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The crystal structure of (*E*)-2-ethyl-*N*-(4-nitrobenzylidene)aniline: three-dimensional supra-molecular assembly mediated by C—H \cdots O hydrogen bonds and nitro $\cdots\pi$ (arene) interactions

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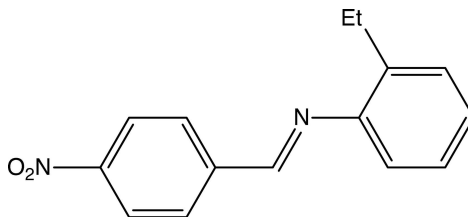
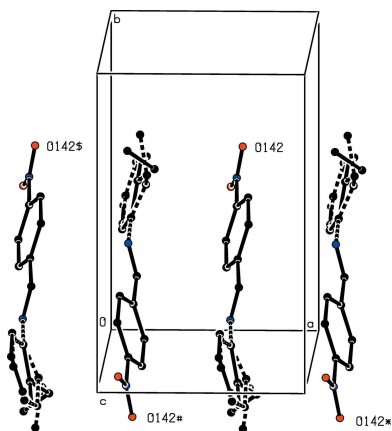
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In the molecule of the title compound, C₁₅H₁₄N₂O₂, the 2-ethylphenyl group is disordered over two sets of atomic sites having occupancies of 0.515 (19) and 0.485 (19), and the dihedral angle between the two partial-occupancy aryl rings is 6(2)°. A combination of C—H \cdots O hydrogen bonds and nitro $\cdots\pi$ (arene) interactions links the molecules into a continuous three-dimensional framework structure. Comparisons are made with the structures of some related compounds.

1. Chemical context

Schiff bases exhibit a very wide range of biological activities (da Silva *et al.*, 2011) and are also of interest because of their photochromic and thermochromic properties (Hadjoudis & Mavridis, 2004; Minkin *et al.*, 2011). In view of the general importance of Schiff bases, and in a continuation of our own structural study of compounds of this type (Girisha *et al.*, 2017, 2018) we report here the molecular and supramolecular structure of (*E*)-2-ethyl-*N*-(4-nitrobenzylidene)aniline (I) (Fig. 1), where the ethyl group turns out to be disordered over two sets of atomic sites and where the molecules are linked into a three-dimensional supramolecular array.



2. Structural commentary

The 2-ethylphenyl group in compound (I) is disordered over two sets of atomic sites having occupancies of 0.515 (19) and 0.485 (19) and it is possible that the ethyl group is simply making full use of an available space within the structure: the dihedral angle between the two components of the disordered aryl ring is 6(2)°. The nitro group is almost coplanar with the

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------|-------|-------------|-------------|---------------|
| $C16-H16\cdots O141^i$ | 0.93 | 2.54 | 3.456 (5) | 167 |

Symmetry code: (i) $-x + \frac{3}{2}, -y + 1, z + \frac{1}{2}$.

adjacent aryl ring, with a dihedral angle of only 8.3 (2)° between these two units; on the other hand, the dihedral angles between the nitrated aryl ring and the major and minor components of the disordered ring are 36.7 (10)° and 42.6 (11)°, respectively. The molecules of (I) are therefore conformationally chiral but, in the absence of significant resonant scattering, it was not possible to determine the absolute configuration of the molecules in the crystal selected for data collection. It is reasonable to assume that, in solution, the two conformational enantiomers will be in rapid equilibrium.

The conformational behaviour of compound (I) may be compared with that of some closely related compounds. In (*E*)-*N*-(4-nitrobenzylidene)-2,3-dimethylaniline, (II) (Tariq *et al.*, 2010), and (*E*)-*N*-(4-nitrobenzylidene)-3,4-dimethoxyaniline, (III) (Akkurt *et al.*, 2008), the dihedral angles between the two aryl rings are 24.52 (5) and 29.52 (8)°, respectively. By contrast, in (*E*)-*N*-(4-nitrobenzylidene)-2-hydroxyaniline, (IV) (Madhuprasad *et al.*, 2014), and (*E*)-*N*-(4-chlorobenzylidene)-2-hydroxyaniline, (V) (Girisha *et al.*, 2018), the dihedral angles between the rings are 0.52° [the atomic coordinates retrieved from the CSD (Groom *et al.*, 2016) carry no s.u.s] and 3.31 (9)° respectively, reflecting the influence of the intramolecular O—H⋯N hydrogen bonds in these two compounds.

3. Supramolecular features

The supramolecular assembly depends upon a combination of one C—H⋯O hydrogen bond (Table 1) and three N—O⋯π(arene) interactions (Kaafarani *et al.*, 2003; Báuza *et al.*,

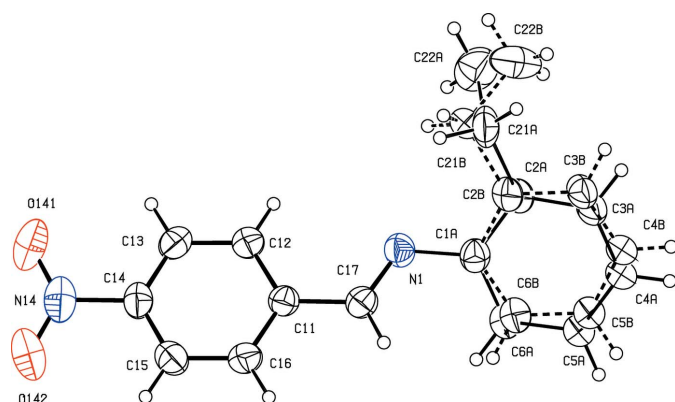


Figure 1
The molecular structure of compound (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level, and for the disordered 2-ethylphenyl group, the major component is drawn using solid lines and the minor component is drawn using dashed lines.

Table 2
Parameters (Å, °) for nitro⋯π(arene) interactions.

| $N-O\cdots Cg$ | $N-O$ | $O\cdots Cg$ | $N\cdots Cg$ | $N-O\cdots Cg$ |
|---------------------------|-----------|--------------|--------------|----------------|
| $N14-O141\cdots Cg1^i$ | 1.215 (4) | 3.88 (2) | 3.91 (2) | 82.5 (3) |
| $N14-O141\cdots Cg2^i$ | 1.215 (4) | 3.82 (2) | 3.79 (2) | 79.4 (3) |
| $N14-O142\cdots Cg1^{ii}$ | 1.220 (4) | 3.97 (2) | 3.85 (2) | 75.1 (3) |

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

2016) (Table 2), and the three-dimensional assembly can readily be analysed in terms of three one-dimensional substructures (Ferguson *et al.*, 1998*a,b*; Gregson *et al.*, 2000). Thus, the action of the C—H⋯O hydrogen bond alone is to link molecules related by the 2_1 screw axis along (0.75, 0.5, z) into a $C(6)$ chain running parallel to the [001] direction (Fig. 2). The action of the two nitro⋯π(arene) interactions links molecules related by the 2_1 screw axis along (x , 0.25, 0.5) into a

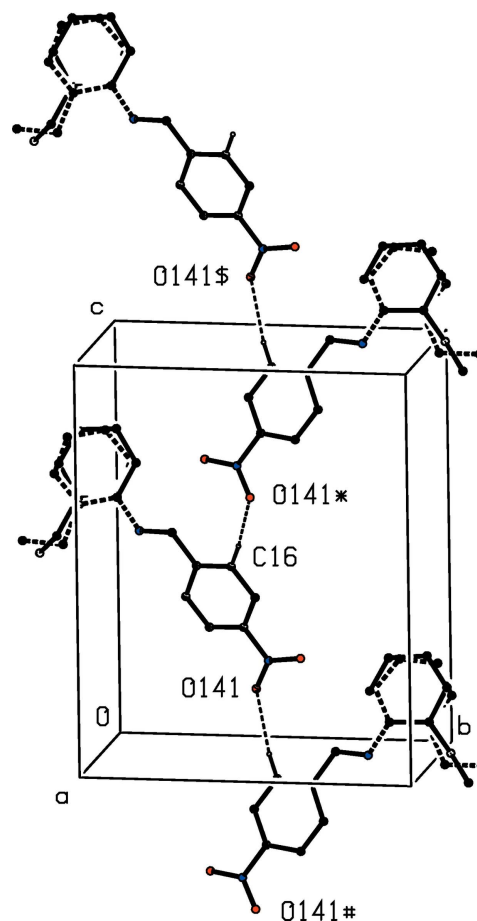
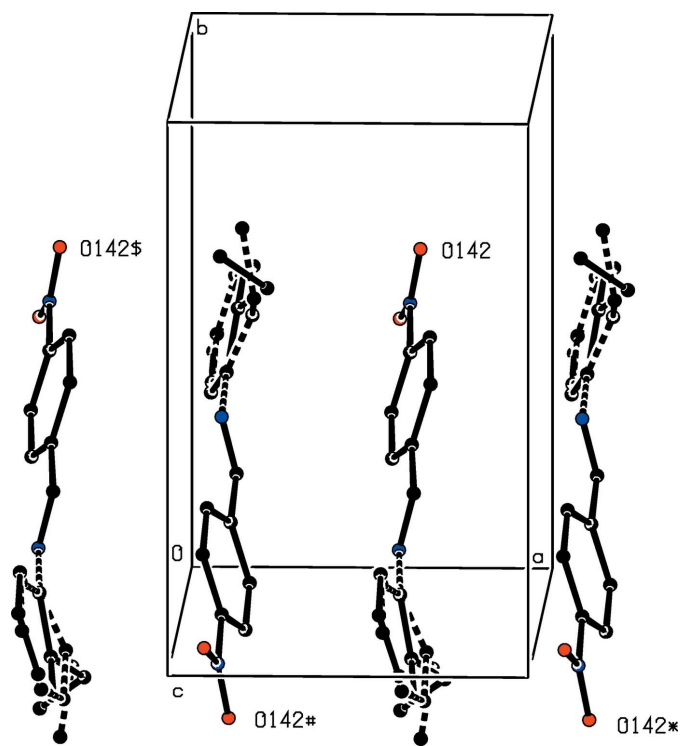
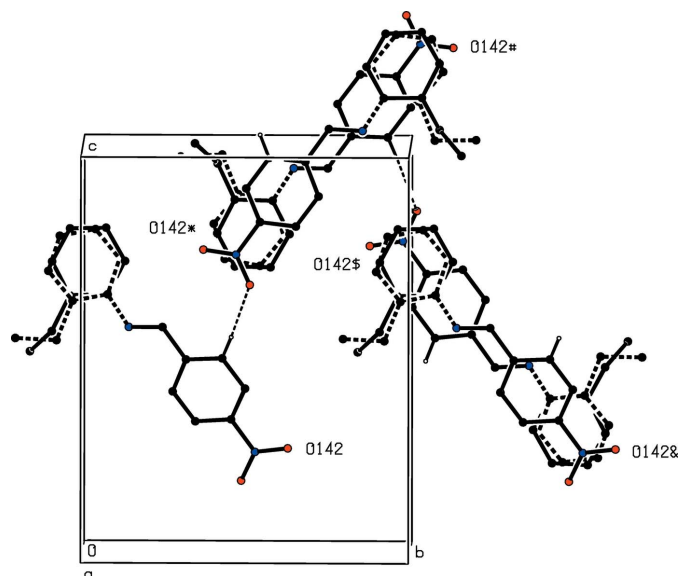


Figure 2
Part of the crystal structure of compound (I) showing the formation of a $C(6)$ hydrogen-bonded chain along [001]. For the sake of clarity, the H atoms not involved in the motif shown have been omitted. The atoms marked with an asterisk (*), a hash (#) or a dollar sign (\$) are at the symmetry positions $(\frac{3}{2} - x, 1 - y, \frac{1}{2} + z)$, $(x, y, 1 + z)$ and $(\frac{3}{2} - x, 1 - y, -\frac{1}{2} + z)$, respectively. For the disordered 2-ethylphenyl group, the major component is drawn using solid lines and the minor component is drawn using dashed lines.


Figure 3

Part of the crystal structure of compound (I) showing the formation of a chain along [100] built from nitro $\cdots\pi$ (arene) interactions. For the sake of clarity, the H atoms have all been omitted. The atoms marked with an asterisk (*), a hash (#) or a dollar sign (\$) are at the symmetry positions $(\frac{1}{2} + x, \frac{1}{2} - y, 1 - z)$, $(-\frac{1}{2} + x, \frac{1}{2} - y, 1 - z)$ and $(-1 + x, y, z)$, respectively. For the disordered 2-ethylphenyl group, the major component is drawn using solid lines and the minor component is drawn using dashed lines.


Figure 4

Part of the crystal structure of compound (I) showing the formation of a chain parallel to the [010] direction built from alternating C–H \cdots O hydrogen bonds and nitro $\cdots\pi$ (arene) interactions. For the sake of clarity, the H atoms not involved in the motif shown have been omitted. The atoms marked with an asterisk (*), a hash (#), a dollar sign (\$) or an ampersand (&) are at the symmetry positions $(\frac{3}{2} - x, 1 - y, \frac{1}{2} + z)$, $(1 - x, \frac{1}{2} + y, \frac{3}{2} - z)$, $(-\frac{1}{2} + x, \frac{3}{2} - y, 1 - z)$ and $(x, 1 + y, z)$, respectively. For the disordered 2-ethylphenyl group, the major component is drawn using solid lines and the minor component is drawn using dashed lines.

chain running parallel to the [100] direction (Fig. 3), while the combined action of the hydrogen bond and the nitro $\cdots\pi$ (arene) interactions links the molecules into a chain running parallel to the [010] direction (Fig. 4). The combination of chain motifs parallel to the [100], [010] and [001] directions then generates a continuous three-dimensional assembly.

4. Database survey

It is of interest to briefly compare the three-dimensional supramolecular assembly in compound (I), with the patterns of aggregation found in related compounds (II)–(V). In compound (II), two independent aromatic π – π stacking interactions combine to link the molecules into chains (Tariq *et al.*, 2010). The structure of compound (III) (Akkurt *et al.*, 2008) contains three short C–H \cdots O contacts, but two of these involve an H atom in a methyl group, while for the third the C–H \cdots O angle is only 131° , so that none of these contacts is likely to be structurally significant (Wood *et al.*, 2009). The molecules of compound (IV) (Madhuprasad *et al.*, 2014) are linked into centrosymmetric dimers by inversion-related O–H \cdots O hydrogen bonds, while those of compound (V) are linked into a three-dimensional framework structure by a combination of C–H \cdots O and C–H $\cdots\pi$ (arene) hydrogen bonds and an aromatic π – π stacking interaction (Girisha *et al.*, 2018).

Other Schiff bases which are derived from nitrobenzaldehydes and whose structures have been reported recently include *N*-(2-nitrobenzylidene)aniline (Naveen *et al.*, 2006), 4-methoxy-*N*-(2-nitrobenzylidene)aniline (Ren & Jian, 2008), 2,3-dimethyl-*N*-(2-nitrobenzylidene)aniline (Tahir *et al.*, 2010) and 2-fluoro-*N*-(3-nitrobenzylidene)-5-(trifluoromethyl)aniline (Yang *et al.*, 2007).

5. Synthesis and crystallization

Solutions of 2-ethylaniline (100 mg, 0.826 mmol) and 4-nitrobenzaldehyde (124 mg, 0.826 mmol), each in ethanol (15 ml), were mixed and a catalytic amount of glacial acetic acid was added. The resulting mixture was heated under reflux for 3 h, when completion of the reaction was confirmed using thin layer chromatography. The solid product was collected by filtration and recrystallized from acetonitrile to give crystals of (I) suitable for single crystal X-ray diffraction; yield 150 mg, 0.590 mmol, 71%; m.p. 369–373 K.

6. Refinement

It was apparent from an early stage in the refinement that the methyl group of the ethyl substituent was disordered over two sets of atomic sites having unequal occupancies, and satisfactory resolution of the disorder required a model in which the whole 2-ethylphenyl unit was disordered over two sets of atomic sites. For the minor disorder component, the bonded distances and the 1,3 non-bonded distances were restrained to be the same as the corresponding distances in the major

disorder component, subject to s.u. values of 0.01 and 0.02 Å, respectively. In addition, the anisotropic displacement parameters for the corresponding pairs of C atoms in the disordered ring were constrained to be identical. All H atoms apart from those in the ethyl unit were located in difference maps and then treated as riding atoms with C–H 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$; the H atoms of the ethyl unit were included in calculated positions with C–H distances of 0.96 Å (CH₃) or 0.97 Å (CH₂) and with $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$, where $k = 1.5$ for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for the CH₂ groups. Subject to these conditions, the occupancies of the two disorder components refined to 0.515 (19) and 0.485 (19). Although the coverage of Friedel pairs was 98%, it was not possible to determine the absolute configuration of the molecules in the crystal selected for study, as the value of the Flack x parameter (Flack, 1983), calculated using 484 quotients of the type $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons *et al.*, 2013), was -0.5 (7), and value calculated for the Hooft y parameter (Hooft *et al.*, 2008) was -0.4 (7). Crystal data, data collection and structure refinement details are summarized in Table 3.

Acknowledgements

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Table 3
Experimental details.

| | |
|--|---|
| Crystal data | |
| Chemical formula | C ₁₅ H ₁₄ N ₂ O ₂ |
| M_r | 254.28 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 296 |
| a, b, c (Å) | 7.6419 (7), 11.8889 (13), 14.8082 (16) |
| V (Å ³) | 1345.4 (2) |
| Z | 4 |
| Radiation type | Mo $K\alpha$ |
| μ (mm ⁻¹) | 0.09 |
| Crystal size (mm) | 0.15 × 0.10 × 0.10 |
| Data collection | |
| Diffractometer | Bruker APEXII CCD |
| Absorption correction | Multi-scan (<i>SADABS</i> ; Bruker, 2012) |
| $T_{\text{min}}, T_{\text{max}}$ | 0.841, 0.992 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 20309, 2535, 1401 |
| R_{int} | 0.055 |
| $(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹) | 0.612 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)], wR(F^2), S$ | 0.040, 0.120, 1.04 |
| No. of reflections | 2535 |
| No. of parameters | 209 |
| No. of restraints | 17 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³) | 0.11, -0.11 |

Computer programs: *APEX2* and *SAIN*T (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

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supporting information

Acta Cryst. (2018). E74, 1071-1074 [https://doi.org/10.1107/S2056989018009544]

The crystal structure of (*E*)-2-ethyl-*N*-(4-nitrobenzylidene)aniline: three-dimensional supramolecular assembly mediated by C—H \cdots O hydrogen bonds and nitro $\cdots\pi$ (arene) interactions

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Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINTE* (Bruker, 2012); data reduction: *SAINTE* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(*E*)-2-Ethyl-*N*-(4-nitrobenzylidene)aniline

Crystal data

C₁₅H₁₄N₂O₂

M_r = 254.28

Orthorhombic, *P*2₁2₁2₁

a = 7.6419 (7) Å

b = 11.8889 (13) Å

c = 14.8082 (16) Å

V = 1345.4 (2) Å³

Z = 4

F(000) = 536

D_x = 1.255 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 2535 reflections

θ = 2.2–25.8°

μ = 0.09 mm⁻¹

T = 296 K

Block, colourless

0.15 × 0.10 × 0.10 mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2012)

T_{min} = 0.841, *T_{max}* = 0.992

20309 measured reflections

2535 independent reflections

1401 reflections with *I* > 2σ(*I*)

R_{int} = 0.055

θ_{max} = 25.8°, θ_{min} = 2.2°

h = -6→9

k = -14→14

l = -17→17

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.040

wR(*F*²) = 0.120

S = 1.04

2535 reflections

209 parameters

17 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0486*P*)² + 0.1129*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

$$\Delta\rho_{\max} = 0.11 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.11 \text{ e } \text{\AA}^{-3}$$

Extinction correction: SHELXL,
 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.009 (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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|------------|--------------|--------------|----------------------------------|------------|
| N1 | 0.6122 (4) | 0.1423 (2) | 0.56000 (17) | 0.0657 (8) | |
| C17 | 0.6533 (5) | 0.2454 (3) | 0.5623 (2) | 0.0643 (9) | |
| H17 | 0.6916 | 0.2765 | 0.6164 | 0.077* | |
| C11 | 0.6423 (4) | 0.3173 (3) | 0.4820 (2) | 0.0582 (9) | |
| C12 | 0.5825 (5) | 0.2760 (3) | 0.3999 (2) | 0.0651 (10) | |
| H12 | 0.5473 | 0.2014 | 0.3951 | 0.078* | |
| C13 | 0.5754 (5) | 0.3451 (3) | 0.3261 (2) | 0.0689 (10) | |
| H13 | 0.5353 | 0.3177 | 0.2710 | 0.083* | |
| C14 | 0.6277 (5) | 0.4549 (3) | 0.3339 (2) | 0.0659 (9) | |
| C15 | 0.6878 (5) | 0.4979 (3) | 0.4132 (3) | 0.0744 (11) | |
| H15 | 0.7234 | 0.5725 | 0.4171 | 0.089* | |
| C16 | 0.6949 (5) | 0.4281 (3) | 0.4880 (2) | 0.0704 (10) | |
| H16 | 0.7355 | 0.4562 | 0.5427 | 0.084* | |
| N14 | 0.6223 (5) | 0.5279 (4) | 0.2537 (3) | 0.0934 (11) | |
| O141 | 0.5892 (5) | 0.4855 (3) | 0.1810 (2) | 0.1275 (13) | |
| O142 | 0.6516 (5) | 0.6280 (3) | 0.2643 (2) | 0.1273 (13) | |
| C1A | 0.6204 (4) | 0.0777 (3) | 0.6407 (2) | 0.0618 (9) | 0.515 (19) |
| C2A | 0.652 (3) | -0.0381 (8) | 0.6399 (9) | 0.060 (3) | 0.515 (19) |
| C21A | 0.739 (2) | -0.0913 (13) | 0.5561 (11) | 0.089 (6) | 0.515 (19) |
| H21A | 0.7893 | -0.0320 | 0.5192 | 0.107* | 0.515 (19) |
| H21B | 0.8340 | -0.1398 | 0.5757 | 0.107* | 0.515 (19) |
| C22A | 0.612 (2) | -0.1593 (16) | 0.4994 (10) | 0.121 (6) | 0.515 (19) |
| H22A | 0.5162 | -0.1121 | 0.4810 | 0.181* | 0.515 (19) |
| H22B | 0.5675 | -0.2210 | 0.5344 | 0.181* | 0.515 (19) |
| H22C | 0.6707 | -0.1878 | 0.4469 | 0.181* | 0.515 (19) |
| C3A | 0.664 (6) | -0.0915 (16) | 0.7237 (12) | 0.073 (3) | 0.515 (19) |
| H3A | 0.6967 | -0.1668 | 0.7255 | 0.087* | 0.515 (19) |
| C4A | 0.628 (5) | -0.0372 (17) | 0.8034 (10) | 0.072 (4) | 0.515 (19) |
| H4A | 0.6420 | -0.0747 | 0.8580 | 0.087* | 0.515 (19) |
| C5A | 0.573 (8) | 0.072 (2) | 0.8022 (10) | 0.075 (3) | 0.515 (19) |
| H5A | 0.5359 | 0.1075 | 0.8550 | 0.090* | 0.515 (19) |
| C6A | 0.572 (13) | 0.130 (3) | 0.7204 (13) | 0.0739 (19) | 0.515 (19) |
| H6A | 0.5383 | 0.2049 | 0.7193 | 0.089* | 0.515 (19) |
| C1B | 0.6204 (4) | 0.0777 (3) | 0.6407 (2) | 0.0618 (9) | 0.485 (19) |
| C2B | 0.693 (3) | -0.0285 (9) | 0.6285 (9) | 0.060 (3) | 0.485 (19) |
| C21B | 0.704 (3) | -0.0772 (10) | 0.5319 (10) | 0.079 (6) | 0.485 (19) |

| | | | | | |
|------|------------|--------------|-------------|-------------|------------|
| H21C | 0.6174 | -0.0412 | 0.4936 | 0.095* | 0.485 (19) |
| H21D | 0.8188 | -0.0627 | 0.5065 | 0.095* | 0.485 (19) |
| C22B | 0.671 (4) | -0.2034 (12) | 0.5351 (15) | 0.179 (11) | 0.485 (19) |
| H22D | 0.6559 | -0.2315 | 0.4748 | 0.268* | 0.485 (19) |
| H22E | 0.5664 | -0.2180 | 0.5694 | 0.268* | 0.485 (19) |
| H22F | 0.7682 | -0.2403 | 0.5631 | 0.268* | 0.485 (19) |
| C3B | 0.691 (6) | -0.1024 (17) | 0.7018 (13) | 0.073 (3) | 0.485 (19) |
| H3B | 0.7289 | -0.1762 | 0.6937 | 0.087* | 0.485 (19) |
| C4B | 0.635 (5) | -0.0684 (17) | 0.7854 (11) | 0.072 (4) | 0.485 (19) |
| H4B | 0.6331 | -0.1195 | 0.8329 | 0.087* | 0.485 (19) |
| C5B | 0.583 (9) | 0.040 (2) | 0.7992 (11) | 0.075 (3) | 0.485 (19) |
| H5B | 0.5538 | 0.0648 | 0.8569 | 0.090* | 0.485 (19) |
| C6B | 0.574 (14) | 0.113 (3) | 0.7262 (14) | 0.0739 (19) | 0.485 (19) |
| H6B | 0.5364 | 0.1871 | 0.7349 | 0.089* | 0.485 (19) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|--------------|--------------|--------------|
| N1 | 0.0710 (19) | 0.0625 (19) | 0.0635 (18) | -0.0032 (15) | -0.0085 (15) | 0.0061 (15) |
| C17 | 0.069 (2) | 0.065 (2) | 0.059 (2) | 0.000 (2) | -0.0101 (19) | -0.0022 (19) |
| C11 | 0.059 (2) | 0.058 (2) | 0.057 (2) | 0.0020 (17) | -0.0041 (17) | -0.0012 (17) |
| C12 | 0.073 (2) | 0.062 (2) | 0.061 (2) | -0.0010 (17) | -0.0050 (19) | -0.0024 (18) |
| C13 | 0.072 (2) | 0.080 (3) | 0.054 (2) | 0.006 (2) | 0.0016 (19) | -0.0050 (19) |
| C14 | 0.064 (2) | 0.072 (2) | 0.062 (2) | 0.0094 (19) | 0.0053 (18) | 0.0149 (19) |
| C15 | 0.081 (3) | 0.059 (2) | 0.082 (3) | -0.001 (2) | 0.002 (2) | 0.004 (2) |
| C16 | 0.082 (3) | 0.066 (2) | 0.063 (2) | 0.001 (2) | -0.0072 (19) | -0.0086 (19) |
| N14 | 0.085 (2) | 0.109 (3) | 0.085 (3) | 0.009 (2) | 0.008 (2) | 0.033 (3) |
| O141 | 0.148 (3) | 0.165 (3) | 0.069 (2) | -0.008 (2) | -0.001 (2) | 0.032 (2) |
| O142 | 0.146 (3) | 0.101 (2) | 0.135 (3) | -0.002 (2) | 0.000 (2) | 0.053 (2) |
| C1A | 0.060 (2) | 0.065 (2) | 0.060 (2) | -0.0052 (19) | -0.0073 (17) | 0.0066 (18) |
| C2A | 0.049 (10) | 0.060 (3) | 0.070 (4) | -0.021 (4) | 0.019 (4) | 0.008 (3) |
| C21A | 0.120 (11) | 0.079 (10) | 0.067 (9) | -0.012 (7) | -0.018 (7) | 0.030 (7) |
| C22A | 0.131 (11) | 0.132 (14) | 0.100 (9) | -0.048 (10) | -0.002 (7) | -0.009 (9) |
| C3A | 0.086 (12) | 0.057 (4) | 0.075 (7) | 0.007 (3) | 0.026 (10) | 0.011 (5) |
| C4A | 0.105 (4) | 0.049 (9) | 0.064 (5) | 0.003 (9) | 0.002 (7) | -0.001 (6) |
| C5A | 0.102 (7) | 0.059 (12) | 0.064 (3) | 0.006 (14) | 0.003 (2) | 0.002 (4) |
| C6A | 0.086 (3) | 0.066 (7) | 0.070 (3) | 0.004 (10) | -0.004 (6) | 0.009 (3) |
| C1B | 0.060 (2) | 0.065 (2) | 0.060 (2) | -0.0052 (19) | -0.0073 (17) | 0.0066 (18) |
| C2B | 0.049 (10) | 0.060 (3) | 0.070 (4) | -0.021 (4) | 0.019 (4) | 0.008 (3) |
| C21B | 0.123 (12) | 0.050 (6) | 0.065 (9) | 0.004 (7) | -0.002 (8) | 0.001 (7) |
| C22B | 0.30 (3) | 0.085 (10) | 0.147 (17) | -0.007 (13) | 0.039 (17) | -0.044 (10) |
| C3B | 0.086 (12) | 0.057 (4) | 0.075 (7) | 0.007 (3) | 0.026 (10) | 0.011 (5) |
| C4B | 0.105 (4) | 0.049 (9) | 0.064 (5) | 0.003 (9) | 0.002 (7) | -0.001 (6) |
| C5B | 0.102 (7) | 0.059 (12) | 0.064 (3) | 0.006 (14) | 0.003 (2) | 0.002 (4) |
| C6B | 0.086 (3) | 0.066 (7) | 0.070 (3) | 0.004 (10) | -0.004 (6) | 0.009 (3) |

Geometric parameters (Å, °)

| | | | |
|-------------|------------|----------------|------------|
| N1—C17 | 1.266 (4) | C22A—H22B | 0.9600 |
| N1—C1A | 1.422 (4) | C22A—H22C | 0.9600 |
| C17—C11 | 1.466 (4) | C3A—C4A | 1.372 (10) |
| C17—H17 | 0.9300 | C3A—H3A | 0.9300 |
| C11—C16 | 1.381 (5) | C4A—C5A | 1.370 (9) |
| C11—C12 | 1.387 (4) | C4A—H4A | 0.9300 |
| C12—C13 | 1.369 (4) | C5A—C6A | 1.390 (8) |
| C12—H12 | 0.9300 | C5A—H5A | 0.9300 |
| C13—C14 | 1.369 (5) | C6A—H6A | 0.9300 |
| C13—H13 | 0.9300 | C2B—C3B | 1.397 (9) |
| C14—C15 | 1.361 (5) | C2B—C21B | 1.547 (11) |
| C14—N14 | 1.471 (5) | C21B—C22B | 1.523 (13) |
| C15—C16 | 1.385 (5) | C21B—H21C | 0.9700 |
| C15—H15 | 0.9300 | C21B—H21D | 0.9700 |
| C16—H16 | 0.9300 | C22B—H22D | 0.9600 |
| N14—O141 | 1.215 (4) | C22B—H22E | 0.9600 |
| N14—O142 | 1.220 (4) | C22B—H22F | 0.9600 |
| C1A—C6A | 1.383 (8) | C3B—C4B | 1.370 (10) |
| C1A—C2A | 1.398 (8) | C3B—H3B | 0.9300 |
| C2A—C3A | 1.397 (9) | C4B—C5B | 1.368 (9) |
| C2A—C21A | 1.545 (11) | C4B—H4B | 0.9300 |
| C21A—C22A | 1.520 (13) | C5B—C6B | 1.391 (9) |
| C21A—H21A | 0.9700 | C5B—H5B | 0.9300 |
| C21A—H21B | 0.9700 | C6B—H6B | 0.9300 |
| C22A—H22A | 0.9600 | | |
| | | | |
| C17—N1—C1A | 119.3 (3) | H22A—C22A—H22B | 109.5 |
| N1—C17—C11 | 121.9 (3) | C21A—C22A—H22C | 109.5 |
| N1—C17—H17 | 119.1 | H22A—C22A—H22C | 109.5 |
| C11—C17—H17 | 119.1 | H22B—C22A—H22C | 109.5 |
| C16—C11—C12 | 119.3 (3) | C4A—C3A—C2A | 122.5 (9) |
| C16—C11—C17 | 119.1 (3) | C4A—C3A—H3A | 118.7 |
| C12—C11—C17 | 121.6 (3) | C2A—C3A—H3A | 118.7 |
| C13—C12—C11 | 120.0 (3) | C5A—C4A—C3A | 119.8 (10) |
| C13—C12—H12 | 120.0 | C5A—C4A—H4A | 120.1 |
| C11—C12—H12 | 120.0 | C3A—C4A—H4A | 120.1 |
| C12—C13—C14 | 119.5 (3) | C4A—C5A—C6A | 118.6 (9) |
| C12—C13—H13 | 120.2 | C4A—C5A—H5A | 120.7 |
| C14—C13—H13 | 120.2 | C6A—C5A—H5A | 120.7 |
| C15—C14—C13 | 121.9 (3) | C1A—C6A—C5A | 121.6 (9) |
| C15—C14—N14 | 118.9 (4) | C1A—C6A—H6A | 119.2 |
| C13—C14—N14 | 119.1 (4) | C5A—C6A—H6A | 119.2 |
| C14—C15—C16 | 118.6 (3) | C3B—C2B—C21B | 118.9 (11) |
| C14—C15—H15 | 120.7 | C22B—C21B—C2B | 109.3 (11) |
| C16—C15—H15 | 120.7 | C22B—C21B—H21C | 109.8 |
| C11—C16—C15 | 120.6 (3) | C2B—C21B—H21C | 109.8 |

| | | | |
|------------------|------------|-------------------|-------------|
| C11—C16—H16 | 119.7 | C22B—C21B—H21D | 109.8 |
| C15—C16—H16 | 119.7 | C2B—C21B—H21D | 109.8 |
| O141—N14—O142 | 123.8 (4) | H21C—C21B—H21D | 108.3 |
| O141—N14—C14 | 118.4 (4) | C21B—C22B—H22D | 109.5 |
| O142—N14—C14 | 117.8 (4) | C21B—C22B—H22E | 109.5 |
| C6A—C1A—C2A | 119.6 (10) | H22D—C22B—H22E | 109.5 |
| C6A—C1A—N1 | 117.7 (7) | C21B—C22B—H22F | 109.5 |
| C2A—C1A—N1 | 122.2 (6) | H22D—C22B—H22F | 109.5 |
| C3A—C2A—C1A | 116.8 (9) | H22E—C22B—H22F | 109.5 |
| C3A—C2A—C21A | 120.0 (10) | C4B—C3B—C2B | 121.3 (10) |
| C1A—C2A—C21A | 118.9 (9) | C4B—C3B—H3B | 119.4 |
| C22A—C21A—C2A | 112.5 (13) | C2B—C3B—H3B | 119.4 |
| C22A—C21A—H21A | 109.1 | C5B—C4B—C3B | 120.4 (10) |
| C2A—C21A—H21A | 109.1 | C5B—C4B—H4B | 119.8 |
| C22A—C21A—H21B | 109.1 | C3B—C4B—H4B | 119.8 |
| C2A—C21A—H21B | 109.1 | C4B—C5B—C6B | 119.3 (10) |
| H21A—C21A—H21B | 107.8 | C4B—C5B—H5B | 120.4 |
| C21A—C22A—H22A | 109.5 | C6B—C5B—H5B | 120.4 |
| C21A—C22A—H22B | 109.5 | C5B—C6B—H6B | 119.8 |
| | | | |
| C1A—N1—C17—C11 | -178.1 (3) | C17—N1—C1A—C2A | -151.9 (12) |
| N1—C17—C11—C16 | -177.7 (3) | C6A—C1A—C2A—C3A | -12 (6) |
| N1—C17—C11—C12 | 1.4 (5) | N1—C1A—C2A—C3A | 178 (2) |
| C16—C11—C12—C13 | -0.3 (5) | C6A—C1A—C2A—C21A | -168 (5) |
| C17—C11—C12—C13 | -179.4 (3) | N1—C1A—C2A—C21A | 21 (2) |
| C11—C12—C13—C14 | 0.0 (5) | C3A—C2A—C21A—C22A | 97 (3) |
| C12—C13—C14—C15 | 0.3 (5) | C1A—C2A—C21A—C22A | -107 (2) |
| C12—C13—C14—N14 | 179.0 (3) | C1A—C2A—C3A—C4A | 7 (5) |
| C13—C14—C15—C16 | -0.4 (5) | C21A—C2A—C3A—C4A | 163 (3) |
| N14—C14—C15—C16 | -179.1 (3) | C2A—C3A—C4A—C5A | 3 (7) |
| C12—C11—C16—C15 | 0.2 (5) | C3A—C4A—C5A—C6A | -7 (9) |
| C17—C11—C16—C15 | 179.4 (3) | C2A—C1A—C6A—C5A | 7 (11) |
| C14—C15—C16—C11 | 0.1 (5) | N1—C1A—C6A—C5A | 179 (6) |
| C15—C14—N14—O141 | 171.1 (4) | C4A—C5A—C6A—C1A | 2 (12) |
| C13—C14—N14—O141 | -7.7 (5) | C3B—C2B—C21B—C22B | 12 (4) |
| C15—C14—N14—O142 | -8.9 (5) | C21B—C2B—C3B—C4B | -162 (4) |
| C13—C14—N14—O142 | 172.3 (4) | C2B—C3B—C4B—C5B | -1 (7) |
| C17—N1—C1A—C6A | 37 (5) | C3B—C4B—C5B—C6B | 5 (10) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H··· <i>A</i> | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| C16—H16···O141 ⁱ | 0.93 | 2.54 | 3.456 (5) | 167 |

Symmetry code: (i) $-x+3/2, -y+1, z+1/2$.