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Crystal structure of 4-[(3-methoxy-2-oxidobenzylidene)azaniumyl]benzoic acid methanol monosolvate

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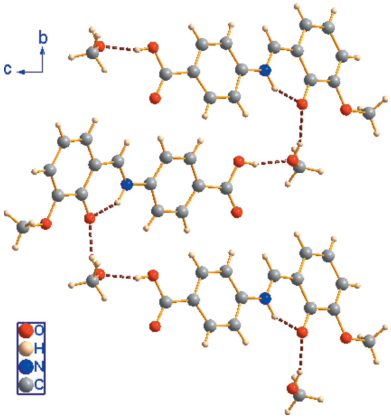
In the crystal of the title compound, $C_{15}H_{13}NO_4\cdot CH_3OH$, the Schiff base molecule exists in the zwitterionic form; an intramolecular N—H···O hydrogen bond stabilizes the molecular structure. The benzene rings are nearly co-planar, subtending a dihedral angle of 5.34 (2)°. In the crystal, classical O—H···O and weak C—H···O hydrogen bonds link the Schiff base molecules and methanol solvent molecules into a three-dimensional supramolecular architecture. The crystal studied was refined as an inversion twin.

1. Chemical context

Vanillin and *o*-vanillin are natural compounds that have both a phenolic OH and an aldehyde group. They are positional isomers, in which *o*-vanillin shows contradictory effects. There are several reports indicating that *o*-vanillin induces mutations and it has also been found to enhance chromosomal aberrations in *in vitro* systems (Barik *et al.*, 2004; Takahashi *et al.*, 1989). Vanillin is also the primary component of the extract of the vanilla bean. Synthetic vanillin rather than natural vanilla extract is now more often used as a flavouring agent in foods, beverages and pharmaceuticals. Schiff bases containing *o*-vanillin possess antifungal and antibacterial properties (Thorat *et al.*, 2012). 4-Aminobenzoic acid (PABA) is an important biological molecule, being an essential bacterial cofactor involved in the synthesis of folic acid (Robinson, 1966). PABA shows polymorphism and so far four polymorphs of PABA are known, all of which are centrosymmetric; a non-centrosymmetric polymorph of 4-aminobenzoic acid has also been reported (Benali-Cherif *et al.*, 2014). Schiff bases derived from 2-hydroxy-3-methoxybenzaldehyde (*o*-vanillin) and PABA have not been investigated so thoroughly. Our research interest focuses on the study of Schiff bases derived from salicylaldehyde. It is well known that Schiff bases of salicylaldehyde derivatives may exhibit thermochromism or photochromism, depending on the planarity or non-planarity of the molecule (Cohen & Schmidt, 1964; Amimoto & Kawato, 2005). Schiff bases often exhibit various biological activities and in many cases have been shown to possess antibacterial, anticancer, anti-inflammatory and antitoxic properties (Lozier *et al.*, 1975). They are used as anion sensors (Dalapati *et al.*, 2011), as non-linear optical compounds (Sun *et al.*, 2012) and as versatile polynuclear ligands for multinuclear magnetic exchange clusters (Moroz *et al.*, 2012). New salicylaldehyde-



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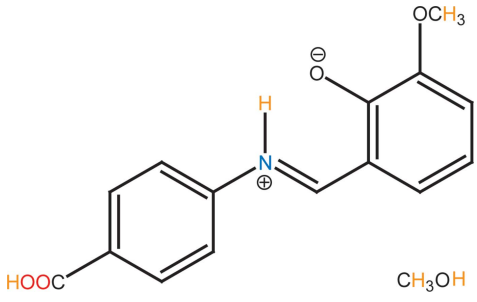
based Schiff bases have also been synthesized and reported (Faizi *et al.*, 2015*a*,*b*; 2016*b*; 2017*a*,*b*,*c*). The present work is a part of an ongoing structural study of Schiff bases and their utilization in the synthesis of new organic, excited state proton-transfer compounds and fluorescent chemosensors (Faizi *et al.*, 2016*a*; Faizi *et al.*, 2018; Kumar *et al.*, 2018; Mukherjee *et al.*, 2018). We report herein the crystal structure of the title compound synthesized by the condensation reaction of 2-hydroxy-3-methoxybenzaldehyde and PABA.



2. Structural commentary

The asymmetric unit of the title compound contains a Schiff base molecule and a methanol molecule of crystallization. In the solid state, the Schiff base molecule (Fig. 1) exists in the zwitterionic form. An intramolecular N—H···O hydrogen bond stabilizes the molecular structure (Table 1). The imine group, which displays a C9—C8—N1—C5 torsion angle of 177.6 (3)°, contributes to the general planarity of the molecule. The Schiff base molecule displays a *trans* configuration with respect to the C=N and C–N bonds. The vanillin ring (C9–C14) is inclined to the central benzene ring (C2–C7) by 5.34 (2)°. A similar value of 5.3 (2)° is observed in 4-chloro-*N*′-(2-hydroxy-4-methoxybenzylidene)benzohydrazide methanol monosolvate (Zhi *et al.*, 2011). All bond lengths are in normal ranges. The O4—C15 bond length is 1.432 (2) Å and similar value of 1.432 (2) Å is observed in (*E*)-2-hydroxy-3-methoxy-5-[(3-methoxyphenyl)diazenyl]benzaldehyde (Karadayı *et al.*, 2006). The methoxy group of the 2-hydroxy-3-methoxyphenyl is almost coplanar with its bound benzene ring, as seen by the C_{methyl}—O—C—C torsion angle of 178.1 (2)°.

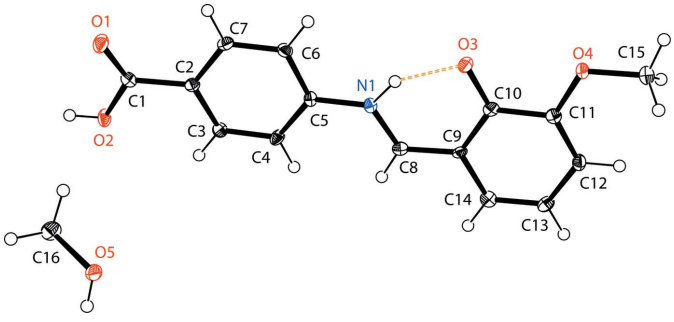


Figure 1
The molecular structure of the title compound, showing the atom labelling and the intramolecular N—H···O hydrogen bond as a dashed line. Displacement ellipsoids are drawn at the 40% probability level.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.86	1.87	2.568 (4)	138
O2—H2···O5^{i}	0.82	1.80	2.598 (4)	164
O5—H5*O*···O3^{ii}	0.96 (5)	1.77 (5)	2.690 (4)	159 (4)
C7—H7···O2^{i}	0.93	2.56	3.233 (5)	130
C8—H8···O1^{iii}	0.93	2.41	3.281 (5)	155

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

3. Supramolecular features

In the crystal, the hydroxyl group of the methanol solvent molecule is linked to the carboxylate group of the neighboring Schiff base molecule and the deprotonated hydroxyl group of the other Schiff base molecule via classical O—H···O hydrogen bonds, forming supramolecular chains propagating along the *b*-axis direction (Fig. 2). Weak C—H···O hydrogen bonds further link the chains into a three-dimensional supramolecular architecture.

4. Database survey

A search of the Cambridge Structural Database (CSD version 5.39, February 2018 update; Groom *et al.*, 2016) for similar systems (benzylidene-phenyl-amine) yielded 285 hits of which ten are similar substituted benzylidene-phenyl-amines: *N*-salicylidene-*p*-chloroaniline (I) (BADDAL01; Kamwaya &

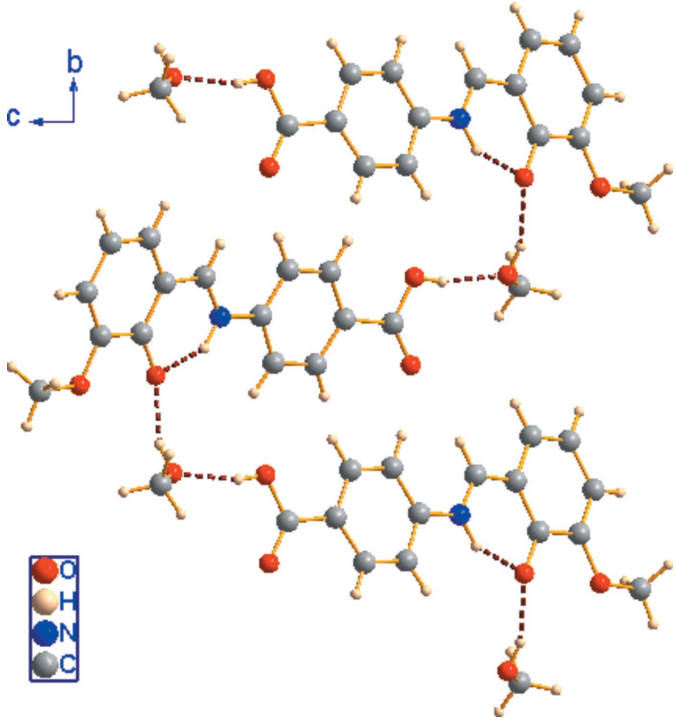


Figure 2
A view of the hydrogen-bonded chain extending along the *b*-axis direction. Hydrogen bonds are shown as dashed lines.

Khoo, 1985), 5-{[(1*E*)-(2-hydroxyphenyl)methylene]amino}-2-hydroxybenzoic acid (II) (CAWJOA; Bourque *et al.*, 2005), 2-(2-hydroxy-5-methylbenzylideneammonio)benzoate (III) (CEXNEZ; Gayathri *et al.*, 2007), *N*,*N*′-bis(2-hydroxy-1-naphthaldimine)-*o*-phenylenediamine methanol solvate (IV) (GETXEJ; Eltayeb *et al.*, 2007), *o*-(salicylideneaminium)-phenol chloride (V) (HALGUW; Ondrácek *et al.*, 1993), *N*-(2-carboxyphenyl)salicylidenimine (VI) (JUTKAK; Ligtenbarg *et al.*, 1999), diisothiocyantotriphenyltin bis[1-(salicyclideneimino)-2-methoxybenzene] (VII) (KIDYOL; Charland *et al.*, 1989), *N*-(2-oxyphenyl)-3-methoxysalicylaldimine (VIII) (NEDMUF; Kannappan *et al.*, 2006), *N*-(5-chloro-2-oxidobenzylidene)-2-hydroxy-5-methylanilinium (IX) (QIKHEX; Elmali *et al.*, 2001) and *N*-(5-chloro-2-hydroxybenzylidene)-4-hydroxyaniline (X) (SAQTOT; Ogawa *et al.*, 1998), 2-[(*E*)-(2-[{(*E*)-2,3-dihydroxybenzylidene]amino}-5-methylphenyl)iminiomethyl]-6-hydroxyphenolate (XI) (HUCQEC; Eltayeb *et al.*, 2009) (see Fig. 3). The dihedral angle between the benzene rings in the title compound [5.34 (2)°] is smaller than those in compounds (III) [5.6 (1)°] (IV [5.84 (9)°], (V) [7.3 (1)°] and (IX) [9.51 (6)°] and (XI) [17.36 (12)°]. In compound (VII), cationic protonated pairs co-crystallize with five-coordinate organotin anions. In the title compound, they form an intramolecular *S*6 ring motif and stabilized by N—H···O hydrogen bonds.

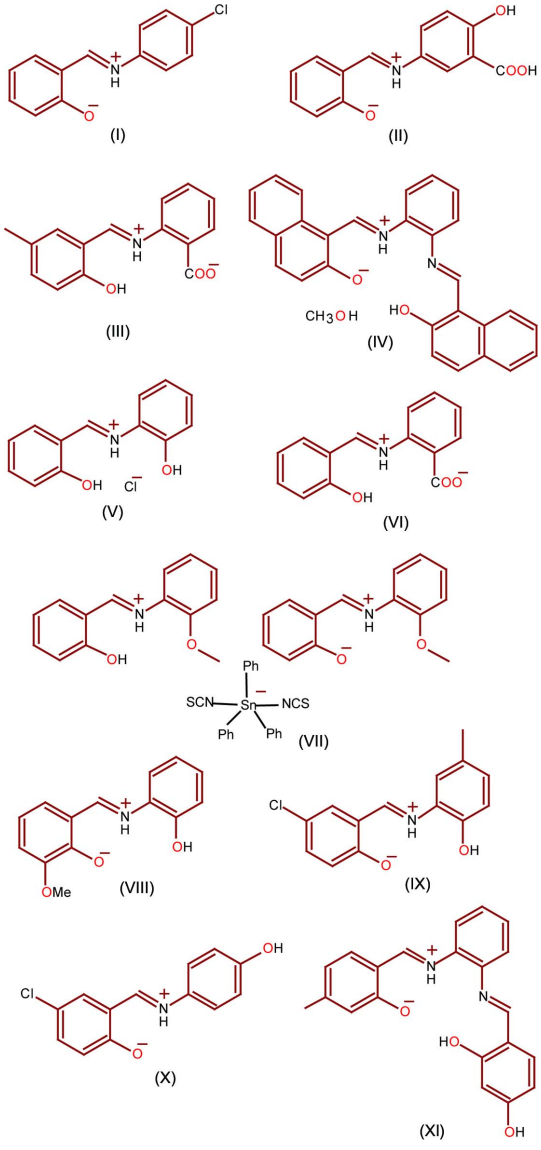


Figure 3
Zwitterionic forms of some closely related compounds.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{13}NO_4·CH_4O$
M_r	303.30
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, b, c (Å)	4.6993 (5), 10.038 (1), 30.155 (3)
V (Å^3)	1422.5 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.61 × 0.36 × 0.17
Data collection	
Diffractometer	Stoe IPDS 2
Absorption correction	Integration (*X-RED32*; Stoe & Cie, 2002)
T_{min}, T_{max}	0.963, 0.988
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17046, 2526, 2117
R_{int}	0.095
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.596
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.112, 1.08
No. of reflections	2526
No. of parameters	206
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.26
Absolute structure	Refined as a perfect inversion twin.
Absolute structure parameter	0.5

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 2002), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2017* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

5. Synthesis and crystallization

To a hot stirred solution of 4-aminobenzoic acid (PABA) (1.00 g, 7.2 mmol) in methanol (15 ml) was added vanillin (1.11 g, 7.2 mmol)). The resulting mixture was then heated under reflux. After an hour, a precipitate formed. The reaction mixture was heated for about another 30 min until the completion of the reaction, which was monitored by TLC. The reaction mixture was cooled to room temperature, filtered and washed with hot methanol. It was then dried under vacuum to give the pure compound in 78% yield. Prismatic colourless single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of a methanol solution.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The N—H and O–H atoms were

located in a difference-Fourier map. Their positional and isotropic thermal parameters were included in further stages of the refinement. All C-bound H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.97 Å and with U_{iso}(H) = 1.2–1.5U_{eq}(C).

Acknowledgements

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References

Amimoto, K. & Kawato, T. (2005). *J. Photochem. Photobiol. Photochem. Rev.* **6**, 207–226.

Barik, A., Priyadarsini, K. I. & Mohan, H. (2004). *Radiat. Phys. Chem.* **70**, 687–696.

Benali-Cherif, R., Takouachet, R., Bendeif, E.-E. & Benali-Cherif, N. (2014). *Acta Cryst.* C**70**, 323–325.

Bourque, T. A., Nelles, M. E., Gullon, T. J., Garon, C. N., Ringer, M. K., Leger, L. J., Mason, J. W., Wheaton, S. L., Baerlocher, F. J., Vogels, C. M., Decken, A. & Westcott, S. A. (2005). *Can. J. Chem.* **83**, 1063–1070.

Charland, J.-P., Gabe, E. J., Khoo, L. E. & Smith, F. E. (1989). *Polyhedron*, **8**, 1897–1901.

Cohen, M. D. & Schmidt, G. M. J. (1964). *J. Chem. Soc.* pp. 1996–2000.

Dalapati, S., Alam, M. A., Jana, S. & Guchhait, N. (2011). *J. Fluor. Chem.* **132**, 536–540.

Elmali, A., Elerman, Y. & Svoboda, I. (2001). *Acta Cryst.* C**57**, 485–486.

Eltayeb, N. E., Teoh, S. G., Teh, J. B.-J., Fun, H.-K. & Ibrahim, K. (2007). *Acta Cryst.* E**63**, o117–o119.

Eltayeb, N. E., Teoh, S. G., Yeap, C. S., Fun, H.-K. & Adnan, R. (2009). *Acta Cryst.* E**65**, o2065–o2066.

Faizi, M. S. H., Ahmad, M., Kapshuk, A. A. & Golenya, I. A. (2017*a*). *Acta Cryst.* E**73**, 38–40.

Faizi, M. S. H., Alam, M. J., Haque, A., Ahmad, S., Shahid, M. & Ahmad, M. (2018). *J. Mol. Struct.* **1156**, 457–464.

Faizi, M. S. H., Ali, A. & Potaskalov, V. A. (2016*a*). *Acta Cryst.* E**72**, 1366–1369.

Faizi, M. S. H., Dege, N., Haque, A., Kalibabchuk, V. A. & Cemberci, M. (2017*b*). *Acta Cryst.* E**73**, 96–98.

Faizi, M. S. H., Gupta, S., Mohan, V. K., Jain, K. V. & Sen, P. (2016*b*). *Sens. Actuators B Chem.* **222**, 15–20.

Faizi, M. S. H., Haque, A. & Kalibabchuk, V. A. (2017*c*). *Acta Cryst.* E**73**, 112–114.

Faizi, M. S. H., Iskenderov, T. S. & Sharkina, N. O. (2015*b*). *Acta Cryst.* E**71**, 28–30.

Faizi, M. S. H., Ohui, K. A. & Golenya, I. A. (2015*a*). *Acta Cryst.* E**71**, 1433–1435.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Gayathri, D., Velmurugan, D., Ravikumar, K., Devaraj, S. & Kandaswamy, M. (2007). *Acta Cryst.* E**63**, o849–o851.

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

Kamwaya, M. E. & Khoo, L. E. (1985). *J. Fiz. Malays.* **6**, 135–140.

Kannappan, R., Tooke, D. M., Spek, A. L. & Reedijk, J. (2006). *Inorg. Chim. Acta*, **359**, 334–338.

Karadayı, N., Albayrak, Ç., Odabaşoğlu, M. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o1727–o1729.

Kumar, M., Kumar, A., Faizi, M. S. H., Kumar, S., Singh, M. K., Sahu, S. K., Kishor, S. & John, R. P. (2018). *Sens. Actuators B Chem.* **260**, 888–899.

Ligtenbarg, A. G. J., Hage, R., Meetsma, A. & Feringa, B. L. (1999). *J. Chem. Soc. Perkin Trans. 2*, pp. 807–812.

Lozier, R. H., Bogomolni, R. A. & Stoeckenius, W. (1975). *Biophys. J.* **15**, 955–962.

Moroz, Y. S., Demeshko, S., Haukka, M., Mokhir, A., Mitra, U., Stocker, M., Müller, P., Meyer, F. & Fritsky, I. O. (2012). *Inorg. Chem.* **51**, 7445–7447.

Mukherjee, P., Das, A., Faizi, M. S. H. & Sen, P. (2018). *ChemistrySelect*, **3**, 3787–3796.

Ogawa, K., Kasahara, Y., Ohtani, Y. & Harada, J. (1998). *J. Am. Chem. Soc.* **120**, 7107–7108.

Ondráček, J., Kovářová, Z., Maixner, J. & Jursık, F. (1993). *Acta Cryst.* C**49**, 1948–1949.

Robinson, F. A. (1966). *The Vitamin Co-factors of Enzyme Systems*, pp. 541–662 London: Pergamon.

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

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

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

Sun, Y., Wang, Y., Liu, Z., Huang, C. & Yu, C. (2012). *Spectrochim. Acta Part A*, **96**, 42–50.

Takahashi, K., Sekiguchi, M. & Kawazoe, Y. (1989). *Biochem. Biophys. Res. Commun.* **162**, 1376–1381.

Thorat, B. R., Mandewale, M., Shelke, S., Kamat, P., Atram, R. G., Bhalerao, M. & Yamgar, R. (2012). *J. Chem. Pharm. Res.* **4**, 14–17.

Zhi, F., Wang, R., Zhang, Y., Wang, Q. & Yang, Y.-L. (2011). *Acta Cryst.* E**67**, o2825.

supporting information

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Saima Kamaal, Md. Serajul Haque Faizi, Akram Ali, Musheer Ahmad and Turganbay Iskenderov

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

4-[(3-Methoxy-2-oxidobenzylidene)azaniumyl]benzoic acid methanol monosolvate

Crystal data

$C_{15}H_{13}NO_4 \cdot CH_4O$
$M_r = 303.30$
Orthorhombic, $P2_12_12_1$
$a = 4.6993$ (5) Å
$b = 10.038$ (1) Å
$c = 30.155$ (3) Å
$V = 1422.5$ (3) Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.416$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8708 reflections
$\theta = 2.4$–$29.9°$
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Prism, colorless
0.61 × 0.36 × 0.17 mm

Data collection

STOE IPDS 2 diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)
$T_{min} = 0.963$, $T_{max} = 0.988$
17046 measured reflections
2526 independent reflections
2117 reflections with $I > 2\sigma(I)$
$R_{int} = 0.095$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.7°$
$h = -5 \rightarrow 5$
$k = -11 \rightarrow 11$
$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.112$
$S = 1.08$
2526 reflections
206 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.7153P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Absolute structure: Refined as a perfect inversion twin.
Absolute structure parameter: 0.5

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 inversion twin

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.9825 (8)	0.5743 (4)	0.20579 (12)	0.0150 (8)
C2	0.8025 (8)	0.5849 (4)	0.24609 (12)	0.0135 (8)
C3	0.6780 (8)	0.7039 (4)	0.25889 (12)	0.0156 (9)
H3	0.723443	0.781513	0.243631	0.019*
C4	0.4888 (8)	0.7099 (4)	0.29364 (12)	0.0168 (9)
H4	0.407880	0.790869	0.301780	0.020*
C5	0.4194 (8)	0.5940 (4)	0.31650 (11)	0.0117 (8)
C6	0.5553 (8)	0.4750 (4)	0.30576 (12)	0.0161 (8)
H6	0.519516	0.398578	0.322296	0.019*
C7	0.7427 (8)	0.4703 (4)	0.27069 (12)	0.0163 (9)
H7	0.830172	0.390229	0.263318	0.020*
C8	0.0577 (8)	0.6914 (4)	0.36404 (12)	0.0137 (8)
H8	0.082942	0.774555	0.351007	0.016*
C9	−0.1484 (8)	0.6767 (4)	0.39755 (12)	0.0133 (8)
C10	−0.1987 (8)	0.5486 (4)	0.41710 (12)	0.0134 (8)
C11	−0.4107 (8)	0.5431 (4)	0.45140 (12)	0.0149 (9)
C12	−0.5604 (9)	0.6534 (4)	0.46335 (12)	0.0162 (8)
H12	−0.697197	0.646746	0.485520	0.019*
C13	−0.5121 (8)	0.7778 (4)	0.44273 (12)	0.0175 (9)
H13	−0.618318	0.851778	0.451168	0.021*
C14	−0.3108 (8)	0.7897 (4)	0.41061 (12)	0.0162 (9)
H14	−0.279196	0.871695	0.397153	0.019*
C15	−0.6548 (9)	0.4036 (4)	0.50242 (13)	0.0216 (10)
H15A	−0.838233	0.422645	0.489935	0.032*
H15B	−0.617086	0.464455	0.526284	0.032*
H15C	−0.652337	0.313983	0.513495	0.032*
C16	0.4392 (9)	1.1681 (4)	0.40083 (13)	0.0226 (9)
H16A	0.377953	1.093074	0.383559	0.034*
H16B	0.323804	1.244117	0.393731	0.034*
H16C	0.420226	1.147815	0.431800	0.034*
N1	0.2152 (6)	0.5912 (3)	0.35065 (10)	0.0128 (7)
H1	0.191331	0.516474	0.364068	0.015*
O1	1.0635 (6)	0.4688 (3)	0.19035 (9)	0.0244 (7)
O2	1.0413 (6)	0.6916 (2)	0.18743 (8)	0.0185 (6)
H2	1.128598	0.680031	0.164203	0.028*
O3	−0.0617 (6)	0.4431 (2)	0.40503 (8)	0.0159 (6)
O4	−0.4410 (6)	0.4183 (3)	0.46896 (8)	0.0185 (6)
O5	0.7301 (6)	1.1970 (3)	0.39101 (9)	0.0192 (6)

H5O	0.795 (11)	1.280 (5)	0.4034 (16)	0.049 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.014 (2)	0.016 (2)	0.0152 (19)	−0.0037 (18)	−0.0028 (17)	0.0022 (16)
C2	0.0111 (19)	0.014 (2)	0.0151 (19)	0.0001 (18)	−0.0019 (16)	−0.0005 (16)
C3	0.016 (2)	0.012 (2)	0.0190 (19)	−0.0024 (18)	0.0022 (17)	0.0026 (16)
C4	0.016 (2)	0.0114 (19)	0.022 (2)	0.0007 (18)	0.0053 (18)	−0.0034 (16)
C5	0.0084 (17)	0.016 (2)	0.0110 (18)	−0.0041 (17)	−0.0013 (16)	−0.0014 (15)
C6	0.0147 (19)	0.015 (2)	0.018 (2)	0.0004 (17)	0.0007 (18)	0.0051 (16)
C7	0.017 (2)	0.014 (2)	0.018 (2)	0.0037 (18)	−0.0006 (18)	−0.0011 (16)
C8	0.0125 (18)	0.0132 (19)	0.0155 (19)	−0.0008 (18)	−0.0037 (16)	−0.0013 (15)
C9	0.0106 (18)	0.015 (2)	0.0145 (19)	0.0034 (16)	−0.0026 (16)	−0.0007 (16)
C10	0.0091 (18)	0.018 (2)	0.0132 (18)	−0.0011 (16)	−0.0057 (16)	−0.0018 (16)
C11	0.012 (2)	0.018 (2)	0.0147 (19)	−0.0022 (17)	−0.0030 (16)	−0.0003 (16)
C12	0.0139 (19)	0.022 (2)	0.0125 (19)	−0.0008 (18)	0.0011 (17)	−0.0004 (16)
C13	0.013 (2)	0.019 (2)	0.021 (2)	0.0011 (17)	−0.0017 (18)	−0.0046 (16)
C14	0.015 (2)	0.016 (2)	0.0175 (19)	−0.0052 (17)	−0.0032 (17)	−0.0011 (17)
C15	0.019 (2)	0.024 (2)	0.021 (2)	0.000 (2)	0.0052 (18)	0.0036 (18)
C16	0.016 (2)	0.026 (2)	0.025 (2)	0.0012 (19)	−0.0024 (19)	0.0013 (18)
N1	0.0126 (16)	0.0129 (17)	0.0130 (16)	−0.0029 (15)	−0.0009 (14)	0.0015 (13)
O1	0.0321 (17)	0.0155 (15)	0.0256 (15)	0.0024 (13)	0.0129 (14)	−0.0001 (12)
O2	0.0237 (15)	0.0145 (14)	0.0174 (14)	−0.0034 (13)	0.0081 (13)	0.0000 (11)
O3	0.0147 (13)	0.0149 (14)	0.0182 (13)	0.0010 (12)	0.0029 (12)	−0.0012 (11)
O4	0.0172 (13)	0.0185 (14)	0.0197 (14)	0.0012 (13)	0.0071 (12)	0.0053 (12)
O5	0.0163 (14)	0.0205 (15)	0.0209 (14)	−0.0015 (14)	0.0047 (12)	−0.0042 (13)

Geometric parameters (Å, º)

C1—O1	1.217 (4)	C8—C9	1.408 (5)
C1—O2	1.331 (4)	C9—C14	1.422 (5)
C1—C2	1.484 (5)	C9—C10	1.434 (5)
C2—C3	1.384 (5)	C10—O3	1.292 (4)
C2—C7	1.397 (5)	C10—C11	1.437 (5)
C3—C4	1.376 (5)	C11—C12	1.360 (5)
C4—C5	1.392 (5)	C11—O4	1.367 (4)
C5—C6	1.393 (5)	C12—C13	1.414 (5)
C5—N1	1.408 (4)	C13—C14	1.359 (5)
C6—C7	1.377 (5)	C15—O4	1.432 (4)
C8—N1	1.312 (5)	C16—O5	1.429 (5)
O1—C1—O2	123.1 (3)	C8—C9—C14	119.0 (3)
O1—C1—C2	123.6 (3)	C8—C9—C10	120.2 (3)
O2—C1—C2	113.3 (3)	C14—C9—C10	120.8 (3)
C3—C2—C7	118.5 (3)	O3—C10—C9	122.5 (3)
C3—C2—C1	122.1 (3)	O3—C10—C11	121.1 (3)
C7—C2—C1	119.4 (3)	C9—C10—C11	116.4 (3)

C4—C3—C2	121.6 (4)	C12—C11—O4	126.1 (3)
C3—C4—C5	119.4 (3)	C12—C11—C10	121.2 (3)
C4—C5—C6	119.7 (3)	O4—C11—C10	112.7 (3)
C4—C5—N1	122.6 (3)	C11—C12—C13	121.3 (4)
C6—C5—N1	117.8 (3)	C14—C13—C12	120.1 (4)
C7—C6—C5	120.1 (3)	C13—C14—C9	120.1 (4)
C6—C7—C2	120.6 (3)	C8—N1—C5	126.5 (3)
N1—C8—C9	121.9 (3)	C11—O4—C15	116.1 (3)

O1—C1—C2—C3	−169.8 (4)	C8—C9—C10—C11	179.3 (3)
O2—C1—C2—C3	8.6 (5)	C14—C9—C10—C11	−2.6 (5)
O1—C1—C2—C7	6.5 (5)	O3—C10—C11—C12	−178.3 (3)
O2—C1—C2—C7	−175.1 (3)	C9—C10—C11—C12	2.0 (5)
C7—C2—C3—C4	−3.0 (6)	O3—C10—C11—O4	1.1 (5)
C1—C2—C3—C4	173.3 (3)	C9—C10—C11—O4	−178.6 (3)
C2—C3—C4—C5	−0.2 (6)	O4—C11—C12—C13	−179.7 (3)
C3—C4—C5—C6	4.0 (6)	C10—C11—C12—C13	−0.4 (6)
C3—C4—C5—N1	−176.2 (3)	C11—C12—C13—C14	−0.7 (6)
C4—C5—C6—C7	−4.5 (6)	C12—C13—C14—C9	0.2 (5)
N1—C5—C6—C7	175.7 (3)	C8—C9—C14—C13	179.7 (3)
C5—C6—C7—C2	1.2 (6)	C10—C9—C14—C13	1.6 (5)
C3—C2—C7—C6	2.5 (6)	C9—C8—N1—C5	177.6 (3)
C1—C2—C7—C6	−173.9 (3)	C4—C5—N1—C8	3.2 (6)
N1—C8—C9—C14	179.7 (3)	C6—C5—N1—C8	−176.9 (3)
N1—C8—C9—C10	−2.2 (5)	C12—C11—O4—C15	1.2 (5)
C8—C9—C10—O3	−0.4 (5)	C10—C11—O4—C15	−178.1 (3)
C14—C9—C10—O3	177.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.86	1.87	2.568 (4)	138
O2—H2···O5^{i}	0.82	1.80	2.598 (4)	164
O5—H5*O*···O3^{ii}	0.96 (5)	1.77 (5)	2.690 (4)	159 (4)
C7—H7···O2^{i}	0.93	2.56	3.233 (5)	130
C8—H8···O1^{iii}	0.93	2.41	3.281 (5)	155

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