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Crystal structure of 2-(2-aminophenyl)-1,3-benzoxazole

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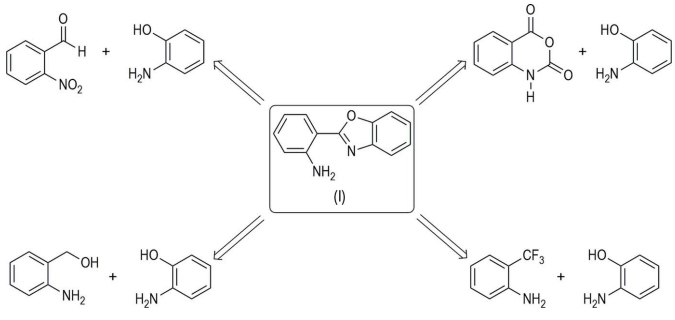
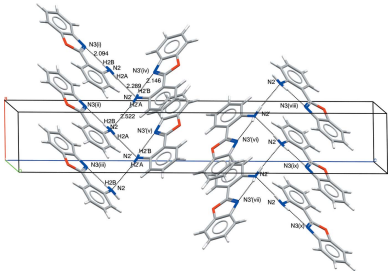
Crystals of the title compound, $C_{13}H_{10}N_2O$, were grown from a dichloromethane/ketone/methanol solvent mixture. It crystallizes with two molecules, *A* and *B*, in the asymmetric unit with very similar almost planar conformations [dihedral angles between the ring planes = 0.74 (8) and 0.67 (6)° for molecules *A* and *B*, respectively; r.m.s. overlay fit = 0.019 Å]. Each molecule features an intramolecular N—H···N hydrogen bond, which closes an *S*(6) ring and therefore establishes a *syn* relationship for the N atoms. In the crystal, molecules are linked by N—H···N hydrogen bonds, generating [100] chains containing alternating *A* and *B* molecules. Weak aromatic π–π stacking [minimum centroid–centroid separation = 3.6212 (9) Å] links the chains into a three-dimensional network.

1. Chemical context

Benzimidazole, benzoxazole, and benzothiazole derivatives are key components in many bioactive compounds of both natural and synthetic origin; many are active components of biocides such as bactericides, fungicides, insecticides and anticarcinogens (Kumar-Samota & Seth, 2010). Benzoxazole derivatives have been used as building blocks for biochemical and pharmaceutical agents, as well as dyes, fluorescent brightening agents, biomarkers and biosensors (Costa *et al.* 2007 and Tong *et al.* 2005).



In this context, 2-(2-aminophenyl)benzoxazole has shown considerable growth inhibition with respect to fungi and gram-positive and gram-negative bacteria (Elnima *et al.* 1981). For this reason, several methods have been described for the synthesis of these heterocyclic compounds, some of which are summarized in the Scheme, which shows the retrosynthesis for the preparation of the title compound, (I). For example, Gajare *et al.* (2000) described a procedure for the preparation of 2-(*o*-aminophenyl)oxazolines from isatoic anhydride and



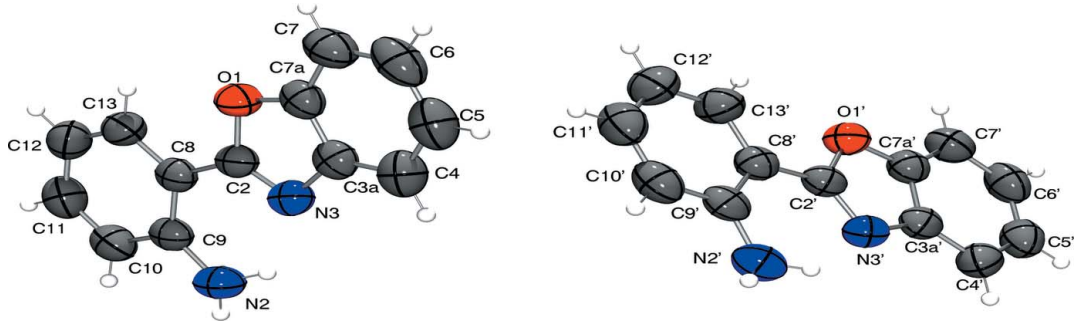


Figure 1
The asymmetric unit of (I) with displacement ellipsoids drawn at the 50% probability level (left: molecule *A* and right: molecule *B*)

2-aminoalcohols at reflux of PhCl mediated *via* a natural kaolinitic clay catalyst; a slightly modified procedure has been describe by Button & Gossage (2003) using zinc chloride as a catalyst. Qiao *et al.* (2011) described the synthesis of benzoxazole *via* the reaction of anionically activated trifluoromethyl groups with amino nucleophiles under mild aqueous conditions. Recently, Khalafi-Nezhad & Panahi (2014) reported an efficient approach for the preparation of benzoxazole derivatives, *via* acceptorless dehydrogenative coupling of alcohols with 2-aminophenol using an Ru catalytic system.

In the present work, as part of our ongoing studies of heterocyclic compounds (López-Ruiz *et al.*, 2011, 2013; de la Cerda-Pedro *et al.*, 2014), we report the synthesis of 2-(2-aminophenyl)benzoxazole, we analyse its molecular structure, as well as its weak intermolecular interactions in molecular packing, which could be useful in the understanding of their mode of action in pharmaceutical science, as well as in the design of materials with specific functions. The title compound has been previously reported by Button & Gossage (2003) from isatoic anhydride and 2-aminophenol but its crystal structure has not been described.

2. Structural commentary

Compound (I) crystallized in the monoclinic space group $P2_1/c$ with two independent molecules (*A* and *B*) in the asymmetric unit (Fig. 1). The orientation of the amino group can be described using as a basis the carbon atom C9, this orientation

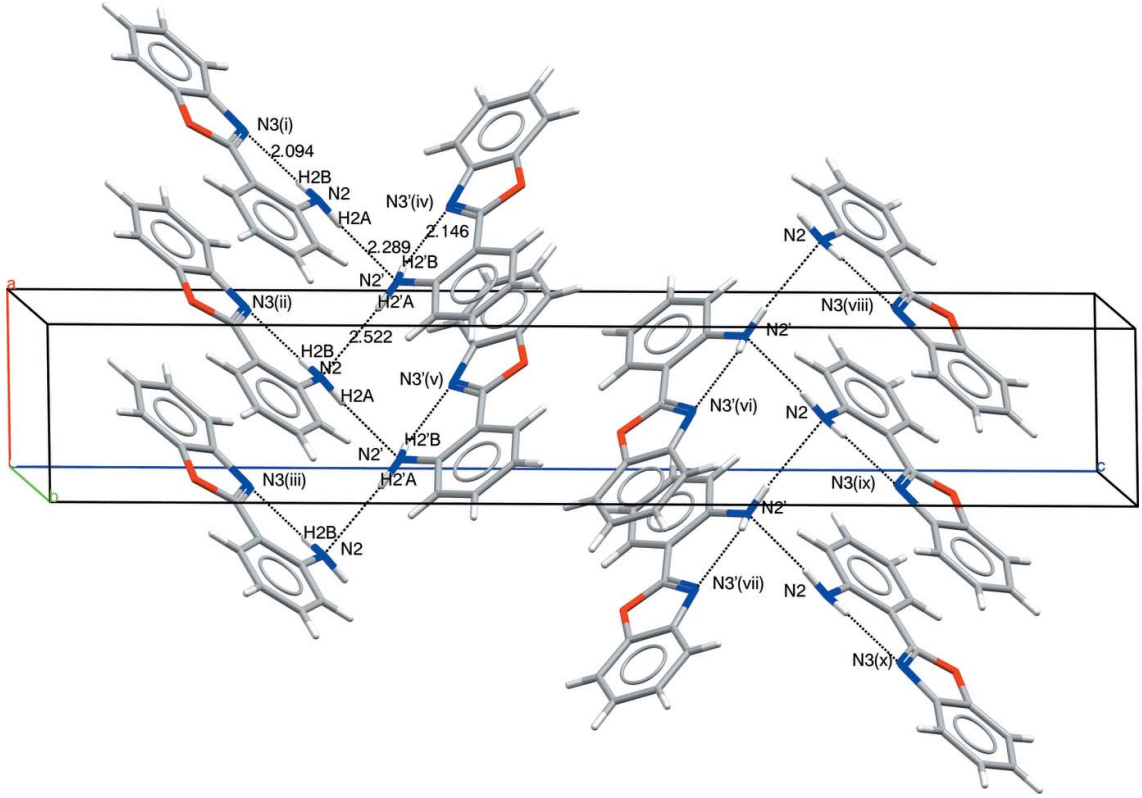


Figure 2
Crystal packing for (I), showing the formation of [100] chains. [Symmetry codes: (i) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (ii) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iii) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iv) $1+x, y, z$; (v) x, y, z; (vi) $1-x, 1-y, 1-z$; (vii) $-x, 1-y, 1-z$; (viii) $1+x, \frac{3}{2}-y, \frac{1}{2}+z$; (ix) $x, \frac{3}{2}-y, \frac{1}{2}+z$; (x) $-1+x, \frac{3}{2}-y, \frac{1}{2}+z$.]

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N2$'^{i}$	0.92 (2)	2.29 (2)	3.202 (2)	175 (2)
N2—H2B···N3	0.92 (1)	2.09 (2)	2.7679 (19)	129 (2)
N2′—H2′A···N2^{ii}	0.86 (2)	2.52 (2)	3.359 (2)	164 (2)
N2′—H2′B···N3′	0.89 (1)	2.15 (2)	2.7913 (19)	129 (2)

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

is *syn* to the nitrogen atom N3 and *anti* for the oxygen atom O1.

The skeleton of each molecule is practically planar: to analyse the planarity of the molecule we use the torsion angle N3—C2—C8—C9, indicating the rotation of the aromatic ring C8—C13: these angles are −1.2 (2) and 0.9 (2)° for molecules *A* and *B*, respectively. The dihedral angles between the benzene ring and the fused ring system are 0.74 (8) and 0.67 (6)° for molecules *A* and *B*, respectively. The two independent molecules are very similar, with an r.m.s. overlay fit of 0.019 Å.

3. Supramolecular features

In the crystal, each NH_2 group forms an intramolecular hydrogen bond of the type N2—H2B···N3 (Table 1) with an H···N distance of 2.094 (18) Å in molecule *A* and 2.146 (18) Å in molecule *B*, and an intermolecular N2—H2A···N2 hydrogen bond with a distance of 2.289 (15) Å for N2—H2A···N2′ and 2.522 (16) Å for N2′—H2A'···N2, forming zigzag chains propagating in the [100] direction and containing alternating *A* and *B* molecules (Fig. 2). Weak aromatic π–π stacking [minimum centroid–centroid separation = 3.6212 (9) Å] links the chains into a three-dimensional network.

4. Synthesis and crystallization

500 mg (3.00 mmol) of isatoic anhydride were dissolved in 50 mL of *m*-xylene then 390 mg (3.60 mmol) of *o*-aminophenol were added followed by the addition of 0.30 ml (10% mol) of a solution of $ZnCl_2$ (1 *M*). The mixture was then stirred and heated slowly to reflux temperature during 18 h. The crude reaction product was concentrated on a rotary evaporator with an azeotropic mixture of AcOEt/xylene to obtain a reddish brown solid which was dissolved in EtOAc and washed with 10% aq. NaCl solution. The crude reaction product was purified by column chromatography to give 356 mg (55%) of the amine (I) as a white solid m.p. = 381–382 K (literature value 379–381 K; Button & Gossage, 2003); IR (film) γ_{max} cm^{-1}: 3408 NH_2, 3051 C—H(arom), 1624 C═N; (literature value IR: 1620 cm^{-1}; Button & Gossage, 2003); ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 6.20 (*br s*, 2H, NH_2), 6.79 (*m*, 2H), 7.29 (*m*, 1H), 7.33 (*m*, 2H), 757 (*m*, 1H), 7.72 (*m*, 1H), 8.09 (*dd*, J = 1.6 Hz, J = 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 108.7, 110.4, 116.3, 116.8, 119.4, 124.3, 124.8, 128.8, 132.5, 141.9, 147.9, 149.3, 163.2 [Literature: Button & Gossage (2003); ^{1}H NMR δ = 6.15 (*br s*, 2H, –NH_2), 6.74 (*m*, 2H, ArH), 7.28 (*m*, 3H, ArH), 7.51 (*m*, 1H, ArH), 7.67 (*m*, 1H, ArH), 8.03 (*m*, 1H, ArH). ^{13}C{^{1}H} NMR δ = 108.7, 110.3, 116.3, 116.8, 119.4, 124.3, 124.7, 128.8, 132.4, 141.9, 147.9, 149.3, 163.2]. Analysis calculated for $C_{13}H_{10}N_2O$: C, 74.27; H, 4.79%; Found: C, 74.43; H, 5.05%.

The single crystal used in the experiment was obtained by the method of liquid–liquid diffusion by slow evaporation. The pure compound was dissolved in the minimum amount of dichloromethane to be added by the walls of the tube the same amount of acetone followed by methanol. The tube was sealed to leave the solution in a vibration-free environment at room temperature. After a few days, the solution had evaporated, leaving colourless blocks of the title compound.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{10}N_2O$
M_r	210.23
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	4.81703 (10), 14.8104 (3), 29.4801 (6)
β (°)	91.3715 (18)
V (Å^3)	2102.57 (7)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.69
Crystal size (mm)	0.38 × 0.14 × 0.11
Data collection	
Diffractometer	Agilent Xcalibur Atlas Gemini
Absorption correction	Analytical [*CrysAlis PRO* (Agilent, 2011), based on expressions derived by Clark & Reid (1995)]
T_{min}, T_{max}	0.742, 0.887
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21894, 4278, 3621
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.121, 1.02
No. of reflections	4278
No. of parameters	301
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.16

Computer programs: *CrysAlis PRO* (Agilent, 2011), *SHELXS97* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bond H atoms were placed in calculated positions and allowed to ride on their carrier atoms, with C—H = 0.93 Å (aromatic CH) and with U_{iso}(H) = 1.2U_{eq}(C). Hydrogen atoms of the amine group were found in a difference map and refined freely.

Acknowledgements

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Crystal structure of 2-(2-aminophenyl)-1,3-benzoxazole

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

2-(2-Aminophenyl)-1,3-benzoxazole

Crystal data

$C_{13}H_{10}N_2O$
M_r = 210.23
Monoclinic, $P2_1/c$
a = 4.81703 (10) Å
b = 14.8104 (3) Å
c = 29.4801 (6) Å
β = 91.3715 (18)°
V = 2102.57 (7) Å^3
Z = 8
$F(000)$ = 880
D_x = 1.328 Mg m^{-3}
Melting point: 381 K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 7503 reflections
θ = 3.0–74.3°
μ = 0.69 mm^{-1}
T = 293 K
Block, colourless
0.38 × 0.14 × 0.11 mm

Data collection

Agilent Xcalibur Atlas Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.3659 pixels mm^{-1}
ω scans
Absorption correction: analytical [*CrysAlis PRO* (Agilent, 2011), based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.742, T_{max} = 0.887
21894 measured reflections
4278 independent reflections
3621 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 74.5°, θ_{min} = 3.0°
h = −6→4
k = −18→18
l = −36→36

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.121
S = 1.02
4278 reflections
301 parameters
4 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.2656P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.35.15 (release 03-08-2011 CrysAlis171 .NET) (compiled Aug 3 2011,13:03:54) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by R.C. Clark & J.S. Reid. (Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0170 (2)	0.92286 (7)	0.34568 (3)	0.0625 (3)
O1′	0.6575 (2)	0.30487 (7)	0.46343 (3)	0.0605 (3)
N2	0.4198 (3)	0.85716 (10)	0.22601 (4)	0.0678 (3)
H2A	0.559 (4)	0.8439 (13)	0.2065 (6)	0.081*
H2B	0.337 (4)	0.8108 (11)	0.2413 (6)	0.081*
N2′	0.1130 (3)	0.31854 (11)	0.34698 (5)	0.0703 (4)
H2′A	−0.034 (4)	0.3172 (13)	0.3298 (6)	0.084*
H2′B	0.180 (4)	0.2642 (11)	0.3545 (7)	0.084*
N3	0.0403 (2)	0.81678 (8)	0.29206 (4)	0.0575 (3)
N3′	0.5227 (3)	0.23223 (8)	0.39963 (4)	0.0576 (3)
C2	0.1126 (3)	0.89555 (9)	0.30691 (4)	0.0532 (3)
C2′	0.4906 (3)	0.30246 (9)	0.42478 (4)	0.0533 (3)
C4	−0.3081 (3)	0.70682 (12)	0.32430 (7)	0.0735 (4)
H4	−0.2870	0.6619	0.3026	0.088*
C4′	0.8489 (4)	0.09914 (11)	0.41168 (6)	0.0683 (4)
H4′	0.7961	0.0673	0.3857	0.082*
C5	−0.4916 (4)	0.69714 (13)	0.35908 (7)	0.0804 (5)
H5	−0.5976	0.6448	0.3607	0.096*
C3A	−0.1563 (3)	0.78613 (10)	0.32287 (5)	0.0587 (3)
C3A′	0.7278 (3)	0.18126 (10)	0.42227 (5)	0.0567 (3)
C5′	1.0510 (4)	0.06674 (12)	0.44134 (6)	0.0752 (5)
H5′	1.1357	0.0118	0.4352	0.090*
C6	−0.5220 (4)	0.76345 (16)	0.39167 (7)	0.0864 (6)
H6	−0.6470	0.7543	0.4148	0.104*
C6′	1.1316 (4)	0.11380 (13)	0.48016 (6)	0.0763 (5)
H6′	1.2695	0.0899	0.4992	0.092*
C7	−0.3698 (4)	0.84388 (14)	0.39075 (6)	0.0785 (5)
H7	−0.3887	0.8890	0.4124	0.094*
C7′	1.0110 (4)	0.19587 (12)	0.49121 (5)	0.0708 (4)
H7′	1.0631	0.2279	0.5172	0.085*
C8	0.3115 (3)	0.95868 (9)	0.28832 (4)	0.0536 (3)
C7A	−0.1899 (3)	0.85106 (11)	0.35532 (5)	0.0607 (3)
C7A′	0.8096 (3)	0.22647 (10)	0.46119 (5)	0.0574 (3)
C8′	0.3042 (3)	0.37874 (10)	0.41810 (5)	0.0564 (3)
C9	0.4607 (3)	0.93665 (10)	0.24915 (4)	0.0553 (3)

C9′	0.1199 (3)	0.38352 (11)	0.38020 (5)	0.0590 (3)
C10	0.6493 (3)	1.00084 (11)	0.23331 (5)	0.0670 (4)
H10	0.7479	0.9883	0.2073	0.080*
C10′	−0.0505 (4)	0.46002 (13)	0.37630 (6)	0.0734 (4)
H10′	−0.1725	0.4652	0.3515	0.088*
C11	0.6923 (4)	1.08145 (12)	0.25509 (6)	0.0734 (4)
H11	0.8203	1.1223	0.2439	0.088*
C11′	−0.0415 (4)	0.52712 (13)	0.40806 (7)	0.0805 (5)
H11′	−0.1578	0.5769	0.4046	0.097*
C12	0.5471 (4)	1.10261 (11)	0.29352 (6)	0.0722 (4)
H12	0.5765	1.1575	0.3082	0.087*
C12′	0.1369 (4)	0.52214 (13)	0.44503 (7)	0.0815 (5)
H12′	0.1420	0.5681	0.4665	0.098*
C13	0.3603 (3)	1.04212 (10)	0.30963 (5)	0.0634 (4)
H13	0.2624	1.0565	0.3355	0.076*
C13′	0.3072 (4)	0.44845 (11)	0.44974 (6)	0.0709 (4)
H13′	0.4280	0.4449	0.4748	0.085*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0665 (6)	0.0656 (6)	0.0558 (5)	0.0094 (5)	0.0078 (4)	−0.0016 (4)
O1′	0.0618 (6)	0.0648 (6)	0.0549 (5)	0.0045 (5)	−0.0013 (4)	−0.0050 (4)
N2	0.0683 (8)	0.0784 (8)	0.0571 (7)	0.0071 (7)	0.0072 (6)	−0.0083 (6)
N2′	0.0623 (8)	0.0896 (9)	0.0586 (7)	−0.0043 (7)	−0.0046 (6)	0.0023 (7)
N3	0.0519 (7)	0.0619 (6)	0.0587 (6)	0.0081 (5)	−0.0023 (5)	−0.0015 (5)
N3′	0.0572 (7)	0.0621 (6)	0.0535 (6)	−0.0006 (5)	0.0038 (5)	−0.0030 (5)
C2	0.0505 (7)	0.0584 (7)	0.0504 (6)	0.0132 (6)	−0.0028 (5)	0.0011 (5)
C2′	0.0502 (7)	0.0615 (7)	0.0485 (6)	−0.0033 (6)	0.0054 (5)	0.0013 (5)
C4	0.0572 (9)	0.0744 (10)	0.0885 (11)	−0.0002 (7)	−0.0072 (8)	0.0117 (8)
C4′	0.0738 (10)	0.0631 (8)	0.0686 (9)	0.0043 (7)	0.0107 (8)	−0.0016 (7)
C5	0.0558 (9)	0.0857 (11)	0.0994 (13)	−0.0001 (8)	−0.0036 (9)	0.0281 (10)
C3A	0.0468 (7)	0.0663 (8)	0.0627 (8)	0.0096 (6)	−0.0060 (6)	0.0087 (6)
C3A′	0.0555 (8)	0.0593 (7)	0.0558 (7)	−0.0024 (6)	0.0100 (6)	0.0028 (6)
C5′	0.0793 (11)	0.0663 (9)	0.0807 (11)	0.0137 (8)	0.0157 (9)	0.0109 (8)
C6	0.0597 (10)	0.1152 (15)	0.0846 (12)	0.0099 (10)	0.0102 (8)	0.0386 (11)
C6′	0.0704 (10)	0.0836 (11)	0.0749 (10)	0.0141 (8)	0.0036 (8)	0.0226 (9)
C7	0.0724 (11)	0.0958 (12)	0.0676 (9)	0.0158 (9)	0.0120 (8)	0.0119 (9)
C7′	0.0714 (10)	0.0821 (10)	0.0587 (8)	0.0035 (8)	−0.0009 (7)	0.0056 (7)
C8	0.0497 (7)	0.0589 (7)	0.0519 (7)	0.0097 (6)	−0.0045 (5)	0.0056 (5)
C7A	0.0516 (8)	0.0693 (8)	0.0612 (8)	0.0105 (6)	−0.0002 (6)	0.0123 (6)
C7A′	0.0553 (8)	0.0608 (8)	0.0565 (7)	0.0016 (6)	0.0068 (6)	0.0046 (6)
C8′	0.0516 (8)	0.0620 (7)	0.0560 (7)	0.0003 (6)	0.0084 (6)	0.0041 (6)
C9	0.0508 (8)	0.0651 (7)	0.0498 (6)	0.0117 (6)	−0.0053 (5)	0.0033 (6)
C9′	0.0491 (8)	0.0746 (9)	0.0538 (7)	−0.0041 (6)	0.0107 (6)	0.0103 (6)
C10	0.0612 (9)	0.0797 (10)	0.0603 (8)	0.0094 (8)	0.0057 (7)	0.0103 (7)
C10′	0.0582 (9)	0.0937 (12)	0.0684 (9)	0.0094 (8)	0.0059 (7)	0.0210 (8)
C11	0.0681 (10)	0.0721 (9)	0.0799 (10)	−0.0009 (8)	0.0014 (8)	0.0171 (8)

C11′	0.0741 (11)	0.0815 (11)	0.0865 (12)	0.0231 (9)	0.0164 (9)	0.0173 (9)
C12	0.0804 (11)	0.0594 (8)	0.0765 (10)	−0.0002 (8)	−0.0034 (8)	0.0037 (7)
C12′	0.0881 (13)	0.0732 (10)	0.0835 (11)	0.0182 (9)	0.0080 (9)	−0.0049 (9)
C13	0.0676 (9)	0.0636 (8)	0.0592 (8)	0.0094 (7)	0.0039 (7)	0.0005 (6)
C13′	0.0731 (11)	0.0717 (9)	0.0677 (9)	0.0098 (8)	−0.0004 (8)	−0.0062 (7)

Geometric parameters (Å, º)

O1—C2	1.3763 (16)	C6—H6	0.9300
O1—C7A	1.3842 (19)	C6—C7	1.399 (3)
O1′—C2′	1.3791 (16)	C6′—H6′	0.9300
O1′—C7A′	1.3756 (17)	C6′—C7′	1.389 (2)
N2—H2A	0.916 (15)	C7—H7	0.9300
N2—H2B	0.919 (14)	C7—C7A	1.377 (2)
N2—C9	1.372 (2)	C7′—H7′	0.9300
N2′—H2′A	0.861 (15)	C7′—C7A′	1.374 (2)
N2′—H2′B	0.894 (14)	C8—C9	1.4129 (19)
N2′—C9′	1.373 (2)	C8—C13	1.404 (2)
N3—C2	1.2910 (18)	C8′—C9′	1.412 (2)
N3—C3A	1.4033 (19)	C8′—C13′	1.391 (2)
N3′—C2′	1.2889 (18)	C9—C10	1.403 (2)
N3′—C3A′	1.4001 (19)	C9′—C10′	1.402 (2)
C2—C8	1.455 (2)	C10—H10	0.9300
C2′—C8′	1.454 (2)	C10—C11	1.369 (3)
C4—H4	0.9300	C10′—H10′	0.9300
C4—C5	1.377 (3)	C10′—C11′	1.365 (3)
C4—C3A	1.385 (2)	C11—H11	0.9300
C4′—H4′	0.9300	C11—C12	1.382 (3)
C4′—C3A′	1.388 (2)	C11′—H11′	0.9300
C4′—C5′	1.379 (2)	C11′—C12′	1.374 (3)
C5—H5	0.9300	C12—H12	0.9300
C5—C6	1.384 (3)	C12—C13	1.363 (2)
C3A—C7A	1.369 (2)	C12′—H12′	0.9300
C3A′—C7A′	1.378 (2)	C12′—C13′	1.370 (2)
C5′—H5′	0.9300	C13—H13	0.9300
C5′—C6′	1.387 (3)	C13′—H13′	0.9300
C2—O1—C7A	103.41 (11)	C7A′—C7′—C6′	115.48 (16)
C7A′—O1′—C2′	103.83 (11)	C7A′—C7′—H7′	122.3
H2A—N2—H2B	118.9 (17)	C9—C8—C2	120.78 (13)
C9—N2—H2A	113.5 (12)	C13—C8—C2	120.11 (13)
C9—N2—H2B	117.1 (12)	C13—C8—C9	119.10 (14)
H2′A—N2′—H2′B	114.5 (19)	C3A—C7A—O1	108.32 (13)
C9′—N2′—H2′A	116.2 (14)	C3A—C7A—C7	124.26 (17)
C9′—N2′—H2′B	116.8 (13)	C7—C7A—O1	127.41 (16)
C2—N3—C3A	104.68 (12)	O1′—C7A′—C3A′	107.95 (13)
C2′—N3′—C3A′	104.70 (12)	C7′—C7A′—O1′	128.04 (14)
O1—C2—C8	116.10 (12)	C7′—C7A′—C3A′	124.01 (15)

N3—C2—O1	115.04 (13)	C9′—C8′—C2′	121.39 (13)
N3—C2—C8	128.86 (13)	C13′—C8′—C2′	119.30 (14)
O1′—C2′—C8′	115.96 (12)	C13′—C8′—C9′	119.31 (14)
N3′—C2′—O1′	114.88 (12)	N2—C9—C8	122.32 (14)
N3′—C2′—C8′	129.16 (13)	N2—C9—C10	120.15 (14)
C5—C4—H4	121.3	C10—C9—C8	117.49 (14)
C5—C4—C3A	117.38 (18)	N2′—C9′—C8′	122.22 (14)
C3A—C4—H4	121.3	N2′—C9′—C10′	120.26 (15)
C3A′—C4′—H4′	121.4	C10′—C9′—C8′	117.45 (15)
C5′—C4′—H4′	121.4	C9—C10—H10	119.1
C5′—C4′—C3A′	117.16 (16)	C11—C10—C9	121.80 (15)
C4—C5—H5	119.2	C11—C10—H10	119.1
C4—C5—C6	121.56 (18)	C9′—C10′—H10′	119.2
C6—C5—H5	119.2	C11′—C10′—C9′	121.53 (16)
C4—C3A—N3	131.24 (15)	C11′—C10′—H10′	119.2
C7A—C3A—N3	108.55 (13)	C10—C11—H11	119.7
C7A—C3A—C4	120.21 (15)	C10—C11—C12	120.57 (16)
C4′—C3A′—N3′	131.37 (14)	C12—C11—H11	119.7
C7A′—C3A′—N3′	108.64 (13)	C10′—C11′—H11′	119.5
C7A′—C3A′—C4′	120.00 (15)	C10′—C11′—C12′	120.94 (17)
C4′—C5′—H5′	119.1	C12′—C11′—H11′	119.5
C4′—C5′—C6′	121.84 (16)	C11—C12—H12	120.4
C6′—C5′—H5′	119.1	C13—C12—C11	119.20 (16)
C5—C6—H6	119.1	C13—C12—H12	120.4
C5—C6—C7	121.76 (17)	C11′—C12′—H12′	120.5
C7—C6—H6	119.1	C13′—C12′—C11′	118.99 (18)
C5′—C6′—H6′	119.2	C13′—C12′—H12′	120.5
C5′—C6′—C7′	121.52 (16)	C8—C13—H13	119.1
C7′—C6′—H6′	119.2	C12—C13—C8	121.83 (15)
C6—C7—H7	122.6	C12—C13—H13	119.1
C7A—C7—C6	114.83 (18)	C8′—C13′—H13′	119.1
C7A—C7—H7	122.6	C12′—C13′—C8′	121.77 (17)
C6′—C7′—H7′	122.3	C12′—C13′—H13′	119.1

O1—C2—C8—C9	178.67 (11)	C5—C4—C3A—N3	−178.83 (15)
O1—C2—C8—C13	−0.43 (18)	C5—C4—C3A—C7A	0.4 (2)
O1′—C2′—C8′—C9′	−179.01 (12)	C5—C6—C7—C7A	−0.2 (3)
O1′—C2′—C8′—C13′	0.9 (2)	C3A—N3—C2—O1	0.01 (15)
N2—C9—C10—C11	178.96 (15)	C3A—N3—C2—C8	179.91 (13)
N2′—C9′—C10′—C11′	−177.83 (16)	C3A—C4—C5—C6	−0.6 (3)
N3—C2—C8—C9	−1.2 (2)	C3A′—N3′—C2′—O1′	0.00 (16)
N3—C2—C8—C13	179.67 (14)	C3A′—N3′—C2′—C8′	−179.94 (13)
N3—C3A—C7A—O1	−0.04 (15)	C3A′—C4′—C5′—C6′	−0.1 (3)
N3—C3A—C7A—C7	179.31 (14)	C5′—C4′—C3A′—N3′	179.39 (15)
N3′—C2′—C8′—C9′	0.9 (2)	C5′—C4′—C3A′—C7A′	−0.4 (2)
N3′—C2′—C8′—C13′	−179.17 (15)	C5′—C6′—C7′—C7A′	−0.1 (3)
N3′—C3A′—C7A′—O1′	0.35 (16)	C6—C7—C7A—O1	179.19 (14)
N3′—C3A′—C7A′—C7′	−179.14 (14)	C6—C7—C7A—C3A	0.0 (2)

C2—O1—C7A—C3A	0.05 (14)	C6′—C7′—C7A′—O1′	−179.79 (15)
C2—O1—C7A—C7	−179.29 (15)	C6′—C7′—C7A′—C3A′	−0.4 (2)
C2—N3—C3A—C4	179.31 (15)	C8—C9—C10—C11	1.0 (2)
C2—N3—C3A—C7A	0.02 (15)	C7A—O1—C2—N3	−0.03 (15)
C2—C8—C9—N2	2.2 (2)	C7A—O1—C2—C8	−179.95 (11)
C2—C8—C9—C10	−179.83 (12)	C7A′—O1′—C2′—N3′	0.21 (15)
C2—C8—C13—C12	179.28 (14)	C7A′—O1′—C2′—C8′	−179.85 (12)
C2′—O1′—C7A′—C3A′	−0.33 (14)	C8′—C9′—C10′—C11′	−0.6 (2)
C2′—O1′—C7A′—C7′	179.14 (15)	C9—C8—C13—C12	0.2 (2)
C2′—N3′—C3A′—C4′	179.99 (15)	C9—C10—C11—C12	−0.7 (3)
C2′—N3′—C3A′—C7A′	−0.21 (16)	C9′—C8′—C13′—C12′	−0.4 (3)
C2′—C8′—C9′—N2′	−2.3 (2)	C9′—C10′—C11′—C12′	0.3 (3)
C2′—C8′—C9′—C10′	−179.43 (13)	C10—C11—C12—C13	0.1 (3)
C2′—C8′—C13′—C12′	179.70 (16)	C10′—C11′—C12′—C13′	0.0 (3)
C4—C5—C6—C7	0.5 (3)	C11—C12—C13—C8	0.2 (3)
C4—C3A—C7A—O1	−179.43 (13)	C11′—C12′—C13′—C8′	0.1 (3)
C4—C3A—C7A—C7	−0.1 (2)	C13—C8—C9—N2	−178.64 (13)
C4′—C3A′—C7A′—O1′	−179.83 (13)	C13—C8—C9—C10	−0.72 (19)
C4′—C3A′—C7A′—C7′	0.7 (2)	C13′—C8′—C9′—N2′	177.80 (15)
C4′—C5′—C6′—C7′	0.4 (3)	C13′—C8′—C9′—C10′	0.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N2^{i}	0.92 (2)	2.29 (2)	3.202 (2)	175 (2)
N2—H2*B*···N3	0.92 (1)	2.09 (2)	2.7679 (19)	129 (2)
N2′—H2′*A*···N2^{ii}	0.86 (2)	2.52 (2)	3.359 (2)	164 (2)
N2′—H2′*B*···N3′	0.89 (1)	2.15 (2)	2.7913 (19)	129 (2)

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