Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-2-[2-(4-Fluorobenzylidene)-hydrazinocarbonyl]-*N*-isopropylbenzamide

Ming Liu,[a]* Yousheng Duan,[a] Yi Wang,[a] Wen-Xiong Zhang[b] and Shangzhong Liu[a]

[a]Department of Applied Chemistry, China Agriculture University, 100193 Beijing, People's Republic of China, and [b]College of Chemistry and Molecular Engineering, Peking University, 100871 Beijing, People's Republic of China
Correspondence e-mail: shangzho@cau.edu.cn

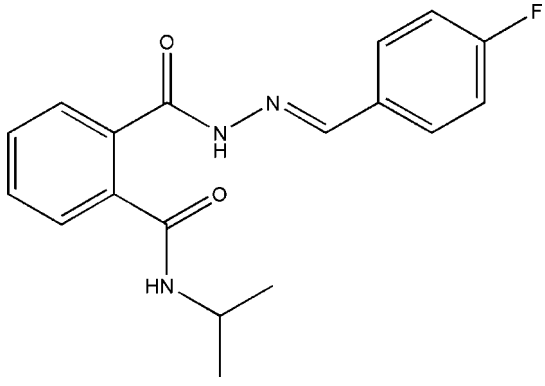
Received 28 March 2009; accepted 10 June 2009

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.069; data-to-parameter ratio = 16.8.

The title compound, $C_{18}H_{18}FN_3O_2$, adopts a *trans* conformation with respect to the C=N double bond. The dihedral angle between the two benzene rings is: 59.73 (6)°. Two independent N—H···O hydrogen bonds link the molecules into layers parallel to (101).

Related literature

For biologically active phthalic diamides, see: Coronado *et al.* (1994); Tohnishi *et al.* (2000). For the preparation of the title compound, see: Zaky (2002); Shigeru *et al.* (2003).



Experimental

Crystal data

$C_{18}H_{18}FN_3O_2$
M_r = 327.35
Monoclinic, $P2_1/n$
a = 13.316 (3) Å
b = 8.8904 (18) Å
c = 14.102 (3) Å
β = 91.10 (3)°
V = 1669.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 123 K
0.30 × 0.30 × 0.30 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi,1995)
T_{min} = 0.944, T_{max} = 0.972
15335 measured reflections
3833 independent reflections
2302 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.069
S = 1.02
3833 reflections
228 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1···O2^{i}	0.875 (15)	2.127 (15)	2.9887 (16)	168.4 (14)
N1—H2···O1^{ii}	0.850 (15)	1.976 (15)	2.8256 (16)	177.8 (15)

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

Data collection: *RAPID-AUTO* (Rigaku, 2000); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Molecular Structure Corporation and Rigaku, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This work was supported by the National Natural Science Foundation of China (NNSFC) (grant No. 20572129), National Basic Research Program of China (2003CB114405) and National Key Project of Scientific and Technical Supporting Programs Funded by Ministry of Science & Technology of China (No. 2006BAE01AE01–11).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YA2090).

References

Coronado, R., Morrissette, J., Sukhareva, M. & Vaughan, D. M. (1994). *Am. J. Physiol.* **266**, 1485–1504.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Molecular Structure Corporation and Rigaku (2000). *CrystalStructure*. MSC, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Rigaku (2000). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shigeru, N., Takeshi, S., Etsuko, M. & Yasuo, K. (2003). *Synth. Commun.* **33**, 87–98.
Tohnishi, M., Nakao, H., Kohno, E., Nishida, T., Furuya, T., Shimizu, T., Seo, A., Sakata, K., Fujioka, S. & Kanno, H. (2000). Eur. Patent No. EP1006107.
Zaky, H. T. (2002). *Heterocycl. Commun.* **8**, 355–360.

supporting information

Acta Cryst. (2009). E**65**, o1599 [doi:10.1107/S1600536809022181]

(*E*)-2-[2-(4-Fluorobenzylidene)hydrazinocarbonyl]-*N*-isopropylbenzamide

Ming Liu, Yousheng Duan, Yi Wang, Wen-Xiong Zhang and Shangzhong Liu

S1. Comment

Phthalic diamides possess insecticidal properties due to their ability to activate ryanodine receptor (Coronado *et al.*, 1994; Tohnishi *et al.*, 2000). The title compound (I), a new phthalic diamide derivative, was synthesized by the condensation of *N*-aminophthalimide with 4-fluorobenzaldehyde followed by a ring-opening reaction using isopropyl amine (Zaky, 2002; Shigeru *et al.*, 2003).

The molecular structure of the title compound is shown in Fig. 1. Molecule was proved to be a *trans* -isomer with respect to the C9=N2 double bond.

There are two independent N—H···O bonds (Table 1), which link molecules into the layers parallel to (101) plane (Fig. 2).

S2. Experimental

To a solution of *N*-aminophthalimide (1.62 g, 10 mmol) and 4-fluorobenzaldehyde (1.24 g, 10 mmol) in 1,4-dioxane (100 ml), 12 N HCl (0.1 ml) was added at room temperature. After stirring for 5–10 min, a solution of isopropyl amine (1.16 g, 20 mmol) in 1,4-dioxane (10 ml) was added; the reaction mixture was stirred overnight at room temperature. After the solvent was evaporated under reduced pressure, the resulting mixture was dissolved in ethyl acetate (80 ml), washed with H_2O (3×30 ml) and dried with anhydrous sodium sulfate to give the title compound (2.01 g, 61.5%). Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of ethanol solution at room temperature over one week.

S3. Refinement

The H atoms bound to N atoms were located in a difference Fourier map and refined isotropically [N—H 0.850 (15), 0.875 (15) Å]. The remaining H atoms were positioned geometrically and included in the refinement in riding model approximation with C—H 0.95 (aromatic), 0.98 (methyl), 1.00 (methyne), and U_{iso}(H) = 1.2U_{eq}(C)[1.5U_{eq}(C) for methyl H atoms].

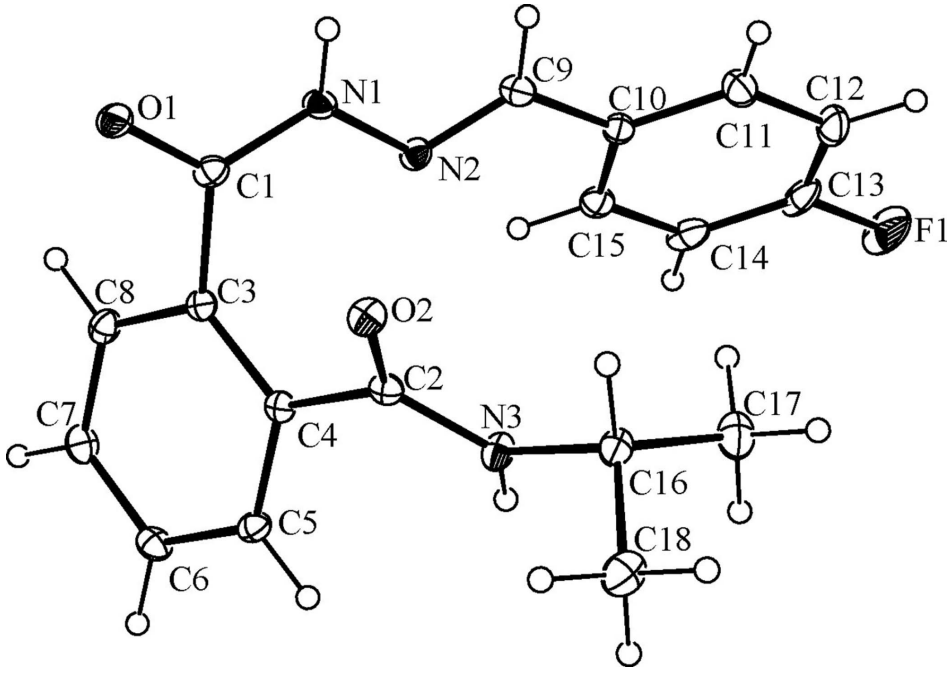


Figure 1

Molecular structure of (I); displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as small circles of arbitrary radius.

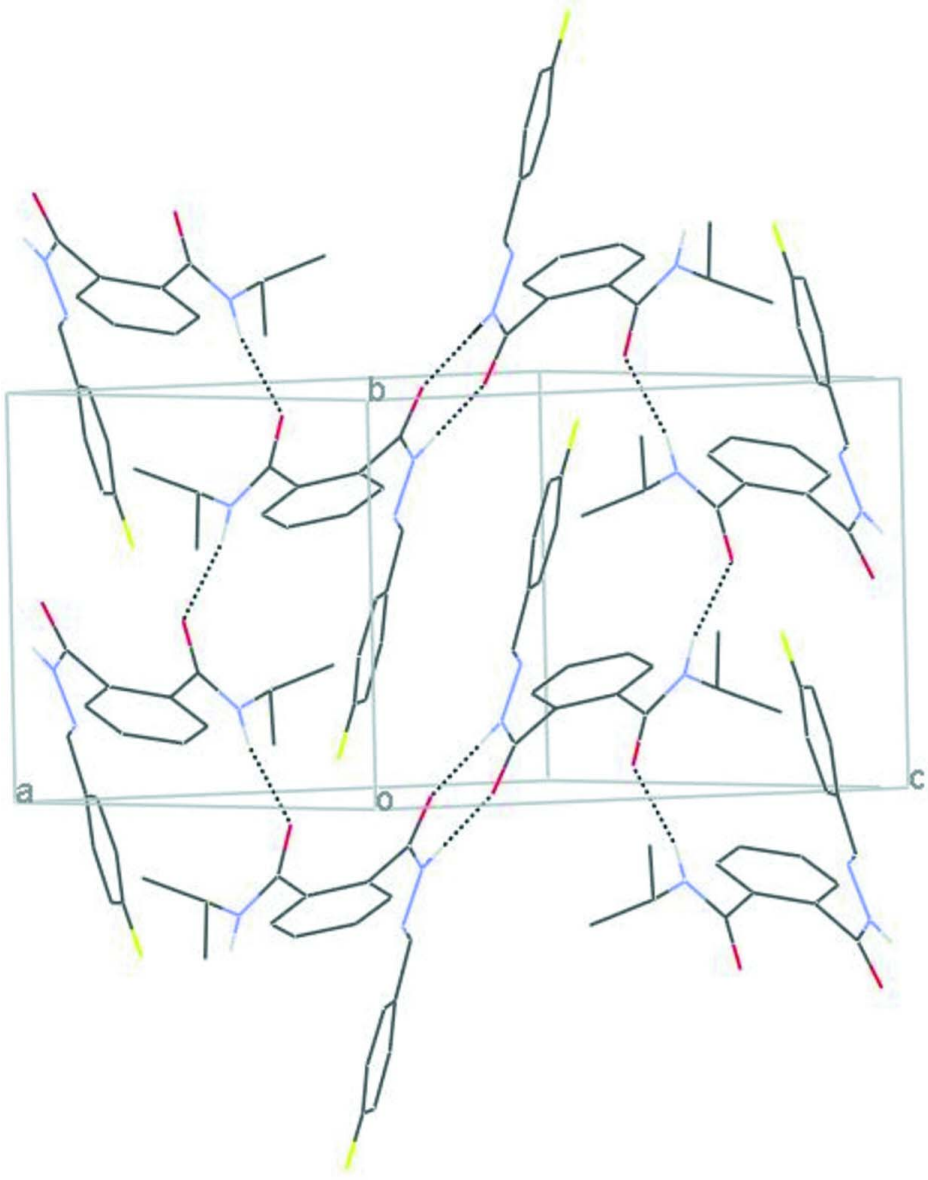


Figure 2

The crystal packing of (I) viewed along the [101] direction; hydrogen bonds are shown as dashed lines.

(*E*)-2-[2-(4-Fluorobenzylidene)hydrazinocarbonyl]-*N*- isopropylbenzamide

Crystal data

$C_{18}H_{18}FN_3O_2$
$M_r = 327.35$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 13.316\ (3)$ Å
$b = 8.8904\ (18)$ Å
$c = 14.102\ (3)$ Å
$\beta = 91.10\ (3)°$
$V = 1669.2\ (6)$ Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.303$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 15335 reflections
$\theta = 2.1–27.5°$
$\mu = 0.09$ mm^{-1}
$T = 123$ K
Block, colourless
0.30 × 0.30 × 0.30 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.00 pixels mm^{-1}
Ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi,1995)
T_{min} = 0.944, T_{max} = 0.972
15335 measured reflections
3833 independent reflections
2302 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −17→17
k = −11→11
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.069
S = 1.02
3833 reflections
228 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
$w = 1/[\sigma^2(F_o^2) + (0.015P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0285 (8)

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.62786 (10)	0.87434 (17)	0.48465 (10)	0.0197 (3)
C2	0.70864 (10)	0.80962 (16)	0.29308 (10)	0.0179 (3)
C3	0.72241 (10)	0.78555 (15)	0.47071 (10)	0.0168 (3)
C4	0.76143 (10)	0.75512 (15)	0.38154 (10)	0.0162 (3)
C5	0.85500 (10)	0.68578 (15)	0.37629 (10)	0.0197 (3)
H5	0.8819	0.6634	0.3160	0.024*
C6	0.90921 (11)	0.64909 (16)	0.45774 (11)	0.0228 (4)
H6	0.9739	0.6050	0.4531	0.027*
C7	0.86928 (10)	0.67651 (16)	0.54567 (11)	0.0236 (4)
H7	0.9057	0.6494	0.6017	0.028*
C8	0.77595 (10)	0.74366 (16)	0.55190 (10)	0.0216 (4)
H8	0.7482	0.7613	0.6124	0.026*
C9	0.44987 (11)	0.65483 (17)	0.36494 (10)	0.0222 (4)
H9	0.3990	0.7297	0.3628	0.027*

C10	0.43139 (10)	0.50842 (17)	0.32041 (10)	0.0215 (4)
C11	0.35688 (11)	0.49509 (19)	0.24980 (11)	0.0299 (4)
H11	0.3173	0.5803	0.2331	0.036*
C12	0.33975 (12)	0.3594 (2)	0.20378 (12)	0.0384 (5)
H12	0.2904	0.3510	0.1545	0.046*
C13	0.39601 (13)	0.2385 (2)	0.23155 (12)	0.0366 (5)
C14	0.46863 (12)	0.24391 (18)	0.30209 (11)	0.0307 (4)
H14	0.5053	0.1565	0.3202	0.037*
C15	0.48647 (11)	0.38099 (17)	0.34587 (11)	0.0237 (4)
H15	0.5371	0.3882	0.3940	0.028*
C16	0.66213 (10)	0.75633 (16)	0.12683 (10)	0.0221 (4)
H16	0.6111	0.8369	0.1373	0.027*
C17	0.60856 (12)	0.61917 (18)	0.08583 (11)	0.0326 (4)
H17A	0.6574	0.5385	0.0758	0.049*
H17B	0.5762	0.6458	0.0251	0.049*
H17C	0.5576	0.5848	0.1301	0.049*
C18	0.74105 (11)	0.81707 (18)	0.05991 (11)	0.0311 (4)
H18A	0.7736	0.9053	0.0886	0.047*
H18B	0.7087	0.8457	−0.0004	0.047*
H18C	0.7915	0.7392	0.0486	0.047*
F1	0.37958 (8)	0.10376 (12)	0.18747 (7)	0.0603 (4)
N1	0.53997 (9)	0.82179 (14)	0.44990 (9)	0.0204 (3)
N2	0.53387 (8)	0.68275 (13)	0.40673 (8)	0.0196 (3)
N3	0.70850 (9)	0.71718 (14)	0.21866 (9)	0.0214 (3)
O1	0.63114 (7)	0.99350 (11)	0.53105 (7)	0.0257 (3)
O2	0.66866 (7)	0.93655 (11)	0.29200 (7)	0.0217 (3)
H1	0.7381 (11)	0.6294 (17)	0.2210 (11)	0.038 (5)*
H2	0.4879 (11)	0.8765 (18)	0.4542 (10)	0.039 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0210 (8)	0.0190 (8)	0.0193 (9)	−0.0014 (7)	0.0050 (6)	0.0005 (7)
C2	0.0172 (8)	0.0152 (8)	0.0215 (9)	−0.0017 (7)	0.0023 (6)	0.0018 (7)
C3	0.0184 (7)	0.0108 (7)	0.0214 (8)	−0.0035 (6)	0.0021 (6)	−0.0015 (6)
C4	0.0178 (8)	0.0110 (7)	0.0197 (8)	−0.0028 (6)	0.0009 (6)	0.0006 (7)
C5	0.0210 (8)	0.0172 (8)	0.0210 (9)	−0.0002 (7)	0.0043 (6)	−0.0024 (7)
C6	0.0191 (8)	0.0187 (8)	0.0305 (10)	0.0035 (7)	−0.0017 (7)	0.0004 (7)
C7	0.0258 (8)	0.0215 (8)	0.0233 (9)	−0.0006 (7)	−0.0052 (7)	0.0030 (7)
C8	0.0260 (8)	0.0222 (8)	0.0168 (8)	−0.0028 (7)	0.0026 (6)	−0.0006 (7)
C9	0.0184 (8)	0.0221 (9)	0.0262 (9)	0.0009 (7)	0.0017 (7)	0.0001 (7)
C10	0.0181 (8)	0.0267 (9)	0.0197 (9)	−0.0052 (7)	0.0038 (6)	−0.0024 (7)
C11	0.0228 (9)	0.0406 (10)	0.0264 (10)	−0.0045 (8)	−0.0005 (7)	−0.0008 (8)
C12	0.0292 (10)	0.0578 (13)	0.0284 (11)	−0.0181 (10)	0.0031 (8)	−0.0149 (10)
C13	0.0406 (11)	0.0358 (11)	0.0339 (11)	−0.0211 (9)	0.0180 (8)	−0.0213 (9)
C14	0.0332 (10)	0.0252 (9)	0.0343 (10)	−0.0042 (8)	0.0161 (8)	−0.0049 (8)
C15	0.0219 (8)	0.0259 (9)	0.0235 (9)	−0.0045 (7)	0.0056 (7)	−0.0009 (8)
C16	0.0262 (9)	0.0204 (8)	0.0197 (9)	0.0081 (7)	−0.0045 (7)	−0.0010 (7)

C17	0.0368 (10)	0.0280 (9)	0.0326 (10)	0.0038 (8)	−0.0088 (8)	−0.0045 (8)
C18	0.0387 (10)	0.0294 (9)	0.0253 (10)	0.0072 (8)	0.0010 (7)	0.0025 (8)
F1	0.0638 (7)	0.0548 (7)	0.0630 (8)	−0.0279 (6)	0.0211 (6)	−0.0401 (6)
N1	0.0160 (7)	0.0170 (7)	0.0283 (8)	0.0009 (6)	0.0017 (6)	−0.0049 (6)
N2	0.0213 (7)	0.0164 (6)	0.0211 (7)	−0.0029 (6)	0.0028 (5)	−0.0033 (6)
N3	0.0283 (8)	0.0166 (7)	0.0192 (7)	0.0067 (6)	−0.0036 (6)	−0.0018 (6)
O1	0.0229 (6)	0.0202 (6)	0.0340 (7)	−0.0007 (5)	0.0037 (5)	−0.0103 (5)
O2	0.0267 (6)	0.0135 (5)	0.0247 (6)	0.0028 (5)	0.0004 (5)	0.0015 (5)

Geometric parameters (Å, º)

C1—O1	1.2455 (16)	C11—H11	0.9500
C1—N1	1.3438 (18)	C12—C13	1.363 (2)
C1—C3	1.5022 (19)	C12—H12	0.9500
C2—O2	1.2476 (16)	C13—F1	1.3656 (18)
C2—N3	1.3330 (18)	C13—C14	1.375 (2)
C2—C4	1.500 (2)	C14—C15	1.385 (2)
C3—C8	1.3879 (19)	C14—H14	0.9500
C3—C4	1.3964 (19)	C15—H15	0.9500
C4—C5	1.3934 (18)	C16—N3	1.4658 (18)
C5—C6	1.3836 (19)	C16—C17	1.521 (2)
C5—H5	0.9500	C16—C18	1.5247 (19)
C6—C7	1.380 (2)	C16—H16	1.0000
C6—H6	0.9500	C17—H17A	0.9800
C7—C8	1.3831 (18)	C17—H17B	0.9800
C7—H7	0.9500	C17—H17C	0.9800
C8—H8	0.9500	C18—H18A	0.9800
C9—N2	1.2786 (17)	C18—H18B	0.9800
C9—C10	1.464 (2)	C18—H18C	0.9800
C9—H9	0.9500	N1—N2	1.3796 (16)
C10—C15	1.393 (2)	N1—H2	0.850 (15)
C10—C11	1.3972 (19)	N3—H1	0.875 (15)
C11—C12	1.386 (2)		

O1—C1—N1	120.58 (13)	C11—C12—H12	121.1
O1—C1—C3	119.65 (13)	C12—C13—F1	118.60 (17)
N1—C1—C3	119.72 (13)	C12—C13—C14	123.75 (16)
O2—C2—N3	123.63 (14)	F1—C13—C14	117.65 (18)
O2—C2—C4	119.68 (13)	C13—C14—C15	117.74 (17)
N3—C2—C4	116.68 (13)	C13—C14—H14	121.1
C8—C3—C4	119.79 (13)	C15—C14—H14	121.1
C8—C3—C1	116.84 (13)	C14—C15—C10	121.07 (15)
C4—C3—C1	123.16 (13)	C14—C15—H15	119.5
C5—C4—C3	118.82 (13)	C10—C15—H15	119.5
C5—C4—C2	120.26 (13)	N3—C16—C17	109.39 (12)
C3—C4—C2	120.64 (12)	N3—C16—C18	110.32 (12)
C6—C5—C4	120.86 (14)	C17—C16—C18	111.86 (13)
C6—C5—H5	119.6	N3—C16—H16	108.4

C4—C5—H5	119.6	C17—C16—H16	108.4
C7—C6—C5	120.02 (14)	C18—C16—H16	108.4
C7—C6—H6	120.0	C16—C17—H17A	109.5
C5—C6—H6	120.0	C16—C17—H17B	109.5
C6—C7—C8	119.69 (14)	H17A—C17—H17B	109.5
C6—C7—H7	120.2	C16—C17—H17C	109.5
C8—C7—H7	120.2	H17A—C17—H17C	109.5
C7—C8—C3	120.76 (13)	H17B—C17—H17C	109.5
C7—C8—H8	119.6	C16—C18—H18A	109.5
C3—C8—H8	119.6	C16—C18—H18B	109.5
N2—C9—C10	120.58 (14)	H18A—C18—H18B	109.5
N2—C9—H9	119.7	C16—C18—H18C	109.5
C10—C9—H9	119.7	H18A—C18—H18C	109.5
C15—C10—C11	118.54 (15)	H18B—C18—H18C	109.5
C15—C10—C9	121.96 (14)	C1—N1—N2	121.06 (13)
C11—C10—C9	119.49 (14)	C1—N1—H2	118.7 (11)
C12—C11—C10	121.04 (16)	N2—N1—H2	120.2 (11)
C12—C11—H11	119.5	C9—N2—N1	114.89 (12)
C10—C11—H11	119.5	C2—N3—C16	122.92 (13)
C13—C12—C11	117.83 (16)	C2—N3—H1	121.8 (10)
C13—C12—H12	121.1	C16—N3—H1	115.3 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H1···O2^{i}	0.875 (15)	2.127 (15)	2.9887 (16)	168.4 (14)
N1—H2···O1^{ii}	0.850 (15)	1.976 (15)	2.8256 (16)	177.8 (15)

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