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3-[2-(3-Nitrophenyl)hydrazono]pentane-2,4-dione

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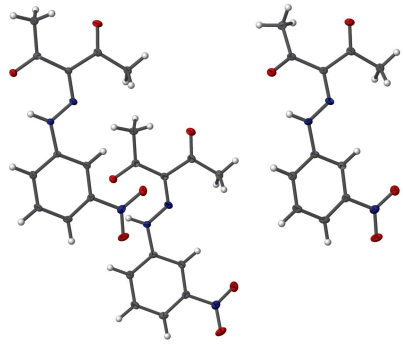
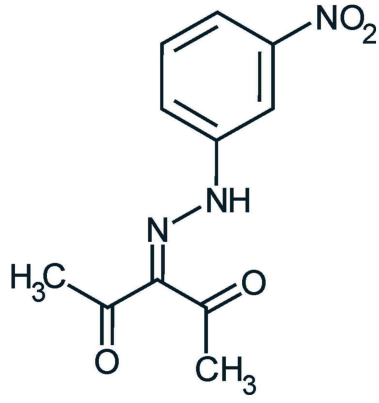
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{11}H_{11}N_3O_4$, crystallizes with three independent but conformationally similar molecules in the asymmetric unit. The plane through the pentane-2,4-dione moiety of the molecule is inclined at angles of 10.1 (1), 10.6 (1) and 17.4 (1)° with regard to the respective arene ring. In the crystal, the molecules are connected *via* C_{arene}—H···O_{nitro} and C_{arene}—H···O=C interactions into supramolecular sheets. Molecules of consecutive sheets are linked by C_{methyl}—H···O bonds.

3D view



Chemical scheme



Structure description

Arylhydrazones of β-diketones have recently become interesting objects of scientific study with regard to complex formation (Marten *et al.*, 2005) and hydrogen bonding, exerting an active influence both on the spectroscopic and electrochemical as well as the structural properties of the β-ketone derivative (Marten *et al.*, 2007, 2011; Sethukumar *et al.*, 2010), including the research question of resonance-assisted hydrogen bonding (RAHB) (Gilli *et al.*, 1989; Alkorta *et al.*, 2004). The title compound belongs to this category of promising molecules and its crystal structure is reported here.

The asymmetric unit contains three independent but conformationally similar molecules (Fig. 1). Each molecule features an intramolecular N—H···O interaction (Table 1) that yields a six-membered hydrogen-bonded ring. In the crystal structure, the molecules exist in an *EZE* configuration which – in this order – represents the alignment of the non-H-bridged O2*x*—C4*x* fragment, the hydrogen-bonded O1*x*—C2*x* fragment, and the aryl group with respect to the N1*x*=C3*x* double bond (*x* = -,*A*,*B*) (Gómez-Sánchez *et al.*, 1987). This geometry agrees with that found in the solid-state structures of other compounds of this kind (Bertolasi *et al.* 1994; Marten *et al.* 2005, 2007). The molecules deviate slightly from planarity, showing tilt angles of 9.0 (2), 6.6 (2) and 9.7 (1)° between the mean planes of the hydrogen-bonded ring and the arene ring. The molecules exhibit a highly distorted geometry along the pentane-2,4-dione fragment, which can be seen from

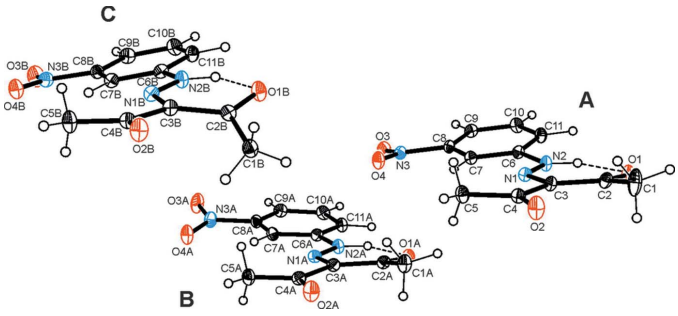


Figure 1
Perspective view of the molecular structure of the title compound with the atom labelling. Displacement ellipsoids of non-H atoms are shown at the 50% probability level.

the enlarged torsion angles of the atomic sequences N1*x*—C3*x*—C2*x*—O1*x* and C2*x*—C3*x*—C4*x*—O2*x* being −10.0 (2) and −5.1 (2)° for molecule *A*, −10.0 (2) and −5.1 (2)° for molecule *B* and 18.5 (2) and 9.7 (2)° for molecule *C*. The six-membered hydrogen-bonded rings, however, are less affected by the distortion. The largest atomic distances from the mean plane are found to be 0.076 (1) and −0.102 (1) Å for atoms O1*B* and C2*B* of molecule *C*. In addition, the nitro groups are tilted slightly with respect to the benzene ring, showing inclination angles of 11.9 (1), 10.7 (1) and 7.8 (1)° for molecules *A*, *B* and *C*, respectively.

In the crystal (Fig. 2), the molecules are connected by means of C_{arene}—H···O_{nitro} and C_{arene}—H···O=C interactions (Desiraju & Steiner, 1999) giving rise to sheets that extend parallel to the *bc* plane. In the stacking direction of

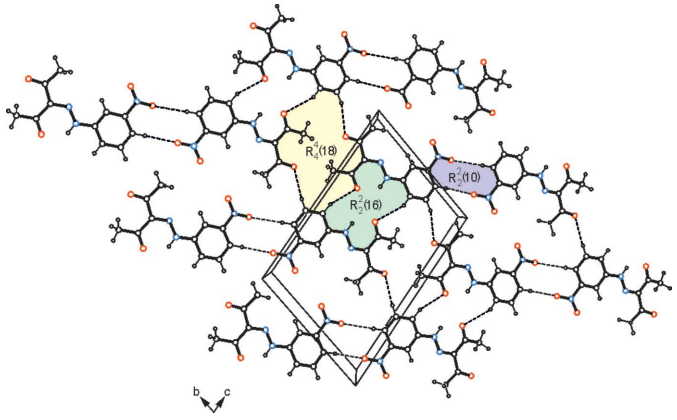


Figure 2
Structure of the molecular sheets including the mode of intermolecular bonding in the crystal of the title compound. Dashed lines represent hydrogen bonds. Particular ring systems of intermolecular interactions are specified by colour highlighting. Specification of the ring pattern corresponds to the graph-set notation introduced by M. C. Etter (Etter *et al.*, 1990).

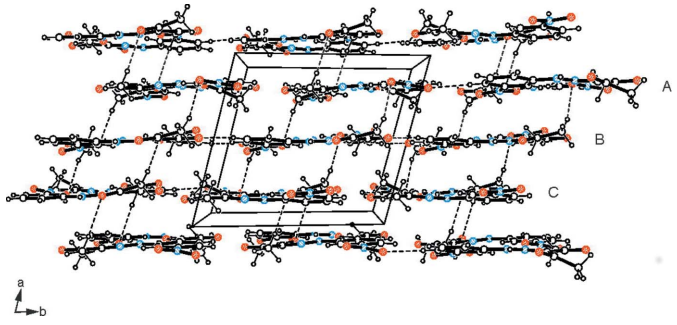


Figure 3
Packing diagram of the title compound viewed along the *c* axis. The intermolecular contacts are shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11*B*—H11*B*···O1*A*^{i}	0.95	2.63	3.4824 (15)	150
C9*B*—H9*B*···O3*A*^{ii}	0.95	2.53	3.4837 (16)	178
N2*B*—H2*B*···O1*B*	0.90 (1)	1.90 (1)	2.5952 (13)	133 (1)
C11*A*—H11*A*···O1*B*^{i}	0.95	2.53	3.4347 (15)	158
C9*A*—H9*A*···O3*B*^{ii}	0.95	2.59	3.5227 (15)	168
C5*A*—H5*A*2···N1^{i}	0.98	2.65	3.5230 (16)	149
C5*A*—H5*A*1···O1*A*^{iii}	0.98	2.51	3.4424 (15)	158
C1*A*—H1*A*2···O4^{i}	0.98	2.49	3.4674 (16)	173
N2*A*—H2*A*···O1*A*	0.90 (1)	1.91 (1)	2.5873 (13)	131 (1)
C11—H11···O1^{iv}	0.95	2.52	3.4114 (14)	156
C9—H9···O3^{v}	0.95	2.63	3.5450 (15)	163
C5—H5*B*···N1*A*^{i}	0.98	2.69	3.5445 (16)	146
C1—H1*B*···O4*A*^{i}	0.98	2.47	3.4237 (18)	165
N2—H2···O1	0.89 (1)	1.89 (1)	2.5834 (12)	134 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{11}N_3O_4$
M_r	249.23
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	93
a, *b*, *c* (Å)	10.1251 (5), 11.1335 (6), 15.9006 (8)
α, β, γ (°)	102.745 (3), 101.100 (3), 101.520 (3)
V (Å^3)	1659.74 (15)
Z	6
Radiation type	Mo *K*α
μ (mm^{-1})	0.12
Crystal size (mm)	0.28 × 0.20 × 0.10
Data collection	
Diffractometer	Bruker APEXII CCD area detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	49810, 8881, 6440
R_{int}	0.033
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.684
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.041, 0.127, 1.00
No. of reflections	8881
No. of parameters	505
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.44, −0.30

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *SHELXTL* (Sheldrick, 2008).

these two-dimensional-supramolecular aggregates, the molecules are linked by $C_{methyl}-H\cdots O_{nitro}$ hydrogen bonds (Table 1, Fig. 3). Moreover, the crystal is stabilized by π–π stacking interactions between the aromatic rings and the six-membered hydrogen-bonded rings with centroid–centroid distances of 4.1043 (8)–4.4867 (8) Å.

Synthesis and crystallization

The title compound was prepared from pentane-2,4-dione and 3-nitrophenyldiazonium chloride (diazotization of 3-nitroaniline) *via* a Japp–Klingemann route (Phillips, 1959) following a described protocol (Sethukumar *et al.*, 2010) to yield yellow crystals (78%, m.p. 412 K) and spectroscopic data correspond to the literature (Bülow & Schlotterbeck, 1902; Sethukumar *et al.*, 2010). Crystals were grown by the slow evaporation technique using ethanol as solvent.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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full crystallographic data

IUCrData (2018). **3**, x181344 [https://doi.org/10.1107/S2414314618013445]

3-[2-(3-Nitrophenyl)hydrazin-1-ylidene]pentane-2,4-dione

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3-[2-(3-Nitrophenyl)hydrazin-1-ylidene]pentane-2,4-dione

Crystal data

$C_{11}H_{11}N_3O_4$
$M_r = 249.23$
Triclinic, $P\overline{1}$
$a = 10.1251$ (5) Å
$b = 11.1335$ (6) Å
$c = 15.9006$ (8) Å
$\alpha = 102.745$ (3)°
$\beta = 101.100$ (3)°
$\gamma = 101.520$ (3)°
$V = 1659.74$ (15) Å^3
$Z = 6$
$F(000) = 780$
$D_x = 1.496$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9958 reflections
$\theta = 2.5$–$38.8°$
$\mu = 0.12$ mm^{-1}
$T = 93$ K
Irregular, yellow
0.28 × 0.20 × 0.10 mm

Data collection

Bruker APEXII CCD area detector diffractometer
φ and ω scans
49810 measured reflections
8881 independent reflections
6440 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 29.1°$, $\theta_{min} = 2.0°$
$h = -13 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.127$
$S = 1.00$
8881 reflections
505 parameters
3 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 0.3903P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-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. The non-hydrogen atoms were refined anisotropically. The hydrogen atoms were positioned geometrically and refined isotropically using the riding model with C—H = 0.98 Å and U_{iso}(H) = 1.5 U_{eq}(C) for methyl and C—H = 0.95 Å and U_{iso}(H) = 1.2 U_{eq}(C) for aryl.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.50272 (9)	0.37266 (8)	0.01900 (6)	0.01728 (18)
O2	0.51679 (11)	0.09300 (8)	0.15551 (6)	0.0236 (2)
O3	0.43473 (10)	0.84492 (9)	0.50143 (6)	0.0226 (2)
O4	0.46928 (9)	0.65656 (9)	0.48900 (6)	0.0202 (2)
N1	0.49937 (10)	0.40849 (9)	0.19988 (6)	0.01255 (19)
N2	0.49547 (10)	0.50955 (9)	0.17125 (6)	0.0129 (2)
H2	0.4964 (19)	0.5041 (16)	0.1147 (7)	0.039 (5)*
N3	0.45970 (10)	0.74844 (10)	0.45984 (7)	0.0151 (2)
C1	0.55504 (17)	0.17248 (13)	0.00493 (9)	0.0269 (3)
H1A	0.4730	0.1000	−0.0146	0.040*
H1B	0.6303	0.1525	0.0439	0.040*
H1C	0.5853	0.1897	−0.0471	0.040*
C2	0.51931 (12)	0.28713 (11)	0.05460 (8)	0.0146 (2)
C3	0.50838 (12)	0.30180 (11)	0.14792 (7)	0.0124 (2)
C4	0.51293 (12)	0.19961 (11)	0.19419 (8)	0.0141 (2)
C5	0.51082 (13)	0.23092 (12)	0.29086 (8)	0.0166 (2)
H5A	0.4200	0.2448	0.2966	0.025*
H5B	0.5839	0.3083	0.3238	0.025*
H5C	0.5272	0.1601	0.3152	0.025*
C6	0.49515 (11)	0.62290 (11)	0.23101 (7)	0.0118 (2)
C7	0.47929 (11)	0.62682 (11)	0.31640 (8)	0.0126 (2)
H7	0.4698	0.5528	0.3375	0.015*
C8	0.47793 (12)	0.74318 (11)	0.36949 (7)	0.0128 (2)
C9	0.49204 (12)	0.85409 (11)	0.34252 (8)	0.0146 (2)
H9	0.4903	0.9320	0.3810	0.017*
C10	0.50894 (12)	0.84719 (11)	0.25684 (8)	0.0155 (2)
H10	0.5189	0.9214	0.2360	0.019*
C11	0.51135 (12)	0.73260 (11)	0.20173 (8)	0.0149 (2)
H11	0.5241	0.7290	0.1437	0.018*
O1A	0.16277 (9)	0.55721 (8)	0.38561 (6)	0.01764 (19)
O2A	0.18248 (10)	0.28004 (8)	0.52201 (6)	0.0236 (2)
O3A	0.18275 (12)	1.04571 (10)	0.88501 (6)	0.0330 (3)
O4A	0.14636 (10)	0.84097 (9)	0.85760 (6)	0.0255 (2)
N1A	0.16065 (10)	0.59407 (9)	0.56645 (7)	0.0140 (2)
N2A	0.15028 (11)	0.69368 (9)	0.53691 (7)	0.0141 (2)
H2A	0.1507 (17)	0.6910 (16)	0.4802 (7)	0.034 (5)*
N3A	0.16103 (11)	0.93940 (11)	0.83415 (7)	0.0200 (2)
C1A	0.22945 (14)	0.36347 (12)	0.37409 (8)	0.0185 (3)
H1A1	0.1526	0.2868	0.3575	0.028*
H1A2	0.3104	0.3507	0.4128	0.028*
H1A3	0.2538	0.3802	0.3203	0.028*
C2A	0.18617 (12)	0.47422 (11)	0.42247 (8)	0.0144 (2)
C3A	0.17495 (12)	0.48892 (11)	0.51580 (8)	0.0140 (2)
C4A	0.17907 (12)	0.38607 (11)	0.56176 (8)	0.0157 (2)
C5A	0.17481 (13)	0.41559 (12)	0.65827 (8)	0.0187 (3)

H5A1	0.0844	0.4311	0.6635	0.028*
H5A2	0.2490	0.4915	0.6924	0.028*
H5A3	0.1882	0.3433	0.6816	0.028*
C6A	0.14666 (12)	0.80636 (11)	0.59629 (8)	0.0135 (2)
C7A	0.15249 (12)	0.81389 (11)	0.68543 (8)	0.0146 (2)
H7A	0.1560	0.7424	0.7084	0.018*
C8A	0.15295 (12)	0.93022 (12)	0.73937 (8)	0.0150 (2)
C9A	0.14790 (12)	1.03754 (12)	0.70965 (8)	0.0170 (2)
H9A	0.1503	1.1161	0.7491	0.020*
C10A	0.13916 (13)	1.02583 (12)	0.61947 (8)	0.0175 (2)
H10A	0.1342	1.0971	0.5965	0.021*
C11A	0.13772 (12)	0.91090 (12)	0.56317 (8)	0.0161 (2)
H11A	0.1306	0.9035	0.5017	0.019*
O1B	0.85907 (9)	0.19177 (8)	0.65642 (6)	0.01866 (19)
O2B	0.85133 (10)	−0.09374 (8)	0.78295 (6)	0.0206 (2)
O3B	0.78511 (11)	0.66059 (9)	1.13961 (6)	0.0259 (2)
O4B	0.80431 (10)	0.46675 (9)	1.12265 (6)	0.0220 (2)
N1B	0.85135 (10)	0.22594 (9)	0.83719 (7)	0.0140 (2)
N2B	0.85919 (11)	0.33023 (9)	0.81086 (7)	0.0145 (2)
H2B	0.8644 (18)	0.3274 (16)	0.7549 (7)	0.036 (5)*
N3B	0.80267 (10)	0.56166 (10)	1.09587 (7)	0.0169 (2)
C1B	0.76785 (13)	−0.03138 (11)	0.61996 (8)	0.0179 (2)
H1B1	0.7221	−0.0223	0.5623	0.027*
H1B2	0.6996	−0.0821	0.6428	0.027*
H1B3	0.8423	−0.0741	0.6126	0.027*
C2B	0.82836 (12)	0.09774 (11)	0.68436 (8)	0.0142 (2)
C3B	0.84300 (12)	0.11612 (11)	0.78164 (8)	0.0137 (2)
C4B	0.84416 (12)	0.01022 (11)	0.82453 (8)	0.0150 (2)
C5B	0.83783 (14)	0.03443 (12)	0.92040 (8)	0.0205 (3)
H5B1	0.8525	−0.0389	0.9420	0.031*
H5B2	0.7464	0.0474	0.9254	0.031*
H5B3	0.9105	0.1106	0.9561	0.031*
C6B	0.85292 (12)	0.44227 (11)	0.86994 (8)	0.0137 (2)
C7B	0.82885 (12)	0.44323 (11)	0.95337 (8)	0.0140 (2)
H7B	0.8165	0.3682	0.9732	0.017*
C8B	0.82363 (12)	0.55825 (11)	1.00622 (8)	0.0142 (2)
C9B	0.83941 (12)	0.66991 (12)	0.98035 (8)	0.0172 (2)
H9B	0.8334	0.7465	1.0183	0.021*
C10B	0.86433 (13)	0.66630 (12)	0.89682 (8)	0.0185 (2)
H10B	0.8760	0.7415	0.8771	0.022*
C11B	0.87222 (12)	0.55351 (11)	0.84220 (8)	0.0164 (2)
H11B	0.8908	0.5520	0.7857	0.020*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0288 (5)	0.0129 (4)	0.0130 (4)	0.0094 (4)	0.0057 (3)	0.0050 (3)
O2	0.0433 (6)	0.0108 (4)	0.0197 (5)	0.0105 (4)	0.0104 (4)	0.0045 (4)

O3	0.0317 (5)	0.0184 (5)	0.0173 (4)	0.0075 (4)	0.0106 (4)	−0.0012 (4)
O4	0.0275 (5)	0.0209 (5)	0.0151 (4)	0.0076 (4)	0.0063 (4)	0.0084 (4)
N1	0.0153 (4)	0.0093 (5)	0.0139 (5)	0.0043 (4)	0.0036 (4)	0.0039 (4)
N2	0.0203 (5)	0.0089 (5)	0.0111 (5)	0.0057 (4)	0.0051 (4)	0.0029 (4)
N3	0.0167 (5)	0.0152 (5)	0.0118 (5)	0.0025 (4)	0.0036 (4)	0.0017 (4)
C1	0.0519 (9)	0.0226 (7)	0.0144 (6)	0.0233 (7)	0.0110 (6)	0.0066 (5)
C2	0.0192 (5)	0.0114 (6)	0.0121 (5)	0.0048 (4)	0.0022 (4)	0.0017 (4)
C3	0.0168 (5)	0.0096 (5)	0.0110 (5)	0.0044 (4)	0.0029 (4)	0.0024 (4)
C4	0.0158 (5)	0.0121 (6)	0.0150 (5)	0.0038 (4)	0.0039 (4)	0.0045 (4)
C5	0.0242 (6)	0.0134 (6)	0.0151 (5)	0.0066 (5)	0.0070 (5)	0.0060 (5)
C6	0.0139 (5)	0.0100 (5)	0.0115 (5)	0.0050 (4)	0.0032 (4)	0.0014 (4)
C7	0.0155 (5)	0.0100 (5)	0.0134 (5)	0.0041 (4)	0.0043 (4)	0.0041 (4)
C8	0.0146 (5)	0.0134 (6)	0.0102 (5)	0.0043 (4)	0.0033 (4)	0.0021 (4)
C9	0.0165 (5)	0.0109 (6)	0.0155 (5)	0.0050 (4)	0.0042 (4)	0.0005 (4)
C10	0.0211 (6)	0.0104 (6)	0.0177 (6)	0.0063 (5)	0.0063 (5)	0.0064 (5)
C11	0.0201 (6)	0.0139 (6)	0.0131 (5)	0.0068 (5)	0.0052 (4)	0.0054 (5)
O1A	0.0258 (5)	0.0146 (4)	0.0151 (4)	0.0090 (4)	0.0052 (3)	0.0055 (3)
O2A	0.0384 (5)	0.0135 (5)	0.0212 (5)	0.0094 (4)	0.0083 (4)	0.0057 (4)
O3A	0.0567 (7)	0.0226 (5)	0.0168 (5)	0.0114 (5)	0.0098 (5)	−0.0025 (4)
O4A	0.0373 (6)	0.0265 (5)	0.0191 (5)	0.0129 (4)	0.0111 (4)	0.0112 (4)
N1A	0.0154 (5)	0.0103 (5)	0.0165 (5)	0.0029 (4)	0.0036 (4)	0.0042 (4)
N2A	0.0209 (5)	0.0100 (5)	0.0125 (5)	0.0054 (4)	0.0057 (4)	0.0027 (4)
N3A	0.0256 (6)	0.0206 (6)	0.0151 (5)	0.0086 (4)	0.0059 (4)	0.0038 (4)
C1A	0.0274 (6)	0.0150 (6)	0.0162 (6)	0.0103 (5)	0.0075 (5)	0.0038 (5)
C2A	0.0157 (5)	0.0121 (6)	0.0143 (5)	0.0030 (4)	0.0024 (4)	0.0030 (4)
C3A	0.0156 (5)	0.0110 (6)	0.0153 (5)	0.0031 (4)	0.0031 (4)	0.0038 (4)
C4A	0.0162 (5)	0.0138 (6)	0.0167 (6)	0.0035 (4)	0.0028 (4)	0.0049 (5)
C5A	0.0238 (6)	0.0186 (6)	0.0156 (6)	0.0062 (5)	0.0050 (5)	0.0078 (5)
C6A	0.0153 (5)	0.0107 (6)	0.0139 (5)	0.0034 (4)	0.0047 (4)	0.0008 (4)
C7A	0.0172 (5)	0.0136 (6)	0.0152 (5)	0.0055 (4)	0.0051 (4)	0.0060 (5)
C8A	0.0163 (5)	0.0171 (6)	0.0127 (5)	0.0054 (5)	0.0055 (4)	0.0033 (5)
C9A	0.0187 (6)	0.0138 (6)	0.0180 (6)	0.0050 (5)	0.0056 (5)	0.0013 (5)
C10A	0.0216 (6)	0.0129 (6)	0.0207 (6)	0.0063 (5)	0.0066 (5)	0.0070 (5)
C11A	0.0207 (6)	0.0156 (6)	0.0139 (5)	0.0060 (5)	0.0057 (4)	0.0051 (5)
O1B	0.0287 (5)	0.0128 (4)	0.0163 (4)	0.0058 (4)	0.0083 (4)	0.0049 (3)
O2B	0.0322 (5)	0.0133 (4)	0.0183 (4)	0.0105 (4)	0.0074 (4)	0.0031 (4)
O3B	0.0384 (6)	0.0204 (5)	0.0207 (5)	0.0114 (4)	0.0138 (4)	0.0000 (4)
O4B	0.0302 (5)	0.0202 (5)	0.0191 (4)	0.0082 (4)	0.0088 (4)	0.0081 (4)
N1B	0.0164 (5)	0.0105 (5)	0.0159 (5)	0.0053 (4)	0.0035 (4)	0.0038 (4)
N2B	0.0219 (5)	0.0103 (5)	0.0121 (5)	0.0057 (4)	0.0054 (4)	0.0020 (4)
N3B	0.0178 (5)	0.0163 (5)	0.0154 (5)	0.0043 (4)	0.0047 (4)	0.0013 (4)
C1B	0.0236 (6)	0.0139 (6)	0.0148 (6)	0.0022 (5)	0.0063 (5)	0.0025 (5)
C2B	0.0161 (5)	0.0126 (6)	0.0154 (5)	0.0057 (4)	0.0055 (4)	0.0033 (5)
C3B	0.0164 (5)	0.0111 (6)	0.0146 (5)	0.0054 (4)	0.0043 (4)	0.0031 (4)
C4B	0.0159 (5)	0.0131 (6)	0.0165 (6)	0.0048 (4)	0.0040 (4)	0.0043 (5)
C5B	0.0326 (7)	0.0166 (6)	0.0154 (6)	0.0090 (5)	0.0082 (5)	0.0063 (5)
C6B	0.0148 (5)	0.0109 (6)	0.0143 (5)	0.0044 (4)	0.0026 (4)	0.0012 (4)
C7B	0.0158 (5)	0.0113 (6)	0.0152 (5)	0.0043 (4)	0.0034 (4)	0.0038 (5)

C8B	0.0146 (5)	0.0151 (6)	0.0128 (5)	0.0040 (4)	0.0039 (4)	0.0028 (4)
C9B	0.0199 (6)	0.0112 (6)	0.0188 (6)	0.0049 (5)	0.0052 (5)	−0.0005 (5)
C10B	0.0242 (6)	0.0121 (6)	0.0209 (6)	0.0052 (5)	0.0072 (5)	0.0057 (5)
C11B	0.0208 (6)	0.0146 (6)	0.0147 (5)	0.0056 (5)	0.0050 (5)	0.0045 (5)

Geometric parameters (Å, º)

O1—C2	1.2331 (14)	C4A—C5A	1.5078 (16)
O2—C4	1.2224 (14)	C5A—H5A1	0.9800
O3—N3	1.2307 (13)	C5A—H5A2	0.9800
O4—N3	1.2258 (13)	C5A—H5A3	0.9800
N1—N2	1.3082 (13)	C6A—C7A	1.3904 (16)
N1—C3	1.3194 (14)	C6A—C11A	1.3907 (16)
N2—C6	1.4033 (14)	C7A—C8A	1.3853 (16)
N2—H2	0.890 (9)	C7A—H7A	0.9500
N3—C8	1.4733 (14)	C8A—C9A	1.3848 (17)
C1—C2	1.4945 (16)	C9A—C10A	1.3947 (17)
C1—H1A	0.9800	C9A—H9A	0.9500
C1—H1B	0.9800	C10A—C11A	1.3860 (17)
C1—H1C	0.9800	C10A—H10A	0.9500
C2—C3	1.4848 (15)	C11A—H11A	0.9500
C3—C4	1.4882 (16)	O1B—C2B	1.2323 (14)
C4—C5	1.5047 (16)	O2B—C4B	1.2234 (14)
C5—H5A	0.9800	O3B—N3B	1.2314 (13)
C5—H5B	0.9800	O4B—N3B	1.2251 (13)
C5—H5C	0.9800	N1B—N2B	1.3116 (14)
C6—C7	1.3899 (15)	N1B—C3B	1.3174 (15)
C6—C11	1.3913 (16)	N2B—C6B	1.4071 (15)
C7—C8	1.3857 (16)	N2B—H2B	0.896 (9)
C7—H7	0.9500	N3B—C8B	1.4746 (15)
C8—C9	1.3841 (16)	C1B—C2B	1.4992 (16)
C9—C10	1.3929 (16)	C1B—H1B1	0.9800
C9—H9	0.9500	C1B—H1B2	0.9800
C10—C11	1.3869 (16)	C1B—H1B3	0.9800
C10—H10	0.9500	C2B—C3B	1.4887 (16)
C11—H11	0.9500	C3B—C4B	1.4875 (16)
O1A—C2A	1.2355 (14)	C4B—C5B	1.5044 (16)
O2A—C4A	1.2205 (15)	C5B—H5B1	0.9800
O3A—N3A	1.2281 (14)	C5B—H5B2	0.9800
O4A—N3A	1.2242 (14)	C5B—H5B3	0.9800
N1A—N2A	1.3109 (14)	C6B—C7B	1.3924 (16)
N1A—C3A	1.3179 (15)	C6B—C11B	1.3943 (16)
N2A—C6A	1.4056 (14)	C7B—C8B	1.3850 (16)
N2A—H2A	0.897 (9)	C7B—H7B	0.9500
N3A—C8A	1.4730 (15)	C8B—C9B	1.3827 (17)
C1A—C2A	1.4937 (16)	C9B—C10B	1.3924 (17)
C1A—H1A1	0.9800	C9B—H9B	0.9500
C1A—H1A2	0.9800	C10B—C11B	1.3867 (17)

C1A—H1A3	0.9800	C10B—H10B	0.9500
C2A—C3A	1.4855 (16)	C11B—H11B	0.9500
C3A—C4A	1.4912 (16)		
N2—N1—C3	121.67 (10)	C4A—C5A—H5A2	109.5
N1—N2—C6	119.22 (9)	H5A1—C5A—H5A2	109.5
N1—N2—H2	118.1 (11)	C4A—C5A—H5A3	109.5
C6—N2—H2	122.6 (11)	H5A1—C5A—H5A3	109.5
O4—N3—O3	123.81 (10)	H5A2—C5A—H5A3	109.5
O4—N3—C8	118.34 (9)	C7A—C6A—C11A	120.79 (11)
O3—N3—C8	117.84 (10)	C7A—C6A—N2A	121.34 (10)
C2—C1—H1A	109.5	C11A—C6A—N2A	117.87 (10)
C2—C1—H1B	109.5	C8A—C7A—C6A	117.09 (11)
H1A—C1—H1B	109.5	C8A—C7A—H7A	121.5
C2—C1—H1C	109.5	C6A—C7A—H7A	121.5
H1A—C1—H1C	109.5	C9A—C8A—C7A	123.98 (11)
H1B—C1—H1C	109.5	C9A—C8A—N3A	118.61 (10)
O1—C2—C3	119.25 (10)	C7A—C8A—N3A	117.40 (11)
O1—C2—C1	119.96 (11)	C8A—C9A—C10A	117.38 (11)
C3—C2—C1	120.74 (10)	C8A—C9A—H9A	121.3
N1—C3—C2	123.61 (10)	C10A—C9A—H9A	121.3
N1—C3—C4	112.75 (10)	C11A—C10A—C9A	120.42 (11)
C2—C3—C4	123.59 (10)	C11A—C10A—H10A	119.8
O2—C4—C3	121.77 (11)	C9A—C10A—H10A	119.8
O2—C4—C5	120.44 (11)	C10A—C11A—C6A	120.29 (11)
C3—C4—C5	117.78 (10)	C10A—C11A—H11A	119.9
C4—C5—H5A	109.5	C6A—C11A—H11A	119.9
C4—C5—H5B	109.5	N2B—N1B—C3B	121.30 (10)
H5A—C5—H5B	109.5	N1B—N2B—C6B	119.20 (10)
C4—C5—H5C	109.5	N1B—N2B—H2B	119.1 (11)
H5A—C5—H5C	109.5	C6B—N2B—H2B	121.6 (11)
H5B—C5—H5C	109.5	O4B—N3B—O3B	123.88 (11)
C7—C6—C11	120.66 (10)	O4B—N3B—C8B	118.42 (10)
C7—C6—N2	121.85 (10)	O3B—N3B—C8B	117.70 (10)
C11—C6—N2	117.49 (10)	C2B—C1B—H1B1	109.5
C8—C7—C6	117.19 (11)	C2B—C1B—H1B2	109.5
C8—C7—H7	121.4	H1B1—C1B—H1B2	109.5
C6—C7—H7	121.4	C2B—C1B—H1B3	109.5
C9—C8—C7	123.93 (11)	H1B1—C1B—H1B3	109.5
C9—C8—N3	118.30 (10)	H1B2—C1B—H1B3	109.5
C7—C8—N3	117.77 (10)	O1B—C2B—C3B	118.94 (10)
C8—C9—C10	117.47 (10)	O1B—C2B—C1B	119.82 (11)
C8—C9—H9	121.3	C3B—C2B—C1B	121.11 (10)
C10—C9—H9	121.3	N1B—C3B—C4B	113.72 (10)
C11—C10—C9	120.38 (11)	N1B—C3B—C2B	123.48 (10)
C11—C10—H10	119.8	C4B—C3B—C2B	122.76 (10)
C9—C10—H10	119.8	O2B—C4B—C3B	120.68 (11)
C10—C11—C6	120.37 (11)	O2B—C4B—C5B	120.87 (11)

C10—C11—H11	119.8	C3B—C4B—C5B	118.45 (10)
C6—C11—H11	119.8	C4B—C5B—H5B1	109.5
N2A—N1A—C3A	122.15 (10)	C4B—C5B—H5B2	109.5
N1A—N2A—C6A	119.27 (10)	H5B1—C5B—H5B2	109.5
N1A—N2A—H2A	120.0 (11)	C4B—C5B—H5B3	109.5
C6A—N2A—H2A	120.6 (11)	H5B1—C5B—H5B3	109.5
O4A—N3A—O3A	123.94 (11)	H5B2—C5B—H5B3	109.5
O4A—N3A—C8A	118.19 (10)	C7B—C6B—C11B	120.72 (11)
O3A—N3A—C8A	117.87 (11)	C7B—C6B—N2B	121.67 (11)
C2A—C1A—H1A1	109.5	C11B—C6B—N2B	117.61 (10)
C2A—C1A—H1A2	109.5	C8B—C7B—C6B	117.14 (11)
H1A1—C1A—H1A2	109.5	C8B—C7B—H7B	121.4
C2A—C1A—H1A3	109.5	C6B—C7B—H7B	121.4
H1A1—C1A—H1A3	109.5	C9B—C8B—C7B	123.82 (11)
H1A2—C1A—H1A3	109.5	C9B—C8B—N3B	118.40 (10)
O1A—C2A—C3A	118.99 (10)	C7B—C8B—N3B	117.77 (10)
O1A—C2A—C1A	119.89 (10)	C8B—C9B—C10B	117.76 (11)
C3A—C2A—C1A	121.09 (10)	C8B—C9B—H9B	121.1
N1A—C3A—C2A	123.52 (11)	C10B—C9B—H9B	121.1
N1A—C3A—C4A	113.14 (10)	C11B—C10B—C9B	120.33 (12)
C2A—C3A—C4A	123.34 (10)	C11B—C10B—H10B	119.8
O2A—C4A—C3A	121.08 (11)	C9B—C10B—H10B	119.8
O2A—C4A—C5A	120.79 (11)	C10B—C11B—C6B	120.20 (11)
C3A—C4A—C5A	118.11 (10)	C10B—C11B—H11B	119.9
C4A—C5A—H5A1	109.5	C6B—C11B—H11B	119.9

C3—N1—N2—C6	−175.82 (10)	N2A—C6A—C7A—C8A	177.91 (10)
N2—N1—C3—C2	1.98 (17)	C6A—C7A—C8A—C9A	0.05 (18)
N2—N1—C3—C4	179.36 (10)	C6A—C7A—C8A—N3A	−179.19 (10)
O1—C2—C3—N1	−9.03 (18)	O4A—N3A—C8A—C9A	170.20 (11)
C1—C2—C3—N1	168.77 (12)	O3A—N3A—C8A—C9A	−10.12 (17)
O1—C2—C3—C4	173.88 (11)	O4A—N3A—C8A—C7A	−10.52 (16)
C1—C2—C3—C4	−8.32 (18)	O3A—N3A—C8A—C7A	169.16 (11)
N1—C3—C4—O2	177.63 (11)	C7A—C8A—C9A—C10A	1.27 (18)
C2—C3—C4—O2	−5.00 (18)	N3A—C8A—C9A—C10A	−179.50 (11)
N1—C3—C4—C5	−1.62 (15)	C8A—C9A—C10A—C11A	−0.89 (18)
C2—C3—C4—C5	175.76 (10)	C9A—C10A—C11A—C6A	−0.77 (18)
N1—N2—C6—C7	−9.15 (16)	C7A—C6A—C11A—C10A	2.16 (18)
N1—N2—C6—C11	170.88 (10)	N2A—C6A—C11A—C10A	−177.53 (11)
C11—C6—C7—C8	1.12 (17)	C3B—N1B—N2B—C6B	173.25 (10)
N2—C6—C7—C8	−178.86 (10)	N2B—N1B—C3B—C4B	176.26 (10)
C6—C7—C8—C9	−0.34 (17)	N2B—N1B—C3B—C2B	−5.93 (17)
C6—C7—C8—N3	179.37 (10)	O1B—C2B—C3B—N1B	18.51 (17)
O4—N3—C8—C9	−168.61 (10)	C1B—C2B—C3B—N1B	−157.16 (11)
O3—N3—C8—C9	11.87 (15)	O1B—C2B—C3B—C4B	−163.87 (11)
O4—N3—C8—C7	11.66 (15)	C1B—C2B—C3B—C4B	20.45 (16)
O3—N3—C8—C7	−167.86 (10)	N1B—C3B—C4B—O2B	−172.49 (11)
C7—C8—C9—C10	−0.19 (17)	C2B—C3B—C4B—O2B	9.68 (17)

N3—C8—C9—C10	−179.90 (10)	N1B—C3B—C4B—C5B	6.87 (15)
C8—C9—C10—C11	−0.04 (17)	C2B—C3B—C4B—C5B	−170.96 (11)
C9—C10—C11—C6	0.82 (18)	N1B—N2B—C6B—C7B	−4.58 (17)
C7—C6—C11—C10	−1.38 (17)	N1B—N2B—C6B—C11B	175.21 (10)
N2—C6—C11—C10	178.60 (10)	C11B—C6B—C7B—C8B	0.68 (17)
C3A—N1A—N2A—C6A	−174.84 (10)	N2B—C6B—C7B—C8B	−179.55 (10)
N2A—N1A—C3A—C2A	2.44 (17)	C6B—C7B—C8B—C9B	0.68 (18)
N2A—N1A—C3A—C4A	−177.70 (10)	C6B—C7B—C8B—N3B	−178.10 (10)
O1A—C2A—C3A—N1A	−9.99 (18)	O4B—N3B—C8B—C9B	−171.93 (11)
C1A—C2A—C3A—N1A	168.04 (11)	O3B—N3B—C8B—C9B	7.75 (16)
O1A—C2A—C3A—C4A	170.15 (11)	O4B—N3B—C8B—C7B	6.92 (16)
C1A—C2A—C3A—C4A	−11.81 (17)	O3B—N3B—C8B—C7B	−173.40 (11)
N1A—C3A—C4A—O2A	175.02 (11)	C7B—C8B—C9B—C10B	−1.14 (18)
C2A—C3A—C4A—O2A	−5.11 (18)	N3B—C8B—C9B—C10B	177.64 (11)
N1A—C3A—C4A—C5A	−3.29 (15)	C8B—C9B—C10B—C11B	0.23 (18)
C2A—C3A—C4A—C5A	176.58 (11)	C9B—C10B—C11B—C6B	1.08 (19)
N1A—N2A—C6A—C7A	−0.64 (16)	C7B—C6B—C11B—C10B	−1.55 (18)
N1A—N2A—C6A—C11A	179.05 (10)	N2B—C6B—C11B—C10B	178.67 (11)
C11A—C6A—C7A—C8A	−1.77 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11*B*—H11*B*···O1*A*^{i}	0.95	2.63	3.4824 (15)	150
C9*B*—H9*B*···O3*A*^{ii}	0.95	2.53	3.4837 (16)	178
N2*B*—H2*B*···O1*B*	0.90 (1)	1.90 (1)	2.5952 (13)	133 (1)
C11*A*—H11*A*···O1*B*^{i}	0.95	2.53	3.4347 (15)	158
C9*A*—H9*A*···O3*B*^{ii}	0.95	2.59	3.5227 (15)	168
C5*A*—H5*A*2···N1^{i}	0.98	2.65	3.5230 (16)	149
C5*A*—H5*A*1···O1*A*^{iii}	0.98	2.51	3.4424 (15)	158
C1*A*—H1*A*2···O4^{i}	0.98	2.49	3.4674 (16)	173
N2*A*—H2*A*···O1*A*	0.90 (1)	1.91 (1)	2.5873 (13)	131 (1)
C11—H11···O1^{iv}	0.95	2.52	3.4114 (14)	156
C9—H9···O3^{v}	0.95	2.63	3.5450 (15)	163
C5—H5*B*···N1*A*^{i}	0.98	2.69	3.5445 (16)	146
C1—H1*B*···O4*A*^{i}	0.98	2.47	3.4237 (18)	165
N2—H2···O1	0.89 (1)	1.89 (1)	2.5834 (12)	134 (2)

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