

supplementary materials

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{*N'*-[(*E*)-1-(5-Bromo-2-oxidophenyl)ethylidene]-4-chlorobenzohydrazidato}pyridinenickel(II)

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Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions (Singh *et al.*, 1982; Salem, 1998; Ali *et al.*, 2004) and their biological activity (Singh *et al.*, 1982; Carcelli *et al.*, 1995). As an extension of work on the structural characterization of aroylhydrazone derivatives, the title compound was synthesized and its crystal structure is reported.

Experimental

A DMF solution (5 ml) of *N'*-[(*E*)-(5-bromo-2-hydroxyphenyl)ethylidene]-4-chlorobenzo-hydrazide (0.25 mmol, 0.092 g) was mixed with a methanol solution (5 ml) of NiCl₂.6H₂O (0.25 mmol, 0.059 g). The mixture was stirred at 298 K for 4 h and then filtered. A green precipitate was produced after about 10 d. A pyridine mixture (5 ml) was used to dissolve the precipitate at 330 K. Dark green block-shaped crystals were obtained after one month (yield 30%).

Refinement

H atoms were placed in calculated positions, with C—H bond lengths fixed to 0.93 (aromatic CH) or 0.96 Å (methyl CH₃). Isotropic displacement parameters for H atoms were computed as $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{carrier C})$ with $x = 1.2$ (aromatic CH) or 1.5 (methyl CH₃).

Figures

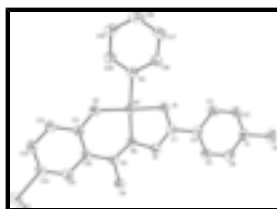


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

{*N'*-[(*E*)-1-(5-Bromo-2-oxidophenyl)ethylidene]-4- chlorobenzohydrazidato}pyridinenickel(II)

Crystal data

[Ni(C₁₅H₁₀BrClNO₂)(C₅H₅N)]

$M_r = 503.42$

Monoclinic, *C*2/*c*

Hall symbol: -*C* 2yc

$a = 32.430 (4) \text{ \AA}$

$F_{000} = 2016$

$D_x = 1.738 \text{ Mg m}^{-3}$

Melting point: 330 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2958 reflections

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$b = 6.0816$ (8) Å	$\theta = 2.6\text{--}25.0^\circ$
$c = 22.865$ (3) Å	$\mu = 3.25$ mm ⁻¹
$\beta = 121.422$ (2)°	$T = 278$ K
$V = 3848.3$ (9) Å ³	Block, green
$Z = 8$	$0.18 \times 0.13 \times 0.10$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	3401 independent reflections
Radiation source: fine-focus sealed tube	2651 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.028$
$T = 278$ K	$\theta_{\text{max}} = 25.1^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.5^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -37 \rightarrow 38$
$T_{\text{min}} = 0.581$, $T_{\text{max}} = 0.723$	$k = -7 \rightarrow 7$
9680 measured reflections	$l = -24 \rightarrow 27$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 2.3403P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
3401 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
253 parameters	$\Delta\rho_{\text{max}} = 0.48$ e Å ⁻³
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.27$ e Å ⁻³
	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.041268 (13)	0.23786 (6)	0.156018 (18)	0.04192 (13)
Br1	-0.211124 (13)	-0.09987 (7)	-0.01369 (2)	0.07519 (15)
Cl1	0.26998 (4)	-0.39055 (19)	0.14682 (6)	0.0893 (3)
O1	0.09933 (7)	0.1537 (3)	0.16792 (10)	0.0483 (5)
O2	-0.01644 (7)	0.3235 (3)	0.14192 (11)	0.0532 (5)
N1	0.05323 (9)	-0.1250 (4)	0.09609 (12)	0.0460 (6)
N2	0.01755 (9)	-0.0048 (4)	0.09955 (11)	0.0418 (6)
N3	0.07253 (9)	0.4809 (4)	0.22016 (12)	0.0449 (6)
C1	0.21951 (12)	-0.2894 (6)	0.14580 (16)	0.0572 (9)
C2	0.22207 (12)	-0.0906 (6)	0.17554 (17)	0.0647 (9)
H2	0.2510	-0.0129	0.1980	0.078*
C3	0.18122 (12)	-0.0071 (6)	0.17171 (16)	0.0578 (8)

H3	0.1827	0.1285	0.1915	0.069*
C4	0.13809 (11)	-0.1220 (5)	0.13888 (14)	0.0454 (7)
C5	0.13736 (13)	-0.3249 (5)	0.11103 (18)	0.0595 (9)
H5	0.1089	-0.4065	0.0899	0.071*
C6	0.17748 (13)	-0.4080 (6)	0.11382 (18)	0.0646 (9)
H6	0.1763	-0.5437	0.0942	0.077*
C7	0.09448 (11)	-0.0276 (5)	0.13425 (14)	0.0437 (7)
C8	-0.02649 (11)	-0.0808 (5)	0.06426 (13)	0.0417 (7)
C9	-0.03642 (12)	-0.2861 (5)	0.02193 (16)	0.0539 (8)
H9A	-0.0094	-0.3164	0.0169	0.081*
H9B	-0.0649	-0.2649	-0.0225	0.081*
H9C	-0.0413	-0.4077	0.0444	0.081*
C10	-0.06526 (10)	0.0294 (5)	0.06629 (13)	0.0415 (7)
C11	-0.05843 (11)	0.2266 (5)	0.10261 (15)	0.0456 (7)
C12	-0.09895 (12)	0.3312 (5)	0.09768 (16)	0.0545 (8)
H12	-0.0947	0.4649	0.1198	0.065*
C13	-0.14427 (12)	0.2416 (6)	0.06125 (17)	0.0564 (8)
H13	-0.1706	0.3138	0.0582	0.068*
C14	-0.15003 (11)	0.0428 (6)	0.02932 (15)	0.0516 (8)
C15	-0.11213 (11)	-0.0603 (5)	0.03060 (14)	0.0487 (7)
H15	-0.1174	-0.1926	0.0074	0.058*
C16	0.12054 (12)	0.4904 (6)	0.25973 (16)	0.0600 (9)
H16	0.1387	0.3768	0.2571	0.072*
C17	0.14431 (14)	0.6615 (6)	0.30425 (19)	0.0701 (10)
H17	0.1779	0.6628	0.3311	0.084*
C18	0.11809 (15)	0.8293 (6)	0.30861 (17)	0.0655 (10)
H18	0.1335	0.9482	0.3375	0.079*
C19	0.06898 (14)	0.8192 (5)	0.26974 (17)	0.0589 (9)
H19	0.0503	0.9294	0.2727	0.071*
C20	0.04720 (12)	0.6437 (5)	0.22580 (15)	0.0525 (8)
H20	0.0136	0.6388	0.1992	0.063*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0420 (2)	0.0397 (2)	0.0444 (2)	-0.00392 (17)	0.02273 (18)	-0.00596 (17)
Br1	0.0486 (2)	0.0968 (3)	0.0797 (3)	-0.0210 (2)	0.0332 (2)	-0.0084 (2)
Cl1	0.0596 (6)	0.1029 (8)	0.1085 (8)	0.0179 (6)	0.0461 (6)	-0.0134 (6)
O1	0.0456 (12)	0.0462 (12)	0.0522 (12)	-0.0061 (9)	0.0248 (10)	-0.0130 (10)
O2	0.0433 (13)	0.0501 (12)	0.0657 (13)	-0.0055 (10)	0.0281 (11)	-0.0161 (11)
N1	0.0454 (15)	0.0431 (15)	0.0514 (15)	0.0007 (12)	0.0265 (13)	-0.0048 (12)
N2	0.0445 (15)	0.0388 (13)	0.0440 (13)	-0.0011 (11)	0.0245 (12)	-0.0025 (11)
N3	0.0486 (16)	0.0436 (14)	0.0464 (14)	-0.0058 (12)	0.0275 (12)	-0.0051 (11)
C1	0.047 (2)	0.067 (2)	0.058 (2)	0.0122 (17)	0.0272 (17)	0.0007 (17)
C2	0.044 (2)	0.074 (3)	0.074 (2)	-0.0059 (17)	0.0297 (18)	-0.0151 (19)
C3	0.054 (2)	0.057 (2)	0.067 (2)	-0.0062 (17)	0.0347 (18)	-0.0163 (17)
C4	0.0454 (18)	0.0493 (19)	0.0432 (16)	0.0000 (14)	0.0242 (15)	-0.0001 (14)
C5	0.050 (2)	0.054 (2)	0.072 (2)	-0.0075 (16)	0.0299 (18)	-0.0163 (17)

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C6	0.065 (2)	0.056 (2)	0.078 (2)	0.0055 (18)	0.040 (2)	-0.0129 (18)
C7	0.0457 (18)	0.0448 (17)	0.0423 (16)	0.0001 (14)	0.0241 (15)	0.0003 (14)
C8	0.0465 (18)	0.0381 (16)	0.0388 (15)	-0.0049 (13)	0.0210 (14)	0.0000 (13)
C9	0.055 (2)	0.0444 (19)	0.0595 (19)	-0.0082 (15)	0.0274 (17)	-0.0105 (15)
C10	0.0414 (17)	0.0421 (17)	0.0425 (15)	-0.0046 (13)	0.0229 (14)	0.0012 (13)
C11	0.0441 (18)	0.0476 (18)	0.0494 (17)	-0.0047 (14)	0.0274 (15)	0.0001 (14)
C12	0.053 (2)	0.0525 (19)	0.064 (2)	-0.0029 (16)	0.0350 (17)	-0.0048 (16)
C13	0.047 (2)	0.066 (2)	0.066 (2)	0.0010 (17)	0.0358 (17)	0.0043 (18)
C14	0.0421 (18)	0.063 (2)	0.0496 (18)	-0.0102 (16)	0.0239 (15)	0.0023 (16)
C15	0.0504 (19)	0.0510 (18)	0.0465 (17)	-0.0095 (15)	0.0266 (15)	-0.0020 (14)
C16	0.048 (2)	0.064 (2)	0.070 (2)	-0.0108 (17)	0.0320 (18)	-0.0193 (18)
C17	0.059 (2)	0.075 (2)	0.074 (2)	-0.025 (2)	0.033 (2)	-0.028 (2)
C18	0.085 (3)	0.059 (2)	0.062 (2)	-0.028 (2)	0.045 (2)	-0.0210 (18)
C19	0.079 (3)	0.0463 (19)	0.059 (2)	-0.0047 (18)	0.042 (2)	-0.0088 (16)
C20	0.056 (2)	0.0511 (19)	0.0510 (18)	-0.0009 (16)	0.0288 (17)	-0.0042 (15)

Geometric parameters (Å, °)

Ni1—O2	1.802 (2)	C8—C10	1.447 (4)
Ni1—O1	1.830 (2)	C8—C9	1.508 (4)
Ni1—N2	1.844 (2)	C9—H9A	0.9600
Ni1—N3	1.953 (2)	C9—H9B	0.9600
Br1—C14	1.901 (3)	C9—H9C	0.9600
Cl1—C1	1.737 (3)	C10—C15	1.407 (4)
O1—C7	1.306 (3)	C10—C11	1.409 (4)
O2—C11	1.317 (3)	C11—C12	1.410 (4)
N1—C7	1.298 (4)	C12—C13	1.369 (4)
N1—N2	1.405 (3)	C12—H12	0.9300
N2—C8	1.305 (4)	C13—C14	1.374 (4)
N3—C16	1.333 (4)	C13—H13	0.9300
N3—C20	1.335 (4)	C14—C15	1.366 (4)
C1—C2	1.368 (5)	C15—H15	0.9300
C1—C6	1.369 (5)	C16—C17	1.377 (4)
C2—C3	1.378 (4)	C16—H16	0.9300
C2—H2	0.9300	C17—C18	1.364 (5)
C3—C4	1.383 (4)	C17—H17	0.9300
C3—H3	0.9300	C18—C19	1.362 (5)
C4—C5	1.383 (4)	C18—H18	0.9300
C4—C7	1.478 (4)	C19—C20	1.381 (4)
C5—C6	1.366 (5)	C19—H19	0.9300
C5—H5	0.9300	C20—H20	0.9300
C6—H6	0.9300		
O2—Ni1—O1	178.39 (9)	C8—C9—H9A	109.5
O2—Ni1—N2	95.20 (10)	C8—C9—H9B	109.5
O1—Ni1—N2	84.35 (9)	H9A—C9—H9B	109.5
O2—Ni1—N3	89.99 (10)	C8—C9—H9C	109.5
O1—Ni1—N3	90.52 (9)	H9A—C9—H9C	109.5
N2—Ni1—N3	174.43 (11)	H9B—C9—H9C	109.5
C7—O1—Ni1	110.50 (18)	C15—C10—C11	117.5 (3)

C11—O2—Ni1	126.92 (19)	C15—C10—C8	119.7 (3)
C7—N1—N2	108.4 (2)	C11—C10—C8	122.7 (3)
C8—N2—N1	116.7 (2)	O2—C11—C10	124.9 (3)
C8—N2—Ni1	129.7 (2)	O2—C11—C12	116.2 (3)
N1—N2—Ni1	113.61 (18)	C10—C11—C12	118.9 (3)
C16—N3—C20	117.5 (3)	C13—C12—C11	122.0 (3)
C16—N3—Ni1	120.5 (2)	C13—C12—H12	119.0
C20—N3—Ni1	122.0 (2)	C11—C12—H12	119.0
C2—C1—C6	121.1 (3)	C12—C13—C14	118.6 (3)
C2—C1—C11	119.6 (3)	C12—C13—H13	120.7
C6—C1—C11	119.3 (3)	C14—C13—H13	120.7
C1—C2—C3	119.1 (3)	C15—C14—C13	121.4 (3)
C1—C2—H2	120.4	C15—C14—Br1	119.0 (3)
C3—C2—H2	120.4	C13—C14—Br1	119.5 (2)
C2—C3—C4	121.0 (3)	C14—C15—C10	121.5 (3)
C2—C3—H3	119.5	C14—C15—H15	119.3
C4—C3—H3	119.5	C10—C15—H15	119.3
C3—C4—C5	118.1 (3)	N3—C16—C17	122.7 (3)
C3—C4—C7	120.1 (3)	N3—C16—H16	118.6
C5—C4—C7	121.8 (3)	C17—C16—H16	118.6
C6—C5—C4	121.4 (3)	C18—C17—C16	119.3 (4)
C6—C5—H5	119.3	C18—C17—H17	120.4
C4—C5—H5	119.3	C16—C17—H17	120.4
C5—C6—C1	119.2 (3)	C19—C18—C17	118.7 (3)
C5—C6—H6	120.4	C19—C18—H18	120.7
C1—C6—H6	120.4	C17—C18—H18	120.7
N1—C7—O1	123.1 (3)	C18—C19—C20	119.4 (3)
N1—C7—C4	118.9 (3)	C18—C19—H19	120.3
O1—C7—C4	118.0 (3)	C20—C19—H19	120.3
N2—C8—C10	120.4 (3)	N3—C20—C19	122.4 (3)
N2—C8—C9	119.3 (3)	N3—C20—H20	118.8
C10—C8—C9	120.4 (3)	C19—C20—H20	118.8
N2—Ni1—O1—C7	-2.06 (18)	N1—N2—C8—C10	178.4 (2)
N3—Ni1—O1—C7	175.76 (18)	Ni1—N2—C8—C10	-0.2 (4)
N2—Ni1—O2—C11	0.0 (2)	N1—N2—C8—C9	-1.0 (4)
N3—Ni1—O2—C11	-178.0 (2)	Ni1—N2—C8—C9	-179.5 (2)
C7—N1—N2—C8	-179.6 (2)	N2—C8—C10—C15	-177.0 (2)
C7—N1—N2—Ni1	-0.8 (3)	C9—C8—C10—C15	2.4 (4)
O2—Ni1—N2—C8	-1.3 (3)	N2—C8—C10—C11	3.4 (4)
O1—Ni1—N2—C8	-179.8 (3)	C9—C8—C10—C11	-177.3 (3)
O2—Ni1—N2—N1	-179.92 (18)	Ni1—O2—C11—C10	2.9 (4)
O1—Ni1—N2—N1	1.62 (17)	Ni1—O2—C11—C12	-177.3 (2)
O2—Ni1—N3—C16	168.9 (2)	C15—C10—C11—O2	175.5 (3)
O1—Ni1—N3—C16	-12.6 (2)	C8—C10—C11—O2	-4.9 (4)
O2—Ni1—N3—C20	-11.7 (2)	C15—C10—C11—C12	-4.3 (4)
O1—Ni1—N3—C20	166.7 (2)	C8—C10—C11—C12	175.4 (3)
C6—C1—C2—C3	-1.5 (5)	O2—C11—C12—C13	-176.7 (3)
C11—C1—C2—C3	177.0 (3)	C10—C11—C12—C13	3.1 (5)
C1—C2—C3—C4	0.5 (5)	C11—C12—C13—C14	0.7 (5)

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C2—C3—C4—C5	1.1 (5)	C12—C13—C14—C15	-3.2 (5)
C2—C3—C4—C7	-178.9 (3)	C12—C13—C14—Br1	173.6 (2)
C3—C4—C5—C6	-1.9 (5)	C13—C14—C15—C10	1.9 (5)
C7—C4—C5—C6	178.1 (3)	Br1—C14—C15—C10	-175.0 (2)
C4—C5—C6—C1	1.0 (5)	C11—C10—C15—C14	2.0 (4)
C2—C1—C6—C5	0.7 (5)	C8—C10—C15—C14	-177.7 (3)
C11—C1—C6—C5	-177.8 (3)	C20—N3—C16—C17	-1.3 (5)
N2—N1—C7—O1	-1.1 (4)	Ni1—N3—C16—C17	178.0 (3)
N2—N1—C7—C4	-179.6 (2)	N3—C16—C17—C18	0.0 (5)
Ni1—O1—C7—N1	2.4 (3)	C16—C17—C18—C19	1.5 (5)
Ni1—O1—C7—C4	-179.08 (19)	C17—C18—C19—C20	-1.8 (5)
C3—C4—C7—N1	171.6 (3)	C16—N3—C20—C19	1.0 (4)
C5—C4—C7—N1	-8.4 (4)	Ni1—N3—C20—C19	-178.3 (2)
C3—C4—C7—O1	-7.0 (4)	C18—C19—C20—N3	0.5 (5)
C5—C4—C7—O1	173.0 (3)		

Fig. 1

