 (1·0)
H′(C8)	0·487 (5)	0·170 (7)	0·124 (3)	4·2 (1·4)
H″(C8)	0·409 (5)	0·101 (7)	0·172 (3)	4·1 (1·4)
H‴(C8)	0·363 (4)	0·233 (6)	0·123 (3)	3·8 (1·2)
H′(C9)	0·063 (6)	0·021 (9)	0·176 (4)	8·6 (2·3)
H″(C9)	0·037 (6)	0·040 (8)	0·377 (4)	7·6 (1·9)
H‴(C9)	0·441 (7)	0·474 (9)	0·397 (5)	11·0 (2·5)

spin complex which associates in concentrated solutions in non-donor solvents. From ethanolic solutions we observed both monoclinic and triclinic red crystalline products. The structure reported here is that of the monoclinic form. It is a normal low-spin bis(hydroxyketo)Ni(II) complex. It resembles other red diamagnetic monomeric square-planar complexes of Ni(II). The average Ni–O distance, 1·84 Å, compares well with the Ni–O distances in other square-planar red complexes of Ni(II): 1·84 Å in bis(salicylaldiminato)Ni(II) (Stewart & Lingafelter, 1959), 1·84 Å in bis-(2,2,6,6-tetramethylheptane-3,5-dionato)Ni(II) (Cotton & Wise, 1966). Octahedrally coordinated nickel complexes all have longer Ni–O bonds: 2·02 Å in diaquobis(salicylaldehydato)Ni(II) (Stewart, Lingafelter & Breazeale, 1961), 2·02 Å in diaquobis(acetylacetonato)-Ni(II) (Montgomery & Lingafelter, 1964), 2·02 Å in dipyridinebis(acetylacetonato)Ni(II) (Elder, 1968) and 2·01–2·12Å in trimeric bis(acetylacetonato)Ni(II) (Bullen, Mason & Pauling, 1965). The entire complex is planar with all non-hydrogen atoms within 0·03 Å of the plane defined by $10{\cdot}46x+3{\cdot}42y-2{\cdot}12z-2{\cdot}39=0$ where x,y,z are fractional coordinates. The hydrogen atoms were located with normal bond distances and angles but were poorly resolved, resulting in a large variability in their thermal parameters.

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