Acta Cryst. (1991). C**47**, 1731–1732

Hexaaquanickel(II) Bis[1,2,3-benzenetricarboxylate(1 −)] Tetrahydrate

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(*Received* 31 *October* 1990; *accepted* 29 *January* 1991)

Abstract. $[Ni(H_2O)_6][C_9H_5O_6]_2.4H_2O$, $M_r = 657{\cdot}13$, triclinic, $P\bar{1}$, $a = 6{\cdot}671\ (1)$, $b = 6{\cdot}683\ (1)$, $c = 16{\cdot}357\ (3)$ Å, $\alpha = 93{\cdot}10\ (2)$, $\beta = 92{\cdot}85\ (1)$, $\gamma = 117{\cdot}69\ (1)°$, $V = 642{\cdot}64$ Å^3, $Z = 1$, $D_m = 1{\cdot}70$, $D_x = 1{\cdot}70$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 7{\cdot}91$ cm^{-1}, $F(000) = 342$, $T = 293$ K, $R = 0{\cdot}038$, $wR = 0{\cdot}035$ [$w = 1/\sigma^2(F_o)$] for 2939 reflections with $|F_o| > 2\sigma(|F_o|)$. Ni atoms are coordinated in a slightly distorted octahedron by six water molecules. The crystal structure is built up of $[Ni(H_2O)_6]^{2+}$ ions, anions of hemimellitic acid and crystal water molecules connected by hydrogen bonds. There are no direct interactions between nickel and carboxylate oxygens.

Experimental. Green acicular crystals by diffusion of an aqueous solution of $Ni(NO_3)_2$ into 1,2,3-benzenetricarboxylic acid in ethanol. D_m by flotation in an aqueous solution of thallium(I) formate/malonate. Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$, graphite monochromator. Crystal of dimensions *ca* 0·25 × 0·10 × 0·08 mm. Lattice parameters from 25 reflections in the range $7{\cdot}1 < \theta < 24{\cdot}7°$, intensity measurement $1 < \theta < 25°$ ($-8 \le h \le 8$; $-8 \le k \le 8$; $0 \le l \le 18$), $\omega/2\theta$ scan, three intensity control reflections every hour of data collection, two reflections for orientation every 200 reflections, no significant decay. 3122 reflections measured, Lp correction, empirical absorption correction with *DIFABS* (Walker & Stuart, 1983), transmission factors range from 0·84 to 1·10. 2939 reflections with

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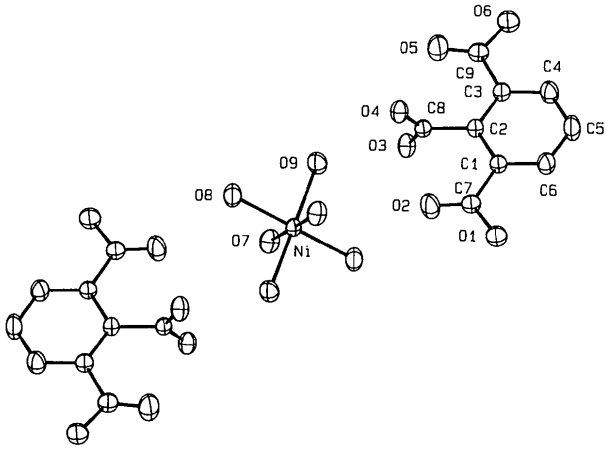

Fig. 1. Surroundings of the Ni atom and the atomic numbering scheme.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$B_{eq} = 8/3\pi^2(\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j)$.

	x	*y*	*z*	B_{eq} (Å^2)
Ni	0·0000	0·0000	0·0000	1·59
O1	−0·0691 (3)	−0·0095 (3)	−0·3587 (1)	2·83
O2	0·1218 (3)	0·1049 (4)	−0·2368 (1)	4.11
O3	0·5718 (3)	0·5056 (2)	−0·1812 (1)	2·26
O4	0·5621 (3)	0·1680 (2)	−0·1780 (1)	2·21
O5	1·0019 (3)	0·5308 (4)	−0·2323 (1)	4·24
O6	1·1350 (3)	0·5972 (3)	−0·3545 (1)	3·00
O7	0·0949 (3)	0·3226 (3)	0·0516 (1)	2·46
O8	0·1094 (3)	−0·0764 (3)	0·1056 (1)	2·89
O9	0·3190 (3)	0·1019 (3)	0·0372 (1)	2·77
O10	0·5547 (3)	0·8177 (3)	−0·2765 (1)	2·10
O11	−0·3098 (3)	0·3869 (3)	0·0687 (1)	3·08
C1	0·3308 (3)	0·1886 (3)	−0·3536 (1)	1·76
C2	0·5432 (3)	0·3022 (3)	−0·3084 (1)	1·54
C3	0·7394 (3)	0·3936 (3)	−0·3511 (1)	1·82
C4	0·7200 (4)	0·3701 (5)	−0·4363 (1)	2·70
C5	0·5106 (4)	0·2581 (5)	−0·4802 (2)	3·12
C6	0·3165 (4)	0·1672 (4)	−0·4389 (1)	2·57
C7	0·1188 (3)	0·0903 (4)	−0·3097 (1)	1·99
C8	0·5605 (3)	0·3271 (3)	−0·2151 (1)	1·68
C9	0·9689 (4)	0·5128 (4)	−0·3050 (1)	2·15

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Ni—O7	2·060 (2)	C3—C9	1·495 (3)
Ni—O8	2·022 (2)	C4—C5	1·378 (3)
Ni—O9	2·046 (2)	C5—C6	1·379 (3)
C1—C2	1·402 (3)	C7—O1	1·313 (3)
C1—C6	1·388 (3)	C7—O2	1·190 (3)
C1—C7	1·495 (3)	C8—O3	1·256 (2)
C2—C3	1·404 (3)	C8—O4	1·257 (2)
C2—C8	1·518 (3)	C9—O5	1·187 (3)
C3—C4	1·386 (3)	C9—O6	1·327 (3)
O8—Ni—O7	90·3 (1)	C5—C4—C3	121·2 (2)
O9—Ni—O7	90·8 (1)	C6—C5—C4	119·5 (2)
O9—Ni—O8	87·4 (1)	C5—C6—C1	120·5 (2)
C6—C1—C2	120·3 (2)	C1—C7—O1	114·1 (2)
C7—C1—C2	119·8 (2)	C1—C7—O2	122·5 (2)
C7—C1—C6	119·9 (2)	O2—C7—O1	123·4 (2)
C3—C2—C1	118·6 (2)	C2—C8—O3	117·2 (2)
C8—C2—C1	120·7 (2)	C2—C8—O4	117·7 (2)
C8—C2—C3	120·7 (2)	O4—C8—O3	125·1 (2)
C4—C3—C2	119·8 (2)	C3—C9—O5	124·6 (2)
C9—C3—C2	120·1 (2)	C3—C9—O6	112·4 (2)
C9—C3—C4	120·0 (2)	O6—C9—O5	123·0 (2)

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$|F_o| > 2\sigma(|F_o|)$ were used for structure refinement. Since $Z = 1$ the Ni atom was located at the centre of symmetry in space group $P\bar{1}$. O and C positions were obtained from subsequent Fourier syntheses. After anisotropic refinement of this model all H atoms were located in a difference Fourier map and added to the model for final refinement with fixed isotropic thermal parameters of 0·08 Å^2. Full-matrix refinement on F (232 parameters) converged at $R = 0{\cdot}038$, $wR = 0{\cdot}035$ [$w = 1/\sigma^2(F_o)$]. Max. Δ/σ in final cycle = 0·003, $\Delta\rho$ fluctuations within +0·50 and −1·06 e Å^{-3}. Calculations were performed with *SHELX*76 (Sheldrick, 1976) using the scattering factors of Cromer & Mann (1968); drawing by *ORTEP* (Johnson, 1976). Atomic coordinates and thermal parameters are given in Table 1,* and bond lengths and angles in Table 2. Fig. 1 shows the Ni-atom surroundings with the atomic numbering scheme.

Related literature. Unlike the title compound there are interactions between metal and carboxylate oxygens in nickel acetate tetrahydrate (Downie, Harrison, Raper & Hepworth, 1971) and in triaquabis[1,2,3-benzenetricarboxylato(1−)]copper(II) trihydrate (Pech & Pickardt, 1990).

We gratefully acknowledge the financial support of the Fonds der Chemischen Industrie.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53959 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
DOWNIE, T. C., HARRISON, W., RAPER, E. S. & HEPWORTH, M. A. (1971). *Acta Cryst.* B**27**, 706–712.
JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
PECH, R. & PICKARDT, J. (1990). *Acta Cryst.* C**46**, 1928–1930.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1991). C**47**, 1732–1734

Structure of (Hydridotrispyrazolylborato)(iodo)(methyl)(triphenylphosphino)-rhodium(III)

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(*Received* 11 *July* 1990; *accepted* 2 *January* 1991)

Abstract. $C_{28}H_{28}BN_6IPRh$, $M_r = 720{\cdot}16$, monoclinic, $C2/c$, $a = 31{\cdot}552$ (6), $b = 9{\cdot}601$ (1), $c = 18{\cdot}606$ (2) Å, $\beta = 93{\cdot}86$ (1)°, $V = 5623$ (2) Å^3, $Z = 8$, $D_m = 1{\cdot}65$, $D_x = 1{\cdot}701$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 17{\cdot}7$ cm^{-1}, $F(000) = 2848$, $T = 298$ K, $R = 0{\cdot}041$, $wR = 0{\cdot}060$, 3072 observed reflections [$I > 3\sigma(I)$]. The Rh^{III} center displays a pseudo-octahedral coordination environment with the trispyrazolylborate ligand capping a trigonal face. The compound crystallizes as a 50:50 mixture of two enantiomers, giving rise to disorder in the relative orientations of the iodine and methyl substituents.

Experimental. The title compound was prepared by J. S. McCallum and R. Bergman of this department as part of an investigation of tris(pyrazolyl)borate analogs of (cyclopentadienyl)- and pentamethylcyclopentadienyl)rhodium complexes. The preparation involved the reaction of the hydrido(trispyrazolylborato)(triphenylphosphino)diiodorhodium(III) complex with methyl iodide (McCallum & Bergman, unpublished results).

Air stable, transparent orange crystal, dimensions 0·24 × 0·18 × 0·08 mm. D_m measured by flotation in a mixture of $CHBr_3$ and CCl_4. Enraf–Nonius CAD-4 diffractometer; 24 reflections ($25{\cdot}8 \le 2\theta \le 30{\cdot}6°$) used to refine cell parameters. Data collection: $3 \le 2\theta \le 45°$; index ranges $0 \le h \le 33$, $0 \le k \le 10$, $-19 \le l \le 19$, excluding (hkl) $h + k = 2n + 1$; θ–2θ scan technique with Mo $K\alpha$ radiation and a graphite monochromator at room temperature. 4001 total reflections; systematic absences: ($h0l$) $l \neq 2n$; 3736 unique reflections; 3072 reflections with $I > 3\sigma(I)$. No indication of crystal decomposition from three standard reflections measured every hour. Empirical absorption correction was performed, ψ-scan variation <5% on I. Structure solved by Patterson methods and refined *via* standard full-matrix least

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0108-2701/91/081732-03$03.00