Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of memantine–carboxyborane

Theppawut I. Ayudhya,[a] Arnold L. Rheingold[b] and Nin N. Dingra[a]*

[a]Department of Chemistry, University of Alaska Anchorage, Anchorage, AK 99508, USA, and [b]Department of Chemistry, University of California–San Diego, La Jolla, CA 92093, USA. *Correspondence e-mail: ndingra@alaska.edu

Received 25 February 2019
Accepted 26 March 2019

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

Keywords: crystal structure; memantine-carboxyborane; CORCB; memantine; adamantane.

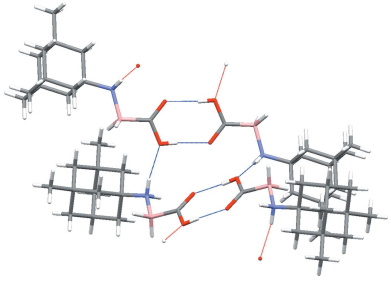
CCDC reference: 1905840

Supporting information: this article has supporting information at journals.iucr.org/e

The synthesis and crystal structure of the title compound, $C_{13}H_{24}BNO_2$ [systematic name: 3,5-dimethyladamantanylamine–boranecarboxylic acid or *N*-(carboxyboranylidene)-3,5-dimethyladamantan-1-amine], derived from the anti-Alzheimer's disease drug memantine is reported. The C—N—B—CO_2 unit is almost planar (r.m.s. deviation = 0.095 Å). The extended structure shows typical carboxylic acid inversion dimers linked by pairwise O—H···O hydrogen bonds [O···O = 2.662 (3) Å]. The amino group forms a weak N—H···O hydrogen bond [N···O = 3.011 (3) Å], linking the dimers into [001] chains in the crystal. Highly disordered solvent molecules were treated using the SQUEEZE routine of *PLATON* [Spek (2015). *Acta Cryst.* C**71**, 9–18], which treats the electron density as a diffuse contribution without assignment of specific atom locations. A scattering contribution of 255 electrons was removed. The crystal studied was refined as a two-component twin.

1. Chemical context

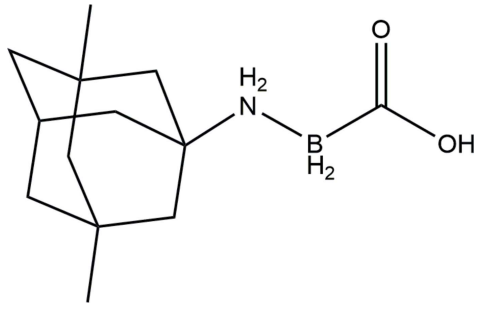
Memantine is a drug used for the treatment of mild and moderate-to-severe Alzheimer's disease as an inhibitor for *N*-methyl-D-aspartate (NMDA) receptors. As a result of its property as a low-affinity, open-channel blocker, memantine does not substantially interfere with normal synaptic activity, thereby reducing side effects. This has led to clinical trials for other neurological disorders (Bullock, 2006; Lipton, 2005; Olivares *et al.*, 2012; Parsons *et al.*, 2007). While memantine in its hydrochloride form is useful in various treatment methods, some modifications were done on this drug to optimize the desired concentration in the system. As a means to preventing drug degradation, memantine has been further processed in a mixture with other compounds (McInnes *et al.*, 2010; Plosker, 2015). The one-week extended release formula by Lyndra Therapeutics is currently under clinical trial phase I (clinicaltrials.gov, NCT03711825). Though efforts to maintain the long-term stability of memantine are underway, chemical modification of the memantine structure itself is rarely reported. Our attempt was to mask the compound with an additional moiety that can be removed under certain conditions, therefore releasing the drug. With this goal, memantine–carboxyborane was synthesized since the carboxyborate group is known to decompose into carbon monoxide and boric acid, leaving the drug molecule itself (Ayudhya *et al.*, 2017, 2018). The single crystal structure of the said compound, (I), was solved and its features are described in this report.



2. Structural commentary

The molecular structure of (I) is shown in Fig. 1. The C2–N1–B1–C1/O1/O2 fragment is almost planar (r.m.s. deviation =

0.095 Å) and the C atoms bonded to the B and N atoms take on an anti orientation [C1—B1—N1—C2 = 173.5 (3)°]. The stereogenic centres in the adamantane unit were assigned as C4 *S* and C8 *R* in the arbitrarily chosen asymmetric unit but crystal symmetry generates a racemic mixture. The bond lengths [C1—O1 = 1.340 (4), C1—O2 = 1.227 (4) Å] of the carboxylic acid group are in agreement with the data for related carboxylic acids and known amine–carboxyboranes (Gavezzotti, 2008; Spielvogel *et al.*, 1980; Vyakaranam *et al.*, 2002; Ayudhya *et al.*, 2017). The C—C—C bond angles of the adamantane cage fall within the expected ranges and the N1—C2 bond length at 1.504 (4) Å is comparable with previously reported values in aminoadamantane structures (Donohue & Goodman, 1967; Chacko & Zand, 1973).



3. Supramolecular features

A dimer is observed between the two memantine–carboxyborane molecules formed through conventional hydrogen bonding between the carboxylic acid moieties (Fig. 2). The hydrogen-bond length listed in Table 1 [O1···O2 = 2.662 (3) Å] is consistent with the hydrogen-bond geometries found in carboxyborane dimers such as ammonia–carboxyborane [2.668 (2) Å; Spielvogel *et al.*, 1980], mophorline–carboxyborane [2.712 (4) Å; Vyakaranam *et al.*, 2002] and trimethylamine–carboxyborane [2.714 Å; Spielvogel *et al.*, 1976]. In (I), these dimers form an extended structure through N1—H1*B*···O1 links (O1 is the protonated oxygen atom of the carboxylic acid), to form [001] chains. This motif has also been reported previously in ammonia–carboxyborane, trimethylamine–carboxyborane, dimethylamine–carboxyborane and methylamine–carboxyborane (Spielvogel *et al.*, 1980). The adjacent dimers shown in Fig. 2 indicates that the planes of the carboxylic acids are not parallel, but twisted by 76.5° from each other.

Assessment of available crystal structures deposited with the Cambridge Structural Database (Version 5.39; Groom *et al.*, 2016) indicates that not all amine–carboxyboranes form dimers during crystallization. While some amine–carboxyboranes described above are dimers, others such as piperidine–carboxyborane and hexamethylenetetramine–carboxyborane do not form dimers, suggesting that the amine-group interaction may influence the overall packing (Rana *et al.*, 2002; Ayudhya *et al.*, 2017). The extended structure of (I) is shown in projection down the *b*- and *c*-axis directions in Fig. 3*a* and 3*b*, respectively. No other contacts beyond the hydrogen bonds already mentioned are observed in this packing. Although the dimers appear to be parallel in Fig. 3*a*, the twisted planes of hydrogen bonds are better represented in Fig. 3*b*.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.84	1.82	2.662 (3)	176
N1—H1*B*···O1^{ii}	0.91	2.11	3.011 (3)	171

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

4. Database survey

The memantine structure in its free (unprotonated) base form is not found in the literature, although the hydrochloride salt with water molecules of crystallization has been solved (Lou *et al.*, 2009). Another memantine crystal structure reported was in a clathrate form with cucurbit[7]uril where memantine is completely bound within the cavity (McInnes *et al.*, 2010).

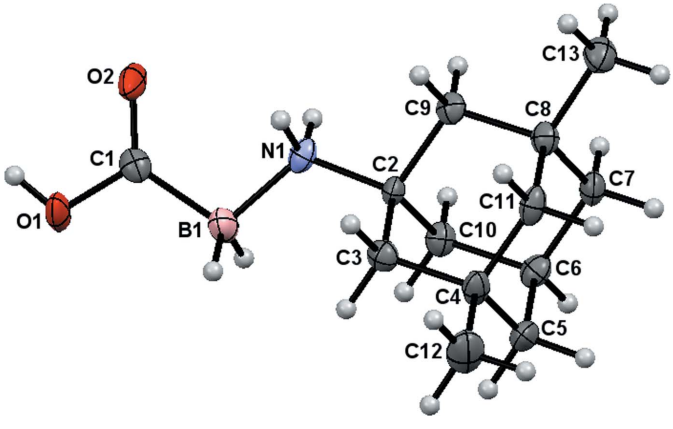


Figure 1
The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

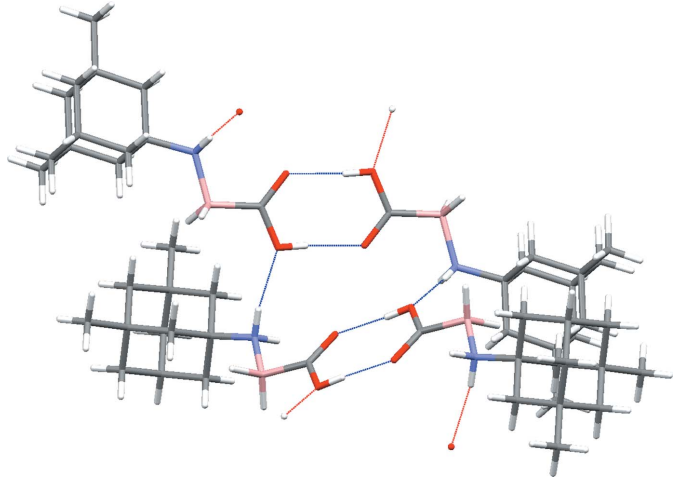


Figure 2
Detail of the hydrogen bonds in (I) showing the carboxylic acid inversion dimers and N—H···O links between dimers.

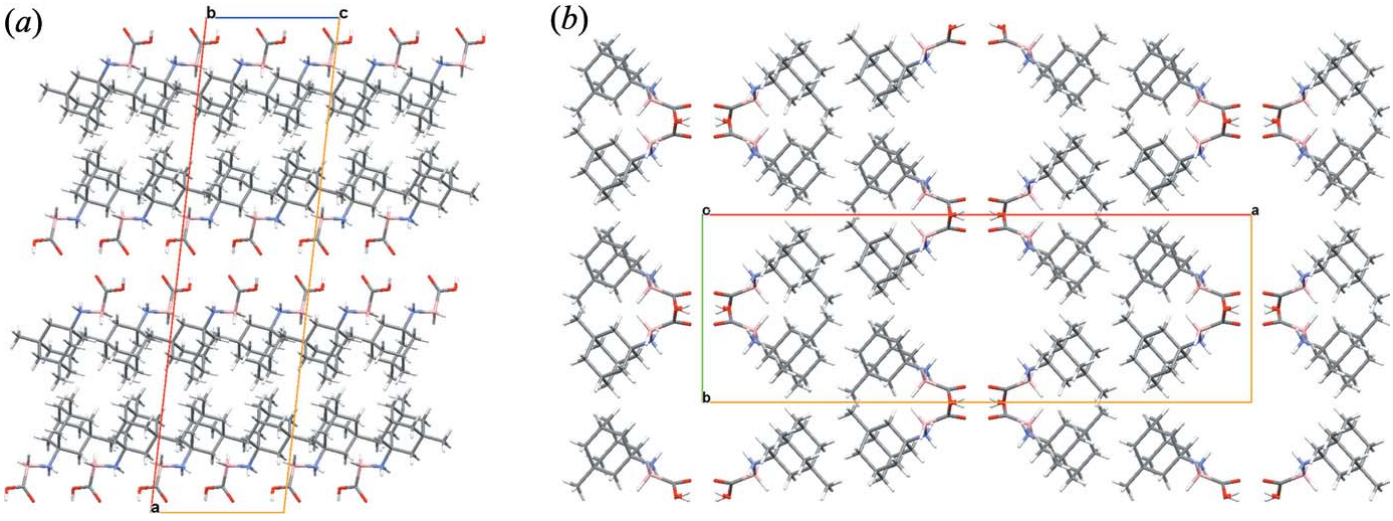


Figure 3
Packing diagrams of (I): (*a*) A view from the *b* axis to show aligned hydrogen-bonding dimers. (*b*) A view down the *c* axis to show the twisted planes.

However, numerous crystal structures of the adamantane cage and its derivatives in various forms have been reported over many years (Nordman & Schmitkons, 1965; Chacko & Zand, 1973; SiMa, 2009; Glaser *et al.*, 2011).

5. Synthesis and crystallization

Memantine, a derivative of adamantine, was first synthesized by Eli Lilly and Company. In an attempt to modify memantine into memantine–carboxyborane, a reaction scheme as shown in Fig. 4 was carried out. Addition of the carboxyborane moiety to memantine was done in a one-step reaction using an amine-exchange process as previously described (Spielvogel *et al.*, 1980). Trimethylamine carboxyborane (117 mg, 1.0 mmol) and memantine (780 mg, 4.4 mmol) were dissolved in tetrahydrofuran (8.0 ml), and maintained at 328 K for 24 h under a nitrogen atmosphere. The solution was concentrated by vacuum distillation and the resulting solid was dissolved in dichloromethane. The product was precipitated from the solvent by using 15 ml of hexane and the white solid crude product (208 mg) was filtered. This residue was purified by multiple recrystallization in dichloromethane/hexane to yield a white solid (15 mg, 6.3%). Crystals suitable for X-ray analysis were prepared by dissolving in toluene and slow cooling of the solution.

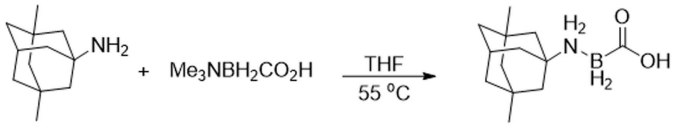


Figure 4
Reaction scheme for the synthesis of (I) through an amine-exchange process.

6. Refinement

Crystal data collection and structure refinement details are summarized in Table 2. H atoms were placed in calculated positions (O—H = 0.84, N—H = 0.91 and C—H = 0.98–0.99 Å) and refined as riding with U_{iso}(eq) = 1.5U_{eq}(C-methyl, O) and 1.2U_{eq}(C, N) for all others. The idealized methyl groups at C12

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{24}BNO_2$
M_r	237.14
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	100
a, *b*, *c* (Å)	34.229 (4), 11.1051 (12), 9.2922 (10)
β (°)	96.526 (5)
V (Å^3)	3509.3 (7)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.06
Crystal size (mm)	0.32 × 0.30 × 0.10
Data collection	
Diffractometer	Bruker APEXII Ultra
Absorption correction	Multi-scan (*TWINABS*; Bruker, 2012)
T_{min}, T_{max}	0.300, 0.333
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10740, 10740, 8531
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.611
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.062, 0.154, 1.04
No. of reflections	10740
No. of parameters	166
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.94, −0.24

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

and C13 and the idealized tetrahedral OH group at O1 were refined as rotating groups. The disordered solvent molecules were treated with the SQUEEZE routine in PLATON (Spek, 2015). The crystal studied was refined as a two-component twin.

Acknowledgements

Funding for this research was provided by an Institutional Development Award (IDeA) from the National Institute of General Medical Sciences of the National Institutes of Health under grant No. P20GM103395. The content is solely the responsibility of the authors and does not necessarily reflect the official views of the NIH.

Funding information

Funding for this research was provided by: National Institutes of Health, National Institute of General Medical Sciences (grant No. P20GM103395).

References

Ayudhya, T. I., Pellechia, P. J. & Dingra, N. N. (2018). *Dalton Trans.* **47**, 538–543.

Ayudhya, T. I., Raymond, C. C. & Dingra, N. N. (2017). *Dalton Trans.* **46**, 882–889.

Bruker (2012). *TWINABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2017). *APEX3* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bullock, R. (2006). *Alzheimer Dis. Assoc. Disord.* **20**, 23–29.

Chacko, K. K. & Zand, R. (1973). *Acta Cryst.* B**29**, 2681–2686.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Donohue, J. & Goodman, S. H. (1967). *Acta Cryst.* **22**, 352–354.

Gavezzotti, A. (2008). *Acta Cryst.* B**64**, 401–403.

Glaser, R., Steinberg, A., Šekutor, M., Rominger, F., Trapp, O. & Mlinarić–Majerski, K. (2011). *Eur. J. Org. Chem.* pp. 3500–3506.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst*. B**72**, 171–179.

Lipton, S. A. (2005). *Curr. Alzheimer Res.* **2**, 155–165.

Lou, W.-J., Hu, X.-R. & Gu, J.-M. (2009). *Acta Cryst.* E**65**, o2191.

McInnes, F. J., Anthony, N. G., Kennedy, A. R. & Wheate, N. J. (2010). *Org. Biomol. Chem.* **8**, 765–773.

Nordman, C. E. & Schmitkons, D. L. (1965). *Acta Cryst.* **18**, 764–767.

Olivares, D., Deshpande, V. K., Shi, Y., Lahiri, D. K., Greig, N. H., Rogers, J. T. & Huang, X. (2012). *Curr. Alzheimer Res.* **9**, 746–758.

Parsons, C. G., Stöffler, A. & Danysz, W. (2007). *Neuropharmacology*, **53**, 699–723.

Plosker, G. L. (2015). *Drugs*, **75**, 887–897.

Rana, G., Vyakaranam, K., Zheng, C., Li, S., Spielvogel, B. F. & Hosmane, N. S. (2002). *Main Group Met. Chem.* **25**, 107–108.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

SiMa, W. (2009). *Acta Cryst*. E**65**, o2492.

Spek, A. L. (2015). *Acta Cryst*. C**71**, 9–18.

Spielvogel, B. F., Das, M. K., McPhail, A. T., Onan, K. D. & Hall, I. H. (1980). *J. Am. Chem. Soc.* **102**, 6343–6344.

Spielvogel, B. F., Wojnowich, L., Das, M. K., McPhail, A. T. & Hargrave, K. D. (1976). *J. Am. Chem. Soc.* **98**, 5702–5703.

Vyakaranam, K., Rana, G., Chong, G., Zheng, S. L., Spielvogel, B. F. & Hosmane, N. S. (2002). *Main Group Met. Chem.* **25**, 181–182.

supporting information

Acta Cryst. (2019). E**75**, 543-546 [https://doi.org/10.1107/S2056989019004092]

Crystal structure of memantine–carboxyborane

Theppawut I. Ayudhya, Arnold L. Rheingold and Nin N. Dingra

Computing details

Data collection: *APEX3* (Bruker, 2017); cell refinement: *SAINT* (Bruker, 2017); data reduction: *SAINT* (Bruker, 2017); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

3,5-Dimethyladamantanylamine–boranecarboxylic acid

Crystal data

$C_{13}H_{24}BNO_2$
M_r = 237.14
Monoclinic, *C*2/*c*
a = 34.229 (4) Å
b = 11.1051 (12) Å
c = 9.2922 (10) Å
β = 96.526 (5)°
V = 3509.3 (7) Å^3
Z = 8
F(000) = 1040
D_x = 0.898 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1799 reflections
θ = 2.9–25.6°
μ = 0.06 mm^{-1}
T = 100 K
Plate, colourless
0.32 × 0.30 × 0.10 mm

Data collection

Bruker APEXII Ultra diffractometer
Radiation source: Micro Focus Rotating Anode, Bruker TXS
Double Bounce Multilayer Mirrors monochromator
Detector resolution: 8.258 pixels mm^{-1}
φ and *ω* scans
Absorption correction: multi-scan (*TWINABS*; Bruker, 2012)
T_{min} = 0.300, T_{max} = 0.333
10740 measured reflections
10740 independent reflections
8531 reflections with $I > 2\sigma(I)$
θ_{max} = 25.7°, θ_{min} = 1.2°
h = −41→41
k = −13→13
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.154
S = 1.04
10740 reflections
166 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 6.9836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.94 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

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

Refinement. Refined as a two-component twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.54884 (6)	0.9933 (2)	1.1021 (3)	0.0296 (6)
H1	0.5259	1.0205	1.0994	0.044*
O2	0.52308 (7)	0.9168 (2)	0.8929 (2)	0.0373 (7)
N1	0.59334 (7)	0.8289 (2)	0.8033 (3)	0.0214 (6)
H1A	0.5763	0.7656	0.7974	0.026*
H1B	0.5825	0.8865	0.7416	0.026*
C9	0.61930 (9)	0.7507 (3)	0.5864 (3)	0.0203 (7)
H9A	0.5993	0.6859	0.5806	0.024*
H9B	0.6077	0.8209	0.5313	0.024*
C2	0.63037 (8)	0.7867 (3)	0.7461 (3)	0.0172 (7)
C10	0.64666 (9)	0.6768 (3)	0.8315 (4)	0.0224 (7)
H10A	0.6267	0.6120	0.8257	0.027*
H10B	0.6536	0.6983	0.9346	0.027*
C8	0.65553 (9)	0.7071 (3)	0.5192 (3)	0.0213 (7)
C11	0.68651 (9)	0.8083 (3)	0.5324 (4)	0.0241 (8)
H11A	0.7103	0.7806	0.4906	0.029*
H11B	0.6759	0.8793	0.4764	0.029*
C6	0.68384 (9)	0.6335 (3)	0.7642 (4)	0.0229 (8)
H6	0.6951	0.5618	0.8193	0.027*
C1	0.55232 (10)	0.9311 (3)	0.9806 (4)	0.0219 (8)
C4	0.69762 (9)	0.8445 (3)	0.6898 (4)	0.0243 (8)
C3	0.66090 (9)	0.8865 (3)	0.7553 (4)	0.0222 (7)
H3A	0.6681	0.9095	0.8578	0.027*
H3B	0.6498	0.9583	0.7023	0.027*
C7	0.67253 (9)	0.5977 (3)	0.6057 (4)	0.0225 (7)
H7A	0.6528	0.5323	0.6002	0.027*
H7B	0.6960	0.5676	0.5639	0.027*
C5	0.71424 (9)	0.7332 (3)	0.7751 (4)	0.0277 (8)
H5A	0.7383	0.7050	0.7354	0.033*
H5B	0.7214	0.7551	0.8780	0.033*
C13	0.64387 (10)	0.6749 (3)	0.3590 (4)	0.0298 (9)
H13A	0.6244	0.6099	0.3522	0.045*
H13B	0.6672	0.6484	0.3157	0.045*
H13C	0.6326	0.7458	0.3071	0.045*
C12	0.72819 (10)	0.9460 (3)	0.6973 (5)	0.0397 (10)
H12A	0.7515	0.9185	0.6543	0.060*
H12B	0.7358	0.9683	0.7988	0.060*
H12C	0.7169	1.0162	0.6438	0.060*

B1	0.59514 (12)	0.8823 (4)	0.9644 (4)	0.0295 (10)
H1C	0.6174 (10)	0.964 (3)	0.966 (4)	0.042 (10)*
H1D	0.6026 (11)	0.810 (3)	1.038 (4)	0.050 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0235 (12)	0.0335 (13)	0.0342 (14)	−0.0008 (11)	0.0140 (12)	−0.0136 (11)
O2	0.0334 (14)	0.0620 (17)	0.0167 (12)	0.0240 (13)	0.0038 (12)	−0.0048 (13)
N1	0.0202 (14)	0.0169 (13)	0.0280 (15)	0.0034 (11)	0.0062 (12)	0.0043 (12)
C9	0.0237 (18)	0.0150 (16)	0.0222 (17)	0.0018 (13)	0.0026 (15)	0.0029 (15)
C2	0.0149 (16)	0.0182 (16)	0.0190 (16)	0.0039 (13)	0.0043 (14)	−0.0018 (14)
C10	0.0236 (18)	0.0213 (17)	0.0230 (17)	0.0000 (14)	0.0062 (15)	0.0018 (15)
C8	0.0238 (18)	0.0177 (16)	0.0227 (18)	0.0022 (14)	0.0046 (15)	0.0007 (15)
C11	0.0225 (18)	0.0206 (17)	0.032 (2)	0.0023 (14)	0.0134 (16)	0.0027 (16)
C6	0.0221 (17)	0.0197 (16)	0.0268 (18)	0.0032 (14)	0.0027 (15)	0.0045 (15)
C1	0.030 (2)	0.0181 (17)	0.0183 (17)	0.0013 (14)	0.0046 (16)	0.0034 (15)
C4	0.0172 (17)	0.0215 (17)	0.035 (2)	−0.0027 (14)	0.0082 (16)	−0.0072 (16)
C3	0.0238 (18)	0.0193 (17)	0.0235 (17)	−0.0012 (14)	0.0024 (15)	−0.0002 (15)
C7	0.0214 (18)	0.0187 (16)	0.0287 (17)	0.0021 (14)	0.0079 (16)	−0.0043 (16)
C5	0.0170 (17)	0.037 (2)	0.0290 (19)	0.0053 (15)	0.0007 (16)	−0.0096 (18)
C13	0.033 (2)	0.033 (2)	0.023 (2)	0.0073 (17)	0.0038 (16)	0.0011 (17)
C12	0.027 (2)	0.036 (2)	0.058 (3)	−0.0085 (17)	0.012 (2)	−0.012 (2)
B1	0.025 (2)	0.037 (3)	0.028 (2)	0.0001 (19)	0.0062 (18)	−0.011 (2)

Geometric parameters (Å, º)

O1—H1	0.8400	C6—H6	1.0000
O1—C1	1.340 (4)	C6—C7	1.532 (5)
O2—C1	1.227 (4)	C6—C5	1.515 (4)
N1—H1A	0.9100	C1—B1	1.586 (5)
N1—H1B	0.9100	C4—C3	1.530 (4)
N1—C2	1.504 (4)	C4—C5	1.541 (4)
N1—B1	1.605 (5)	C4—C12	1.535 (4)
C9—H9A	0.9900	C3—H3A	0.9900
C9—H9B	0.9900	C3—H3B	0.9900
C9—C2	1.542 (4)	C7—H7A	0.9900
C9—C8	1.530 (4)	C7—H7B	0.9900
C2—C10	1.525 (4)	C5—H5A	0.9900
C2—C3	1.520 (4)	C5—H5B	0.9900
C10—H10A	0.9900	C13—H13A	0.9800
C10—H10B	0.9900	C13—H13B	0.9800
C10—C6	1.557 (4)	C13—H13C	0.9800
C8—C11	1.541 (4)	C12—H12A	0.9800
C8—C7	1.535 (4)	C12—H12B	0.9800
C8—C13	1.539 (5)	C12—H12C	0.9800
C11—H11A	0.9900	B1—H1C	1.19 (3)
C11—H11B	0.9900	B1—H1D	1.07 (4)

C11—C4	1.523 (5)		
C1—O1—H1	109.5	O2—C1—O1	118.8 (3)
H1A—N1—H1B	106.9	O2—C1—B1	125.8 (3)
C2—N1—H1A	107.3	C11—C4—C3	109.6 (3)
C2—N1—H1B	107.3	C11—C4—C5	108.6 (3)
C2—N1—B1	120.0 (3)	C11—C4—C12	109.4 (3)
B1—N1—H1A	107.3	C3—C4—C5	108.2 (3)
B1—N1—H1B	107.3	C3—C4—C12	110.1 (3)
H9A—C9—H9B	108.1	C12—C4—C5	110.8 (3)
C2—C9—H9A	109.5	C2—C3—C4	110.2 (2)
C2—C9—H9B	109.5	C2—C3—H3A	109.6
C8—C9—H9A	109.5	C2—C3—H3B	109.6
C8—C9—H9B	109.5	C4—C3—H3A	109.6
C8—C9—C2	110.7 (2)	C4—C3—H3B	109.6
N1—C2—C9	107.2 (2)	H3A—C3—H3B	108.1
N1—C2—C10	109.8 (2)	C8—C7—H7A	109.7
N1—C2—C3	110.8 (2)	C8—C7—H7B	109.7
C10—C2—C9	109.2 (2)	C6—C7—C8	109.7 (3)
C3—C2—C9	109.6 (3)	C6—C7—H7A	109.7
C3—C2—C10	110.2 (2)	C6—C7—H7B	109.7
C2—C10—H10A	110.1	H7A—C7—H7B	108.2
C2—C10—H10B	110.1	C6—C5—C4	109.9 (3)
C2—C10—C6	107.8 (3)	C6—C5—H5A	109.7
H10A—C10—H10B	108.5	C6—C5—H5B	109.7
C6—C10—H10A	110.1	C4—C5—H5A	109.7
C6—C10—H10B	110.1	C4—C5—H5B	109.7
C9—C8—C11	108.6 (3)	H5A—C5—H5B	108.2
C9—C8—C7	108.3 (3)	C8—C13—H13A	109.5
C9—C8—C13	109.6 (3)	C8—C13—H13B	109.5
C7—C8—C11	108.6 (3)	C8—C13—H13C	109.5
C7—C8—C13	111.4 (3)	H13A—C13—H13B	109.5
C13—C8—C11	110.3 (3)	H13A—C13—H13C	109.5
C8—C11—H11A	109.4	H13B—C13—H13C	109.5
C8—C11—H11B	109.4	C4—C12—H12A	109.5
H11A—C11—H11B	108.0	C4—C12—H12B	109.5
C4—C11—C8	111.3 (3)	C4—C12—H12C	109.5
C4—C11—H11A	109.4	H12A—C12—H12B	109.5
C4—C11—H11B	109.4	H12A—C12—H12C	109.5
C10—C6—H6	109.0	H12B—C12—H12C	109.5
C7—C6—C10	109.7 (3)	N1—B1—H1C	104.6 (18)
C7—C6—H6	109.0	N1—B1—H1D	107 (2)
C5—C6—C10	109.5 (3)	C1—B1—N1	106.1 (3)
C5—C6—H6	109.0	C1—B1—H1C	109.7 (16)
C5—C6—C7	110.5 (3)	C1—B1—H1D	111 (2)
O1—C1—B1	115.4 (3)	H1C—B1—H1D	118 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.84	1.82	2.662 (3)	176
N1—H1*B*···O1^{ii}	0.91	2.11	3.011 (3)	171

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