Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[2-(1*H*-benzimidazol-2-yl-κN^3)-4,6-dibromophenolato-κO]nickel(II)

Yu Wu,[a,b] Bin Xie,[a] Like Zou,[a] Jian-Shen Feng[a] and Zongqiu Hu[b]*

[a]Department of Chemistry, Sichuan University of Science and Engineering, Zigong, Sichuan 643000, People's Republic of China, and [b]Department of Chemistry, Central China Normal University, Wuhan, Hubei 430079, People's Republic of China
Correspondence e-mail: solicitor.wu@126.com

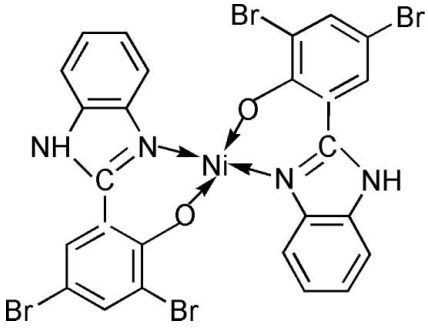
Received 24 July 2007; accepted 3 August 2007

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.013 Å; R factor = 0.065; wR factor = 0.180; data-to-parameter ratio = 14.6.

The title compound, $[Ni(C_{13}H_7Br_2N_2O)_2]$, an Ni^{II} complex of the Schiff base 2-(3,5-dibromo-2-hydroxyphenyl)benzimidazole, was synthesized by the reaction of 3,5-dibromosalicylaldehyde and 1,2-phenylenediamine. The molecule resides on a twofold rotation axis. The Ni^{II} atom exists in a distorted tetrahedral geometry and is coordinated by one O and one N atom from each of two 2-(3,5-dibromo-2-hydroxyphenyl)benzimidazole ligands. The crystal structure is stabilized by N—H···Br and N—H···O hydrogen bonds, which link the molecules into a chain along the *b* axis.

Related literature

For ligand synthesis, see: Elzbieta *et al.* (1964). For a related structure, see: Wang *et al.* (2003).



Experimental

Crystal data

$[Ni(C_{13}H_7Br_2N_2O)_2]$
M_r = 792.76
Tetragonal, $I4_1/a$
a = 12.4177 (14) Å
c = 33.389 (6) Å
V = 5148.6 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 7.00 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.228, T_{max} = 0.335
(expected range = 0.168–0.247)
13205 measured reflections
2510 independent reflections
1324 reflections with $I > 2\sigma(I)$
R_{int} = 0.127

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.180
S = 0.95
2510 reflections
172 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.90 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	1.957 (6)	Ni1—O1	1.984 (6)
N1—Ni1—N1^{i}	117.8 (4)	N1—Ni1—O1^{i}	116.8 (2)
N1—Ni1—O1	94.4 (2)	O1—Ni1—O1^{i}	118.7 (3)

Symmetry code: (i) $-x+2, -y+\frac{1}{2}, z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Br1^{ii}	0.86 (3)	2.74 (7)	3.464 (7)	143 (9)
N2—H2A···O1^{ii}	0.86 (3)	2.11 (8)	2.811 (9)	138 (9)

Symmetry code: (ii) $x, y+\frac{1}{2}, -z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Sichuan Province Education Department, Sichuan, China (grant No. 2006 A110).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2430).

References

Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Elzbieta, C., Zygmunt, E. & Romuald, K. (1964). *Diss. Pharm.* **15**, 369–378.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wang, J., Bei, F.-L. & Ma, W.-X. (2003). *Chin. J. Inorg. Chem.* **19**, 609–612.

  doi:10.1107/S1600536807038214

supplementary materials

Acta Cryst. (2007). E**63**, m2298 [**doi:10.1107/S1600536807038214**]

Bis[2-(1*H*-benzimidazol-2-yl-κN^3)-4,6-dibromophenolato-κO]nickel(II)

Y. Wu, B. Xie, L. Zou, J.-S. Feng and Z. Hu

Comment

Crystal structure and properties of 1,2-*N*,*N*-disallicydene-phenylamineato nickel(II) has been reported (Wang *et al.*, 2003). We report here the synthesis and crystal structure of bis[2-(3,5-dibromo-2-hydroxyphenyl) benzimidazole]nickel(II).

The asymmetric unit of the title compound consists of a half-molecule, with the Ni^{II} atom lying on a crystallographic twofold axis; the other half of the molecule is generated by the twofold axis (Fig. 1). The Ni^{II} atom exists in a distorted tetrahedral geometry (Table 1) and is coordinated by the O and one N atom each from two 3,5-dibromo-2-hydroxyphenyl benzimidazole ligands.

The crystal structure is stabilized by N—H···Br and N—H···O type hydrogen bonds which link the molecules into a chain along the *b* axis.

Experimental

3,5-Dibromosalicylaldehyde was prepared according to the literature method (Elzbieta *et al.*, 1964). To a solution of 1,2-phenylenediamine (1 g) in pyridine (30 ml), one mole equivalent of 3,5-dibromosalicylaldehyde in pyridine (30 ml) was added slowly under continuous stirring and refluxed for 1 h. Then $Ni(Ac)_2$ (10 mmol) in DMF (10 ml) was added and the solution were refluxed for 1 h. The hot solution was filtered and allowed to stand at room temperature undisturbed for about three weeks, resulting in yellow crystals.

Refinement

The N-bound H atom was located in a difference map and refined with a N—H distance restraint of 0.86 (2) Å. C-bound H atoms were placed at calculated positions and refined using a riding model, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

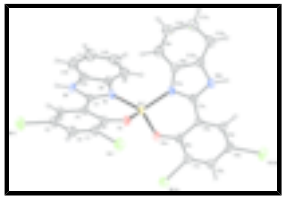


Fig. 1. The molecular structure of the title compound, showing labelling of the non-H atoms and 20% probability displacement ellipsoids. Atoms labelled with the suffixe a are generated by the symmetry operation $(2 - x, 1/2 - y, z)$.

Bis[2-(1*H*-benzimidazol-2-yl-κN^3)-4,6-dibromophenolato-κ*O*]nickel(II)

Crystal data

$[Ni(C_{13}H_7Br_2N_2O)_2]$	$Z = 8$
$M_r = 792.76$	$F_{000} = 3056$
Tetragonal, $I4_1/a$	$D_x = 2.045$ Mg m^{-3}
Hall symbol: -I 4ad	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 12.4177$ (14) Å	Cell parameters from 1302 reflections
$b = 12.4177$ (14) Å	$\theta = 2.4$–16.7º
$c = 33.389$ (6) Å	$\mu = 7.00$ mm^{-1}
$\alpha = 90$º	$T = 292$ (2) K
$\beta = 90$º	Block, yellow
$\gamma = 90$º	0.30 × 0.20 × 0.20 mm
$V = 5148.6$ (12) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2510 independent reflections
Radiation source: fine-focus sealed tube	1324 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.127$
$T = 292(2)$ K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 2.5$º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -15 \rightarrow 9$
$T_{min} = 0.228$, $T_{max} = 0.335$	$k = -15 \rightarrow 14$
13205 measured reflections	$l = -41 \rightarrow 41$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.065$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.180$	$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{max} = 0.001$
2510 reflections	$\Delta\rho_{max} = 0.90$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.48$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	1.0000	0.2500	0.00587 (4)	0.0486 (4)
Br1	0.69106 (11)	0.20197 (9)	−0.08406 (3)	0.0957 (5)
Br2	0.54458 (11)	0.61501 (10)	−0.04228 (4)	0.1101 (6)
N1	0.9689 (5)	0.3813 (5)	0.03615 (18)	0.0519 (17)
C1	0.7060 (8)	0.3281 (7)	−0.0523 (2)	0.068 (3)
C2	0.6348 (8)	0.4068 (8)	−0.0575 (3)	0.069 (3)
H2	0.5784	0.3992	−0.0756	0.083*
C3	0.6467 (8)	0.5014 (8)	−0.0351 (3)	0.073 (3)
C4	0.7292 (8)	0.5120 (7)	−0.0096 (3)	0.066 (2)
H4	0.7366	0.5759	0.0046	0.080*
C5	0.8040 (7)	0.4306 (6)	−0.0038 (2)	0.054 (2)
C6	0.7982 (7)	0.3334 (7)	−0.0261 (2)	0.054 (2)
C7	0.8901 (6)	0.4497 (6)	0.0259 (2)	0.0455 (18)
C8	1.0267 (6)	0.4270 (6)	0.0663 (2)	0.0498 (19)
C9	1.1152 (8)	0.3939 (7)	0.0879 (3)	0.073 (3)
H9	1.1459	0.3267	0.0833	0.087*
C10	1.1577 (9)	0.4622 (9)	0.1164 (3)	0.085 (3)
H10	1.2195	0.4418	0.1302	0.102*
C11	1.1105 (10)	0.5600 (9)	0.1249 (3)	0.094 (3)
H11	1.1388	0.6018	0.1454	0.112*
C12	1.0223 (9)	0.5978 (8)	0.1038 (3)	0.082 (3)
H12	0.9906	0.6641	0.1092	0.098*
C13	0.9851 (7)	0.5306 (7)	0.0744 (2)	0.057 (2)
O1	0.8626 (5)	0.2535 (4)	−0.02442 (16)	0.0602 (15)
N2	0.8956 (6)	0.5388 (6)	0.0481 (2)	0.0577 (18)
H2A	0.867 (8)	0.601 (5)	0.052 (3)	0.11 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0612 (10)	0.0366 (8)	0.0480 (7)	0.0184 (7)	0.000	0.000
Br1	0.1218 (10)	0.0805 (8)	0.0850 (7)	0.0258 (7)	−0.0409 (7)	−0.0212 (6)

Br2	0.1109 (10)	0.0997 (9)	0.1197 (10)	0.0604 (8)	−0.0217 (7)	0.0026 (7)
N1	0.066 (4)	0.033 (3)	0.056 (4)	0.014 (3)	0.003 (3)	−0.001 (3)
C1	0.085 (7)	0.064 (6)	0.056 (5)	0.021 (5)	−0.005 (5)	−0.003 (4)
C2	0.073 (6)	0.073 (7)	0.061 (5)	0.020 (5)	−0.021 (4)	−0.004 (5)
C3	0.082 (7)	0.064 (6)	0.072 (6)	0.041 (5)	−0.003 (5)	0.012 (5)
C4	0.081 (7)	0.058 (6)	0.060 (5)	0.027 (5)	0.001 (5)	0.005 (4)
C5	0.067 (6)	0.051 (5)	0.042 (4)	0.013 (4)	0.000 (4)	−0.001 (4)
C6	0.066 (6)	0.051 (5)	0.046 (4)	0.013 (4)	0.001 (4)	0.009 (4)
C7	0.059 (5)	0.031 (4)	0.047 (4)	0.001 (4)	0.017 (4)	0.009 (3)
C8	0.045 (5)	0.055 (5)	0.049 (4)	0.006 (4)	0.003 (4)	0.010 (4)
C9	0.091 (7)	0.047 (5)	0.080 (6)	−0.004 (5)	−0.007 (5)	0.007 (5)
C10	0.098 (8)	0.098 (9)	0.060 (6)	−0.018 (7)	−0.020 (5)	0.004 (5)
C11	0.118 (10)	0.073 (8)	0.090 (7)	0.002 (7)	−0.015 (7)	−0.016 (6)
C12	0.099 (8)	0.057 (6)	0.090 (7)	0.001 (6)	−0.004 (6)	−0.021 (5)
C13	0.070 (6)	0.044 (5)	0.057 (5)	0.003 (4)	0.010 (4)	−0.001 (4)
O1	0.080 (4)	0.038 (3)	0.062 (3)	0.016 (3)	−0.009 (3)	−0.005 (3)
N2	0.067 (5)	0.041 (4)	0.065 (4)	0.015 (4)	0.006 (4)	−0.004 (3)

Geometric parameters (Å, °)

Ni1—N1	1.957 (6)	C5—C7	1.480 (11)
Ni1—N1^{i}	1.957 (6)	C6—O1	1.276 (9)
Ni1—O1	1.984 (6)	C7—N2	1.333 (10)
Ni1—O1^{i}	1.984 (6)	C8—C9	1.378 (11)
Br1—C1	1.902 (9)	C8—C13	1.413 (11)
Br2—C3	1.913 (8)	C9—C10	1.379 (12)
N1—C7	1.340 (9)	C9—H9	0.93
N1—C8	1.359 (10)	C10—C11	1.378 (14)
C1—C2	1.329 (11)	C10—H10	0.93
C1—C6	1.442 (12)	C11—C12	1.385 (15)
C2—C3	1.400 (12)	C11—H11	0.93
C2—H2	0.93	C12—C13	1.367 (12)
C3—C4	1.338 (13)	C12—H12	0.93
C4—C5	1.385 (11)	C13—N2	1.421 (11)
C4—H4	0.93	N2—H2A	0.86 (3)
C5—C6	1.419 (11)		
N1—Ni1—N1^{i}	117.8 (4)	C5—C6—C1	113.3 (7)
N1—Ni1—O1	94.4 (2)	N2—C7—N1	110.3 (7)
N1^{i}—Ni1—O1	116.8 (2)	N2—C7—C5	122.9 (7)
N1—Ni1—O1^{i}	116.8 (2)	N1—C7—C5	126.7 (7)
N1^{i}—Ni1—O1^{i}	94.4 (2)	N1—C8—C9	133.1 (8)
O1—Ni1—O1^{i}	118.7 (3)	N1—C8—C13	109.3 (7)
C7—N1—C8	108.0 (6)	C9—C8—C13	117.5 (8)
C7—N1—Ni1	122.7 (5)	C8—C9—C10	118.9 (9)
C8—N1—Ni1	128.8 (5)	C8—C9—H9	120.6
C2—C1—C6	125.0 (8)	C10—C9—H9	120.6
C2—C1—Br1	117.9 (7)	C11—C10—C9	121.4 (10)

C6—C1—Br1	117.0 (6)	C11—C10—H10	119.3
C1—C2—C3	118.5 (8)	C9—C10—H10	119.3
C1—C2—H2	120.8	C10—C11—C12	122.1 (10)
C3—C2—H2	120.8	C10—C11—H11	119.0
C4—C3—C2	120.2 (8)	C12—C11—H11	119.0
C4—C3—Br2	121.0 (7)	C13—C12—C11	115.1 (9)
C2—C3—Br2	118.8 (7)	C13—C12—H12	122.5
C3—C4—C5	122.0 (8)	C11—C12—H12	122.5
C3—C4—H4	119.0	C12—C13—C8	124.9 (9)
C5—C4—H4	119.0	C12—C13—N2	131.6 (9)
C4—C5—C6	120.9 (8)	C8—C13—N2	103.4 (7)
C4—C5—C7	117.5 (7)	C6—O1—Ni1	125.3 (5)
C6—C5—C7	121.6 (7)	C7—N2—C13	108.9 (7)
O1—C6—C5	127.4 (8)	C7—N2—H2A	145 (8)
O1—C6—C1	119.3 (7)	C13—N2—H2A	106 (8)
$N1^{i}$—Ni1—N1—C7	−137.8 (6)	C6—C5—C7—N2	−177.9 (7)
O1—Ni1—N1—C7	−14.4 (6)	C4—C5—C7—N1	179.3 (7)
$O1^{i}$—Ni1—N1—C7	110.9 (6)	C6—C5—C7—N1	−1.4 (11)
$N1^{i}$—Ni1—N1—C8	51.4 (6)	C7—N1—C8—C9	−179.7 (9)
O1—Ni1—N1—C8	174.9 (6)	Ni1—N1—C8—C9	−7.9 (13)
$O1^{i}$—Ni1—N1—C8	−59.8 (7)	C7—N1—C8—C13	−2.3 (8)
C6—C1—C2—C3	−2.4 (15)	Ni1—N1—C8—C13	169.5 (5)
Br1—C1—C2—C3	−178.2 (7)	N1—C8—C9—C10	178.0 (9)
C1—C2—C3—C4	1.1 (14)	C13—C8—C9—C10	0.8 (12)
C1—C2—C3—Br2	−179.9 (7)	C8—C9—C10—C11	3.0 (15)
C2—C3—C4—C5	−1.2 (14)	C9—C10—C11—C12	−3.9 (17)
Br2—C3—C4—C5	179.8 (6)	C10—C11—C12—C13	0.8 (16)
C3—C4—C5—C6	2.5 (13)	C11—C12—C13—C8	3.3 (15)
C3—C4—C5—C7	−178.2 (8)	C11—C12—C13—N2	177.0 (9)
C4—C5—C6—O1	178.4 (8)	N1—C8—C13—C12	178.1 (8)
C7—C5—C6—O1	−0.9 (13)	C9—C8—C13—C12	−4.1 (13)
C4—C5—C6—C1	−3.3 (11)	N1—C8—C13—N2	2.9 (8)
C7—C5—C6—C1	177.4 (7)	C9—C8—C13—N2	−179.3 (7)
C2—C1—C6—O1	−178.1 (9)	C5—C6—O1—Ni1	−7.4 (11)
Br1—C1—C6—O1	−2.3 (10)	C1—C6—O1—Ni1	174.4 (6)
C2—C1—C6—C5	3.4 (13)	N1—Ni1—O1—C6	12.9 (6)
Br1—C1—C6—C5	179.2 (6)	$N1^{i}$—Ni1—O1—C6	137.1 (6)
C8—N1—C7—N2	0.7 (8)	$O1^{i}$—Ni1—O1—C6	−111.0 (6)
Ni1—N1—C7—N2	−171.7 (5)	N1—C7—N2—C13	1.1 (8)
C8—N1—C7—C5	−176.1 (7)	C5—C7—N2—C13	178.1 (7)
Ni1—N1—C7—C5	11.5 (10)	C12—C13—N2—C7	−177.1 (9)
C4—C5—C7—N2	2.8 (11)	C8—C13—N2—C7	−2.4 (8)

Symmetry codes: (i) −*x*+2, −*y*+1/2, *z*.

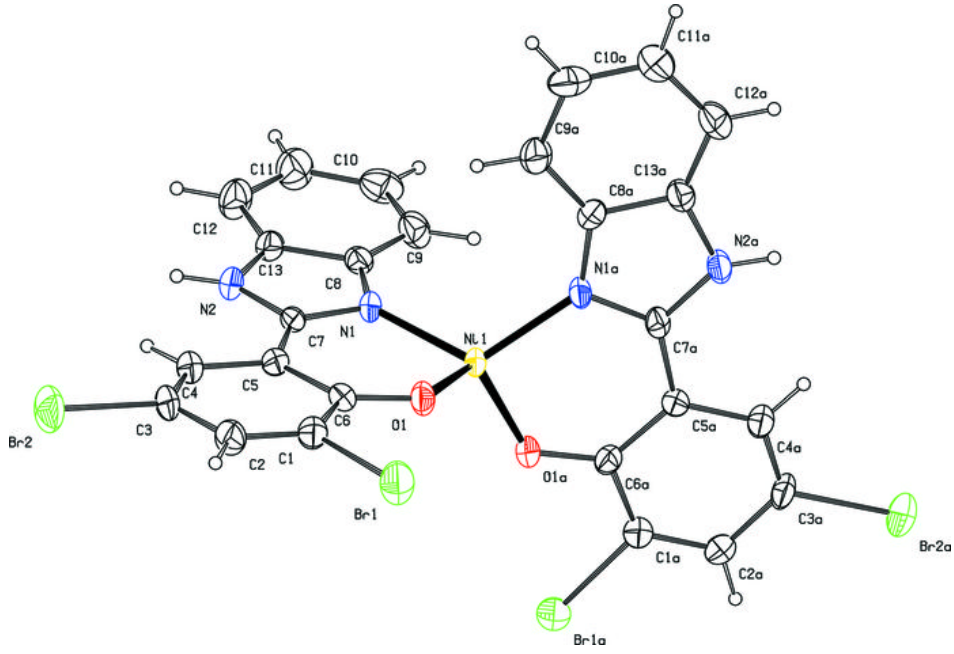
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N2—H2A···Br1^{ii}	0.86 (3)	2.74 (7)	3.464 (7)	143 (9)
N2—H2A···O1^{ii}	0.86 (3)	2.11 (8)	2.811 (9)	138 (9)

Symmetry codes: (ii) x, y+1/2, −z.

Fig. 1


C11a
C10a
C12a
C9a
C13a
C8a
N2a
N1a
C7a
C5a
C4a
C3a
Br2a
C1a
C2a
Br1a
O1a
C6a
C10
C11
C12
C13
C8
C9
N2
N1
C7
C5
C4
C3
Br2
C6
O1
C2
C1
Br1
NL1