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$N^2,N^{2'}$-Bis(2,2-dimethylpropanoyl)-benzene-1,3-dicarbohydrazide

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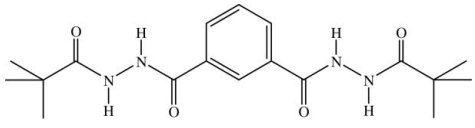
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.055; wR factor = 0.141; data-to-parameter ratio = 20.6.

In the molecular structure of the title hydrazide derivative, $C_{18}H_{26}N_4O_4$, the conformations of the two units of 2-(2,2-dimethyl-1-oxopropyl)hydrazide substituents are not planar; these two units are attached axially to the benzene ring with C(*ortho*)—C—C(=O)—N torsion angles of 28.1 (2) and 31.0 (2)° [where C(*ortho*) is the C atom at position 4 of the benzene ring relative to the substituent at position 3 or the C atom at position 6 of the benzene ring relative to the substituent at position 1, as appropriate]. The dihedral angles between the hydrazide units and the benzene ring are 62.66 (7) and 63.84 (7)°. In the crystal structure, molecules are arranged in an anti-parallel manner and are linked by N—H···O intermolecular hydrogen bonds and weak C—H···O intermolecular interactions into a three-dimensional network. The structure is further stabilized by a weak C—H···N intramolecular interaction.

Related literature

For values of bond lengths, see: Allen *et al.* (1987). For related literature on the applications and bioactivities of hydrazide derivatives, see for example: Feng *et al.* (2006); Fernández *et al.* (2004); Hołtra *et al.* (2007); Imramovský *et al.* (2007); Kim *et al.* (2007); Lemay *et al.* (2007); Liu *et al.* (2006); Nica *et al.* (2007); Raveendran & Pal (2007); Rivero & Buchwald (2007); Sicardi *et al.* (1980); Yang *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{26}N_4O_4$
M_r = 362.43
Monoclinic, $P2_1/c$
a = 7.1853 (2) Å
b = 14.8928 (4) Å
c = 17.1656 (5) Å
β = 96.050 (2)°
V = 1826.65 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100.0 (1) K
0.56 × 0.10 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.949, T_{max} = 0.993
34290 measured reflections
5301 independent reflections
3858 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.141
S = 1.06
5301 reflections
257 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···O3^{i}	0.90 (2)	2.12 (2)	3.0193 (16)	176.7 (16)
N2—H1*N*2···O4^{ii}	0.845 (18)	1.993 (19)	2.8262 (17)	169 (2)
N3—H1*N*3···O1^{iii}	0.876 (18)	1.969 (18)	2.8307 (16)	167.3 (16)
N4—H1*N*4···O2^{iv}	0.878 (19)	2.059 (19)	2.9320 (16)	172.3 (19)
C1—H1*A*···O4^{ii}	0.93	2.48	3.1879 (17)	133
C3—H3*A*···O1^{iii}	0.93	2.56	3.2854 (17)	135
C12—H12*C*···O3^{i}	0.96	2.52	3.433 (2)	159
C18—H18*C*···N4	0.96	2.61	2.9347 (19)	100

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2255).

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N^2,$N^{2'}$-Bis(2,2-dimethylpropanoyl)benzene-1,3-dicarbohydrazide

Hoong-Kun Fun, Suchada Chantrapromma, Subrata Jana, Anita Hazra and Shyamaprosad Goswami

S1. Comment

Hydrazide derivatives of different compounds are very important units in *host–guest* chemistry due to their special arrangement of donor-acceptors (Feng *et al.*, 2006; Yang *et al.*, 2007). These types of compounds are also important for the metal coordinations and related studies (Hołtra *et al.*, 2007; Nica *et al.*, 2007; Raveendran & Pal, 2007). Hydrazide-based compounds are also involved in different synthetic applications (Fernández *et al.*, 2004; Lemay *et al.*, 2007; Kim *et al.*, 2007; Rivero & Buchwald, 2007) as well as in medicinal activities (Imramovský *et al.*, 2007; Liu *et al.*, 2006; Sicardi *et al.*, 1980). We synthesized the title compound for being a host of *host–guest* complexes syntheses. The single-crystal X-ray structural study of the title compound was undertaken in order to establish the three-dimensional structure and to gain more details of conformations of the various groups.

In the molecular structure of the title compound (Fig. 1), the conformations of the two units of 2-(2,2-dimethyl-1-oxo-propyl)hydrazide substituents are not planar which can be indicated by the dihedral angles between the mean planes of C6/C7/O2/N2 and O1/N1/N2/C8/C9 = 87.77 (8)° and C4/C13/O3/N3 and O4/N3/N4/C14/C15 = 87.90 (8)°. These two units are axially attached to the benzene ring with the torsion angles C1–C6–C7–N2 = 28.1 (2)° and C3–C4–C13–N3 = 31.0 (2)°. The orientations of the two hydrazide moieties with respect to the benzene ring can be indicated by the dihedral angles between the mean planes of N1/N2/C8/C9 and N3/N4/C14/C15 and the benzene ring being 62.66 (7) and 63.84 (7)°, respectively. The torsion angles of N1–N2–C7–C6 = -165.58 (12)° and N4–N3–C13–C14 = -160.69 (12)° indicate that the two substituents are in (-)-anti-periplanar conformations. All bond lengths and angles are in normal values (Allen *et al.*, 1987).

In the crystal packing in Fig. 2, the molecules are arranged in an anti-parallel manner and linked by N—H···O intermolecular hydrogen bonds and weak C—H···O intermolecular interactions (Table 1) into three dimensional networks. The crystal is further stabilized by a weak C—H···N intramolecular interaction.

S2. Experimental

Initially isophthalic acid was converted to its methyl ester under refluxing condition with methanol and a catalytic amount of concentrated sulfuric acid. This ester was then refluxed with excess hydrazine hydrate and ethanol for three hours. After completion of the reaction, excess ethanol was evaporated out and the solid substance was washed well with water and dried under reduced pressure. The properly dried intermediate compound was treated with pivalic anhydride at 353 K for seven hours. The crude compound was extracted with chloroform after neutralizing the reaction mixture with aqueous sodium bicarbonate solution. The title compound was purified by column chromatography (Silica gel 100–200 mesh) using ethyl acetate as eluent to afford an off-white colored solid compound. Single crystals were grown by slow evaporation of $CHCl_3$/MeOH solution (*v*/*v* 1:1) (m.p. over 523 K).

S3. Refinement

Hydrazide H atoms were located in a difference map and isotropically refined. The remaining H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å for aromatic and 0.96 Å for CH_3. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups.

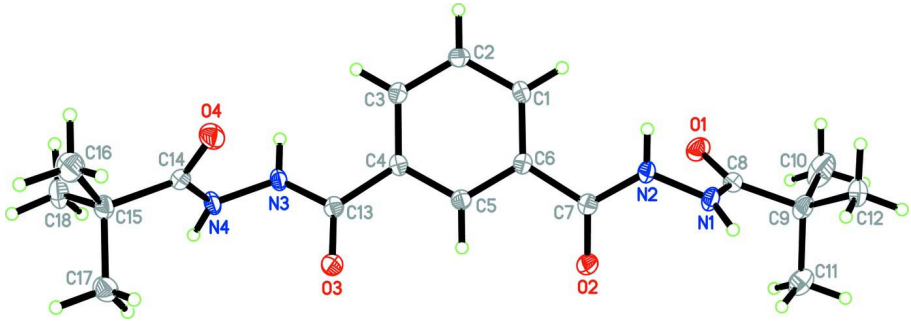

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering.

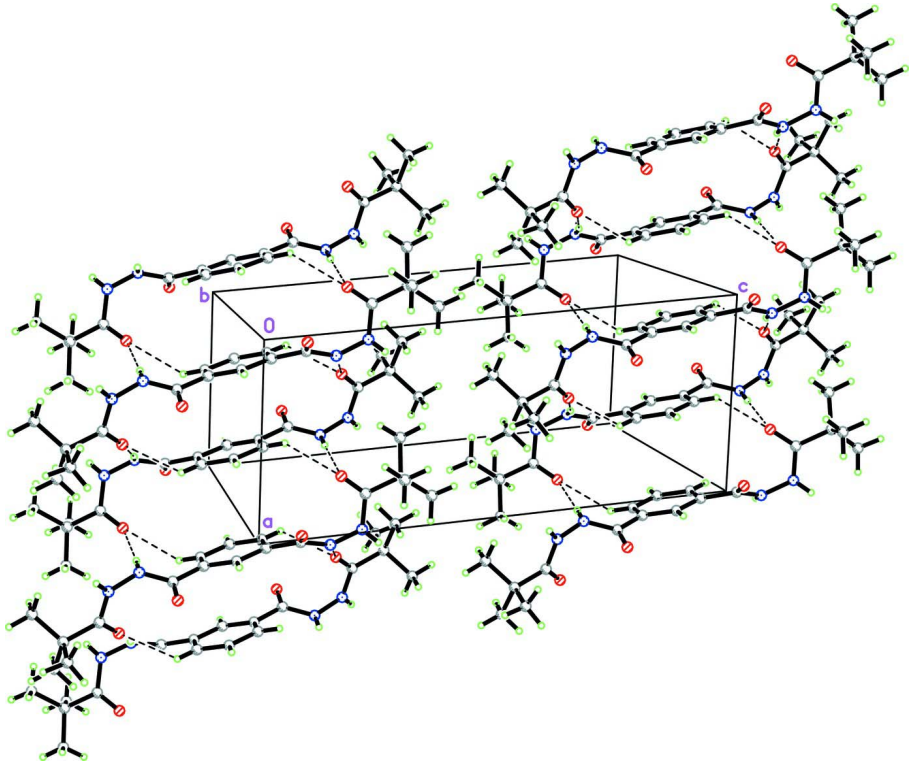

Figure 2

The crystal packing of the title compound. Hydrogen bonds were shown as dash lines.

N^2,$N^{2'}$—Bis(2,2-dimethylpropanoyl)benzene-1,3-dicarbohydrazide

Crystal data

$C_{18}H_{26}N_4O_4$
$M_r = 362.43$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.1853$ (2) Å
$b = 14.8928$ (4) Å
$c = 17.1656$ (5) Å
$\beta = 96.050$ (2)°
$V = 1826.65$ (9) Å^3
$Z = 4$
$F(000) = 776$
$D_x = 1.318$ Mg m^{-3}
Melting point: over 523 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5301 reflections
$\theta = 2.4$–30.0°
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Needle, colorless
0.56 × 0.10 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.33 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.949$, $T_{max} = 0.993$
34290 measured reflections
5301 independent reflections
3858 reflections with $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 30.0°$, $\theta_{min} = 2.4°$
$h = -10 \rightarrow 10$
$k = -20 \rightarrow 20$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.141$
$S = 1.06$
5301 reflections
257 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
$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 0.4134P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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}
O1	0.60769 (16)	0.05613 (7)	0.25206 (6)	0.0237 (3)
O2	0.38220 (15)	0.21786 (7)	0.36444 (6)	0.0184 (2)

O3	0.11704 (15)	0.22624 (7)	0.62797 (6)	0.0187 (2)
O4	−0.10708 (16)	0.07187 (7)	0.73429 (6)	0.0240 (3)
N1	0.33903 (19)	0.13157 (8)	0.22251 (7)	0.0184 (3)
N2	0.27315 (19)	0.09916 (9)	0.29066 (7)	0.0184 (3)
N3	0.23552 (18)	0.11418 (8)	0.70784 (7)	0.0172 (3)
N4	0.15571 (19)	0.14796 (8)	0.77239 (7)	0.0173 (3)
C1	0.2816 (2)	−0.00484 (9)	0.43413 (8)	0.0159 (3)
H1A	0.3021	−0.0379	0.3899	0.019*
C2	0.2495 (2)	−0.04858 (9)	0.50312 (8)	0.0171 (3)
H2A	0.2474	−0.1110	0.5046	0.021*
C3	0.2207 (2)	0.00018 (9)	0.56966 (8)	0.0157 (3)
H3A	0.1998	−0.0295	0.6156	0.019*
C4	0.2231 (2)	0.09396 (9)	0.56759 (7)	0.0142 (3)
C5	0.25445 (19)	0.13769 (9)	0.49847 (7)	0.0150 (3)
H5A	0.2563	0.2001	0.4969	0.018*
C6	0.2831 (2)	0.08878 (9)	0.43155 (7)	0.0143 (3)
C7	0.3208 (2)	0.14076 (9)	0.36016 (8)	0.0152 (3)
C8	0.5108 (2)	0.10397 (9)	0.20601 (8)	0.0177 (3)
C9	0.5743 (2)	0.13472 (10)	0.12760 (9)	0.0219 (3)
C10	0.7443 (3)	0.07870 (14)	0.11231 (11)	0.0404 (5)
H10A	0.8442	0.0897	0.1529	0.061*
H10B	0.7840	0.0950	0.0625	0.061*
H10C	0.7118	0.0162	0.1119	0.061*
C11	0.6266 (3)	0.23490 (11)	0.13166 (10)	0.0281 (4)
H11A	0.7375	0.2430	0.1673	0.042*
H11B	0.5257	0.2687	0.1496	0.042*
H11C	0.6494	0.2555	0.0805	0.042*
C12	0.4177 (3)	0.11974 (11)	0.06080 (9)	0.0270 (4)
H12A	0.3870	0.0570	0.0573	0.040*
H12B	0.4593	0.1394	0.0123	0.040*
H12C	0.3090	0.1534	0.0711	0.040*
C13	0.1843 (2)	0.15065 (9)	0.63621 (8)	0.0151 (3)
C14	−0.0226 (2)	0.12316 (9)	0.78142 (8)	0.0166 (3)
C15	−0.1087 (2)	0.16126 (10)	0.85232 (8)	0.0185 (3)
C16	−0.2991 (3)	0.11803 (12)	0.85580 (10)	0.0298 (4)
H16A	−0.2844	0.0542	0.8613	0.045*
H16B	−0.3778	0.1312	0.8085	0.045*
H16C	−0.3554	0.1414	0.8999	0.045*
C17	−0.1340 (2)	0.26337 (10)	0.84314 (9)	0.0242 (3)
H17A	−0.2175	0.2759	0.7970	0.036*
H17B	−0.0148	0.2908	0.8384	0.036*
H17C	−0.1853	0.2871	0.8883	0.036*
C18	0.0197 (2)	0.14110 (12)	0.92760 (8)	0.0259 (4)
H18A	0.0268	0.0774	0.9357	0.039*
H18B	−0.0301	0.1690	0.9714	0.039*
H18C	0.1426	0.1643	0.9228	0.039*
H1N1	0.269 (3)	0.1724 (13)	0.1938 (11)	0.032 (5)*
H1N2	0.235 (3)	0.0455 (12)	0.2878 (10)	0.019 (4)*

H1N3	0.276 (3)	0.0588 (12)	0.7132 (10)	0.023 (5)*
H1N4	0.215 (3)	0.1885 (12)	0.8028 (11)	0.025 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0249 (6)	0.0224 (5)	0.0232 (5)	0.0051 (4)	−0.0001 (5)	0.0054 (4)
O2	0.0197 (6)	0.0175 (5)	0.0182 (5)	−0.0023 (4)	0.0031 (4)	0.0015 (4)
O3	0.0208 (6)	0.0174 (5)	0.0184 (5)	0.0020 (4)	0.0041 (4)	0.0004 (4)
O4	0.0255 (6)	0.0245 (5)	0.0217 (5)	−0.0061 (5)	0.0013 (5)	−0.0053 (4)
N1	0.0213 (7)	0.0212 (6)	0.0133 (5)	0.0034 (5)	0.0050 (5)	0.0042 (5)
N2	0.0246 (7)	0.0188 (6)	0.0127 (5)	−0.0039 (5)	0.0057 (5)	0.0006 (4)
N3	0.0217 (7)	0.0184 (6)	0.0123 (5)	0.0038 (5)	0.0049 (5)	−0.0008 (4)
N4	0.0192 (7)	0.0203 (6)	0.0131 (5)	−0.0009 (5)	0.0050 (5)	−0.0044 (4)
C1	0.0166 (7)	0.0180 (6)	0.0133 (6)	0.0001 (5)	0.0024 (5)	−0.0026 (5)
C2	0.0183 (7)	0.0148 (6)	0.0183 (6)	−0.0005 (5)	0.0020 (5)	0.0001 (5)
C3	0.0157 (7)	0.0176 (6)	0.0139 (6)	−0.0008 (5)	0.0021 (5)	0.0008 (5)
C4	0.0113 (7)	0.0180 (6)	0.0132 (6)	0.0003 (5)	0.0012 (5)	−0.0015 (5)
C5	0.0142 (7)	0.0151 (6)	0.0155 (6)	−0.0009 (5)	0.0015 (5)	−0.0002 (5)
C6	0.0116 (7)	0.0177 (6)	0.0133 (6)	−0.0011 (5)	0.0005 (5)	0.0006 (5)
C7	0.0123 (7)	0.0179 (6)	0.0157 (6)	0.0018 (5)	0.0025 (5)	0.0008 (5)
C8	0.0215 (8)	0.0157 (6)	0.0160 (6)	−0.0003 (5)	0.0024 (5)	−0.0004 (5)
C9	0.0231 (8)	0.0247 (7)	0.0192 (7)	0.0043 (6)	0.0078 (6)	0.0055 (6)
C10	0.0371 (12)	0.0497 (11)	0.0387 (10)	0.0206 (9)	0.0236 (9)	0.0158 (9)
C11	0.0261 (9)	0.0297 (8)	0.0287 (8)	−0.0028 (7)	0.0037 (7)	0.0079 (7)
C12	0.0373 (10)	0.0286 (8)	0.0157 (7)	−0.0005 (7)	0.0059 (6)	0.0005 (6)
C13	0.0126 (7)	0.0172 (6)	0.0158 (6)	−0.0019 (5)	0.0026 (5)	−0.0003 (5)
C14	0.0197 (8)	0.0153 (6)	0.0148 (6)	−0.0008 (5)	0.0028 (5)	0.0010 (5)
C15	0.0183 (8)	0.0201 (7)	0.0180 (6)	−0.0006 (6)	0.0061 (5)	−0.0014 (5)
C16	0.0243 (9)	0.0319 (9)	0.0350 (9)	−0.0069 (7)	0.0121 (7)	−0.0048 (7)
C17	0.0245 (9)	0.0225 (7)	0.0265 (7)	0.0012 (6)	0.0067 (6)	−0.0031 (6)
C18	0.0290 (9)	0.0340 (9)	0.0155 (7)	0.0056 (7)	0.0057 (6)	−0.0006 (6)

Geometric parameters (Å, º)

O1—C8	1.2252 (17)	C8—C9	1.536 (2)
O2—C7	1.2295 (17)	C9—C10	1.525 (2)
O3—C13	1.2273 (17)	C9—C12	1.536 (2)
O4—C14	1.2261 (17)	C9—C11	1.538 (2)
N1—C8	1.359 (2)	C10—H10A	0.9600
N1—N2	1.3935 (16)	C10—H10B	0.9600
N1—H1N1	0.90 (2)	C10—H10C	0.9600
N2—C7	1.3559 (17)	C11—H11A	0.9600
N2—H1N2	0.844 (18)	C11—H11B	0.9600
N3—C13	1.3592 (17)	C11—H11C	0.9600
N3—N4	1.3942 (16)	C12—H12A	0.9600
N3—H1N3	0.876 (18)	C12—H12B	0.9600
N4—C14	1.357 (2)	C12—H12C	0.9600

N4—H1N4	0.879 (19)	C14—C15	1.5314 (19)
C1—C2	1.3920 (18)	C15—C16	1.519 (2)
C1—C6	1.3951 (19)	C15—C18	1.536 (2)
C1—H1A	0.9300	C15—C17	1.538 (2)
C2—C3	1.3874 (19)	C16—H16A	0.9600
C2—H2A	0.9300	C16—H16B	0.9600
C3—C4	1.3973 (19)	C16—H16C	0.9600
C3—H3A	0.9300	C17—H17A	0.9600
C4—C5	1.3926 (18)	C17—H17B	0.9600
C4—C13	1.4990 (19)	C17—H17C	0.9600
C5—C6	1.3937 (18)	C18—H18A	0.9600
C5—H5A	0.9300	C18—H18B	0.9600
C6—C7	1.4979 (19)	C18—H18C	0.9600

C8—N1—N2	117.82 (12)	C9—C10—H10C	109.5
C8—N1—H1N1	123.8 (13)	H10A—C10—H10C	109.5
N2—N1—H1N1	118.3 (13)	H10B—C10—H10C	109.5
C7—N2—N1	120.25 (12)	C9—C11—H11A	109.5
C7—N2—H1N2	122.2 (12)	C9—C11—H11B	109.5
N1—N2—H1N2	114.5 (12)	H11A—C11—H11B	109.5
C13—N3—N4	118.68 (12)	C9—C11—H11C	109.5
C13—N3—H1N3	121.8 (12)	H11A—C11—H11C	109.5
N4—N3—H1N3	114.6 (12)	H11B—C11—H11C	109.5
C14—N4—N3	117.68 (12)	C9—C12—H12A	109.5
C14—N4—H1N4	121.6 (12)	C9—C12—H12B	109.5
N3—N4—H1N4	120.4 (12)	H12A—C12—H12B	109.5
C2—C1—C6	119.81 (12)	C9—C12—H12C	109.5
C2—C1—H1A	120.1	H12A—C12—H12C	109.5
C6—C1—H1A	120.1	H12B—C12—H12C	109.5
C3—C2—C1	120.54 (13)	O3—C13—N3	122.45 (12)
C3—C2—H2A	119.7	O3—C13—C4	121.99 (12)
C1—C2—H2A	119.7	N3—C13—C4	115.51 (12)
C2—C3—C4	119.93 (12)	O4—C14—N4	120.13 (13)
C2—C3—H3A	120.0	O4—C14—C15	122.80 (14)
C4—C3—H3A	120.0	N4—C14—C15	117.07 (12)
C5—C4—C3	119.52 (12)	C16—C15—C14	108.38 (12)
C5—C4—C13	117.80 (12)	C16—C15—C18	110.34 (13)
C3—C4—C13	122.63 (12)	C14—C15—C18	109.82 (12)
C4—C5—C6	120.60 (13)	C16—C15—C17	109.03 (13)
C4—C5—H5A	119.7	C14—C15—C17	109.77 (12)
C6—C5—H5A	119.7	C18—C15—C17	109.48 (12)
C5—C6—C1	119.60 (12)	C15—C16—H16A	109.5
C5—C6—C7	117.34 (12)	C15—C16—H16B	109.5
C1—C6—C7	123.03 (12)	H16A—C16—H16B	109.5
O2—C7—N2	122.34 (12)	C15—C16—H16C	109.5
O2—C7—C6	121.90 (12)	H16A—C16—H16C	109.5
N2—C7—C6	115.66 (12)	H16B—C16—H16C	109.5
O1—C8—N1	120.54 (13)	C15—C17—H17A	109.5

O1—C8—C9	122.55 (14)	C15—C17—H17B	109.5
N1—C8—C9	116.91 (12)	H17A—C17—H17B	109.5
C10—C9—C12	109.22 (14)	C15—C17—H17C	109.5
C10—C9—C8	107.75 (13)	H17A—C17—H17C	109.5
C12—C9—C8	110.45 (13)	H17B—C17—H17C	109.5
C10—C9—C11	109.98 (15)	C15—C18—H18A	109.5
C12—C9—C11	109.34 (12)	C15—C18—H18B	109.5
C8—C9—C11	110.08 (12)	H18A—C18—H18B	109.5
C9—C10—H10A	109.5	C15—C18—H18C	109.5
C9—C10—H10B	109.5	H18A—C18—H18C	109.5
H10A—C10—H10B	109.5	H18B—C18—H18C	109.5

C8—N1—N2—C7	85.19 (17)	O1—C8—C9—C10	13.0 (2)
C13—N3—N4—C14	76.61 (17)	N1—C8—C9—C10	−166.35 (14)
C6—C1—C2—C3	−0.6 (2)	O1—C8—C9—C12	132.24 (15)
C1—C2—C3—C4	0.2 (2)	N1—C8—C9—C12	−47.12 (17)
C2—C3—C4—C5	0.0 (2)	O1—C8—C9—C11	−106.92 (16)
C2—C3—C4—C13	177.12 (13)	N1—C8—C9—C11	73.72 (17)
C3—C4—C5—C6	0.1 (2)	N4—N3—C13—O3	21.9 (2)
C13—C4—C5—C6	−177.15 (13)	N4—N3—C13—C4	−160.69 (12)
C4—C5—C6—C1	−0.5 (2)	C5—C4—C13—O3	25.5 (2)
C4—C5—C6—C7	−178.47 (13)	C3—C4—C13—O3	−151.62 (15)
C2—C1—C6—C5	0.7 (2)	C5—C4—C13—N3	−151.89 (13)
C2—C1—C6—C7	178.61 (13)	C3—C4—C13—N3	31.0 (2)
N1—N2—C7—O2	17.9 (2)	N3—N4—C14—O4	0.9 (2)
N1—N2—C7—C6	−165.58 (12)	N3—N4—C14—C15	−179.64 (12)
C5—C6—C7—O2	22.5 (2)	O4—C14—C15—C16	5.10 (19)
C1—C6—C7—O2	−155.42 (14)	N4—C14—C15—C16	−174.36 (13)
C5—C6—C7—N2	−154.00 (13)	O4—C14—C15—C18	125.70 (15)
C1—C6—C7—N2	28.1 (2)	N4—C14—C15—C18	−53.76 (17)
N2—N1—C8—O1	−3.9 (2)	O4—C14—C15—C17	−113.89 (16)
N2—N1—C8—C9	175.47 (12)	N4—C14—C15—C17	66.65 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···O3^{i}	0.90 (2)	2.12 (2)	3.0193 (16)	176.7 (16)
N2—H1*N*2···O4^{ii}	0.845 (18)	1.993 (19)	2.8262 (17)	169 (2)
N3—H1*N*3···O1^{iii}	0.876 (18)	1.969 (18)	2.8307 (16)	167.3 (16)
N4—H1*N*4···O2^{iv}	0.878 (19)	2.059 (19)	2.9320 (16)	172.3 (19)
C1—H1*A*···O4^{ii}	0.93	2.48	3.1879 (17)	133
C3—H3*A*···O1^{iii}	0.93	2.56	3.2854 (17)	135
C12—H12*C*···O3^{i}	0.96	2.52	3.433 (2)	159
C18—H18*C*···N4	0.96	2.61	2.9347 (19)	100

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