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Crystal structures of two hydrazide derivatives of mefenamic acid, 3-(2,3-dimethylanilino)-*N*′-[(*E*)-(furan-2-yl)methylidene]benzohydrazide and *N*′-[(*E*)-benzylidene]-2-(2,3-dimethylanilino)benzohydrazide

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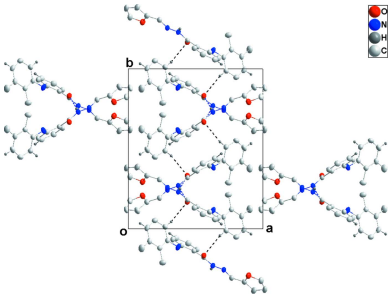
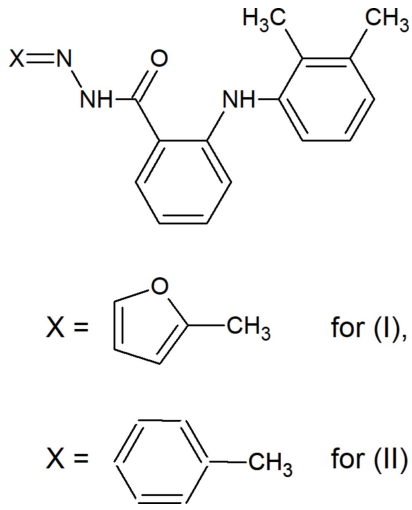
The conformation about the central benzene ring in the molecule of (I), $C_{20}H_{19}N_3O_2$, is partially determined by an intramolecular N—H···O hydrogen bond. In the crystal, chains parallel to the *c* axis are generated by intermolecular N—H···O hydrogen bonds with the chains assembled into a three-dimensional network structure by intermolecular C—H···O hydrogen bonds and C—H···π(ring) interactions. The molecule of (II), $C_{22}H_{21}N_3O$, differs from (I) only in the substituent at the hydrazide N atom where a phenylmethylene moiety for (II) is present instead of a furanmethylene moiety for (I). Hence, molecules of (I) and (II) show similarities in their molecular and crystal structures. The conformation of the central portion of the molecule of (II) is also therefore partially determined by an intramolecular N—H···O hydrogen bond and intermolecular N—H···O hydrogen bonds form chains parallel to the *c* axis. Likewise, the chains are connected into a three-dimensional network by C—H···O hydrogen bonds and C—H···π(ring) interactions.

1. Chemical context

Hydrazones possess a wide variety of biological activities such as anticonvulsant (Kumar *et al.*, 2010), anti-depressant (Mohareb *et al.*, 2010), analgesic, anti-inflammatory (Hernandez *et al.*, 2012), antimicrobial (Maguene *et al.*, 2011), anticancer (Al-Said *et al.*, 2011) or antiparasitic (Siddiqui *et al.*, 2012) properties. A better tolerated and potent non-steroidal anti-inflammatory drug (NSAID) with fewer side effect characteristic is mefenamic acid. This drug belongs to the most commonly prescribed medications worldwide for treatment of painful inflammatory conditions such as rheumatic arthritis, traumatic injuries, pain and fever (Abbas, 2017). It is also used to treat mild to moderate pain, including menstrual pain and the associated migraines (Pringsheim *et al.*, 2008). With this background in mind, we report here the synthesis and crystal structural determination of two hydrazide derivatives of mefenamic acid, (I) and (II).





2. Structural commentary

In the molecule of (I) (Fig. 1), the dihedral angles between the central C9–C14 benzene ring and the C1–C6 and C17–C20/O2 rings are, respectively, 51.90 (6) and 43.32 (8)°. The conformation about the central portion of the molecule is partially determined by the intramolecular N1—H1···O1 hydrogen bond (Table 1).

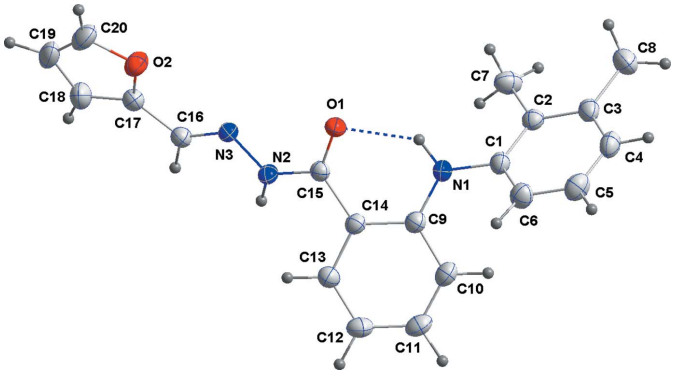


Figure 1
The molecule of (I) with atom-labeling scheme and displacement ellipsoids drawn at the 50% probability level. The intramolecular N—H···O hydrogen bond is shown as a dashed line.

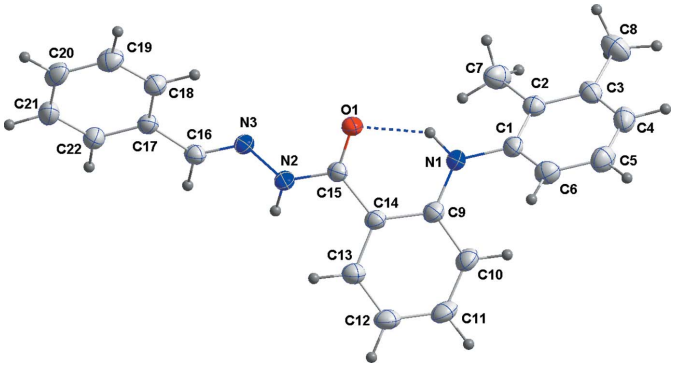


Figure 2
The molecule of (II) with atom-labeling scheme and displacement ellipsoids drawn at the 50% probability level. The intramolecular N—H···O hydrogen bond is shown by a dashed line.

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

*Cg*1 and *Cg*2 are the centroids of the C17–C20/O2 and C1–C6 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.910 (19)	1.977 (18)	2.7045 (14)	135.8 (15)
N2—H2···O1^{i}	0.883 (18)	2.014 (18)	2.8458 (13)	156.4 (15)
C4—H4···*Cg*1^{ii}	0.955 (17)	2.941 (17)	3.7248 (15)	140.1 (17)
C6—H6···O1^{iii}	0.976 (18)	2.556 (18)	3.3434 (16)	137.7 (14)
C11—H11···*Cg*2^{iv}	0.996 (16)	2.765 (16)	3.6231 (14)	144.8 (12)

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

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

*Cg*1 and *Cg*3 are the centroids of the C1–C6 and C17–C22 benzene rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.908 (18)	1.946 (18)	2.6920 (13)	138.2 (15)
N2—H2···O1^{i}	0.913 (16)	1.974 (17)	2.8564 (13)	162.0 (14)
C4—H4···*Cg*3^{ii}	1.001 (17)	2.796 (17)	3.6141 (15)	139.4 (13)
C6—H6···O1^{iii}	0.980 (18)	2.583 (19)	3.4815 (16)	152.5 (13)
C20—H20···*Cg*1^{iv}	1.000 (18)	2.838 (17)	3.6644 (15)	140.5 (13)

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, -y+1, -z+1$; (iv) $x-1, -y-\frac{1}{2}, z-\frac{3}{2}$.

Molecule (II) (Fig. 2) differs from molecule (I) only by the substituent at N3, *i.e.* a phenylmethylene moiety for (II) instead of a furanmethylene moiety for (I). Hence, the structural characteristics for most parts of the two molecules are very similar, as exemplified by the dihedral angles between the central C9–C14 benzene ring and the C1–C6 and C17–C22 benzene rings of 57.38 (6) and 43.48 (6)°, respectively, observed in molecule (II). Likewise, in the crystal of (II), the conformation of the central portion of the molecule is also partially determined by the intramolecular N1—H1···O1 hydrogen bond (Table 2; Fig. 2).

3. Supramolecular features

In the crystal structure of (I), chains of molecules extending parallel to the *c*-axis direction are generated by N2—H2···O2 hydrogen bonds (Table 1; Fig. 3). These chains are linked into a three-dimensional network structure by a combination of C6—H6···O1 hydrogen bonds and C4—H4···*Cg*1 and C11—H11···*Cg*2 interactions (Table 1; Fig. 4).

In the crystal structure of (II), intermolecular N2—H2···O1 hydrogen bonds form chains parallel the *c*-axis direction (Table 2; Fig. 5), which are connected through C6—H6···O1 hydrogen bonds and C4—H4···*Cg*3 and C20—H20···*Cg*1 interactions to form a three-dimensional network (Table 2; Fig. 6).

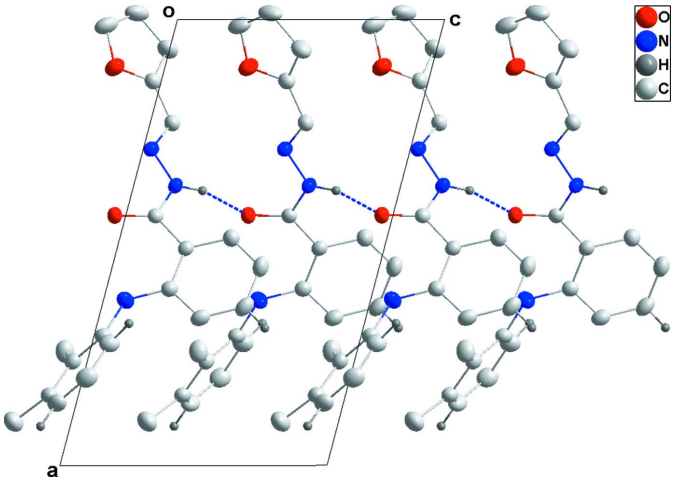


Figure 3
A portion of one N—H···O hydrogen-bonded chain viewed along the *b* axis of (I) with hydrogen bonds shown as dashed lines.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.42, November 2020; Groom *et al.*, 2016) gave six hits for structures with a 2-(2,3-dimethylanilino)-*N*′-methylidenebenzohydrazide skeleton: *N*′-[(4-chlorophenyl)methylidene]-2-[(2,3-dimethylphenyl)amino]benzohydrazide (VEDBAK; Jasinski *et al.*, 2017), *N*′-[1-(4-chlorophenyl)ethylidene]-2-[(2,3-dimethylphenyl)amino]benzohydrazide (LEBSET; Mohamed *et al.*, 2017), 2-[(2,3-dimethylphenyl)amino]-*N*′-(2-hydroxybenzylidene)benzohydrazide (DABREG; Mohamed *et al.*, 2015), 2-[(2,3-dimethylphenyl)amino]-*N*′-(2-thienylmethylene)benzohydrazide (LEGHAI; Fun *et al.*, 2012*a*), 2-[(2,3-dimethylphenyl)amino]benzohydrazide (LEGHIQ; Fun *et al.*, 2012*b*) and (*E*)-2-[(2,3-dimethylphenyl)amino]-*N*′-(2-methyl-5-(prop-1-en-2-yl)cyclohex-2-en-1-ylidene)benzohydrazide (YAXJUE; Bhat *et al.*, 2012).

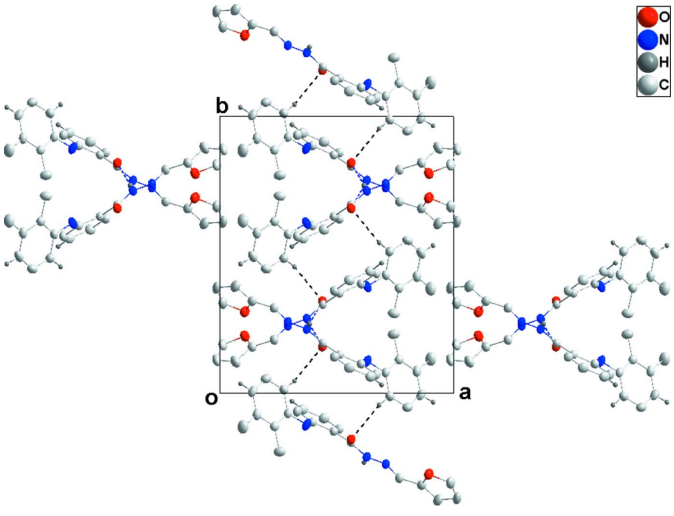


Figure 4
Packing view of (I) along the *c* axis with intermolecular C—H···O hydrogen bonds shown as dashed lines.

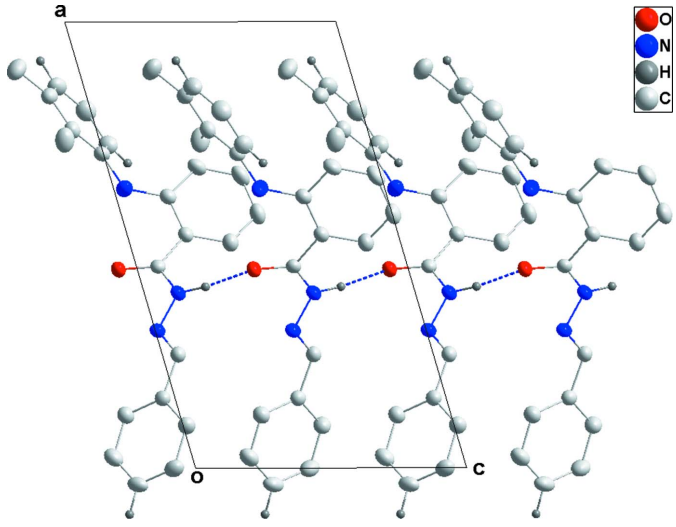


Figure 5
A portion of the N—H···O hydrogen-bonded chain viewed along the *b* axis of (II) with hydrogen bonds shown as dashed lines..

In the structure of VEDBAK, the dihedral angle between the planes of the chlorophenyl and dimethylphenyl rings is 66.50 (9)°. These rings make dihedral angles of 47.79 (8) and 69.24 (9)°, respectively, with the central benzene ring. In the crystal structure of VEDBAK, molecules are linked into a three-dimensional supramolecular network by N—H···O, C—H···O hydrogen bonds and weak C—H···π interactions.

In the crystal structure of LEBSET, molecules are linked into a three-dimensional supramolecular network by N—H···N, N—H···O, C—H···O hydrogen bonds and weak C—H···π interactions.

The asymmetric unit of DABREG consists of two molecules (*A* and *B*) having differing conformations that mainly concern the dihedral angles between the hydroxyphenyl and dimethylphenyl rings relative to the central phenylene ring, with values of 30.16 (6) and 58.60 (6)° in molecule *A* and of

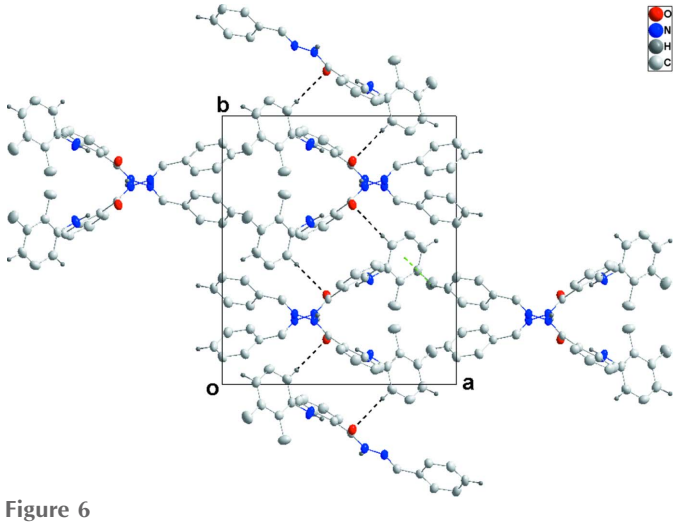


Figure 6
Packing view of (II) along the *c* axis with intermolecular C—H···O hydrogen bonds shown as dashed lines.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{20}H_{19}N_3O_2$	$C_{22}H_{21}N_3O$
M_r	333.38	343.42
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	150	150
a, b, c (Å)	13.8467 (3), 15.8409 (3), 8.0225 (2)	14.3493 (8), 15.7501 (9), 8.3737 (5)
β (°)	104.814 (1)	106.285 (2)
V (Å^3)	1701.20 (7)	1816.55 (18)
Z	4	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	0.69	0.62
Crystal size (mm)	0.19 × 0.11 × 0.07	0.19 × 0.13 × 0.08
Data collection		
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.88, 0.95	0.86, 0.95
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13222, 3372, 2939	13875, 3665, 3140
R_{int}	0.031	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625	0.625
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.095, 1.05	0.037, 0.092, 1.04
No. of reflections	3372	3665
No. of parameters	303	320
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.19	0.17, −0.16

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *publCIF* (Westrip, 2010).

13.42 (7) and 60.31 (7)° in molecule *B*. With the exception of the dimethylphenyl substituent, the conformations of the rest of each molecule are largely determined by intramolecular O—H···N and N—H···O hydrogen bonds. In the crystal structure, N—H···O hydrogen bonds link the molecules into chains extending parallel to the *a* axis where the types of molecules alternate in an ···*A*···*B*···*A*···*B*··· fashion.

In LEGHAI, the central benzene ring makes dihedral angles of 45.36 (9) and 55.33 (9)° with the thiophene ring and the dimethyl-substituted benzene ring, respectively. The dihedral angle between the thiophene ring and dimethyl-substituted benzene ring is 83.60 (9)°. The thiophene ring and the benzene ring are twisted from the mean plane of the C(=O)—N—N=C bridge [maximum deviation = 0.0860 (13) Å], with dihedral angles of 23.86 (9) and 24.77 (8)°, respectively. An intramolecular N—H···O hydrogen bond generates an *S*(6) ring motif. In the crystal structure of LEGHAI, molecules are linked by N—H···O and C—H···O hydrogen bonds to the same acceptor atom, forming sheets lying parallel to the *bc* plane. The crystal packing also features C—H···π interactions.

In LEGHIQ, the dihedral angle between the benzene rings is 58.05 (9)°. The non-H atoms of the hydrazide group lie in a common plane (r.m.s. deviation = 0.0006 Å) and are close to co-planar with their attached benzene ring [dihedral angle = 8.02 (9)°]. An intramolecular N—H···O hydrogen bond generates an *S*(6) ring motif in the molecule, and a short intramolecular contact (H···H = 1.88 Å) is also observed. In the crystal structure of LEGHIQ, molecules are linked by pairs of N—H···N hydrogen bonds into inversion dimers. The crystal packing also features C—H···π interactions.

The asymmetric unit of the compound YAXJUE comprises two molecules. The dihedral angles between the benzene rings in the two molecules are 59.7 (2) and 61.27 (18)°. The cyclohexene rings adopt sofa and half-chair conformations. In the crystal structure of YAXJUE, molecules are connected *via* N—H···O and weak C—H···O hydrogen bonds, forming chains along the *a*-axis direction. In each molecule, there is an intramolecular N—H···O hydrogen bond.

5. Synthesis and crystallization

Synthesis of (I): A mixture of 1 mmol of 2-furaldehyde (96 mg) and 1 mmol of 2-[(2,3-dimethylphenyl)amino]-benzohydrazide (255 mg) in 20 ml of ethanol was refluxed and monitored by TLC until completion. The reaction mixture was cooled to room temperature when the solid product was obtained. The crude product was filtered off, dried and recrystallized from ethanol to afford crystals suitable for X-ray diffraction. M.p. 479–483 K.

Synthesis of (II): In a solution of 20 ml of ethanol, a mixture of 106 mg (1 mmol) of benzaldehyde (106 mg) and 255 mg (1 mmol) of 2-[(2,3-dimethylphenyl)amino]benzohydrazide was refluxed for 4 h. The solid product was obtained after the reaction mixture was cooled to room temperature. The crude product was filtered off, dried and recrystallized from ethanol to afford crystals suitable for X-ray diffraction. M.p. 466–469 K.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For (I) and (II), all H atoms were located in a difference-Fourier map and were refined freely.

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supporting information

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Crystal structures of two hydrazide derivatives of mefenamic acid, 3-(2,3-dimethylanilino)-*N*′-[(*E*)-(furan-2-yl)methylidene]benzohydrazide and *N*′-[(*E*)-benzylidene]-2-(2,3-dimethylanilino)benzohydrazide

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Computing details

For both structures, data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2016/6* (Sheldrick, 2015*b*); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010).

3-(2,3-Dimethylanilino)-*N*′-[(*E*)-(furan-2-yl)methylidene]benzohydrazide (I)

Crystal data

$C_{20}H_{19}N_3O_2$
M_r = 333.38
Monoclinic, $P2_1/c$
a = 13.8467 (3) Å
b = 15.8409 (3) Å
c = 8.0225 (2) Å
β = 104.814 (1)°
V = 1701.20 (7) Å^3
Z = 4
$F(000)$ = 704
D_x = 1.302 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9755 reflections
θ = 3.3–74.5°
μ = 0.69 mm^{-1}
T = 150 K
Column, colourless
0.19 × 0.11 × 0.07 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.88, T_{max} = 0.95
13222 measured reflections
3372 independent reflections
2939 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 74.5°, θ_{min} = 3.3°
h = −14→16
k = −18→19
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.095
S = 1.05
3372 reflections
303 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.3981P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL* 2016/6 (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0049 (4)

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. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.43892 (6)	0.33178 (5)	0.45837 (10)	0.0275 (2)
O2	0.10644 (7)	0.20592 (6)	0.31256 (12)	0.0371 (2)
N1	0.62923 (8)	0.38535 (8)	0.58252 (15)	0.0353 (3)
H1	0.5810 (13)	0.3629 (11)	0.495 (2)	0.051 (5)*
N2	0.37222 (7)	0.26862 (7)	0.65665 (13)	0.0262 (2)
H2	0.3836 (12)	0.2485 (11)	0.763 (2)	0.043 (4)*
N3	0.29029 (7)	0.24278 (6)	0.52997 (12)	0.0260 (2)
C1	0.70907 (9)	0.42610 (8)	0.53437 (15)	0.0279 (3)
C2	0.76645 (9)	0.37935 (8)	0.44626 (15)	0.0276 (3)
C3	0.84199 (9)	0.42133 (9)	0.39033 (15)	0.0313 (3)
C4	0.85989 (10)	0.50619 (9)	0.42738 (17)	0.0361 (3)
H4	0.9119 (12)	0.5334 (11)	0.389 (2)	0.047 (5)*
C5	0.80281 (11)	0.55168 (9)	0.51433 (19)	0.0378 (3)
H5	0.8130 (13)	0.6122 (12)	0.533 (2)	0.052 (5)*
C6	0.72631 (10)	0.51191 (9)	0.56546 (18)	0.0342 (3)
H6	0.6815 (13)	0.5435 (11)	0.618 (2)	0.052 (5)*
C7	0.74397 (12)	0.28753 (9)	0.40596 (19)	0.0381 (3)
H7A	0.6963 (16)	0.2816 (13)	0.295 (3)	0.073 (6)*
H7B	0.8049 (17)	0.2542 (14)	0.398 (3)	0.078 (6)*
H7C	0.7169 (13)	0.2599 (12)	0.493 (2)	0.055 (5)*
C8	0.90166 (13)	0.37716 (12)	0.2834 (2)	0.0470 (4)
H8A	0.8587 (14)	0.3622 (12)	0.170 (3)	0.056 (5)*
H8B	0.957 (2)	0.4145 (17)	0.265 (3)	0.105 (8)*
H8C	0.9298 (16)	0.3242 (15)	0.331 (3)	0.075 (6)*
C9	0.60374 (9)	0.39480 (8)	0.73618 (15)	0.0275 (3)
C10	0.66889 (10)	0.43330 (8)	0.88024 (16)	0.0326 (3)
H10	0.7324 (13)	0.4574 (11)	0.866 (2)	0.045 (4)*
C11	0.64394 (10)	0.44053 (9)	1.03499 (16)	0.0350 (3)

H11	0.6916 (11)	0.4702 (10)	1.131 (2)	0.037 (4)*
C12	0.55432 (10)	0.40856 (9)	1.05579 (16)	0.0343 (3)
H12	0.5341 (11)	0.4161 (10)	1.165 (2)	0.037 (4)*
C13	0.48990 (10)	0.36959 (8)	0.91722 (16)	0.0293 (3)
H13	0.4250 (12)	0.3490 (10)	0.9272 (19)	0.038 (4)*
C14	0.51281 (9)	0.36090 (7)	0.75761 (15)	0.0251 (3)
C15	0.44003 (8)	0.32000 (7)	0.61193 (14)	0.0238 (2)
C16	0.23183 (9)	0.19224 (8)	0.58218 (15)	0.0280 (3)
H16	0.2460 (11)	0.1729 (10)	0.701 (2)	0.038 (4)*
C17	0.13770 (9)	0.16726 (8)	0.46938 (16)	0.0297 (3)
C18	0.06614 (11)	0.11334 (10)	0.4926 (2)	0.0410 (3)
H18	0.0716 (14)	0.0788 (12)	0.597 (2)	0.059 (5)*
C19	−0.01455 (11)	0.11896 (10)	0.3418 (2)	0.0440 (4)
H19	−0.0782 (15)	0.0886 (12)	0.319 (2)	0.061 (5)*
C20	0.01305 (10)	0.17477 (10)	0.2391 (2)	0.0425 (4)
H20	−0.0217 (15)	0.1968 (13)	0.124 (3)	0.063 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0276 (4)	0.0337 (5)	0.0215 (4)	−0.0028 (3)	0.0067 (3)	−0.0006 (3)
O2	0.0308 (5)	0.0430 (5)	0.0328 (5)	−0.0046 (4)	−0.0004 (4)	0.0003 (4)
N1	0.0289 (6)	0.0477 (7)	0.0315 (6)	−0.0131 (5)	0.0118 (4)	−0.0092 (5)
N2	0.0236 (5)	0.0337 (5)	0.0197 (5)	−0.0034 (4)	0.0026 (4)	0.0016 (4)
N3	0.0225 (5)	0.0315 (5)	0.0229 (5)	−0.0018 (4)	0.0040 (4)	−0.0016 (4)
C1	0.0224 (6)	0.0326 (6)	0.0280 (6)	−0.0021 (5)	0.0051 (4)	0.0011 (5)
C2	0.0266 (6)	0.0307 (6)	0.0239 (6)	−0.0007 (5)	0.0037 (4)	0.0010 (5)
C3	0.0236 (6)	0.0426 (7)	0.0269 (6)	−0.0005 (5)	0.0050 (5)	0.0022 (5)
C4	0.0288 (7)	0.0433 (8)	0.0352 (7)	−0.0101 (6)	0.0065 (5)	0.0057 (6)
C5	0.0387 (8)	0.0292 (7)	0.0428 (8)	−0.0044 (5)	0.0056 (6)	0.0025 (6)
C6	0.0295 (7)	0.0321 (7)	0.0402 (7)	0.0025 (5)	0.0078 (5)	−0.0009 (5)
C7	0.0498 (9)	0.0314 (7)	0.0344 (7)	−0.0032 (6)	0.0130 (6)	−0.0027 (5)
C8	0.0392 (9)	0.0645 (11)	0.0414 (9)	−0.0004 (7)	0.0176 (7)	−0.0055 (7)
C9	0.0250 (6)	0.0297 (6)	0.0271 (6)	0.0004 (5)	0.0052 (4)	0.0000 (5)
C10	0.0260 (6)	0.0356 (7)	0.0327 (7)	−0.0025 (5)	0.0011 (5)	−0.0009 (5)
C11	0.0358 (7)	0.0358 (7)	0.0275 (6)	−0.0008 (5)	−0.0028 (5)	−0.0015 (5)
C12	0.0411 (7)	0.0370 (7)	0.0235 (6)	−0.0008 (6)	0.0060 (5)	−0.0024 (5)
C13	0.0301 (7)	0.0313 (6)	0.0268 (6)	−0.0012 (5)	0.0079 (5)	0.0003 (5)
C14	0.0232 (6)	0.0275 (6)	0.0233 (6)	0.0009 (4)	0.0033 (4)	0.0001 (4)
C15	0.0217 (6)	0.0260 (6)	0.0236 (6)	0.0023 (4)	0.0055 (4)	0.0000 (4)
C16	0.0264 (6)	0.0335 (6)	0.0249 (6)	−0.0010 (5)	0.0081 (5)	−0.0001 (5)
C17	0.0272 (6)	0.0341 (6)	0.0286 (6)	−0.0013 (5)	0.0088 (5)	−0.0029 (5)
C18	0.0342 (7)	0.0482 (8)	0.0433 (8)	−0.0102 (6)	0.0150 (6)	−0.0046 (6)
C19	0.0250 (7)	0.0502 (9)	0.0563 (9)	−0.0075 (6)	0.0097 (6)	−0.0170 (7)
C20	0.0285 (7)	0.0471 (8)	0.0451 (8)	−0.0014 (6)	−0.0033 (6)	−0.0108 (7)

Geometric parameters (Å, º)

O1—C15	1.2422 (14)	C7—H7C	0.978 (19)
O2—C17	1.3663 (16)	C8—H8A	0.98 (2)
O2—C20	1.3687 (16)	C8—H8B	1.01 (3)
N1—C9	1.3744 (16)	C8—H8C	0.96 (2)
N1—C1	1.4165 (16)	C9—C10	1.4102 (17)
N1—H1	0.910 (19)	C9—C14	1.4191 (17)
N2—C15	1.3582 (15)	C10—C11	1.3759 (19)
N2—N3	1.3777 (13)	C10—H10	0.993 (17)
N2—H2	0.883 (18)	C11—C12	1.389 (2)
N3—C16	1.2826 (16)	C11—H11	0.996 (16)
C1—C6	1.3915 (18)	C12—C13	1.3804 (18)
C1—C2	1.4023 (17)	C12—H12	0.989 (16)
C2—C3	1.4063 (17)	C13—C14	1.4024 (17)
C2—C7	1.5053 (18)	C13—H13	0.978 (16)
C3—C4	1.385 (2)	C14—C15	1.4829 (15)
C3—C8	1.507 (2)	C16—C17	1.4392 (17)
C4—C5	1.383 (2)	C16—H16	0.973 (16)
C4—H4	0.955 (17)	C17—C18	1.3566 (19)
C5—C6	1.3818 (19)	C18—C19	1.424 (2)
C5—H5	0.975 (19)	C18—H18	0.990 (19)
C6—H6	0.976 (18)	C19—C20	1.330 (2)
C7—H7A	0.97 (2)	C19—H19	0.98 (2)
C7—H7B	1.01 (2)	C20—H20	0.99 (2)
C17—O2—C20	106.08 (11)	H8B—C8—H8C	108.9 (19)
C9—N1—C1	126.32 (11)	N1—C9—C10	121.67 (11)
C9—N1—H1	115.6 (11)	N1—C9—C14	120.45 (11)
C1—N1—H1	115.8 (11)	C10—C9—C14	117.77 (11)
C15—N2—N3	118.53 (10)	C11—C10—C9	121.32 (12)
C15—N2—H2	120.4 (11)	C11—C10—H10	120.6 (10)
N3—N2—H2	120.9 (11)	C9—C10—H10	118.0 (10)
C16—N3—N2	114.45 (10)	C10—C11—C12	121.07 (12)
C6—C1—C2	120.85 (11)	C10—C11—H11	118.0 (9)
C6—C1—N1	120.43 (11)	C12—C11—H11	120.9 (9)
C2—C1—N1	118.62 (11)	C13—C12—C11	118.64 (12)
C1—C2—C3	118.33 (11)	C13—C12—H12	119.6 (9)
C1—C2—C7	120.39 (11)	C11—C12—H12	121.7 (9)
C3—C2—C7	121.21 (12)	C12—C13—C14	121.92 (12)
C4—C3—C2	119.82 (12)	C12—C13—H13	120.1 (9)
C4—C3—C8	118.60 (13)	C14—C13—H13	118.0 (9)
C2—C3—C8	121.53 (13)	C13—C14—C9	119.23 (11)
C5—C4—C3	121.32 (12)	C13—C14—C15	119.71 (11)
C5—C4—H4	120.2 (10)	C9—C14—C15	121.00 (10)
C3—C4—H4	118.5 (10)	O1—C15—N2	121.19 (10)
C6—C5—C4	119.50 (13)	O1—C15—C14	123.33 (10)
C6—C5—H5	119.7 (10)	N2—C15—C14	115.47 (10)

C4—C5—H5	120.7 (10)	N3—C16—C17	120.80 (11)
C5—C6—C1	120.11 (13)	N3—C16—H16	122.0 (9)
C5—C6—H6	121.1 (11)	C17—C16—H16	117.0 (9)
C1—C6—H6	118.7 (11)	C18—C17—O2	109.77 (12)
C2—C7—H7A	110.2 (13)	C18—C17—C16	131.50 (13)
C2—C7—H7B	112.8 (13)	O2—C17—C16	118.59 (11)
H7A—C7—H7B	106.1 (17)	C17—C18—C19	106.60 (14)
C2—C7—H7C	112.1 (11)	C17—C18—H18	124.4 (11)
H7A—C7—H7C	108.4 (16)	C19—C18—H18	129.0 (11)
H7B—C7—H7C	106.9 (17)	C20—C19—C18	106.30 (13)
C3—C8—H8A	110.5 (11)	C20—C19—H19	126.7 (11)
C3—C8—H8B	111.0 (15)	C18—C19—H19	127.0 (11)
H8A—C8—H8B	108.1 (18)	C19—C20—O2	111.25 (13)
C3—C8—H8C	113.8 (13)	C19—C20—H20	131.6 (12)
H8A—C8—H8C	104.2 (17)	O2—C20—H20	117.1 (12)

C15—N2—N3—C16	177.81 (11)	C11—C12—C13—C14	0.5 (2)
C9—N1—C1—C6	−44.52 (19)	C12—C13—C14—C9	−1.49 (19)
C9—N1—C1—C2	139.08 (13)	C12—C13—C14—C15	−178.79 (12)
C6—C1—C2—C3	0.10 (18)	N1—C9—C14—C13	178.37 (11)
N1—C1—C2—C3	176.49 (11)	C10—C9—C14—C13	2.26 (17)
C6—C1—C2—C7	−177.09 (12)	N1—C9—C14—C15	−4.36 (18)
N1—C1—C2—C7	−0.70 (18)	C10—C9—C14—C15	179.53 (11)
C1—C2—C3—C4	1.90 (18)	N3—N2—C15—O1	−12.62 (17)
C7—C2—C3—C4	179.07 (12)	N3—N2—C15—C14	166.53 (10)
C1—C2—C3—C8	−175.36 (12)	C13—C14—C15—O1	157.42 (11)
C7—C2—C3—C8	1.81 (19)	C9—C14—C15—O1	−19.84 (18)
C2—C3—C4—C5	−1.98 (19)	C13—C14—C15—N2	−21.70 (16)
C8—C3—C4—C5	175.36 (13)	C9—C14—C15—N2	161.04 (11)
C3—C4—C5—C6	0.0 (2)	N2—N3—C16—C17	173.06 (11)
C4—C5—C6—C1	2.0 (2)	C20—O2—C17—C18	0.28 (15)
C2—C1—C6—C5	−2.09 (19)	C20—O2—C17—C16	−175.78 (12)
N1—C1—C6—C5	−178.41 (12)	N3—C16—C17—C18	177.42 (14)
C1—N1—C9—C10	−14.2 (2)	N3—C16—C17—O2	−7.54 (18)
C1—N1—C9—C14	169.88 (12)	O2—C17—C18—C19	−0.22 (16)
N1—C9—C10—C11	−178.26 (12)	C16—C17—C18—C19	175.15 (13)
C14—C9—C10—C11	−2.20 (19)	C17—C18—C19—C20	0.08 (17)
C9—C10—C11—C12	1.3 (2)	C18—C19—C20—O2	0.10 (18)
C10—C11—C12—C13	−0.4 (2)	C17—O2—C20—C19	−0.23 (16)

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*2 are the centroids of the C17–C20/O2 and C1–C6 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.910 (19)	1.977 (18)	2.7045 (14)	135.8 (15)
N2—H2···O1^{i}	0.883 (18)	2.014 (18)	2.8458 (13)	156.4 (15)
C4—H4···*Cg*1^{ii}	0.955 (17)	2.941 (17)	3.7248 (15)	140.1 (17)

C6—H6···O1^{iii}	0.976 (18)	2.556 (18)	3.3434 (16)	137.7 (14)
C11—H11···*Cg*2^{iv}	0.996 (16)	2.765 (16)	3.6231 (14)	144.8 (12)

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

N′-[(*E*)-Benzylidene]-2-(2,3-dimethylanilino)-benzohydrazide (II)

Crystal data

$C_{22}H_{21}N_3O$
M_r = 343.42
Monoclinic, $P2_1/c$
a = 14.3493 (8) Å
b = 15.7501 (9) Å
c = 8.3737 (5) Å
β = 106.285 (2)°
V = 1816.55 (18) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.256 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9904 reflections
θ = 4.3–74.6°
μ = 0.62 mm^{-1}
T = 150 K
Block, pale yellow
0.19 × 0.13 × 0.08 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.86, T_{max} = 0.95
13875 measured reflections
3665 independent reflections
3140 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 74.6°, θ_{min} = 4.3°
h = −17→16
k = −19→18
l = −10→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.092
S = 1.04
3665 reflections
320 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 0.444P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL* 2016/6 (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0033 (3)

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.44514 (6)	0.33126 (6)	0.43024 (9)	0.0300 (2)
N1	0.62636 (8)	0.39413 (8)	0.53993 (14)	0.0392 (3)
H1	0.5771 (13)	0.3712 (11)	0.458 (2)	0.051 (5)*
N2	0.39227 (7)	0.26174 (7)	0.62519 (12)	0.0287 (2)
H2	0.4042 (11)	0.2421 (10)	0.732 (2)	0.040 (4)*
N3	0.30815 (7)	0.23802 (7)	0.50842 (12)	0.0278 (2)
C1	0.70441 (8)	0.43293 (8)	0.49433 (14)	0.0301 (3)
C2	0.75817 (9)	0.38475 (8)	0.41121 (14)	0.0301 (3)
C3	0.83421 (9)	0.42441 (9)	0.36395 (15)	0.0330 (3)
C4	0.85496 (10)	0.50931 (9)	0.40197 (17)	0.0386 (3)
H4	0.9098 (13)	0.5351 (11)	0.367 (2)	0.052 (5)*
C5	0.80049 (11)	0.55653 (9)	0.48198 (18)	0.0411 (3)
H5	0.8132 (13)	0.6202 (12)	0.504 (2)	0.053 (5)*
C6	0.72458 (10)	0.51889 (9)	0.52686 (16)	0.0365 (3)
H6	0.6833 (13)	0.5527 (12)	0.578 (2)	0.054 (5)*
C7	0.73544 (13)	0.29257 (9)	0.3725 (2)	0.0451 (4)
H7A	0.7008 (18)	0.2847 (16)	0.256 (3)	0.095 (7)*
H7B	0.7995 (19)	0.2567 (16)	0.388 (3)	0.096 (8)*
H7C	0.6979 (15)	0.2671 (13)	0.443 (3)	0.072 (6)*
C8	0.89350 (12)	0.37677 (12)	0.2713 (2)	0.0490 (4)
H8A	0.8504 (15)	0.3577 (13)	0.155 (3)	0.068 (6)*
H8B	0.9499 (16)	0.4120 (13)	0.259 (2)	0.074 (6)*
H8C	0.9197 (17)	0.3238 (15)	0.328 (3)	0.083 (7)*
C9	0.60826 (8)	0.39908 (8)	0.69193 (14)	0.0300 (3)
C10	0.67446 (9)	0.43734 (9)	0.83041 (16)	0.0356 (3)
H10	0.7358 (12)	0.4613 (10)	0.8156 (19)	0.045 (4)*
C11	0.65461 (10)	0.44332 (9)	0.98135 (16)	0.0391 (3)
H11	0.7018 (12)	0.4732 (10)	1.073 (2)	0.045 (4)*
C12	0.56938 (10)	0.41042 (9)	1.00387 (16)	0.0386 (3)
H12	0.5526 (12)	0.4186 (11)	1.110 (2)	0.053 (5)*
C13	0.50518 (9)	0.36998 (8)	0.87248 (15)	0.0323 (3)
H13	0.4448 (11)	0.3485 (10)	0.8855 (18)	0.037 (4)*
C14	0.52311 (8)	0.36209 (8)	0.71679 (14)	0.0273 (3)
C15	0.45190 (8)	0.31809 (8)	0.57935 (14)	0.0259 (2)
C16	0.25463 (8)	0.18507 (8)	0.55850 (14)	0.0283 (3)
H16	0.2745 (11)	0.1595 (10)	0.6724 (19)	0.039 (4)*
C17	0.15643 (8)	0.16629 (8)	0.45380 (14)	0.0270 (2)
C18	0.11897 (9)	0.20627 (8)	0.30028 (15)	0.0325 (3)
H18	0.1624 (12)	0.2442 (11)	0.258 (2)	0.046 (4)*
C19	0.02375 (10)	0.19387 (9)	0.20915 (17)	0.0387 (3)
H19	−0.0003 (12)	0.2241 (11)	0.098 (2)	0.051 (5)*
C20	−0.03663 (9)	0.14166 (9)	0.26957 (17)	0.0377 (3)
H20	−0.1059 (13)	0.1325 (11)	0.205 (2)	0.050 (4)*
C21	−0.00080 (9)	0.10191 (9)	0.42071 (17)	0.0373 (3)
H21	−0.0449 (13)	0.0660 (11)	0.467 (2)	0.051 (4)*

C22	0.09561 (9)	0.11354 (9)	0.51326 (16)	0.0333 (3)
H22	0.1219 (11)	0.0868 (10)	0.6234 (19)	0.038 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0270 (4)	0.0408 (5)	0.0213 (4)	−0.0033 (3)	0.0051 (3)	−0.0017 (3)
N1	0.0290 (5)	0.0598 (8)	0.0297 (5)	−0.0158 (5)	0.0096 (4)	−0.0104 (5)
N2	0.0229 (5)	0.0392 (6)	0.0212 (5)	−0.0030 (4)	0.0019 (4)	0.0015 (4)
N3	0.0219 (5)	0.0362 (6)	0.0235 (5)	−0.0016 (4)	0.0034 (4)	−0.0018 (4)
C1	0.0235 (5)	0.0390 (7)	0.0261 (6)	−0.0033 (5)	0.0041 (4)	0.0003 (5)
C2	0.0309 (6)	0.0328 (6)	0.0253 (6)	−0.0003 (5)	0.0053 (5)	0.0017 (5)
C3	0.0282 (6)	0.0424 (7)	0.0281 (6)	0.0030 (5)	0.0076 (5)	0.0056 (5)
C4	0.0333 (7)	0.0454 (8)	0.0364 (7)	−0.0080 (6)	0.0085 (5)	0.0067 (6)
C5	0.0463 (8)	0.0333 (7)	0.0417 (7)	−0.0066 (6)	0.0092 (6)	0.0012 (6)
C6	0.0354 (7)	0.0362 (7)	0.0368 (7)	0.0037 (5)	0.0083 (5)	−0.0027 (5)
C7	0.0632 (10)	0.0346 (7)	0.0402 (8)	−0.0068 (7)	0.0189 (7)	−0.0025 (6)
C8	0.0463 (8)	0.0616 (10)	0.0453 (9)	0.0114 (8)	0.0229 (7)	0.0053 (7)
C9	0.0248 (5)	0.0366 (7)	0.0263 (6)	−0.0003 (5)	0.0034 (4)	−0.0009 (5)
C10	0.0292 (6)	0.0416 (7)	0.0306 (6)	−0.0046 (5)	−0.0005 (5)	−0.0013 (5)
C11	0.0416 (7)	0.0412 (7)	0.0268 (6)	−0.0047 (6)	−0.0030 (5)	−0.0025 (5)
C12	0.0467 (7)	0.0436 (8)	0.0234 (6)	−0.0018 (6)	0.0065 (5)	−0.0032 (5)
C13	0.0336 (6)	0.0367 (7)	0.0260 (6)	−0.0004 (5)	0.0074 (5)	−0.0008 (5)
C14	0.0246 (5)	0.0329 (6)	0.0226 (5)	0.0012 (5)	0.0037 (4)	−0.0007 (4)
C15	0.0205 (5)	0.0326 (6)	0.0237 (5)	0.0029 (4)	0.0050 (4)	0.0003 (4)
C16	0.0260 (6)	0.0352 (6)	0.0236 (5)	0.0009 (5)	0.0066 (4)	0.0004 (5)
C17	0.0251 (6)	0.0307 (6)	0.0252 (5)	0.0008 (5)	0.0070 (4)	−0.0030 (4)
C18	0.0300 (6)	0.0369 (7)	0.0292 (6)	0.0000 (5)	0.0057 (5)	0.0012 (5)
C19	0.0330 (7)	0.0451 (8)	0.0326 (7)	0.0035 (6)	0.0001 (5)	0.0003 (6)
C20	0.0244 (6)	0.0447 (8)	0.0402 (7)	0.0007 (5)	0.0028 (5)	−0.0106 (6)
C21	0.0298 (6)	0.0425 (7)	0.0411 (7)	−0.0077 (6)	0.0123 (5)	−0.0075 (6)
C22	0.0306 (6)	0.0381 (7)	0.0306 (6)	−0.0049 (5)	0.0076 (5)	−0.0009 (5)

Geometric parameters (Å, º)

O1—C15	1.2424 (14)	C9—C10	1.4115 (17)
N1—C9	1.3707 (16)	C9—C14	1.4208 (17)
N1—C1	1.4189 (16)	C10—C11	1.3745 (19)
N1—H1	0.908 (18)	C10—H10	0.997 (16)
N2—C15	1.3607 (16)	C11—C12	1.389 (2)
N2—N3	1.3750 (13)	C11—H11	0.990 (16)
N2—H2	0.913 (16)	C12—C13	1.3770 (18)
N3—C16	1.2814 (16)	C12—H12	0.988 (17)
C1—C6	1.3954 (19)	C13—C14	1.4033 (16)
C1—C2	1.3987 (17)	C13—H13	0.965 (16)
C2—C3	1.4068 (17)	C14—C15	1.4796 (15)
C2—C7	1.5034 (19)	C16—C17	1.4659 (16)
C3—C4	1.387 (2)	C16—H16	1.000 (15)

C3—C8	1.5030 (19)	C17—C22	1.3941 (17)
C4—C5	1.381 (2)	C17—C18	1.3966 (17)
C4—H4	1.001 (17)	C18—C19	1.3793 (18)
C5—C6	1.382 (2)	C18—H18	0.997 (17)
C5—H5	1.026 (18)	C19—C20	1.389 (2)
C6—H6	0.980 (18)	C19—H19	1.013 (18)
C7—H7A	0.97 (3)	C20—C21	1.376 (2)
C7—H7B	1.06 (3)	C20—H20	1.000 (18)
C7—H7C	0.99 (2)	C21—C22	1.3952 (18)
C8—H8A	1.04 (2)	C21—H21	1.004 (18)
C8—H8B	1.01 (2)	C22—H22	0.988 (15)
C8—H8C	0.98 (2)		

C9—N1—C1	126.50 (11)	C11—C10—C9	121.30 (12)
C9—N1—H1	114.2 (11)	C11—C10—H10	120.4 (9)
C1—N1—H1	118.4 (11)	C9—C10—H10	118.3 (9)
C15—N2—N3	118.21 (10)	C10—C11—C12	121.11 (12)
C15—N2—H2	122.4 (10)	C10—C11—H11	118.3 (9)
N3—N2—H2	119.3 (10)	C12—C11—H11	120.6 (9)
C16—N3—N2	115.55 (10)	C13—C12—C11	118.77 (12)
C6—C1—C2	120.68 (11)	C13—C12—H12	120.0 (10)
C6—C1—N1	120.10 (12)	C11—C12—H12	121.2 (10)
C2—C1—N1	119.17 (12)	C12—C13—C14	121.83 (12)
C1—C2—C3	118.60 (12)	C12—C13—H13	119.5 (9)
C1—C2—C7	120.96 (12)	C14—C13—H13	118.6 (9)
C3—C2—C7	120.44 (12)	C13—C14—C9	119.28 (11)
C4—C3—C2	119.79 (12)	C13—C14—C15	119.84 (11)
C4—C3—C8	118.91 (13)	C9—C14—C15	120.85 (10)
C2—C3—C8	121.30 (13)	O1—C15—N2	120.95 (10)
C5—C4—C3	121.08 (12)	O1—C15—C14	123.08 (10)
C5—C4—H4	121.5 (10)	N2—C15—C14	115.96 (10)
C3—C4—H4	117.4 (10)	N3—C16—C17	119.96 (11)
C4—C5—C6	119.84 (13)	N3—C16—H16	122.5 (9)
C4—C5—H5	121.3 (9)	C17—C16—H16	117.3 (9)
C6—C5—H5	118.8 (10)	C22—C17—C18	118.65 (11)
C5—C6—C1	119.96 (12)	C22—C17—C16	119.98 (11)
C5—C6—H6	120.4 (10)	C18—C17—C16	121.12 (11)
C1—C6—H6	119.6 (10)	C19—C18—C17	120.75 (12)
C2—C7—H7A	111.1 (15)	C19—C18—H18	120.5 (9)
C2—C7—H7B	111.2 (13)	C17—C18—H18	118.7 (9)
H7A—C7—H7B	103.8 (19)	C18—C19—C20	120.31 (12)
C2—C7—H7C	112.7 (12)	C18—C19—H19	118.0 (10)
H7A—C7—H7C	109.5 (19)	C20—C19—H19	121.7 (10)
H7B—C7—H7C	108.2 (18)	C21—C20—C19	119.62 (12)
C3—C8—H8A	110.6 (11)	C21—C20—H20	119.3 (10)
C3—C8—H8B	111.4 (12)	C19—C20—H20	121.0 (10)
H8A—C8—H8B	110.3 (15)	C20—C21—C22	120.51 (12)
C3—C8—H8C	111.8 (13)	C20—C21—H21	119.9 (10)

H8A—C8—H8C	104.6 (17)	C22—C21—H21	119.6 (10)
H8B—C8—H8C	107.9 (18)	C17—C22—C21	120.16 (12)
N1—C9—C10	121.87 (11)	C17—C22—H22	118.3 (9)
N1—C9—C14	120.49 (10)	C21—C22—H22	121.5 (9)
C10—C9—C14	117.58 (11)		

C15—N2—N3—C16	−179.78 (11)	C12—C13—C14—C9	−2.03 (19)
C9—N1—C1—C6	−49.49 (19)	C12—C13—C14—C15	−179.98 (12)
C9—N1—C1—C2	132.87 (14)	N1—C9—C14—C13	−178.39 (12)
C6—C1—C2—C3	1.20 (17)	C10—C9—C14—C13	4.26 (18)
N1—C1—C2—C3	178.83 (11)	N1—C9—C14—C15	−0.46 (18)
C6—C1—C2—C7	−178.68 (12)	C10—C9—C14—C15	−177.81 (11)
N1—C1—C2—C7	−1.05 (18)	N3—N2—C15—O1	−18.17 (17)
C1—C2—C3—C4	0.75 (18)	N3—N2—C15—C14	161.30 (10)
C7—C2—C3—C4	−179.36 (12)	C13—C14—C15—O1	156.06 (12)
C1—C2—C3—C8	−178.75 (12)	C9—C14—C15—O1	−21.86 (18)
C7—C2—C3—C8	1.13 (19)	C13—C14—C15—N2	−23.40 (16)
C2—C3—C4—C5	−1.70 (19)	C9—C14—C15—N2	158.68 (11)
C8—C3—C4—C5	177.82 (13)	N2—N3—C16—C17	169.95 (10)
C3—C4—C5—C6	0.7 (2)	N3—C16—C17—C22	−175.28 (12)
C4—C5—C6—C1	1.3 (2)	N3—C16—C17—C18	−1.04 (18)
C2—C1—C6—C5	−2.25 (19)	C22—C17—C18—C19	−0.02 (19)
N1—C1—C6—C5	−179.86 (12)	C16—C17—C18—C19	−174.33 (12)
C1—N1—C9—C10	−7.4 (2)	C17—C18—C19—C20	0.4 (2)
C1—N1—C9—C14	175.32 (12)	C18—C19—C20—C21	−0.3 (2)
N1—C9—C10—C11	178.76 (13)	C19—C20—C21—C22	−0.1 (2)
C14—C9—C10—C11	−3.9 (2)	C18—C17—C22—C21	−0.45 (19)
C9—C10—C11—C12	1.2 (2)	C16—C17—C22—C21	173.93 (12)
C10—C11—C12—C13	1.2 (2)	C20—C21—C22—C17	0.5 (2)
C11—C12—C13—C14	−0.7 (2)		

Hydrogen-bond geometry (Å, º)

*Cg*1 and *Cg*3 are the centroids of the C1–C6 and C17–C22 benzene rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.908 (18)	1.946 (18)	2.6920 (13)	138.2 (15)
N2—H2···O1^{i}	0.913 (16)	1.974 (17)	2.8564 (13)	162.0 (14)
C4—H4···*Cg*3^{ii}	1.001 (17)	2.796 (17)	3.6141 (15)	139.4 (13)
C6—H6···O1^{iii}	0.980 (18)	2.583 (19)	3.4815 (16)	152.5 (13)
C20—H20···*Cg*1^{iv}	1.000 (18)	2.838 (17)	3.6644 (15)	140.5 (13)

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) *x*−1, −*y*−1/2, *z*−3/2.