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Crystal structure of (1*S*,2*R*)-2-hydroxy-1,2-diphenylethan-1-aminium (*S*)-2-azaniumylbutanedioate monohydrate

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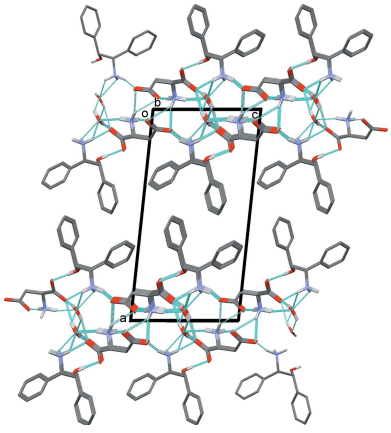
The title diastereomeric salt, formed between 2-amino-1,2-diphenylethanol (ADE) and aspartic acid (ASP), $C_{14}H_{16}NO^{+}\cdot C_4H_6NO_4^{-}\cdot H_2O$, crystallizes as a monohydrate. The 1,2-diphenylethyl group in the cation has a *cis* conformation, and the aspartic acid anion is in the zwitterionic form. In the crystal, the ASP anions are linked *via* N—H···O hydrogen bonds to form a 2_1 helix along the *b*-axis direction. The helices are linked by the ADE cations *via* O—H···O and N—H···O hydrogen bonds, forming layers parallel to the *bc* plane. There are channels in the layers that are occupied by water molecules, which link to both the anions and cations *via* O_{water}—H···O and N—H···O_{water} hydrogen bonds. There are also C—H···O and C—H···π interactions present within the layers.

1. Chemical context

The production of chiral compounds has great importance in the pharmaceutical industry, and diastereomeric salt separation is still widely applied in the process. A synthetic optical resolving agent, chiral 2-amino-1,2-diphenylethanol (ADE) (Read & Steele, 1927), has been widely tried and used in diastereomeric salt-separation methods for chiral alcohols or organic acids. L-(*S*)-aspartic acid (ASP) is a known neurotransmitter, and D-(*R*)-ASP is a non-essential amino acid, one of two D-amino acids commonly found in mammals. D-ASP has also attracted attention as residue in the antifungal bacitracin, while *N*-methyl-D-aspartic acid (NMDA) acts as a specific agonist at the NMDA receptor. D-amino acids are mainly resolved enzymatically with D-aminoacylase (EC 3.5.1.14) in industrial applications. The optical separation of ASP with *cis*-ADE was introduced without chemical modification. The crystal structure of the title molecular salt, formed between L-(*S*)-ASP and (*1R*,*2S*)-*cis*-ADE, is reported herein.



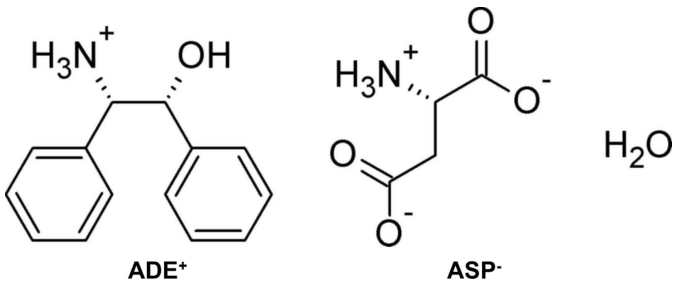


Table 1
Selected torsion angles (°).

O1*A*—C1*A*—C2*A*—N1*A*	−65.0 (2)	N1*B*—C2*B*—C3*B*—C4*B*	73.0 (2)
C3*A*—C1*A*—C2*A*—C9*A*	−66.1 (2)	C1*B*—C2*B*—C3*B*—C4*B*	−53.0 (2)
O1*B*—C1*B*—C2*B*—N1*B*	17.4 (2)	C2*B*—C3*B*—C4*B*—O3*B*	1.4 (3)

2. Structural commentary

The molecular structures of the components of the title salt are shown in Fig. 1, and selected torsion angles are given in Table 1. It can be seen that the hydroxy and protonated amino groups of *cis*-ADE form a tweezer-like motif. The dihedral angle between the phenyl rings (*A* and *B*; Fig.1) is 48.71 (9)° and the torsion angle O1*A*—C1*A*—C2*A*—N1*A* is −65.0 (2)°. The hydroxy group adopts a *gauche* conformation [O1*A*—C1*A*—C2*A*—C9*A* = 60.1 (2)°] with respect to phenyl ring *B*. Thus, the tweezer-like motif is twisted with respect to the phenyl groups. This arrangement is similar to that found in racemic *cis*-ADE (Fujii, 2015) and the diastereomeric salts formed with *cis*-enantiomers.

L-(*S*)-ASP crystallizes as a deprotonated zwitterion. The succinate group adopts a *cis* conformation [C1*B*—C2*B*—C3*B*—C4*B* = −53.0 (2)°], which is the motif commonly found in L-ASP salts; for example L-His·L-ASP monohydrate (Suresh & Vijayan, 1987). The amino and residual carboxy groups have a slightly right-handed helical-shape; torsion angles N1*B*—C2*B*—C3*B*—C4*B* and C2*B*—C3*B*—C4*B*—O3*B* are 73.0 (2) and 1.4 (3)°, respectively.

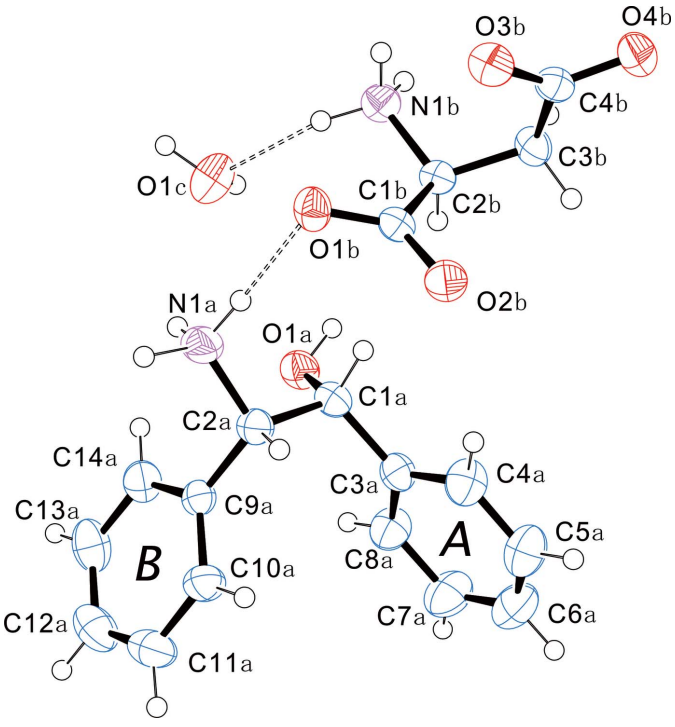


Figure 1
A view of the molecular structure of (1*S*,2*R*)-*cis*-ADE·(*S*)-ASP monohydrate, with the atom and ring labelling. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate the hydrogen bonds (see Table 2).

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

CgB is the centroid of phenyl ring *B* (C9–C14).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*B*—H1*B*1···O3*B*^{i}	0.92 (4)	1.92 (4)	2.819 (2)	168 (3)
N1*B*—H1*B*3···O3*B*^{ii}	0.87 (3)	2.46 (3)	3.112 (2)	132 (3)
N1*B*—H1*B*3···O4*B*^{ii}	0.87 (3)	2.20 (3)	2.868 (2)	134 (3)
O1*A*—H1*O*1···O2*B*^{i}	0.88 (3)	1.88 (3)	2.752 (2)	171 (3)
N1*A*—H1*A*1···O4*B*^{iii}	1.08 (4)	1.70 (4)	2.742 (3)	162 (3)
N1*A*—H1*A*2···O1*B*	0.95 (3)	1.92 (3)	2.862 (2)	173 (3)
O1*C*—H1*OB*···O1*B*^{i}	0.92 (4)	1.82 (4)	2.734 (2)	173 (3)
O1*C*—H1*OA*···O1*B*^{iv}	0.90 (4)	2.01 (4)	2.840 (2)	153 (4)
N1*A*—H1*A*3···O1*C*	0.87 (4)	2.45 (3)	2.926 (3)	115 (2)
N1*B*—H1*B*2···O1*C*	0.96 (2)	2.01 (3)	2.938 (2)	163 (2)
C2*A*—H2*A*···O1*A*^{v}	0.98	2.42	3.304 (3)	150
C14*A*—H14*A*···O4*B*^{vi}	0.93	2.53	3.371 (3)	150
C3*B*—H3*B*2···Cg*B*^{iii}	0.97	2.87	3.6127 (16)	134

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

3. Supramolecular features

In the crystal, the (*S*)-ASP anions correlated with crystallographic symmetry are linked *via* N1*B*—H1*B*3···O4*B*^{ii} [2.868 (2) Å] hydrogen bonds into *C*(6) chains to form a right-handed 2_1-helix along the *b*-axis direction (Fig. 2 and Table 2). The helices are linked by the (*1R,2S*)-*cis*-ADE cations *via* N—H···O hydrogen bonds [N1*A*—H1*A*2···O1*B* = 2.862 (2) Å and N1*A*—H1*A*1···O4*B*^{iii} = 2.742 (3) Å] and O—H···O hydrogen bonds [O1*A*—H1*O*1···O2*B*^{i} = 2.752 (2) Å], forming layers parallel to the *bc* plane (Fig. 3, Table 2). There are channels in the layers that are occupied by water molecules which link to both the anions and cations *via* tetrahedrally placed hydrogen bonds; O_{water}—H···O hydrogen bonds

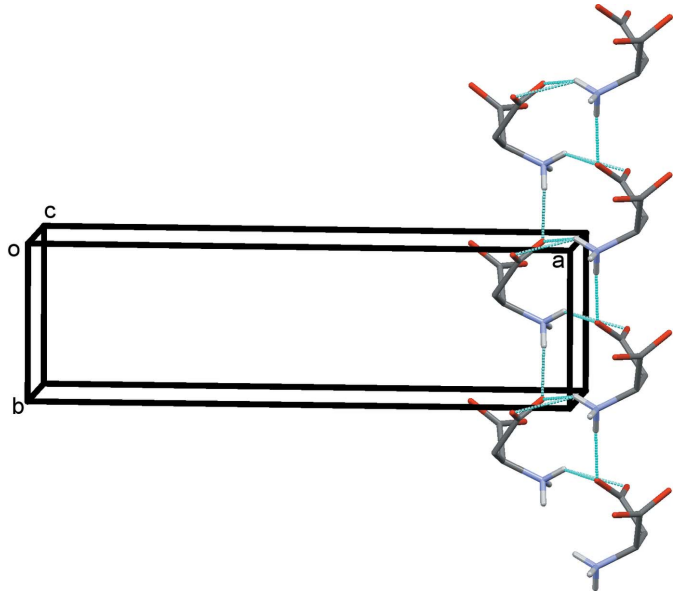


Figure 2
A view along the *c* axis of the right-handed 2_1-helix of ASP anions. Hydrogen bonds are shown as dashed lines (see Table 2) and C-bound H atoms have been omitted.

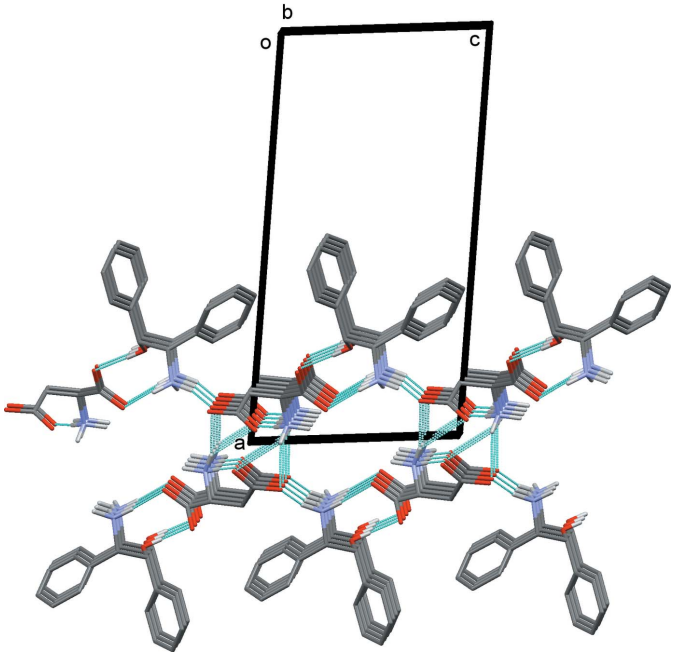


Figure 3
A partial view along the *b* axis of the crystal packing of the ASP helices linked by the ADE cations. Hydrogen bonds are shown as dashed lines (see Table 2) and C-bound H atoms have been omitted.

[O1*C*—H1*OB*···O1*B*^{i} = 2.734 (2) Å and O1*C*—H1*OA*···O1*B*^{iv} = 2.840 (2) Å] and N—H···O_{water} hydrogen bonds [N1*B*—H1*B*2···O1*C* = 2.938 (2) Å and N1*A*—H1*A*3···O1*C* = 2.926 (3) Å], shown in Fig. 4; see also Table 2. There are also C—H···O and C—H···π interactions present within the layers (Table 2). Finally, the hydrophobic and hydrophilic layers are well separated along the *a*-axis direction.

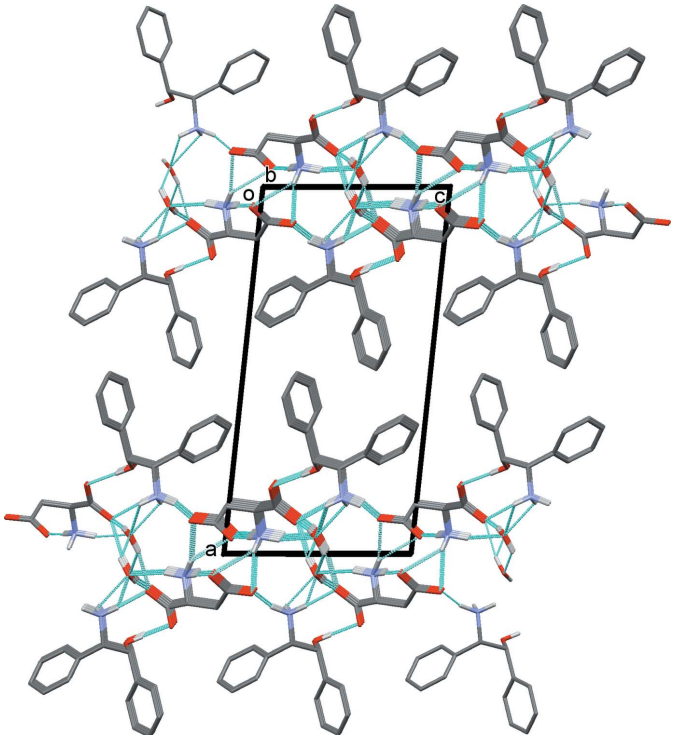


Figure 4
A view along the *b* axis of the crystal packing of (1*S*,2*R*)-*cis*-ADE·(*S*)-ASP monohydrate. Hydrogen bonds are shown as dashed lines (see Table 2) and C-bound H atoms have been omitted.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{16}NO^{+}\cdot C_4H_6NO_4^{-}\cdot H_2O$
M_r	364.39
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	297
a, *b*, *c* (Å)	18.310 (8), 5.2661 (10), 9.2792 (10)
β (°)	96.070 (4)
V (Å^3)	889.7 (4)
Z	2
Radiation type	Cu *K*α
μ (mm^{-1})	0.86
Crystal size (mm)	0.4 × 0.2 × 0.2
Data collection	
Diffractometer	Enraf–Nonius CAD-4
Absorption correction	ψ scan (North *et al.*, 1968)
T_{min}, T_{max}	0.74, 0.86
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2114, 2051, 1907
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.626
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.028, 0.079, 1.06
No. of reflections	2051
No. of parameters	272
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.17, −0.13
Absolute structure	No quotients, so Flack (1983) parameter determined by classical intensity fit
Absolute structure parameter	0.1 (2)

Computer programs: CAD4 (Enraf–Nonius, 1994), *XCAD4* (Harms & Wocadlo, 1995), *SHELXS86* (Sheldrick, 2008), *SHELXL2017* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

4. Database survey

The author has reported the crystal structures of several amino acids without chemical modification including the chiral resolving agents; 1,1′-binaphthalene-2,2′-diyl hydrogen phosphate, 2-phenoxypropionic acid and mandelic acid (Fujii & Hirayama, 2002; Fujii *et al.*, 2005, 2006). The crystal structures of racemic *trans*- and *cis*-ADE have been reported (GAQXON: Bari *et al.*, 2012; RUTROP: Fujii, 2015, respectively). Recently, the solvent-induced chirality switching in optical resolution between mandelic acid and *cis*-ADE has been demonstrated (Shitara *et al.*, 2013). Moreover, a database search (CSD Version 5.28, last update May 2017; Groom *et al.*, 2016) yielded other comparable structures, *viz.* L-aspartic acid (LASPRT: Derissen *et al.*, 1968), L-aspartic acid monohydrate (IJEQET: Umadevi *et al.*, 2003) and *N*-methyl-D-aspartic acid monohydrate (KEWGUO: Sawka-Dobrowolska *et al.*, 1990).

5. Synthesis and crystallization

(1*R*,2*S*)-*cis*-2-Amino-1,2-diphenylethanol (ADE) and aspartic acid (ASP) were purchased from Sigma–Aldrich Co. Ltd. The title molecular salt was obtained from an aqueous ethanol solution of racemic-ASP and (1*R*,2*S*)-*cis*-ADE in a 2:1 molar ratio, heated to 333 K under stirring. On slow cooling to ambient temperature and slow evaporation of the solvent, colourless rod-shaped crystals were obtained.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All the H atoms were located in difference-Fourier maps. The NH_3^+, OH, and water H atoms were freely refined. The C-bound H atoms were included in calculated positions and treated as riding atoms: C—H = 0.93–0.98 Å with $U_{iso}(H) = 1.2U_{eq}(C)$.

Acknowledgements

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References

Bari, A., Al-Obaid, A. M. & Ng, S. W. (2012). *Acta Cryst.* E**68**, o491.
Derissen, J. L., Endeman, H. J. & Peerdeman, A. F. (1968). *Acta Cryst.* B**24**, 1349–1354.
Enraf–Nonius (1994). CAD4. Enraf-Nonius, Delft, The Netherlands.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fujii, I. (2015). *Acta Cryst.* E**71**, 1539–1541.
Fujii, I., Baba, H. & Takahashi, Y. (2005). *Anal. Sci. X-ray Struct. Anal. Online*, **21**, x175–176.
Fujii, I. & Hirayama, N. (2002). *Helv. Chim. Acta*, **85**, 2946–2960.
Fujii, I., Watadani, T., Nunomura, S. & Takahashi, Y. (2006). *Anal. Sci. X-ray Struct. Anal. Online*, **22**, x75–x76.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Harms, K. & Wocadlo, S. (1995). *XCAD4.* University of Marburg, Germany.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Read, J. & Steele, C. C. (1927). *J. Chem. Soc.* pp. 910–918.
Sawka-Dobrowolska, W., Głowiak, T., Kozłowski, H. & Masterlerz, P. (1990). *Acta Cryst.* C**46**, 1679–1681.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Shitara, H., Shintani, T., Kodama, K. & Hirose, T. (2013). *J. Org. Chem.* **78**, 9309–9316.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suresh, C. G. & Vijayan, M. (1987). *J. Biosci.* **12**, 13–21.
Umadevi, K., Anitha, K., Sridhar, B., Srinivasan, N. & Rajaram, R. K. (2003). *Acta Cryst.* E**59**, o1073–o1075.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

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Crystal structure of (1*S*,2*R*)-2-hydroxy-1,2-diphenylethan-1-aminium (*S*)-2-azaniumylbutanedioate monohydrate

Isao Fujii

Computing details

Data collection: CAD4 (Enraf–Nonius, 1994); cell refinement: CAD4 (Enraf–Nonius, 1994); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

(1*S*,2*R*)-2-Hydroxy-1,2-diphenylethan-1-aminium (*S*)-2-azaniumylbutanedioate monohydrate

Crystal data

$C_{14}H_{16}NO^{+}\cdot C_4H_6NO_4^{-}\cdot H_2O$
M_r = 364.39
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 18.310 (8) Å
b = 5.2661 (10) Å
c = 9.2792 (10) Å
β = 96.070 (4)°
V = 889.7 (4) Å^3
Z = 2
$F(000)$ = 388
D_x = 1.36 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 28.2–34.6°
μ = 0.86 mm^{-1}
T = 297 K
Rod, colorless
0.4 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: Enraf Nonius FR590
Graphite monochromator
non–profiled $\omega/2\tau$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.74, T_{max} = 0.86
2114 measured reflections
2051 independent reflections
1907 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 74.9°, θ_{min} = 2.4°
h = 0→22
k = 0→6
l = −11→11
3 standard reflections every 60 min
intensity decay: 5%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.079
S = 1.06
2051 reflections
272 parameters
1 restraint
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.0743P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}

Extinction correction: (SHELXL2017; Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0104 (12)
Absolute structure: No quotients, so Flack (1983) parameter determined by classical intensity fit
Absolute structure parameter: 0.1 (2)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1B	0.86265 (9)	0.1336 (4)	0.25943 (17)	0.0285 (3)
C2B	0.87228 (9)	0.3556 (4)	0.15574 (17)	0.0281 (3)
H2B	0.834662	0.481729	0.171984	0.034*
C3B	0.86125 (10)	0.2862 (4)	−0.00305 (18)	0.0331 (4)
H3B1	0.872821	0.433561	−0.059083	0.040*
H3B2	0.809703	0.247484	−0.028525	0.040*
C4B	0.90650 (9)	0.0630 (4)	−0.04800 (18)	0.0309 (4)
N1B	0.94481 (9)	0.4807 (4)	0.19193 (17)	0.0344 (3)
O1B	0.90523 (7)	0.1301 (3)	0.37604 (13)	0.0363 (3)
O2B	0.81052 (7)	−0.0126 (3)	0.22709 (14)	0.0410 (3)
O3B	0.94788 (7)	−0.0504 (3)	0.04607 (14)	0.0378 (3)
O4B	0.89947 (7)	0.0126 (3)	−0.18182 (14)	0.0437 (4)
H1B1	0.9480 (15)	0.620 (7)	0.134 (3)	0.064 (8)*
H1B2	0.9537 (12)	0.517 (5)	0.294 (3)	0.044 (6)*
H1B3	0.9841 (16)	0.405 (7)	0.168 (3)	0.069 (9)*
C1A	0.73892 (10)	0.5069 (4)	0.44043 (19)	0.0329 (4)
H1A	0.768063	0.436672	0.367158	0.039*
C2A	0.75763 (9)	0.3549 (4)	0.58118 (18)	0.0326 (4)
H2A	0.741273	0.179814	0.561992	0.039*
C3A	0.65856 (10)	0.4782 (4)	0.38426 (18)	0.0343 (4)
C4A	0.63715 (12)	0.2766 (5)	0.2934 (2)	0.0458 (5)
H4A	0.672227	0.164185	0.265603	0.055*
C5A	0.56362 (13)	0.2416 (5)	0.2438 (3)	0.0547 (6)
H5A	0.549539	0.104493	0.184080	0.066*
C6A	0.51174 (12)	0.4086 (5)	0.2827 (3)	0.0562 (6)
H6A	0.462589	0.385662	0.248470	0.067*
C7A	0.53231 (12)	0.6099 (6)	0.3721 (3)	0.0578 (6)
H7A	0.497042	0.722986	0.398421	0.069*
C8A	0.60593 (11)	0.6449 (5)	0.4233 (2)	0.0450 (5)
H8A	0.619641	0.781071	0.484053	0.054*
C9A	0.72103 (9)	0.4489 (4)	0.70990 (17)	0.0300 (4)
C10A	0.66288 (10)	0.3106 (4)	0.7532 (2)	0.0403 (4)

H10A	0.646344	0.166838	0.701394	0.048*
C11A	0.62902 (13)	0.3850 (6)	0.8737 (2)	0.0551 (6)
H11A	0.589386	0.292688	0.900760	0.066*
C12A	0.65359 (13)	0.5929 (6)	0.9524 (2)	0.0541 (6)
H12A	0.631510	0.639855	1.034136	0.065*
C13A	0.71124 (14)	0.7326 (5)	0.9101 (2)	0.0503 (5)
H13A	0.728153	0.873940	0.963729	0.060*
C14A	0.74423 (11)	0.6637 (4)	0.7880 (2)	0.0397 (4)
H14A	0.782096	0.762143	0.758481	0.048*
N1A	0.83958 (9)	0.3495 (5)	0.6135 (2)	0.0445 (4)
O1A	0.76176 (8)	0.7615 (3)	0.46671 (15)	0.0392 (3)
H1A1	0.8579 (18)	0.240 (8)	0.709 (3)	0.087 (11)*
H1A2	0.8600 (15)	0.288 (7)	0.530 (3)	0.067 (9)*
H1A3	0.8561 (15)	0.501 (7)	0.638 (3)	0.053 (8)*
H1O1	0.7784 (15)	0.818 (7)	0.387 (3)	0.062 (8)*
O1C	0.94824 (9)	0.6688 (3)	0.49070 (19)	0.0476 (4)
H1OB	0.9297 (17)	0.820 (9)	0.453 (3)	0.077 (10)*
H1OA	0.991 (2)	0.707 (9)	0.543 (4)	0.090 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1B	0.0317 (7)	0.0269 (8)	0.0277 (7)	−0.0005 (7)	0.0073 (6)	0.0007 (7)
C2B	0.0292 (7)	0.0260 (8)	0.0297 (7)	0.0032 (7)	0.0054 (6)	0.0030 (7)
C3B	0.0374 (8)	0.0338 (10)	0.0280 (7)	0.0067 (8)	0.0025 (6)	0.0061 (7)
C4B	0.0312 (7)	0.0305 (9)	0.0318 (7)	−0.0023 (7)	0.0075 (6)	0.0018 (7)
N1B	0.0367 (8)	0.0300 (8)	0.0367 (8)	−0.0035 (7)	0.0050 (6)	0.0064 (7)
O1B	0.0440 (7)	0.0328 (7)	0.0312 (6)	−0.0031 (6)	−0.0005 (5)	0.0062 (6)
O2B	0.0437 (7)	0.0411 (8)	0.0387 (6)	−0.0143 (7)	0.0061 (5)	0.0035 (6)
O3B	0.0425 (7)	0.0308 (7)	0.0406 (7)	0.0073 (6)	0.0068 (5)	0.0072 (6)
O4B	0.0458 (7)	0.0528 (10)	0.0328 (6)	0.0030 (7)	0.0054 (5)	−0.0066 (6)
C1A	0.0359 (9)	0.0326 (10)	0.0310 (8)	0.0018 (8)	0.0077 (6)	0.0030 (8)
C2A	0.0317 (8)	0.0334 (9)	0.0330 (8)	0.0023 (8)	0.0041 (6)	0.0039 (8)
C3A	0.0389 (9)	0.0338 (9)	0.0300 (7)	0.0022 (8)	0.0028 (6)	0.0045 (8)
C4A	0.0485 (11)	0.0399 (12)	0.0479 (11)	0.0043 (10)	0.0003 (8)	−0.0053 (10)
C5A	0.0568 (12)	0.0483 (13)	0.0561 (12)	−0.0057 (12)	−0.0075 (10)	−0.0066 (11)
C6A	0.0409 (11)	0.0594 (17)	0.0655 (14)	−0.0038 (11)	−0.0074 (10)	0.0067 (12)
C7A	0.0395 (10)	0.0593 (15)	0.0733 (15)	0.0108 (12)	−0.0004 (10)	−0.0058 (13)
C8A	0.0412 (10)	0.0426 (11)	0.0506 (11)	0.0057 (10)	0.0015 (8)	−0.0067 (10)
C9A	0.0299 (8)	0.0297 (9)	0.0298 (8)	0.0018 (7)	0.0013 (6)	0.0039 (7)
C10A	0.0378 (9)	0.0414 (11)	0.0427 (10)	−0.0083 (9)	0.0083 (7)	−0.0013 (9)
C11A	0.0513 (12)	0.0670 (17)	0.0506 (12)	−0.0069 (12)	0.0213 (9)	0.0014 (12)
C12A	0.0658 (13)	0.0590 (16)	0.0399 (10)	0.0156 (13)	0.0171 (9)	−0.0001 (11)
C13A	0.0746 (14)	0.0369 (11)	0.0380 (10)	0.0047 (11)	−0.0003 (9)	−0.0051 (9)
C14A	0.0471 (10)	0.0332 (10)	0.0382 (9)	−0.0060 (9)	0.0016 (7)	0.0010 (9)
N1A	0.0338 (8)	0.0613 (13)	0.0392 (9)	0.0097 (9)	0.0076 (6)	0.0119 (9)
O1A	0.0455 (7)	0.0349 (7)	0.0378 (7)	−0.0054 (6)	0.0065 (5)	0.0053 (6)
O1C	0.0472 (8)	0.0297 (8)	0.0625 (9)	−0.0008 (6)	−0.0098 (7)	−0.0011 (7)

Geometric parameters (Å, º)

C1B—O2B	1.239 (2)	C5A—C6A	1.370 (4)
C1B—O1B	1.265 (2)	C5A—H5A	0.9300
C1B—C2B	1.536 (2)	C6A—C7A	1.374 (4)
C2B—N1B	1.488 (2)	C6A—H6A	0.9300
C2B—C3B	1.511 (2)	C7A—C8A	1.393 (3)
C2B—H2B	0.9800	C7A—H7A	0.9300
C3B—C4B	1.522 (3)	C8A—H8A	0.9300
C3B—H3B1	0.9700	C9A—C10A	1.384 (3)
C3B—H3B2	0.9700	C9A—C14A	1.386 (3)
C4B—O3B	1.246 (2)	C10A—C11A	1.391 (3)
C4B—O4B	1.263 (2)	C10A—H10A	0.9300
N1B—H1B1	0.92 (4)	C11A—C12A	1.365 (4)
N1B—H1B2	0.96 (2)	C11A—H11A	0.9300
N1B—H1B3	0.87 (3)	C12A—C13A	1.377 (4)
C1A—O1A	1.418 (2)	C12A—H12A	0.9300
C1A—C3A	1.516 (3)	C13A—C14A	1.387 (3)
C1A—C2A	1.539 (2)	C13A—H13A	0.9300
C1A—H1A	0.9800	C14A—H14A	0.9300
C2A—N1A	1.499 (2)	N1A—H1A1	1.08 (4)
C2A—C9A	1.513 (2)	N1A—H1A2	0.95 (3)
C2A—H2A	0.9800	N1A—H1A3	0.87 (4)
C3A—C8A	1.380 (3)	O1A—H1O1	0.88 (3)
C3A—C4A	1.386 (3)	O1C—H1OB	0.92 (4)
C4A—C5A	1.388 (3)	O1C—H1OA	0.90 (4)
C4A—H4A	0.9300		

O2B—C1B—O1B	125.99 (17)	C3A—C4A—H4A	119.8
O2B—C1B—C2B	117.27 (14)	C5A—C4A—H4A	119.8
O1B—C1B—C2B	116.47 (15)	C6A—C5A—C4A	120.2 (2)
N1B—C2B—C3B	110.56 (14)	C6A—C5A—H5A	119.9
N1B—C2B—C1B	110.74 (13)	C4A—C5A—H5A	119.9
C3B—C2B—C1B	114.48 (15)	C5A—C6A—C7A	120.0 (2)
N1B—C2B—H2B	106.9	C5A—C6A—H6A	120.0
C3B—C2B—H2B	106.9	C7A—C6A—H6A	120.0
C1B—C2B—H2B	106.9	C6A—C7A—C8A	120.1 (2)
C2B—C3B—C4B	115.70 (14)	C6A—C7A—H7A	119.9
C2B—C3B—H3B1	108.4	C8A—C7A—H7A	119.9
C4B—C3B—H3B1	108.4	C3A—C8A—C7A	120.2 (2)
C2B—C3B—H3B2	108.4	C3A—C8A—H8A	119.9
C4B—C3B—H3B2	108.4	C7A—C8A—H8A	119.9
H3B1—C3B—H3B2	107.4	C10A—C9A—C14A	118.70 (17)
O3B—C4B—O4B	125.39 (18)	C10A—C9A—C2A	118.39 (18)
O3B—C4B—C3B	119.12 (15)	C14A—C9A—C2A	122.89 (16)
O4B—C4B—C3B	115.47 (16)	C9A—C10A—C11A	120.5 (2)
C2B—N1B—H1B1	109.3 (17)	C9A—C10A—H10A	119.8
C2B—N1B—H1B2	111.4 (14)	C11A—C10A—H10A	119.8

H1B1—N1B—H1B2	114 (3)	C12A—C11A—C10A	120.4 (2)
C2B—N1B—H1B3	119 (2)	C12A—C11A—H11A	119.8
H1B1—N1B—H1B3	96 (3)	C10A—C11A—H11A	119.8
H1B2—N1B—H1B3	107 (2)	C11A—C12A—C13A	119.7 (2)
O1A—C1A—C3A	114.28 (16)	C11A—C12A—H12A	120.2
O1A—C1A—C2A	108.10 (15)	C13A—C12A—H12A	120.2
C3A—C1A—C2A	111.15 (14)	C12A—C13A—C14A	120.4 (2)
O1A—C1A—H1A	107.7	C12A—C13A—H13A	119.8
C3A—C1A—H1A	107.7	C14A—C13A—H13A	119.8
C2A—C1A—H1A	107.7	C9A—C14A—C13A	120.3 (2)
N1A—C2A—C9A	111.47 (15)	C9A—C14A—H14A	119.8
N1A—C2A—C1A	107.92 (15)	C13A—C14A—H14A	119.8
C9A—C2A—C1A	114.98 (16)	C2A—N1A—H1A1	113.1 (18)
N1A—C2A—H2A	107.4	C2A—N1A—H1A2	108.5 (16)
C9A—C2A—H2A	107.4	H1A1—N1A—H1A2	111 (3)
C1A—C2A—H2A	107.4	C2A—N1A—H1A3	110.3 (18)
C8A—C3A—C4A	119.11 (18)	H1A1—N1A—H1A3	102 (3)
C8A—C3A—C1A	121.73 (18)	H1A2—N1A—H1A3	112 (3)
C4A—C3A—C1A	119.16 (18)	C1A—O1A—H1O1	107 (2)
C3A—C4A—C5A	120.4 (2)	H1OB—O1C—H1OA	106 (4)

O1A—C1A—C2A—N1A	−65.0 (2)	C5A—C6A—C7A—C8A	0.1 (4)
O1A—C1A—C2A—C9A	60.14 (19)	C6A—C7A—C8A—C3A	0.2 (4)
C3A—C1A—C2A—N1A	168.86 (17)	C2A—C9A—C10A—C11A	177.95 (19)
C3A—C1A—C2A—C9A	−66.1 (2)	C14A—C9A—C10A—C11A	−0.6 (3)
O1A—C1A—C3A—C4A	149.76 (18)	C2A—C9A—C14A—C13A	−176.29 (19)
O1A—C1A—C3A—C8A	−31.4 (2)	C10A—C9A—C14A—C13A	2.2 (3)
C2A—C1A—C3A—C4A	−87.6 (2)	C9A—C10A—C11A—C12A	−1.2 (3)
C2A—C1A—C3A—C8A	91.3 (2)	C10A—C11A—C12A—C13A	1.4 (4)
N1A—C2A—C9A—C10A	−131.49 (19)	C11A—C12A—C13A—C14A	0.2 (4)
N1A—C2A—C9A—C14A	47.0 (3)	C12A—C13A—C14A—C9A	−2.0 (3)
C1A—C2A—C9A—C10A	105.3 (2)	O1B—C1B—C2B—N1B	17.4 (2)
C1A—C2A—C9A—C14A	−76.2 (2)	O1B—C1B—C2B—C3B	143.24 (16)
C1A—C3A—C4A—C5A	178.1 (2)	O2B—C1B—C2B—N1B	−168.28 (16)
C8A—C3A—C4A—C5A	−0.7 (3)	O2B—C1B—C2B—C3B	−42.5 (2)
C1A—C3A—C8A—C7A	−178.7 (2)	N1B—C2B—C3B—C4B	73.0 (2)
C4A—C3A—C8A—C7A	0.2 (3)	C1B—C2B—C3B—C4B	−53.0 (2)
C3A—C4A—C5A—C6A	1.0 (4)	C2B—C3B—C4B—O3B	1.4 (3)
C4A—C5A—C6A—C7A	−0.6 (4)	C2B—C3B—C4B—O4B	−177.08 (16)

Hydrogen-bond geometry (Å, º)

CgB is the centroid of phenyl ring *B* (C9–C14).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*B*—H1*B*1···O3*B*^{i}	0.92 (4)	1.92 (4)	2.819 (2)	168 (3)
N1*B*—H1*B*3···O3*B*^{ii}	0.87 (3)	2.46 (3)	3.112 (2)	132 (3)
N1*B*—H1*B*3···O4*B*^{ii}	0.87 (3)	2.20 (3)	2.868 (2)	134 (3)
O1*A*—H1*O*1···O2*B*^{i}	0.88 (3)	1.88 (3)	2.752 (2)	171 (3)

N1*A*—H1*A*1···O4*B*^{iii}	1.08 (4)	1.70 (4)	2.742 (3)	162 (3)
N1*A*—H1*A*2···O1*B*	0.95 (3)	1.92 (3)	2.862 (2)	173 (3)
O1*C*—H1*OB*···O1*B*^{i}	0.92 (4)	1.82 (4)	2.734 (2)	173 (3)
O1*C*—H1*OA*···O1*B*^{iv}	0.90 (4)	2.01 (4)	2.840 (2)	153 (4)
N1*A*—H1*A*3···O1*C*	0.87 (4)	2.45 (3)	2.926 (3)	115 (2)
N1*B*—H1*B*2···O1*C*	0.96 (2)	2.01 (3)	2.938 (2)	163 (2)
C2*A*—H2*A*···O1*A*^{v}	0.98	2.42	3.304 (3)	150
C14*A*—H14*A*···O4*B*^{vi}	0.93	2.53	3.371 (3)	150
C3*B*—H3*B*2···CgB^{iii}	0.97	2.87	3.6127 (16)	134

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