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Key indicators

Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
 Disorder in solvent or counterion
 R factor = 0.039
 wR factor = 0.085
 Data-to-parameter ratio = 19.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Di- μ -pyridyl-1:2 $\kappa^2\text{N}:\text{C}^2$;2:1 $\kappa^2\text{N}:\text{C}^2$ - μ -tetrahydrofuran- $\kappa^2\text{O}:\text{O}$ -bis[bromo(tetrahydrofuran)-magnesium(II)] tetrahydrofuran hemisolvate

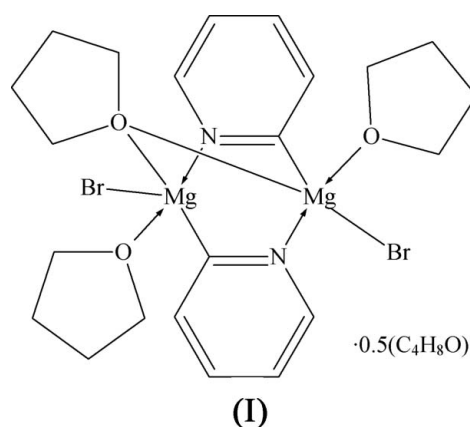
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The title compound, $[\text{Mg}_2\text{Br}_2(\text{C}_5\text{H}_4\text{N})_2(\text{C}_4\text{H}_8\text{O})_3] \cdot 0.5\text{C}_4\text{H}_8\text{O}$, contains dimeric associations of Mg atoms bridged by tetrahydrofuran (THF) molecules. The coordination polyhedron of the Mg atom is a slightly distorted MgCNO_2Br trigonal bipyramid with two THF molecules in the axial positions. One O atom occupies a site with symmetry 2.

Comment

The main molecule of the title Grignard reagent, $(\mu\text{-C}_4\text{H}_8\text{O})\text{-}[\text{Br}(\text{C}_4\text{H}_8\text{O})(\mu\text{-}\eta^2\text{-C,N-C}_5\text{H}_4\text{N-2})\text{Mg}]_2$, (I) (Fig. 1), is dimeric $[\text{Mg}1 \cdots \text{Mg}1^i = 3.3237 (18) \text{ \AA}$; symmetry code: (i) $-y, -x, \frac{1}{2} - z$] and is generated by twofold symmetry with O2 lying on a twofold rotation axis. The coordination polyhedron of the Mg atom is a slightly distorted MgCNO_2Br trigonal bipyramid (Table 1) with two tetrahydrofuran (THF) molecules in the axial (ax) positions. Bromine, pyridyl N and C atoms occupy equatorial (eq) sites. The eq–Mg1–eq angles lie within the range 115.52 (11)–121.74 (8)° and the ax–Mg1–eq angles are close to 90° [83.16 (8)–95.07 (10)°].



This coordination environment of Mg is rather characteristic for adducts of Grignard reagents with THF, as was observed for MeMgBr (Vallino, 1969) and EtMgCl (Toney & Stucky, 1971). The $\text{Mg}1\text{-Br}1$, $\text{Mg}1\text{-O}1$ and $\text{Mg}1\text{-C}1$ bond lengths are normal and consistent with related structures (Cambridge Structural Database; Version 5.27 of January 2006; Allen, 2002). The $\text{Mg}1\text{-N}1$ distance in (I) is close to that found previously for pyridyl substituted alkylmagnesiumbromide [2.122 (4) \AA ; Al-Juaid *et al.*, 2001].

The sum of valence angles around O1, 358.0°, corresponds to sp^2 -hybridization. Analysis of data in the CSD showed that the latter is common for structures with $\text{Hal-Mg(C)-O(THF, terminal)}$ fragments where the sum of angles varies

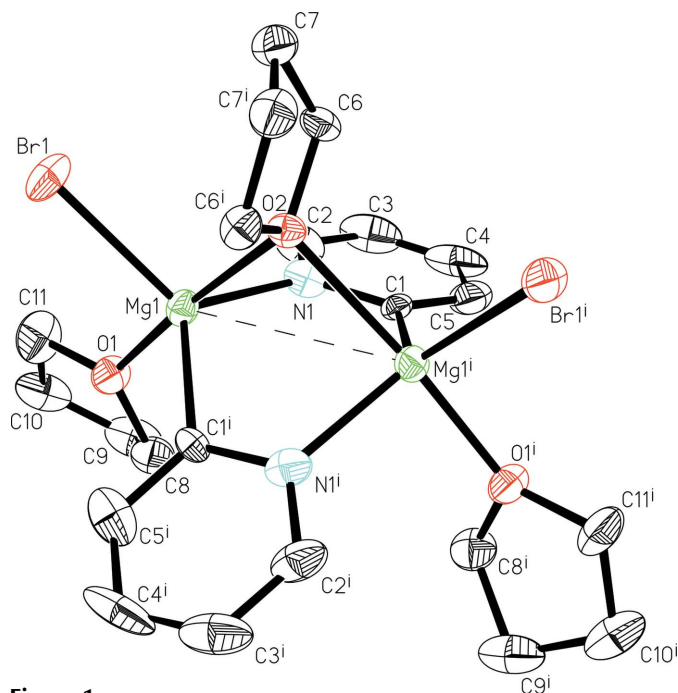


Figure 1
Molecular structure of the main molecule of (I), showing 50% probability displacement ellipsoids with H atoms omitted for clarity. [Symmetry code: (i) $-y, -x, \frac{1}{2} - z$.]

from 351.8 to 360.0°. The second (O2) THF molecule is bridging and the $\text{Mg1}-\text{O2}-\text{Mg1}^i$ angle is 88.86 (10)° [symmetry code: (i) $-y, -x, \frac{1}{2} - z$]. As expected, the $\text{Mg}-\text{O2}$ bond length is much longer than $\text{Mg}-\text{O1}$.

To the best of our knowledge, (I) is only the second example of an Mg complex with a bridging THF molecule. Previously, the dinuclear complex $[(\text{THF})(\eta^2\text{-PhNCNPh})_2\text{Mg}_2(\mu\text{-Cl})_2(\mu\text{-THF})]$, (II), was structurally investigated (Cotton *et al.*, 1997); for comparison, the $\text{Mg}-\text{O}(\mu\text{-THF})$ distances in (II) are 2.322 (6) and 2.357 (6) Å, while the $\text{Mg}-\text{O}-\text{Mg}$ angle is 84.3 (2)°. However, the bridging THF ligand is well known in the structures of alkali and rare earth metals complexes; there are 70 entries in the CSD, of which 29 are Li derivatives.

Compound (I) is the first structurally characterized example of an Mg complex with bridging ($\mu\text{-C}, N$ -pyridyl-2) ligands. However, this bridging ligand is common for di- and polynuclear complexes of other metals (110 entries in the CSD, of which 85 are compounds of 8B group metals).

In the dimeric structure of (I), the Br atoms are terminal. In contrast, an analysis of the CSD demonstrates that in all previously investigated di- and polymeric structures of Grignard reagents, the halogen atoms serve as bridges forming $[\text{Mg}_2(\mu\text{-Hal})_2]$ fragments (16 entries).

Previously, the synthesis of closely related Grignard reagents (2-pyridyl) $\text{MgX}\cdot 2\text{THF}$ ($X = \text{Br}$ and I) was reported and their unit-cell parameters were determined (Paradies, 1974). However, no information on their molecular structures was published.

The crystals of (I) contain disordered solvent THF molecules lying on a fourfold axis. These THF molecules occupy the cavities between the main molecules.

Experimental

The synthetic procedure for (I) reported by Paradies & Görbing (1969) was found to be non-reproducible. This fact was mentioned by Furukava *et al.* (1987). Compound (I) was prepared by treatment of *i*-PrMgBr with 2-bromopyridine (Trécourt *et al.*, 1999) and for the first time isolated in pure form (yield 58%). The crystals of (I) decompose rapidly in open air.

Crystal data

$[\text{Mg}_2\text{Br}_2(\text{C}_5\text{H}_4\text{N})_2(\text{C}_4\text{H}_8\text{O})_3]\cdot 0.5\text{C}_4\text{H}_8\text{O}$
 $M_r = 616.99$
 Tetragonal, $P4/ncc$
 $a = 17.3368$ (3) Å
 $c = 18.8696$ (4) Å
 $V = 5671.53$ (18) Å³

$Z = 8$
 $D_x = 1.445$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 2.93$ mm⁻¹
 $T = 120$ (2) K
 Block, colourless
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART 1K diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1997)
 $T_{\min} = 0.473$, $T_{\max} = 0.758$

30363 measured reflections
 3108 independent reflections
 2022 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.093$
 $\theta_{\text{max}} = 27.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.085$
 $S = 1.00$
 3108 reflections
 161 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 1.9833P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.36$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

$\text{Mg1}-\text{Br1}$	2.4887 (9)	$\text{Mg1}-\text{C1}^i$	2.149 (3)
$\text{Mg1}-\text{O1}$	2.089 (2)	$\text{Mg1}-\text{O2}$	2.374 (2)
$\text{Mg1}-\text{N1}$	2.129 (3)		
$\text{O1}-\text{Mg1}-\text{N1}$	93.15 (9)	$\text{C1}^i-\text{Mg1}-\text{Br1}$	120.98 (8)
$\text{O1}-\text{Mg1}-\text{C1}^i$	95.07 (10)	$\text{O2}-\text{Mg1}-\text{Br1}$	89.57 (5)
$\text{N1}-\text{Mg1}-\text{C1}^i$	115.52 (11)	$\text{C6}-\text{O2}-\text{C6}^i$	108.2 (3)
$\text{O1}-\text{Mg1}-\text{O2}$	175.18 (8)	$\text{C6}-\text{O2}-\text{Mg1}^i$	115.36 (12)
$\text{N1}-\text{Mg1}-\text{O2}$	83.16 (8)	$\text{C6}^i-\text{O2}-\text{Mg1}^i$	114.16 (12)
$\text{C1}^i-\text{Mg1}-\text{O2}$	83.78 (8)	$\text{C6}-\text{O2}-\text{Mg1}$	114.16 (12)
$\text{O1}-\text{Mg1}-\text{Br1}$	95.02 (6)	$\text{C6}^i-\text{O2}-\text{Mg1}$	115.36 (12)
$\text{N1}-\text{Mg1}-\text{Br1}$	121.74 (8)	$\text{Mg1}^i-\text{O2}-\text{Mg1}$	88.86 (10)

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

The possibility of partial positional disorder of C1 and N1 was checked; no evidence for such disorder was found. The disordered solvent (THF) molecule was refined isotropically with restrained C—C and C—O distances. The position of the O atom in the five-membered ring of the solvent THF molecule was assigned by analysis of isotropic displacement parameters and confirmed by the fact that the methylene group could not be placed in the O22 site without forming unusually short intermolecular H...H contacts (1.90–1.94 Å). All H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics:

SHELXTL-Plus (Bruker, 2000); software used to prepare material for publication: *SHELXTL-Plus*.

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supporting information

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Di- μ -pyridyl-1:2 κ^2 N:C²;2:1 κ^2 N:C²- μ -tetrahydrofuran- κ^2 O:O-bis[bromo(tetrahydrofuran)magnesium(II)] tetrahydrofuran hemisolvate

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Crystal data

[Mg₂Br₂(C₅H₄N)₂(C₄H₈O)₃].0.5C₄H₈O

$M_r = 616.99$

Tetragonal, *P4/ncc*

Hall symbol: -P 4a 2ac

$a = 17.3368$ (3) Å

$c = 18.8696$ (4) Å

$V = 5671.53$ (18) Å³

$Z = 8$

$F(000) = 2528$

$D_x = 1.445$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5951 reflections

$\theta = 2.4$ – 27.6°

$\mu = 2.93$ mm⁻¹

$T = 120$ K

Block, colourless

0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART 1K
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1997)

$T_{\min} = 0.473$, $T_{\max} = 0.758$

30363 measured reflections

3108 independent reflections

2022 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.093$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 1.7^\circ$

$h = -22 \rightarrow 15$

$k = -20 \rightarrow 22$

$l = -17 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.085$

$S = 1.00$

3108 reflections

161 parameters

6 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 1.9833P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.47$ e Å⁻³

$\Delta\rho_{\min} = -0.36$ e Å⁻³

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Br1	0.00140 (2)	0.130532 (18)	0.410058 (15)	0.03633 (12)	
Mg1	0.03748 (6)	0.02350 (5)	0.32866 (5)	0.0227 (2)	
N1	-0.02342 (14)	-0.08313 (15)	0.32806 (14)	0.0306 (6)	
C1	-0.04365 (15)	-0.11090 (15)	0.26120 (14)	0.0185 (6)	
C2	-0.0382 (2)	-0.1275 (2)	0.38650 (18)	0.0391 (8)	
H2A	-0.0231	-0.1082	0.4315	0.047*	
C3	-0.0731 (2)	-0.1975 (2)	0.3840 (2)	0.0504 (10)	
H3A	-0.0842	-0.2252	0.4262	0.060*	
C4	-0.0918 (2)	-0.22714 (19)	0.3187 (2)	0.0512 (11)	
H4A	-0.1148	-0.2767	0.3148	0.061*	
C5	-0.07646 (18)	-0.18353 (19)	0.2586 (2)	0.0408 (9)	
H5A	-0.0891	-0.2046	0.2136	0.049*	
O1	0.12524 (11)	-0.01943 (12)	0.39346 (11)	0.0297 (5)	
C8	0.17086 (19)	-0.08419 (19)	0.36970 (18)	0.0354 (8)	
H8A	0.2209	-0.0667	0.3502	0.043*	
H8B	0.1431	-0.1139	0.3328	0.043*	
C9	0.1827 (2)	-0.1326 (2)	0.4360 (2)	0.0499 (10)	
H9A	0.1386	-0.1679	0.4439	0.060*	
H9B	0.2308	-0.1633	0.4328	0.060*	
C10	0.18817 (19)	-0.0729 (2)	0.49443 (18)	0.0469 (10)	
H10A	0.1694	-0.0941	0.5400	0.056*	
H10B	0.2420	-0.0551	0.5006	0.056*	
C11	0.1372 (2)	-0.0084 (2)	0.46924 (17)	0.0461 (9)	
H11A	0.0873	-0.0096	0.4946	0.055*	
H11B	0.1622	0.0420	0.4782	0.055*	
O2	-0.06215 (10)	0.06215 (10)	0.2500	0.0217 (6)	
C6	-0.14031 (16)	0.05431 (17)	0.27933 (16)	0.0256 (7)	
H6A	-0.1384	0.0318	0.3275	0.031*	
H6B	-0.1722	0.0206	0.2488	0.031*	
C7	-0.17336 (18)	0.13479 (18)	0.28166 (16)	0.0339 (8)	
H7A	-0.1598	0.1612	0.3265	0.041*	
H7B	-0.2302	0.1340	0.2763	0.041*	
C21	0.7304 (14)	0.751 (2)	-0.0028 (6)	0.073 (7)*	0.25
H21A	0.6801	0.7243	-0.0069	0.087*	0.25
H21B	0.7420	0.7770	-0.0483	0.087*	0.25

C24	0.7638 (17)	0.7662 (10)	0.1203 (6)	0.064 (6)*	0.25
H24A	0.8090	0.7944	0.1392	0.076*	0.25
H24B	0.7251	0.7604	0.1585	0.076*	0.25
C23	0.7875 (12)	0.6884 (11)	0.0919 (8)	0.066 (5)*	0.25
H23A	0.8383	0.6732	0.1113	0.079*	0.25
H23B	0.7491	0.6487	0.1050	0.079*	0.25
C25	0.7297 (18)	0.8081 (12)	0.0576 (10)	0.111 (10)*	0.25
H25A	0.7609	0.8541	0.0457	0.133*	0.25
H25B	0.6763	0.8248	0.0681	0.133*	0.25
O22	0.7917 (7)	0.6964 (7)	0.0158 (5)	0.065 (3)*	0.25

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0532 (2)	0.03296 (19)	0.02287 (16)	0.01342 (15)	-0.00432 (15)	-0.00763 (14)
Mg1	0.0277 (6)	0.0229 (5)	0.0176 (5)	-0.0017 (4)	0.0006 (4)	-0.0012 (4)
N1	0.0280 (15)	0.0321 (15)	0.0317 (15)	0.0020 (11)	0.0009 (12)	0.0085 (12)
C1	0.0185 (15)	0.0150 (15)	0.0220 (15)	0.0005 (11)	0.0058 (12)	-0.0039 (11)
C2	0.0325 (19)	0.051 (2)	0.0335 (18)	0.0004 (17)	0.0006 (15)	0.0196 (17)
C3	0.038 (2)	0.046 (3)	0.067 (3)	0.0085 (17)	0.017 (2)	0.030 (2)
C4	0.032 (2)	0.0179 (18)	0.103 (4)	0.0012 (14)	0.032 (2)	0.012 (2)
C5	0.033 (2)	0.036 (2)	0.053 (2)	-0.0024 (15)	0.0146 (17)	-0.0169 (17)
O1	0.0308 (12)	0.0336 (13)	0.0246 (11)	0.0063 (9)	-0.0043 (9)	0.0000 (9)
C8	0.0290 (19)	0.037 (2)	0.040 (2)	0.0061 (15)	0.0008 (15)	0.0010 (16)
C9	0.037 (2)	0.042 (2)	0.070 (3)	0.0078 (17)	0.0093 (19)	0.025 (2)
C10	0.033 (2)	0.072 (3)	0.036 (2)	0.0074 (18)	0.0024 (16)	0.0212 (19)
C11	0.057 (2)	0.055 (2)	0.0258 (17)	0.0040 (19)	-0.0140 (16)	0.0038 (17)
O2	0.0206 (9)	0.0206 (9)	0.0238 (15)	0.0000 (11)	0.0027 (8)	0.0027 (8)
C6	0.0174 (16)	0.0322 (18)	0.0273 (16)	-0.0017 (13)	0.0028 (12)	0.0018 (13)
C7	0.0302 (19)	0.038 (2)	0.0336 (19)	0.0065 (15)	-0.0006 (14)	-0.0055 (14)

Geometric parameters (Å, °)

Mg1—Br1	2.4887 (9)	C10—H10A	0.9900
Mg1—O1	2.089 (2)	C10—H10B	0.9900
Mg1—N1	2.129 (3)	C11—H11A	0.9900
Mg1—C1 ⁱ	2.149 (3)	C11—H11B	0.9900
Mg1—O2	2.374 (2)	O2—C6	1.470 (3)
Mg1—Mg1 ⁱ	3.3237 (18)	O2—C6 ⁱ	1.470 (3)
N1—C2	1.369 (4)	O2—Mg1 ⁱ	2.374 (2)
N1—C1	1.395 (3)	C6—C7	1.509 (4)
C1—C5	1.383 (4)	C6—H6A	0.9900
C1—Mg1 ⁱ	2.149 (3)	C6—H6B	0.9900
C2—C3	1.356 (5)	C7—C7 ⁱ	1.524 (6)
C2—H2A	0.9500	C7—H7A	0.9900
C3—C4	1.373 (5)	C7—H7B	0.9900
C3—H3A	0.9500	C21—O22	1.46 (2)
C4—C5	1.390 (5)	C21—C25	1.511 (19)

C4—H4A	0.9500	C21—H21A	0.9900
C5—H5A	0.9500	C21—H21B	0.9900
O1—C8	1.445 (3)	C24—C23	1.507 (16)
O1—C11	1.458 (4)	C24—C25	1.508 (15)
C8—C9	1.521 (5)	C24—H24A	0.9900
C8—H8A	0.9900	C24—H24B	0.9900
C8—H8B	0.9900	C23—O22	1.444 (16)
C9—C10	1.515 (5)	C23—H23A	0.9900
C9—H9A	0.9900	C23—H23B	0.9900
C9—H9B	0.9900	C25—H25A	0.9900
C10—C11	1.502 (4)	C25—H25B	0.9900
O1—Mg1—N1	93.15 (9)	C9—C10—H10B	111.0
O1—Mg1—C1 ⁱ	95.07 (10)	H10A—C10—H10B	109.0
N1—Mg1—C1 ⁱ	115.52 (11)	O1—C11—C10	107.3 (3)
O1—Mg1—O2	175.18 (8)	O1—C11—H11A	110.3
N1—Mg1—O2	83.16 (8)	C10—C11—H11A	110.3
C1 ⁱ —Mg1—O2	83.78 (8)	O1—C11—H11B	110.3
O1—Mg1—Br1	95.02 (6)	C10—C11—H11B	110.3
N1—Mg1—Br1	121.74 (8)	H11A—C11—H11B	108.5
C1 ⁱ —Mg1—Br1	120.98 (8)	C6—O2—C6 ⁱ	108.2 (3)
O2—Mg1—Br1	89.57 (5)	C6—O2—Mg1 ⁱ	115.36 (12)
O1—Mg1—Mg1 ⁱ	129.87 (6)	C6 ⁱ —O2—Mg1 ⁱ	114.16 (12)
N1—Mg1—Mg1 ⁱ	63.98 (7)	C6—O2—Mg1	114.16 (12)
C1 ⁱ —Mg1—Mg1 ⁱ	62.31 (8)	C6 ⁱ —O2—Mg1	115.36 (12)
O2—Mg1—Mg1 ⁱ	45.57 (5)	Mg1 ⁱ —O2—Mg1	88.86 (10)
Br1—Mg1—Mg1 ⁱ	135.11 (2)	O2—C6—C7	106.0 (2)
C2—N1—C1	119.2 (3)	O2—C6—H6A	110.5
C2—N1—Mg1	125.2 (2)	C7—C6—H6A	110.5
C1—N1—Mg1	115.41 (18)	O2—C6—H6B	110.5
C5—C1—N1	116.8 (3)	C7—C6—H6B	110.5
C5—C1—Mg1 ⁱ	125.3 (2)	H6A—C6—H6B	108.7
N1—C1—Mg1 ⁱ	117.92 (18)	C6—C7—C7 ⁱ	102.49 (18)
C3—C2—N1	123.9 (4)	C6—C7—H7A	111.3
C3—C2—H2A	118.0	C7 ⁱ —C7—H7A	111.3
N1—C2—H2A	118.0	C6—C7—H7B	111.3
C2—C3—C4	118.1 (3)	C7 ⁱ —C7—H7B	111.3
C2—C3—H3A	120.9	H7A—C7—H7B	109.2
C4—C3—H3A	120.9	O22—C21—C25	104.4 (13)
C3—C4—C5	118.9 (3)	O22—C21—H21A	110.9
C3—C4—H4A	120.5	C25—C21—H21A	110.9
C5—C4—H4A	120.5	O22—C21—H21B	110.9
C1—C5—C4	123.0 (3)	C25—C21—H21B	110.9
C1—C5—H5A	118.5	H21A—C21—H21B	108.9
C4—C5—H5A	118.5	C23—C24—C25	105.0 (9)
C8—O1—C11	109.2 (2)	C23—C24—H24A	110.7
C8—O1—Mg1	119.60 (18)	C25—C24—H24A	110.7
C11—O1—Mg1	129.16 (18)	C23—C24—H24B	110.7

O1—C8—C9	104.3 (3)	C25—C24—H24B	110.7
O1—C8—H8A	110.9	H24A—C24—H24B	108.8
C9—C8—H8A	110.9	O22—C23—C24	106.3 (11)
O1—C8—H8B	110.9	O22—C23—H23A	110.5
C9—C8—H8B	110.9	C24—C23—H23A	110.5
H8A—C8—H8B	108.9	O22—C23—H23B	110.5
C10—C9—C8	103.3 (3)	C24—C23—H23B	110.5
C10—C9—H9A	111.1	H23A—C23—H23B	108.7
C8—C9—H9A	111.1	C24—C25—C21	105.8 (11)
C10—C9—H9B	111.1	C24—C25—H25A	110.6
C8—C9—H9B	111.1	C21—C25—H25A	110.6
H9A—C9—H9B	109.1	C24—C25—H25B	110.6
C11—C10—C9	104.0 (3)	C21—C25—H25B	110.6
C11—C10—H10A	111.0	H25A—C25—H25B	108.7
C9—C10—H10A	111.0	C23—O22—C21	105.3 (12)
C11—C10—H10B	111.0		

Symmetry code: (i) $-y, -x, -z+1/2$.