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Ethyl 4-anilino-3-nitrobenzoate

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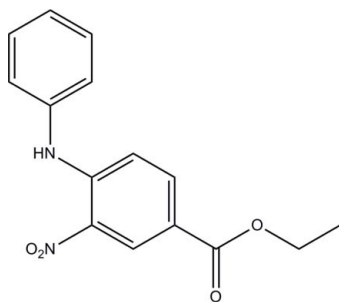
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.047; wR factor = 0.124; data-to-parameter ratio = 23.8.

In the title compound, $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_4$, the dihedral angle between the benzene and phenyl rings is $73.20(6)^\circ$. An intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond forms an $S(6)$ ring motif. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds into a layer parallel to the bc plane.

Related literature

For applications of nitrophenyleneamines, see: Stephane (2006); Glebowska *et al.* (2009); Remusat *et al.* (2004). For related structures, see: Mohd. Maidin *et al.* (2008); Zhang *et al.* (2009). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_4$ $M_r = 286.28$ Monoclinic, $P2_1/c$ $a = 10.6464(2)$ Å $b = 9.9178(2)$ Å $c = 14.7885(2)$ Å $\beta = 120.244(1)^\circ$ $V = 1348.96(4)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹

$T = 100$ K
 $0.35 \times 0.20 \times 0.16$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.965$, $T_{\max} = 0.984$

17988 measured reflections
4639 independent reflections
3276 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.124$
 $S = 1.04$
4639 reflections
195 parameters

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\text{max}} = 0.39$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{O4}$	0.859 (18)	2.00 (2)	2.6375 (17)	130.6 (17)
$\text{N1}-\text{H1N1}\cdots\text{O2}^i$	0.858 (17)	2.288 (16)	2.9411 (14)	133.0 (14)
$\text{C15}-\text{H15A}\cdots\text{O2}^{ii}$	0.93	2.45	3.342 (2)	160

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5128).

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Ethyl 4-anilino-3-nitrobenzoate

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S1. Comment

In chemistry, nitrophenyleneamines are important building blocks for many pharmaceutical compounds. Phenylenediamine themselves are also used as composition in making dyes (Stephane, 2006), metallomesogens (Glebowska *et al.*, 2009) as well as ligand precursors. Condensation of substituted *o*-phenylenediamine with various diketones is then used in the preparation of a variety of pharmaceuticals (Remusat *et al.*, 2004).

In the molecular structure (Fig. 1), an intramolecular N1—H1N1···O4 hydrogen bond generates an *S*(6) ring motif (Bernstein *et al.*, 1995). The C1—C6 benzene ring [maximum deviation of 0.007 (1) Å at atoms C5 and C6] and C10—C15 phenyl ring [maximum deviation of 0.005 (2) Å at atom C12] make a dihedral angle of 73.20 (6)° with each other. Bond lengths and angles are within normal ranges and are comparable to related structures (Mohd. Maidin *et al.*, 2008; Zhang *et al.*, 2009).

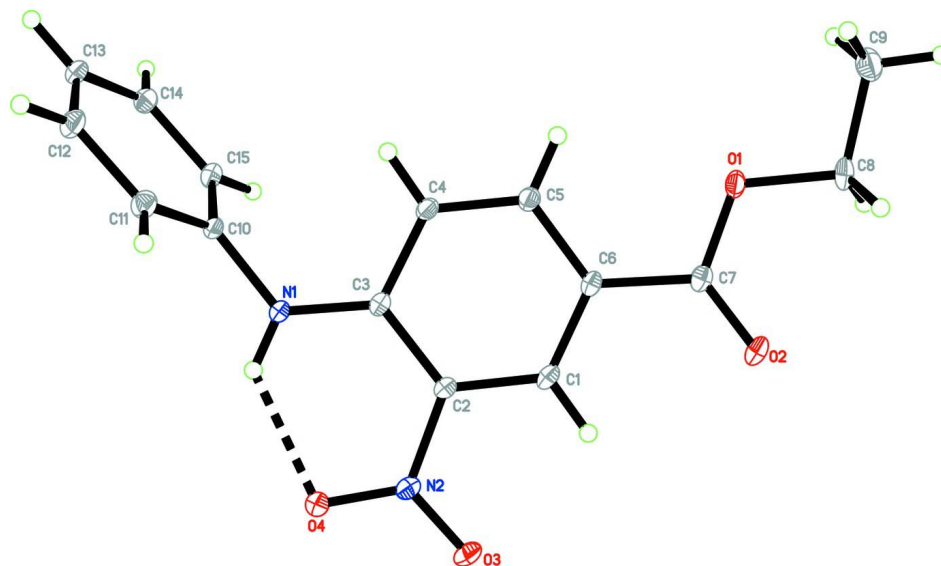
The crystal packing is shown in Fig. 2. The intermolecular N1—H1N1···O2 and C15—H15A···O2 (Table 1) hydrogen bonds linked the molecules into a two-dimensional network parallel to the *bc* plane.

S2. Experimental

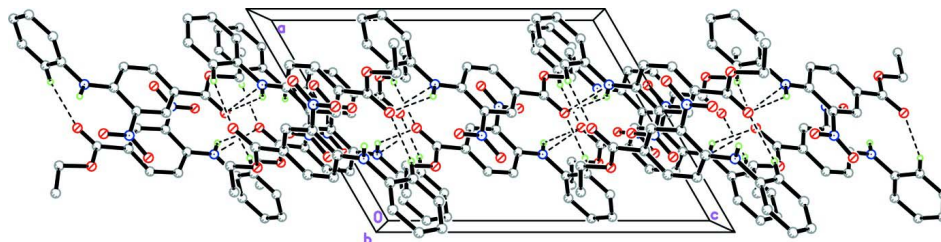
Ethyl-4-fluro-3-nitro benzoate (1 g) in dichloromethane (20 ml) was added into the solution of aniline (0.51 g) and *N,N*-diisopropylethylamine (0.72 g) in dichloromethane (20 ml). The reaction mixture was stirred overnight at room temperature. After completion of the reaction, evidenced by TLC analysis. The reaction mixture was washed with water (10 ml × 2) and 10% Na₂CO₃ (10 ml × 2). The dichloromethane layer was collected and dried over Na₂SO₄ and evaporated in vacuo to yield the product. The product was recrystallised from ethyl acetate.

S3. Refinement

N-bound H atom was located in a difference Fourier map and refined freely [N—H = 0.858 (17) Å]. Other H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$. A rotating group model was applied to the methyl group.

**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. The intramolecular N—H···O hydrogen bond is shown by a dashed line.

**Figure 2**

A crystal packing diagram of the title compound, viewed along the *b* axis. The H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

Ethyl 4-anilino-3-nitrobenzoate

Crystal data

$C_{15}H_{14}N_2O_4$

$M_r = 286.28$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 10.6464(2)\ \text{\AA}$

$b = 9.9178(2)\ \text{\AA}$

$c = 14.7885(2)\ \text{\AA}$

$\beta = 120.244(1)^\circ$

$V = 1348.96(4)\ \text{\AA}^3$

$Z = 4$

$F(000) = 600$

$D_x = 1.410\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3629 reflections

$\theta = 2.9\text{--}31.4^\circ$

$\mu = 0.10\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Block, yellow

$0.35 \times 0.20 \times 0.16\ \text{mm}$

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.965$, $T_{\max} = 0.984$

17988 measured reflections
 4639 independent reflections
 3276 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

$\theta_{\text{max}} = 31.9^\circ$, $\theta_{\text{min}} = 2.2^\circ$
 $h = -15 \rightarrow 15$
 $k = -14 \rightarrow 14$
 $l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.124$
 $S = 1.04$
 4639 reflections
 195 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.2709P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.26 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.28632 (10)	-0.12818 (8)	0.76247 (7)	0.01976 (19)
O2	0.46453 (10)	0.00161 (9)	0.76921 (7)	0.0212 (2)
O3	0.66947 (10)	0.34436 (10)	1.03221 (8)	0.0273 (2)
O4	0.55970 (10)	0.42674 (9)	1.10945 (8)	0.0228 (2)
N1	0.33488 (11)	0.29963 (10)	1.09858 (8)	0.0181 (2)
N2	0.56820 (11)	0.34349 (10)	1.05006 (8)	0.0184 (2)
C1	0.46344 (13)	0.16060 (11)	0.92632 (9)	0.0165 (2)
H1A	0.5353	0.1764	0.9095	0.020*
C2	0.45671 (12)	0.24143 (11)	1.00074 (9)	0.0154 (2)
C3	0.34771 (13)	0.22195 (11)	1.02854 (9)	0.0153 (2)
C4	0.24935 (13)	0.11392 (12)	0.97700 (9)	0.0169 (2)
H4A	0.1767	0.0968	0.9929	0.020*
C5	0.25813 (13)	0.03416 (12)	0.90452 (10)	0.0173 (2)
H5A	0.1924	-0.0362	0.8730	0.021*
C6	0.36523 (13)	0.05748 (11)	0.87723 (9)	0.0161 (2)
C7	0.37913 (13)	-0.02319 (12)	0.79882 (10)	0.0173 (2)
C8	0.29118 (15)	-0.20792 (13)	0.68146 (11)	0.0239 (3)
H8A	0.3826	-0.2563	0.7107	0.029*

H8B	0.2825	-0.1497	0.6259	0.029*
C9	0.16782 (18)	-0.30447 (14)	0.63980 (12)	0.0324 (3)
H9A	0.1677	-0.3582	0.5858	0.049*
H9B	0.0780	-0.2555	0.6114	0.049*
H9C	0.1780	-0.3620	0.6953	0.049*
C10	0.23046 (13)	0.27774 (12)	1.13077 (10)	0.0175 (2)
C11	0.11792 (14)	0.37011 (13)	1.10012 (11)	0.0227 (3)
H11A	0.1090	0.4418	1.0567	0.027*
C12	0.01887 (15)	0.35537 (14)	1.13428 (12)	0.0281 (3)
H12A	-0.0559	0.4176	1.1143	0.034*
C13	0.03126 (15)	0.24804 (14)	1.19810 (12)	0.0287 (3)
H13A	-0.0359	0.2374	1.2202	0.034*
C14	0.14412 (16)	0.15626 (14)	1.22907 (12)	0.0269 (3)
H14A	0.1528	0.0846	1.2724	0.032*
C15	0.24447 (15)	0.17090 (12)	1.19555 (11)	0.0218 (3)
H15A	0.3202	0.1095	1.2165	0.026*
H1N1	0.3899 (19)	0.3693 (16)	1.1227 (13)	0.030 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0247 (5)	0.0208 (4)	0.0188 (5)	0.0008 (3)	0.0147 (4)	-0.0030 (3)
O2	0.0237 (5)	0.0249 (4)	0.0222 (5)	0.0049 (3)	0.0169 (4)	0.0034 (4)
O3	0.0211 (5)	0.0350 (5)	0.0342 (6)	-0.0065 (4)	0.0202 (4)	-0.0053 (4)
O4	0.0259 (5)	0.0229 (4)	0.0250 (5)	-0.0052 (4)	0.0169 (4)	-0.0054 (4)
N1	0.0189 (5)	0.0194 (5)	0.0218 (6)	-0.0037 (4)	0.0146 (4)	-0.0049 (4)
N2	0.0176 (5)	0.0211 (5)	0.0193 (5)	-0.0010 (4)	0.0113 (4)	0.0013 (4)
C1	0.0172 (5)	0.0188 (5)	0.0176 (6)	0.0039 (4)	0.0119 (5)	0.0048 (4)
C2	0.0151 (5)	0.0172 (5)	0.0163 (6)	0.0000 (4)	0.0096 (5)	0.0011 (4)
C3	0.0153 (5)	0.0176 (5)	0.0153 (6)	0.0018 (4)	0.0092 (4)	0.0011 (4)
C4	0.0157 (5)	0.0211 (5)	0.0172 (6)	-0.0009 (4)	0.0107 (5)	-0.0005 (4)
C5	0.0176 (5)	0.0191 (5)	0.0167 (6)	-0.0001 (4)	0.0098 (5)	-0.0005 (4)
C6	0.0182 (5)	0.0187 (5)	0.0146 (6)	0.0036 (4)	0.0107 (5)	0.0025 (4)
C7	0.0195 (6)	0.0183 (5)	0.0162 (6)	0.0050 (4)	0.0106 (5)	0.0037 (4)
C8	0.0314 (7)	0.0249 (6)	0.0208 (7)	0.0044 (5)	0.0171 (6)	-0.0038 (5)
C9	0.0428 (9)	0.0279 (7)	0.0307 (8)	-0.0050 (6)	0.0217 (7)	-0.0078 (6)
C10	0.0157 (5)	0.0222 (5)	0.0183 (6)	-0.0038 (4)	0.0113 (5)	-0.0063 (5)
C11	0.0181 (6)	0.0273 (6)	0.0227 (7)	0.0004 (5)	0.0104 (5)	-0.0031 (5)
C12	0.0181 (6)	0.0356 (7)	0.0334 (8)	-0.0002 (5)	0.0151 (6)	-0.0101 (6)
C13	0.0256 (7)	0.0362 (7)	0.0356 (8)	-0.0118 (6)	0.0239 (6)	-0.0167 (6)
C14	0.0348 (8)	0.0266 (6)	0.0314 (8)	-0.0086 (5)	0.0256 (7)	-0.0073 (6)
C15	0.0245 (6)	0.0217 (6)	0.0259 (7)	-0.0012 (5)	0.0176 (6)	-0.0033 (5)

Geometric parameters (Å, °)

O1—C7	1.3470 (15)	C6—C7	1.4764 (16)
O1—C8	1.4582 (14)	C8—C9	1.485 (2)
O2—C7	1.2165 (13)	C8—H8A	0.9700

O3—N2	1.2325 (12)	C8—H8B	0.9700
O4—N2	1.2415 (13)	C9—H9A	0.9600
N1—C3	1.3518 (14)	C9—H9B	0.9600
N1—C10	1.4293 (14)	C9—H9C	0.9600
N1—H1N1	0.858 (17)	C10—C15	1.3852 (17)
N2—C2	1.4468 (15)	C10—C11	1.3904 (17)
C1—C6	1.3789 (17)	C11—C12	1.3875 (17)
C1—C2	1.3923 (15)	C11—H11A	0.9300
C1—H1A	0.9300	C12—C13	1.384 (2)
C2—C3	1.4262 (15)	C12—H12A	0.9300
C3—C4	1.4218 (16)	C13—C14	1.388 (2)
C4—C5	1.3727 (16)	C13—H13A	0.9300
C4—H4A	0.9300	C14—C15	1.3935 (17)
C5—C6	1.4071 (15)	C14—H14A	0.9300
C5—H5A	0.9300	C15—H15A	0.9300
C7—O1—C8	115.13 (9)	O1—C8—H8A	110.2
C3—N1—C10	124.35 (10)	C9—C8—H8A	110.2
C3—N1—H1N1	117.8 (11)	O1—C8—H8B	110.2
C10—N1—H1N1	117.8 (11)	C9—C8—H8B	110.2
O3—N2—O4	122.02 (11)	H8A—C8—H8B	108.5
O3—N2—C2	118.75 (10)	C8—C9—H9A	109.5
O4—N2—C2	119.23 (9)	C8—C9—H9B	109.5
C6—C1—C2	121.06 (10)	H9A—C9—H9B	109.5
C6—C1—H1A	119.5	C8—C9—H9C	109.5
C2—C1—H1A	119.5	H9A—C9—H9C	109.5
C1—C2—C3	121.55 (11)	H9B—C9—H9C	109.5
C1—C2—N2	116.46 (10)	C15—C10—C11	120.29 (11)
C3—C2—N2	121.97 (10)	C15—C10—N1	121.04 (11)
N1—C3—C4	120.59 (10)	C11—C10—N1	118.58 (11)
N1—C3—C2	123.68 (11)	C12—C11—C10	120.00 (13)
C4—C3—C2	115.72 (10)	C12—C11—H11A	120.0
C5—C4—C3	122.11 (10)	C10—C11—H11A	120.0
C5—C4—H4A	118.9	C13—C12—C11	120.03 (13)
C3—C4—H4A	118.9	C13—C12—H12A	120.0
C4—C5—C6	120.86 (11)	C11—C12—H12A	120.0
C4—C5—H5A	119.6	C12—C13—C14	119.90 (11)
C6—C5—H5A	119.6	C12—C13—H13A	120.0
C1—C6—C5	118.69 (10)	C14—C13—H13A	120.0
C1—C6—C7	117.78 (10)	C13—C14—C15	120.36 (13)
C5—C6—C7	123.53 (11)	C13—C14—H14A	119.8
O2—C7—O1	122.94 (11)	C15—C14—H14A	119.8
O2—C7—C6	124.18 (11)	C10—C15—C14	119.41 (12)
O1—C7—C6	112.88 (9)	C10—C15—H15A	120.3
O1—C8—C9	107.49 (10)	C14—C15—H15A	120.3
C6—C1—C2—C3	-0.58 (18)	C4—C5—C6—C7	-179.04 (11)
C6—C1—C2—N2	177.85 (11)	C8—O1—C7—O2	-2.29 (17)

O3—N2—C2—C1	-7.34 (16)	C8—O1—C7—C6	177.49 (10)
O4—N2—C2—C1	173.15 (11)	C1—C6—C7—O2	-5.67 (18)
O3—N2—C2—C3	171.07 (11)	C5—C6—C7—O2	174.66 (12)
O4—N2—C2—C3	-8.44 (17)	C1—C6—C7—O1	174.56 (10)
C10—N1—C3—C4	3.33 (18)	C5—C6—C7—O1	-5.11 (17)
C10—N1—C3—C2	-177.07 (11)	C7—O1—C8—C9	-171.43 (11)
C1—C2—C3—N1	-178.51 (11)	C3—N1—C10—C15	72.36 (17)
N2—C2—C3—N1	3.15 (18)	C3—N1—C10—C11	-110.92 (14)
C1—C2—C3—C4	1.12 (17)	C15—C10—C11—C12	-0.1 (2)
N2—C2—C3—C4	-177.22 (10)	N1—C10—C11—C12	-176.86 (12)
N1—C3—C4—C5	179.17 (12)	C10—C11—C12—C13	-0.6 (2)
C2—C3—C4—C5	-0.47 (17)	C11—C12—C13—C14	0.9 (2)
C3—C4—C5—C6	-0.72 (19)	C12—C13—C14—C15	-0.5 (2)
C2—C1—C6—C5	-0.65 (18)	C11—C10—C15—C14	0.49 (19)
C2—C1—C6—C7	179.67 (11)	N1—C10—C15—C14	177.15 (12)
C4—C5—C6—C1	1.29 (18)	C13—C14—C15—C10	-0.2 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N1 \cdots O4	0.859 (18)	2.00 (2)	2.6375 (17)	130.6 (17)
N1—H1N1 \cdots O2 ⁱ	0.858 (17)	2.288 (16)	2.9411 (14)	133.0 (14)
C15—H15A \cdots O2 ⁱⁱ	0.93	2.45	3.342 (2)	160

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