

$|F_o| > 2\sigma(|F_o|)$ 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 \AA^2 . Full-matrix refinement on F (232 parameters) converged at $R = 0.038$, $wR = 0.035$ [$w = 1/\sigma^2(F_o)$]. Max. Δ/σ in final cycle = 0.003 , $\Delta\rho$ fluctuations within $+0.50$ and -1.06 e \AA^{-3} . Calculations were performed with *SHELX76* (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

* 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.

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).

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

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Abstract. C₂₈H₂₈BN₆IPRh, $M_r = 720.16$, monoclinic, $C2/c$, $a = 31.552$ (6), $b = 9.601$ (1), $c = 18.606$ (2) Å, $\beta = 93.86$ (1)°, $V = 5623$ (2) Å³, $Z = 8$, $D_m = 1.65$, $D_x = 1.701 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 17.7 \text{ cm}^{-1}$, $F(000) = 2848$, $T = 298 \text{ K}$, $R = 0.041$, $wR = 0.060$, 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 prepara-

tion involved the reaction of the hydrido(trispyrazolylborato)(triphenylphosphino)diodorhodium(III) complex with methyl iodide (McCallum & Bergman, unpublished results).

Air stable, transparent orange crystal, dimensions $0.24 \times 0.18 \times 0.08 \text{ mm}$. D_m measured by flotation in a mixture of CHBr₃ and CCl₄. Enraf–Nonius CAD-4 diffractometer; 24 reflections ($25.8 \leq 2\theta \leq 30.6^\circ$) used to refine cell parameters. Data collection: $3 \leq 2\theta \leq 45^\circ$; index ranges $0 \leq h \leq 33$, $0 \leq k \leq 10$, $-19 \leq l \leq 19$, 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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squares (on F) and Fourier synthesis techniques with a Digital Equipment MicroVAX computer using *SDP-Plus* software (Frenz, 1985) modified locally by F. Hollander. Function minimized was $\sum w(F_o - |F_c|)^2$, $w = 1/\sigma^2(F_o)$; p factor = 0.03; Non-H atoms with the exception of the disordered coordinated methyl C atoms refined with anisotropic thermal parameters, methyl C atoms refined with isotropic thermal parameters. Disorder of the iodine and methyl positions was modeled as a 1:1 mixture of the two enantiomers, *i.e.*, two iodine and two methyl positions were refined at 50% occupancy. Variation of the multiplicities around 50% led to an increased R factor. H atoms located on difference Fourier map, positions of H atoms bound to C atoms (except the disordered methyl C atoms) calculated using idealized geometry and C—H distances of 0.95 Å, but not refined; isotropic thermal parameters assigned by $B(H) = 1.2 \times B_{eq}(C)$. Final number of parameters refined = 351, $R = 0.041$, $wR = 0.060$, $S = 2.487$; unit weights; max. shift/e.s.d. on final cycle = 0.01; largest peak on final difference Fourier map $0.722 \text{ e } \text{Å}^{-3}$; no secondary-extinction coefficient included in refinement. Atomic scattering factors and anomalous corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV).

An *ORTEP* (Johnson, 1965) drawing of enantiomer (*A*) of the title compound with the atom-numbering scheme is shown in Fig. 1. Enantiomer (*B*) has a similar structure, with the positions of the I atom and methyl groups interchanged. Atomic coordinates and final equivalent isotropic temperature

Table 1. *Positional parameters with their e.s.d.'s in parentheses*

$$B_{eq} = (1/3) \sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Rh	0.38788 (2)	0.19600 (5)	0.43632 (3)	2.44 (1)
I(A)	0.39791 (3)	0.2356 (1)	0.57266 (5)	4.19 (2)
I(B)	0.33132 (4)	0.0295 (1)	0.48125 (7)	5.43 (3)
P	0.34340 (5)	0.3855 (2)	0.41273 (9)	2.74 (4)
N(1)	0.4294 (2)	0.0252 (5)	0.4497 (3)	2.8 (1)
N(2)	0.4581 (2)	0.0053 (5)	0.4010 (3)	2.9 (1)
N(3)	0.4435 (2)	0.3093 (5)	0.4122 (3)	2.8 (1)
N(4)	0.4694 (2)	0.2527 (6)	0.3647 (3)	3.2 (1)
N(5)	0.3810 (2)	0.1289 (5)	0.3268 (3)	2.5 (1)
N(6)	0.4170 (2)	0.1013 (5)	0.2938 (3)	2.9 (1)
C(A)	0.3343 (8)	0.074 (3)	0.433 (1)	9.5 (6)†
C(B)	0.408 (1)	0.257 (4)	0.527 (2)	12.8 (9)†
C(1)	0.4809 (2)	-0.1090 (7)	0.4182 (4)	4.1 (2)
C(2)	0.4666 (2)	-0.1642 (7)	0.4794 (4)	4.3 (2)
C(3)	0.4351 (2)	-0.0792 (7)	0.4970 (4)	3.7 (2)
C(4)	0.5012 (2)	0.3383 (8)	0.3526 (4)	3.8 (2)
C(5)	0.4598 (2)	0.4363 (7)	0.4288 (3)	3.5 (2)
C(6)	0.4969 (2)	0.4556 (8)	0.3921 (4)	4.1 (2)
C(7)	0.4071 (2)	0.0601 (7)	0.2256 (4)	3.6 (2)
C(9)	0.3490 (2)	0.1043 (7)	0.2776 (4)	3.4 (2)
C(10)	0.2927 (2)	0.3506 (7)	0.3609 (4)	3.1 (1)
C(11)	0.2835 (2)	0.4052 (8)	0.2939 (4)	4.1 (2)
C(12)	0.2458 (3)	0.378 (1)	0.2563 (4)	5.6 (2)
C(13)	0.2162 (3)	0.2950 (9)	0.2853 (5)	6.0 (2)
C(14)	0.2233 (2)	0.2383 (9)	0.3547 (5)	5.1 (2)
C(15)	0.2630 (2)	0.2672 (8)	0.3910 (4)	4.0 (2)
C(16)	0.3681 (2)	0.5137 (7)	0.3551 (3)	2.9 (1)
C(17)	0.3875 (2)	0.4683 (7)	0.2942 (4)	3.6 (2)
C(18)	0.4062 (3)	0.5599 (8)	0.2525 (4)	4.5 (2)
C(19)	0.4081 (3)	0.6999 (8)	0.2688 (4)	4.7 (2)
C(20)	0.3885 (3)	0.7462 (7)	0.3268 (4)	4.3 (2)
C(21)	0.3689 (2)	0.6545 (7)	0.3704 (4)	3.4 (2)
C(22)	0.3278 (2)	0.4939 (7)	0.4871 (3)	3.3 (1)
C(23)	0.3600 (3)	0.5517 (8)	0.5334 (4)	4.6 (2)
C(24)	0.3500 (3)	0.6427 (9)	0.5882 (4)	5.7 (2)
C(25)	0.3070 (3)	0.6742 (9)	0.5980 (4)	5.7 (2)
C(26)	0.2759 (3)	0.619 (1)	0.5518 (5)	6.2 (2)
C(27)	0.2862 (3)	0.5273 (8)	0.4982 (4)	4.4 (2)
B	0.4604 (3)	0.1014 (9)	0.3373 (4)	3.5 (2)

† Refined with isotropic temperature factors.

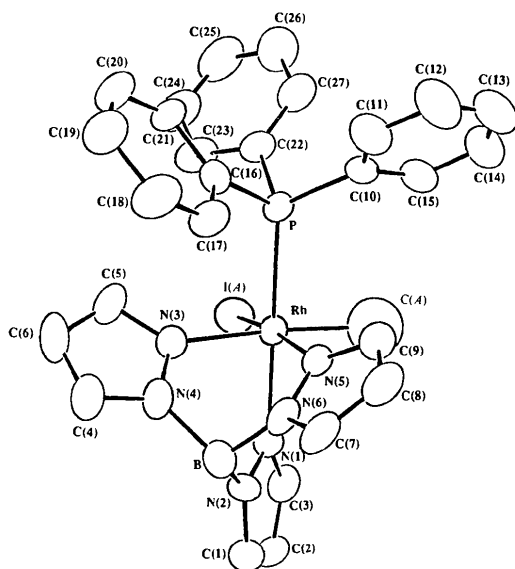


Fig. 1. *ORTEP* (Johnson, 1965) drawing of enantiomer (*A*) of the title compound showing the atom-numbering scheme. Ellipsoids are drawn at the 50% probability level.

Table 2. *Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses*

Rh—I(A)*	2.565 (1)	Rh—C(B)	1.86 (3)
Rh—I(B)	2.577 (1)	P—C(10)	1.844 (6)
Rh—P	2.322 (2)	P—C(16)	1.839 (7)
Rh—N(1)	2.103 (5)	P—C(22)	1.825 (7)
Rh—N(3)	2.138 (5)	B—N(2)	1.508 (10)
Rh—N(5)	2.135 (5)	B—N(4)	1.560 (10)
Rh—C(A)	2.05 (3)	B—N(6)	1.543 (9)
I(A)—Rh—P	95.96 (5)	N(3)—Rh—N(5)	89.2 (2)
I(B)—Rh—P	97.17 (5)	N(3)—Rh—C(A)	166.0 (7)
I(A)—Rh—N(1)	87.9 (1)	N(5)—Rh—C(B)	166 (1)
I(B)—Rh—N(3)	85.2 (1)	N(5)—Rh—C(A)	76.8 (7)
I(A)—Rh—N(5)	94.8 (1)	N(3)—Rh—C(B)	78 (1)
I(B)—Rh—N(5)	95.4 (1)	Rh—P—C(10)	116.7 (2)
I(A)—Rh—N(3)	170.9 (1)	Rh—P—C(16)	111.1 (2)
I(B)—Rh—N(3)	168.5 (1)	Rh—P—C(22)	119.6 (2)
I(A)—Rh—C(A)	99.1 (7)	Rh—N(1)—N(2)	118.4 (4)
I(B)—Rh—C(B)	96 (1)	B—N(2)—N(1)	121.0 (5)
P—Rh—N(1)	175.8 (1)	B—N(2)—C(1)	129.6 (6)
P—Rh—N(3)	93.2 (1)	B—N(4)—N(3)	118.8 (5)
P—Rh—N(5)	92.0 (1)	B—N(4)—C(4)	130.1 (6)
P—Rh—C(A)	87.5 (7)	B—N(6)—N(5)	120.3 (5)
P—Rh—C(B)	95 (1)	B—N(6)—C(7)	129.6 (6)
N(1)—Rh—N(3)	84.8 (2)	P—C(10)—C(11)	122.1 (5)
N(1)—Rh—N(5)	84.3 (2)	P—C(16)—C(17)	119.4 (5)
N(1)—Rh—C(A)	93.6 (7)	P—C(16)—C(21)	122.5 (5)
N(1)—Rh—C(B)	88 (1)	P—C(22)—C(23)	118.1 (5)

* Atoms designated (*A*) and (*B*) occur in the (*A*) and (*B*) enantiomers of the crystallized compound.

factors of all non-H atoms are given in Table 1.* Selected interatomic distances and angles for both enantiomers are listed in Table 2.

Related literature. Other structures containing trispyrazolylborate groups coordinated to rhodium have been reported, including [Rh^{III}(Bpz₄)(CO)(I)₂] (Cocivera, Desmond, Ferguson, Kaitner, Lalor & O'Sullivan, 1982), [Rh^{III}(C₅Me₅)(HBpz₃)] [PF₆] (Restivo, Ferguson, O'Sullivan & Lalor, 1975), and [Rh^{III}{HB(3,5-Me₂pz)₃}(C₂H₅)(C₆H₅)(CO)] (3,5-Me₂pz = 3,5-dimethylpyrazolyl) (Ghosh & Graham, 1989).

* 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 54020 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

This structure was solved at the X-ray Crystallographic Facility (CHEXRAY) at the Department of Chemistry, University of California, Berkeley, as part of the requirements of a graduate chemistry course on structure analysis by X-ray diffraction. We thank Mr Liang Tong, Dr F. Hollander and Professor D. H. Templeton for their guidance.

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Structure of Manganese Acetate Dihydrate

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Abstract. Hexaacetatotetraaquatrimanganese dihydrate, [Mn₃(CH₃COO)₆(H₂O)₄].2H₂O, *M_r* = 627.3, monoclinic, *P*2₁/*c*, *a* = 8.982 (4), *b* = 9.155 (4), *c* = 16.514 (5) Å, β = 114.18 (3)°, *V* = 1239 (1) Å³, *Z* = 2, *D_x* = 1.678 Mg m⁻³, λ = 0.71073 Å, μ = 1.518 mm⁻¹, *F*(000) = 642, *T* = 297 K, *R* = 0.044, *wR* = 0.044 for 1271 unique reflections with *I* > 3.0σ(*I*). The title compound consists of MnO₆ octahedra which are linked by acetate bridges to form Mn₃(OAc)₆(H₂O)₄ units in the *bc* plane. This structure is closely related to that of the octahydrate Mn₃(CH₃COO)₆(H₂O)₄.8H₂O [Bertaut, Duc, Burlet, Burlet, Thomas & Moreau (1974). *Acta Cryst.* **B30**, 2234–2236]. In the crystal structure of the title compound, two thirds of the water molecules are in the manganese coordination sphere and the rest reside in the interlayer region. Two adjacent layers are 8.2 Å apart and held by hydrogen bonds through water and acetate moieties.

Experimental. The sample was obtained as pink crystals by heating a reaction mixture of Mn₃O₄

(0.6g) and 14.0 ml 12.5 *M* acetic acid in a 23 ml teflon-lined autoclave at 503 K for 2 d followed by slow cooling to room temperature. The structure of Mn₃(CH₃COO)₆(H₂O)₄.2H₂O was determined from single-crystal X-ray diffraction. Peak profile analysis (*ω* scan) on the pink plate crystals using a Nicolet *R3m/V* diffractometer with Mo *Kα* radiation indicated that most of them were not suitable for indexing and intensity data collection. Many had to be selected from the reaction products before a satisfactory crystal was obtained. Finally a piece of dimensions 0.06 × 0.10 × 0.12 mm was chosen. Some of its reflections also showed broad peak profiles but the quality of the intensity data was considered to be adequate for elucidation of the structure. The unit-cell parameters were determined by a least-squares fit of 15 reflections with 2θ ranging from 12.8 to 27.9°. The intensity data were collected up to 2θ = 50° (sinθ_{max}/λ = 0.594 Å⁻¹) with θ/2θ scans. A periodic check of three standard reflections did not reveal any significant variation in intensity. A total of 3587 reflections were measured in the range -10 ≤ *h* ≤ 10, 0 ≤ *k* ≤ 10, 0 ≤ *l* ≤ 19. Scan width 1.20° plus *Kα* separation and scan speed 2.93–14.95° min⁻¹. The

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