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Involvement of conformational isomerism in the complexity of the crystal network of 1-(4-nitrophenyl)-1*H*-1,3-benzimidazole derivatives driven by C—H···*A* (*A* = NO_2, N_{py} and π) and orthogonal $N_{py}\cdots NO_2$ and $ONO\cdots Csp^2$ interactions

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A detailed structural analysis of the benzimidazole nitroarenes 1-(4-nitrophenyl)-1*H*-1,3-benzimidazole, $C_{13}H_9N_3O_2$, (I), 1-(4-nitrophenyl)-2-phenyl-1*H*-1,3-benzimidazole, $C_{19}H_{13}N_3O_2$, (II), and 2-(3-methylphenyl)-1-(4-nitrophenyl)-1*H*-1,3-benzimidazole, $C_{20}H_{15}N_3O_2$, (III), has been performed. They are nonplanar structures whose crystal arrangement is governed by Csp^2—H···*A* (*A* = NO_2, N_{py} and π) hydrogen bonding. The inherent complexity of the supramolecular arrangements of compounds (I) (Z' = 2) and (II) (Z' = 4) into tapes, helices and sheets is the result of the additional participation of π–π_{NO_2} and *n*–π^* (*n* = O and N_{py}; π^* = Csp^2 and N_{NO_2}) interactions that contribute to the stabilization of the equi-energetic conformations adopted by each of the independent molecules in the asymmetric unit. In contrast, compound (III) (Z' = 1) is self-paired, probably due to the effect of the steric demand of the methyl group on the crystal packing. Theoretical *ab initio* calculations confirmed that the presence of the arene ring at the benzimidazole 2-position increases the rotational barrier of the nitrobenzene ring and also supports the electrostatic nature of the orthogonal $ONO\cdots Csp^2$ and $N_{py}\cdots NO_2$ interactions.

1. Introduction

Benzimidazoles are recognized as essential chemical motifs present in a variety of natural products, agrochemicals and bioactive molecules (Keri *et al.*, 2015). Particularly, C2-aryl-substituted benzimidazoles are often found as a key unit in various natural compounds, biologically active agents, potent pharmacophores and functional chemicals (Horton *et al.*, 2003; Kumar, 2004; Candeias *et al.*, 2009; Gupta & Rawat, 2010; Carvalho *et al.*, 2011). In addition, *N*-arylbenzimidazoles are a class of prominent heterocyclic compounds that exhibit a wide range of biological properties (Sabat *et al.*, 2006; Elias *et al.*, 2011). In particular, 1,2-diarylbenzimidazoles have been reported as strong inhibitors of human cyclooxygenases with a skewed selectivity towards the COX-2 (cyclooxygenase 2) isoform at the micromolar level (Secci *et al.*, 2012).

It is worth mentioning the case of 1-(4-nitrophenyl)-1*H*-1,3-benzimidazole, (I), which has been reported as an inhibitor of platelet-derived growth factor receptor (PDGFR), which is highly expressed in tumour cells (Zhong *et al.*, 2004; Katritzky *et al.*, 2005). Experimental evidence indicates that the inhibi-

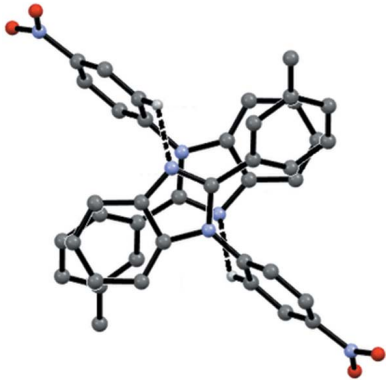
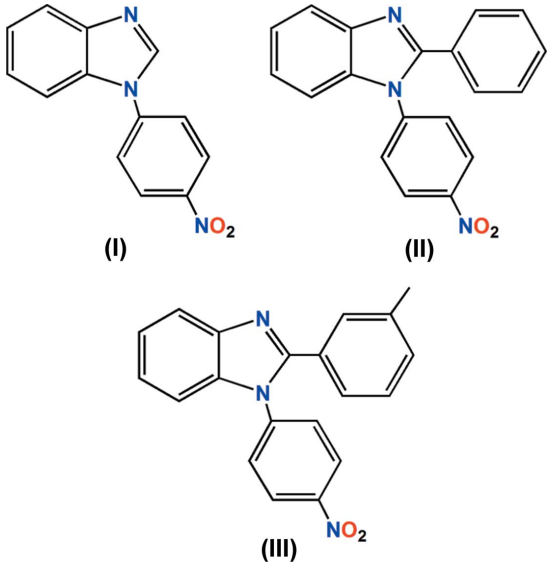


Table 1
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{13}H_9N_3O_2$	$C_{19}H_{13}N_3O_2$	$C_{20}H_{15}N_3O_2$
M_r	239.23	315.32	329.35
Crystal system, space group	Monoclinic, $C2/c$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	293	100	273
a, b, c (Å)	25.074 (3), 7.1422 (8), 24.283 (3)	10.2685 (7), 15.1411 (10), 19.4521 (14)	8.186 (4), 9.806 (4), 11.264 (5)
α, β, γ (°)	90, 96.599 (2), 90	91.886 (1), 95.725 (1), 90.118 (1)	112.825 (7), 98.468 (7), 94.276 (7)
V (Å^3)	4319.9 (9)	3007.6 (4)	815.6 (6)
Z	16	8	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.10	0.09	0.09
Crystal size (mm)	0.30 × 0.28 × 0.24	0.38 × 0.34 × 0.32	0.40 × 0.30 × 0.25
Data collection			
Diffractometer	Bruker APEXII area detector	Bruker APEXII area detector	Bruker APEXII area detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19923, 3811, 3303	16980, 10449, 8103	9543, 3787, 3177
R_{int}	0.047	0.029	0.022
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.595	0.595	0.666
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.072, 0.153, 1.20	0.054, 0.120, 1.05	0.050, 0.132, 1.04
No. of reflections	3811	10449	3787
No. of parameters	325	865	228
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.27	0.26, −0.22	0.19, −0.25

Computer programs: *APEX2* (Bruker, 2004), *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008), *SHELXL97* (Sheldrick, 2008) and *WinGX* (Farrugia, 2012).

tory activity involves discrete noncovalent dipolar protein–ligand interactions, which significantly contribute to the binding affinity and to intermolecular recognition. On the other hand, little is known about the nature of the noncovalent interactions of nitroarenes with hydrophobic aromatic protein



areas and their contribution to binding affinities, which might be relevant for the interaction with different receptors (An *et al.*, 2015). In this context, 1-(4-nitrophenyl)-2-phenyl-1*H*-1,3-benzimidazole, (II), and 2-(4-methylphenyl)-1-(4-nitrophenyl)-1*H*-1,3-benzimidazole, (III), were also synthesized and their molecular structures analysed with the aim of further understanding their pharmacophore properties, as well their use in the design of materials with specific functions. Moreover, compounds (I)–(III) are characterized by the presence of strong hydrogen-bond acceptor groups but weak hydrogen-bond donors, allowing us to further expand our knowledge of the roles of noncovalent intermolecular forces in crystal engineering and supramolecular chemistry.

2. Experimental

2.1. Instrumental

The uncorrected melting points were measured in open-ended capillary tubes in an Electrothermal apparatus IA 9100. ^{1}H (300.01 MHz) and ^{13}C NMR (75.46 MHz) spectra were recorded on a Varian Mercury-300 spectrometer using $CDCl_3$ as solvent and tetramethylsilane (TMS) as internal reference; chemical shift values (δ) are in parts per million (ppm) and coupling constants (J values) are in Hertz (Hz). IR spectra was obtained with a 3100 FT–IR Excalibur Series spectrophotometer.

2.2. Theoretical calculations

Geometry optimizations at the B3LYP/6-31G(d,p) level of theory were performed without any symmetry restraints using the *GAUSSIAN09* package (Frisch *et al.*, 2009). Relaxed linear potential energy surface scans for the N1—C10 and C2—C16 rotations were performed using direct inversion of iterative subspace (GDIIS) (Farkas & Schlegel, 2002).

2.3. Synthesis and crystallization

2.3.1. 1-(4-Nitrophenyl)-1*H*-1,3-benzimidazole, (I). Compound (I) was prepared from benzimidazole (1.00 g, 8.47 mmol) and 1-fluoro-4-nitrobenzene (1.19 g, 8.47 mol) in a basic medium of K_2CO_3 (2.34 g, 16.9 mmol) in dimethyl sulfoxide (13 ml) at 393 K for 20 h, as reported for 2-(4-bromophenyl)-1-(4-nitrophenyl)-1*H*-benzimidazole (González-Padilla *et al.*, 2014). The compound was obtained as a pale-yellow solid in 96% yield (m.p. 453–454 K). Crystals of (I) were obtained after crystallization from an ethanol solution. ^{1}H NMR: δ 8.48 (*m*, 2H, H-12,14), 8.20 (*s*, 1H, H-2), 7.92 (*m*, 1H, H-4), 7.41 (*m*, 2H, H-5,6), 7.62 (*m*, 1H, H-7), 7.77 (*m*, 2H, H-11,15). ^{13}C NMR: δ 146.7 (C-13), 144.6 (C-9), 141.9 (C-10), 141.8 (C-2), 133.0 (C-8), 126.1 (C-12,14), 124.8 (C-6), 124.0 (C5), 123.9 (C-11,15), 121.4 (C-4), 110.5 (C-7). IR (neat, ν, cm^1): 1595, 1507 (C═C Ar), 1347 (NO_2), 848, 754 (C—H out of plane).

2.3.2. 1-(4-Nitrophenyl)-2-phenyl-1*H*-1,3-benzimidazole, (II). Compound (II) was prepared from 2-phenyl-1*H*-1,3-benzimidazole (0.216 g, 1.11 mmol) and 1-fluoro-4-nitrobenzene (0.157 g, 1.11 mmol) in a basic medium of K_2CO_3 (0.155 g, 1.11 mmol), dimethylformamide (2 ml) and CuCl (11 mg) as catalyst, as a yellow solid in 72% yield (m.p. 421–423 K). Crystals suitable for X-ray diffraction were obtained from a hexane/ethyl acetate solution. ^{1}H NMR: δ 8.34 (*m*, 2H, H-12,14), 7.88 (*d*, 1H, H-4, ^3J = 7.6 Hz), 7.48 (*m*, 4H, H-11,15,17,21), 7.34 (*m*, 6H, H5-7, 18-20). ^{13}C NMR: δ 152.4 (C2), 147.2 (C9), 143.4 (C13), 142.8 (C10), 136.4 (C8), 130.2 (C19), 129.7 (C16), 129.5 (C18,20), 128.9 (C17), 128.1 (C12,14), 125.5 (C11,15), 124.3 (C6), 124.0 (C5), 120.6 (C4), 110.1 (C7). IR (neat, ν, cm^1): 1590, 1526 (C═C Ar), 1350 (NO_2), 778, 746, 694 (C—H out of plane).

2.3.3. 2-(4-Methylphenyl)-1-(4-nitrophenyl)-1*H*-1,3-benzimidazole, (III). Compound (III) was prepared from 2-*m*-tolyl-1*H*-1,3-benzimidazole (0.300 mg, 1.44 mmol) as a yellow solid in 20% yield after silica-gel chromatography (m.p. 447.7–449.0 K). Crystals suitable for X-ray diffraction were obtained from an hexane/ethyl acetate solution. ^{1}H NMR: δ 8.38 (*m*, 2H, H-18,20), 7.90 (*d*, 1H, H-4, ^3J = 7.8 Hz), 7.54 (*s*, 1H, H15), 7.50 (*m*, 2H, H-17,21), 7.34 (*m*, 6H, H5-7, 11-13), 2.34 (*s*, 3H, Me). ^{13}C NMR: δ 152.7 (C2), 147.2 (C9), 143.4 (C10), 142.8 (C13), 139.0 (C20), 136.3 (C8), 131.1 (C18), 130.5 (C17), 129.3 (C16), 128.6 (C19), 128.1 (C12,14), 126.7 (C21), 125.1 (C11,15), 124.2 (C6), 124.0 (C5), 120.6 (C4), 110.1 (C7), 21.6 (Me). IR (neat, ν, cm^1): 1678 (C═N), 1590, 1515 (C═C Ar), 1347, 1303 (NO_2), 791, 756, 745, 695 (C—H out of plane).

2.4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms on C atoms were positioned geometrically and treated as riding atoms, with C—H = 0.95–0.99 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms or 1.2U_{eq}(C) otherwise.

3. Results and discussion

3.1. Molecular and supramolecular structure of compound (I)

Two independent molecules, *i.e.* *A* (atoms N1/C2/N3/C4–C15/N13/O13*A*/O13*B*) and *B* (N21/C22/N23/C24–C35/N33/O33*A*/O33*B*), appear in the asymmetric unit of compound (I) (Fig. 1), which crystallizes in the monoclinic space group *C*2/*c*. Molecules (I*A*) and (I*B*) are related by a second-order pseudo-helicoidal axis. The nitrobenzene ring (denoted N-nitroBz) is twisted from the mean benzimidazole (Bzm) plane by 35.71 (9) and 40.11 (7)° in molecules (I*A*) and (I*B*), respectively (Spek, 2009). The first value is very close to that observed in 6-methoxy-1-(4-nitrophenyl)-1*H*-1,3-benzimidazole (36.15°; Kumar *et al.*, 2013). The NO_2 group is in the plane of the nitrobenzene ring in (I*A*) [C14—C13—N13—O13*B* = −2.2 (4)°] and twisted in (I*B*) [C34—C33—N33—O33*B* = −22.4 (4)°]. However, the C—NO_2 bond lengths are equal in both molecules and are also in the expected range (Allen *et al.*, 1987), suggesting a limited conjugation between them. The N-nitroBz ring in the reported crystal structures of 1-(4-nitrophenyl)pyrazole and 1-(4-nitrophenyl)pyrrole (Ishihara *et al.*, 1992) is almost coplanar with the heterocyclic ring. Thus, the observed twist of the N-nitroBz ring from the Bzm plane in compound (I) is the result of steric repulsion between the fused benzene and N-nitroBz rings. This last ring can adopt a

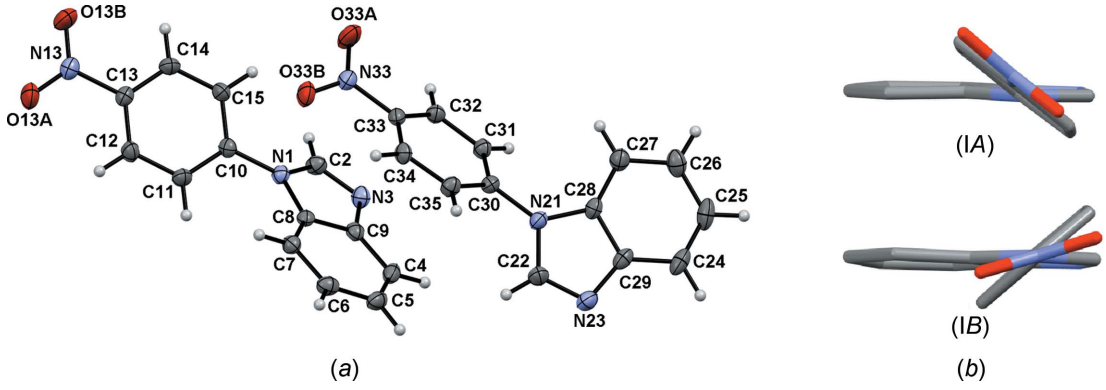


Figure 1
(*a*) The molecular structure of compound (I), with displacement ellipsoids drawn at the 30% probability level. Two independent molecules, *i.e.* (I*A*) (atoms N1/C2/N3/C4–C15/N13/O13*A*/O13*B*) and (I*B*) (N21/C22/N23/C24-C35/N33/O33*A*/O33*B*), are present in the asymmetric unit. (*b*) A view of rotamers (I*A*) and (I*B*) along the N13···N3 and N33···N23 imaginary axes, respectively.

perpendicular disposition, in relation to the Bzm heterocycle, similar to those structures with high steric demand such as phenanthroimidazoles (Zhang *et al.*, 2016).

Soft Csp^2—H···O interactions give shape to the crystal packing, with the participation of a nitro O atom, as the acceptor, in a monocoordination fashion (Allen *et al.*, 1997). Two (I*A*) molecules form centrosymmetric dimers, *i.e.* A_2, through C12—H12···O13A^{i} interactions, describing a twisted $R_2^2(10)$ motif (Bernstein *et al.*, 1995) (Fig. 2*a*). Furthermore, a *meso* helix is developed along the [030] direction through C7—H7···*Cg*2^{v} T-shaped interactions linking the A_2 dimers [*Cg*2 is the centroid of the C4–C9 ring; symmetry code: (v) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$]. (I*B*) molecules self-associate into *C*(11) chains through C24—H24···O33B^{iii} interactions, which propagate within the $(\bar{1},1,11)$ and $(\bar{1},\bar{1},11)$ families of planes. *A* and *B* molecules of compound (I) are connected through C15—H15···O33A^{ii} interactions. Chains of (I*B*) running in the $[\bar{1},1,11]$ direction and *n* molecules of (I*B*), each belonging to an infinite number of (I*B*) chains running within the $(\bar{1},\bar{1},11)$ family of planes, are linked to A_2 helices, forming the *M* and *P* strands B_nnA_2nB. The second dimension is given by the interlinkage of the strands through C35—H35···O13A^{iv} interactions (Figs. 2*b* and 2*c*). The geometric features and symmetry codes associated with these interactions are listed in Table 2. Molecule (I*B*) displays a twist of 22.4 (4)° of the NO_2 group, which is comparable to that seen in high-energy molecules such as TNT (Landenberger & Matzger, 2010). This torsion, together with an N-nitroBz torsion of 40.11 (7)° from the Bzm plane, favour the helical arrangement of (I) in the solid (Ramírez-Milanés *et al.*, 2017).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O13A^{i}	0.93	2.45	3.350 (4)	164
C15—H15···O33A^{ii}	0.93	2.59	3.515 (4)	173
C24—H24···O33B^{iii}	0.93	2.55	3.375 (4)	149
C35—H35···O13A^{iv}	0.93	2.64	3.281 (4)	127

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

The three-dimensional structure is developed by nitro–π and π–π_{NO_2} dispersive interactions, *viz.* the nitro group of

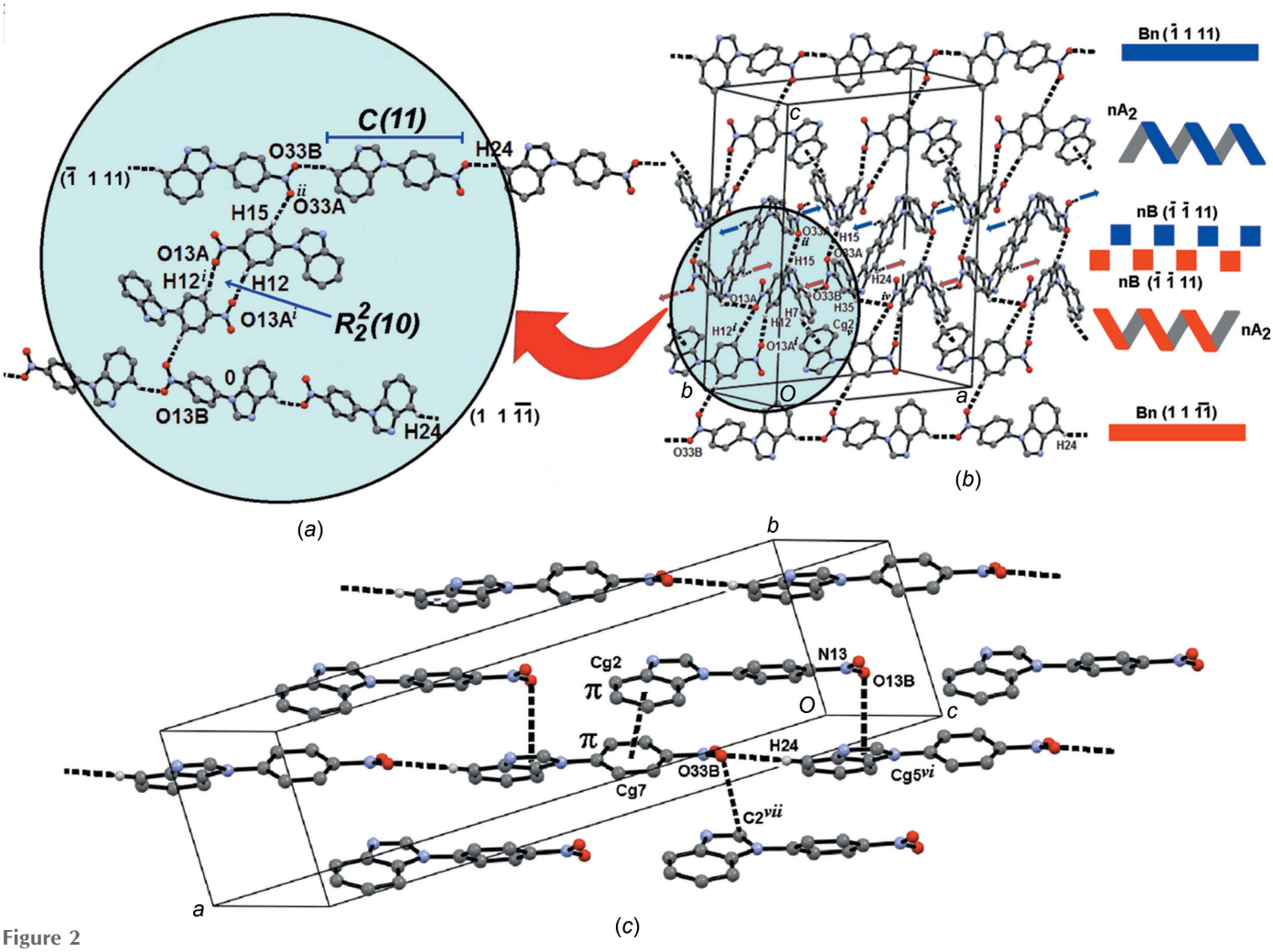


Figure 2
The two-dimensional supramolecular architecture of compound (I), built up by C—H···O interactions. (*a*) Dimers A_2 and tapes B_n are shown. (*b*) The *M* and *P* strands formed by the interlinkage of tapes, and helices B_nnA_2nB. (*c*) Dispersive interactions ONO···*Cg*, *Cg*···*Cg* and ONO···C2, giving rise to the three-dimensional network of compound (I).

Table 3
Experimental angles (°) between the planes of the Bzm, N-nitroPh and C2-Ph rings in molecules *A*–*D* of compound (II).

Planes		Angles (°)					
1	2	(II*A*)	(II*B*)	Mean value (II*A*) and (II*B*)	(II*C*)	(II*D*)	Mean value (II*C*) and (II*D*)
Bzm	N-nitroBz	54.20 (5)	54.40 (5)	54.30 (7)	60.47 (5)	58.02 (5)	59.25 (7)
Bzm	C2-Ph	29.05 (6)	29.10 (6)	29.08 (8)	31.22 (6)	32.74 (6)	26.98 (8)
N-nitroBz	C2-Ph	58.65 (5)	59.16 (5)	58.91 (7)	68.44 (6)	66.70 (5)	67.57 (8)

molecule (I*A*) to the centroid of the heterocyclic ring of molecule (I*B*), *i.e.* N13···O13*B*···*Cg*5^{vi} [O13*B*···*Cg*5 = 3.346 (3) Å, N13···*Cg*5 = 3.431 (3) Å and N13···O13*B*···*Cg*5 = 83.68 (19)°; *Cg*5 is the centroid of the N21/C22/N23/C29/C28 ring; symmetry code: (vi) $x-\frac{1}{2}, y-\frac{1}{2}, z$], and *Cg*2···*Cg*7^{v}, between the aromatic ring (*Cg*2 is the centroid of the C4—C9 ring and *Cg*7 is the centroid of the C30–C35 ring) of the Bzm moiety of molecule (I*A*) and the nitrobenzene ring of molecule (I*B*). The intercentroid *Cg*2···*Cg*7^{v} distance [3.6123 (17) Å] is very close to the interplanar distance [3.4361 (12) Å], in agreement with a face-to-face interaction (García-Báez *et al.*, 2003). It is worth mentioning that the calculated value of the gas-phase binding energy of π–π_{NO_2} stacking has been reported as −6.7 kcal mol^{-1} (1 kcal mol^{-1} = 4.184 kJ mol^{-1}) between phenylalanine and nitrobenzene (An *et al.*, 2015), pointing to the relevance of this interaction in the crystal lattice arrangement.

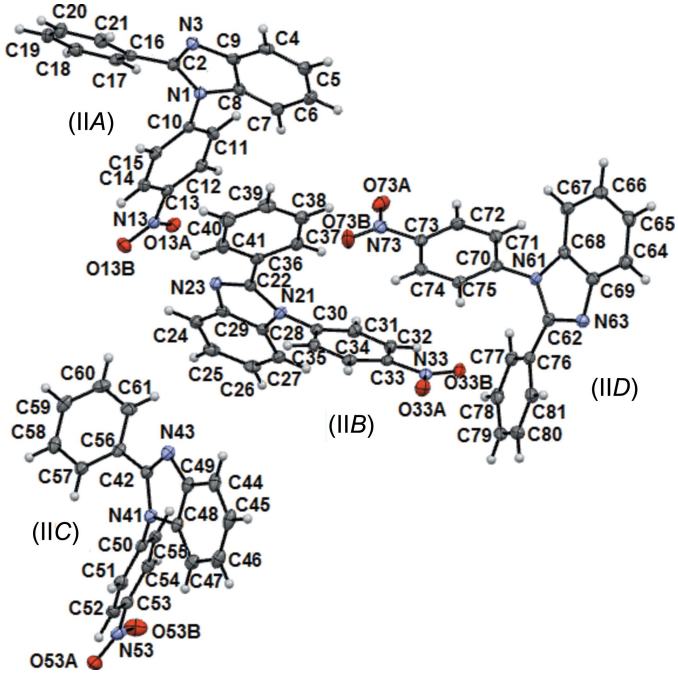


Figure 3
The molecular structure of compound (II), with displacement ellipsoids drawn at the 30% probability level. Four independent molecules are present in the asymmetric unit, namely *A* (atoms N1/C2/N3/C4–C15/N13/O13*A*/O13*B*), *B* (N21/C22/N23/C24–C35/N33/O33*A*/O33*B*), *C* (N41/C42/N43/C44–C55/N53/O53*A*/O53*B*) and *D* (N61/C62/N63/C64–C75/N73/O73*A*/O73*B*).

In addition, an intermolecular $NO_2\cdots Csp^2$ interaction is observed between a nitro O atom as donor and the C2 atom of the NCN fragment of the heterocyclic Bzm ring. The geometric parameters associated with this last n–π^* interaction are O33*B*···C2^{vii} = 3.213 (3) Å and N33···O33*B*···C2 = 93.7 (2)° [symmetry code: (vii) $x, y-1, z$] (Fig. 2*c*). This interaction has been described in 3,3′-dinitro-2,2′-bipyridine *N*-oxides, with distances in the range 2.762 (4)–2.789 (3) Å (O'Leary & Wallis, 2007), clearly shorter than in (I) because of its intramolecular nature. Additionally, an analogous interaction of the nitrile group with the C2 atom of the Bzm ring, CN···Csp^2, occurred in (*Z*)-3-(4-nitrophenyl)-2-(1-phenyl-1*H*-benzimidazol-2-yl)acrylonitrile (Hranjec *et al.*, 2012).

3.2. Molecular and supramolecular structure of compound (II)

Compound (II) crystallizes in the triclinic space group $P\overline{1}$, with four independent molecules in the asymmetric unit (Fig. 3), namely (II*A*) (atoms N1/C2/N3/C4–C15/N13/O13*A*/O13*B*), (II*B*) (N21/C22/N23/C24–C35/N33/O33*A*/O33*B*), (II*C*) (N41/C42/N43/C44–C55/N53/O53*A*/O53*B*) and (II*D*) (N61/C62/N63/C64–C75/N73/O73*A*/O73*B*). Molecules (II*A*) and (II*C*), as well as (II*B*) and (II*D*), are related by a local pseudocentre of inversion located at the fractional coordinates (0.276, 0.376, 0.626) and (0.272, 0.876, 0.626), respectively, in the asymmetric unit. This condition has frequently been observed in $P\overline{1}$ crystals of high-Z' structures (Desiraju, 2007). The N-nitroBz and C2-Ph rings are both twisted from the mean Bzm plane; the angles between the planes of the Bzm, N-nitroBz and C2-Ph rings are listed in Table 3. In spite of their inherent crystallographic differences, molecules (II*A*) and (II*B*) have similar angles, as have molecules (II*C*) and (II*D*), judged by the mean values of the angles between the planes. The N-nitroBz ring in compound (II) deviates more

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O13*A*^{i}	0.95	2.52	3.298 (3)	139
C7—H7···O33*B*^{ii}	0.95	2.54	3.357 (3)	145
C15—H15···O53*B*^{iii}	0.95	2.61	3.468 (3)	151
C26—H26···O33*B*^{iv}	0.95	2.52	3.302 (3)	140
C27—H27···O13*A*^{v}	0.95	2.53	3.355 (3)	145
C35—H35···O73*B*^{v}	0.95	2.64	3.527 (3)	156
C44—H44···O73*A*^{v}	0.95	2.66	3.309 (3)	126
C52—H52···O53*A*^{vi}	0.95	2.46	3.299 (3)	148
C54—H54···N3^{vii}	0.95	2.47	3.412 (3)	171
C55—H55···O33*A*^{iv}	0.95	2.33	3.184 (3)	149
C64—H64···O53*A*^{viii}	0.95	2.51	3.227 (3)	132
C74—H74···N23^{v}	0.95	2.50	3.437 (3)	167
C75—H75···O13*B*^{v}	0.95	2.34	3.217 (3)	153

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

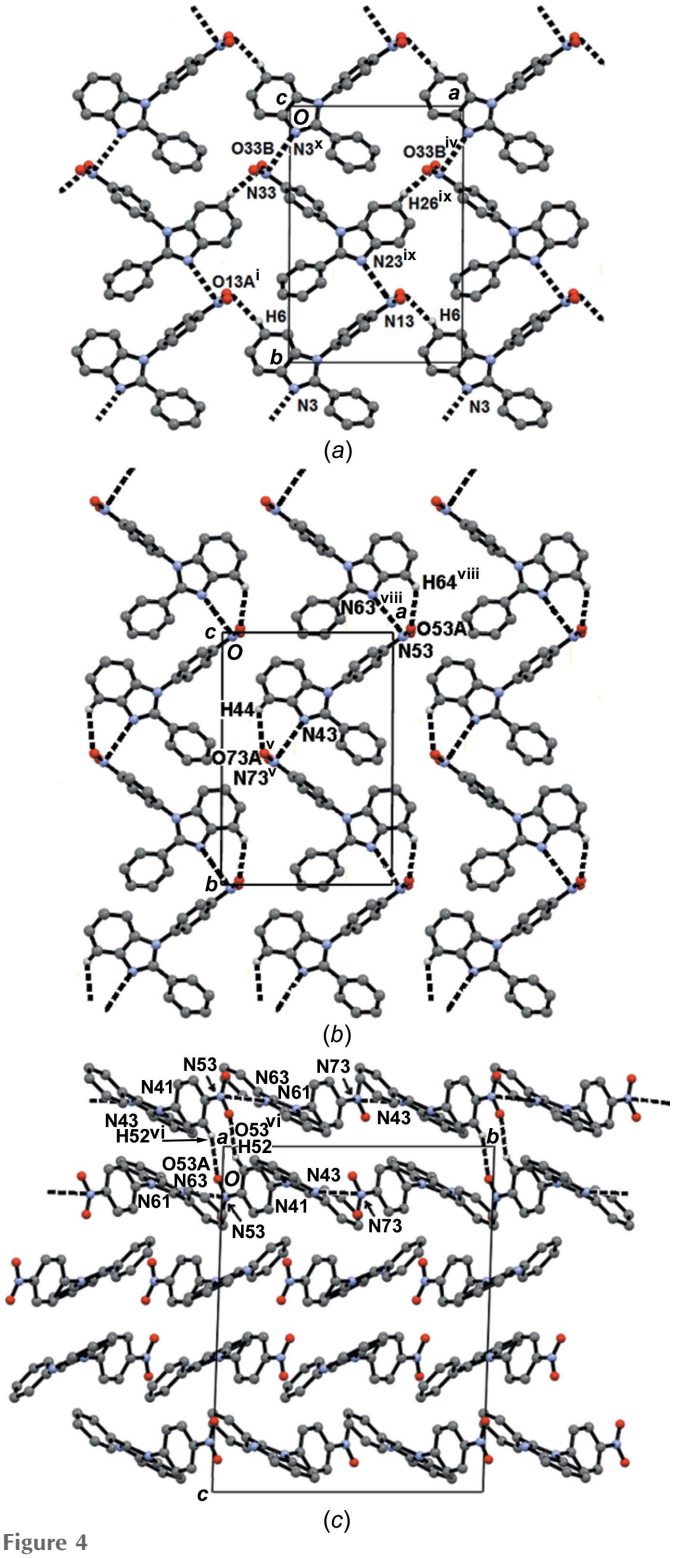


Figure 4
The supramolecular architecture of compound (II). (*a*) The interlinkage of $C_2^2(16)$ chains propagating along the *b* axis developing the two-dimensional arrangement of molecules (II*A*) and (II*B*) in the *ab* plane. (*b*) Ribbons of (II*C*)/(II*D*) developing along the *b* axis; the (II*D*) molecule with symmetry code (*x*, *y*, *z*) is not shown for clarity. (*c*) $(DC_2D)_n$ ribbons within the *bc* plane.

from coplanarity with the Bzm ring than the C2-Ph ring, but within the range found for 1,2-diphenylbenzimidazole compounds (González-Padilla *et al.*, 2014) and 1-(4-nitrophenyl)-2-phenylimidazole (Ishihara *et al.*, 1992).

Table 5
N···NO_2 geometric parameters (Å, °) for (II).

C—N···N	N···N	C—N···N
C13—N13···N23^{ix}	3.050 (2)	88.49 (19)
C33—N33···N3^{x}	3.053 (2)	88.33 (19)
C53···N53···N63^{viii}	3.102 (2)	90.58 (19)
C73···N73···N43^{v}	3.134 (2)	91.59 (19)

Symmetry codes: (v) $-x+1, -y+1, -z+1$; (viii) $-x+1, -y, -z+1$; (ix) x, y, z; (*x*) $x, y-1, z$.

Molecules (II*A*) and (II*B*) are linked through N···NO_2 interactions (N13···N23 and N33···N3), with the participation of the pyridine-like N atom as the donor and the N atom of the nitro group as the acceptor, forming $C_2^2(16)$ chains propagating along the *b* axis. Chains of (II*A*) and (II*B*) molecules are linked through C6—H6···O13*A*^{i} and C26—H26···O33*B*^{iv} soft hydrogen bonds to develop a sheet within the *ab* plane. C7—H7···O33*B*^{ii} and C27—H27···O13*A*^{v} soft hydrogen bonds are responsible for linking two (II*A*)/(II*B*) planes along the *c*-axis direction, *i.e.* $(A_2B_2)_n$ (Fig. 4*a*). These C—H···O interactions are of the bifurcated type with respect to the acceptor O atoms, *i.e.* H6···O13*A*···H27 and H7···O33*B*···H26. The geometrical parameters and symmetry codes of the hydrogen-bonding and N···NO_2 interactions are listed in Tables 4 and 5, respectively.

Molecules (II*C*) and (II*D*) develop a $(DC_2D)_n$ ribbon within the $(10\overline{1})$ family of planes (Figs. 4*b* and 4*c*), also through N···NO_2 and C—H···O interactions (N63···N53^{viii}, N43···N73^{v}, C44—H44···O73*A*^{i}, C64—H64···O53*A*^{viii} and C52—H52···O53*A*^{vi}). The $(A_2B_2)_n$ double sheets and $(DC_2D)_n$ ribbons are interleaved to develop the three-dimensional structure along the *c*-axis direction through C—H···*X* (*X* = N and O) interactions (C54—H54···N3^{vii}, C74—H74···N23^{v}, C15—H15···O53*B*^{iii}, C35—H35···O73*B*^{v}, C55—H55···O33*A*^{iv} and C75—H75···O13*B*^{v}), with the participation of the pyridine-like N and nitro O atoms as acceptors.

Remarkably, the intermolecular N_{py}···NO_2 (n–π^*) interaction plays a crucial role in the molecular self-assembly and crystal packing of compound (II). The nitro N atoms have been observed to interact with electron-rich centres, such as an O atom of another nitro group (Daszkiewicz, 2013), the N atom of a dimethylamino group in perinaphthalenes (Egli *et al.*, 1986; Ciechanowicz-Rutkowska, 1977) and the pyridine-like N atom of azole compounds (Yap *et al.*, 2005). The geometric parameters of the intermolecular N···NO_2 interactions in (II) are similar to the values found in the crystal structure of 2-methyl-4,6-dinitro-1-(2,4,6-trinitrophenyl)benzimidazole (Freyer *et al.*, 1992), with N···N = 3.089 Å and C···N···N = 95.4°.

The NO_2···Csp^2 and N_{py}···NO_2 interactions present in (I) and (II), respectively, are of the orthogonal n–π^* type, since the donor atom approaches in a perpendicular manner to the plane that includes the acceptor. N···NO_2 interactions have been envisaged as an entry to supramolecular cages without using metals to make orthogonal corners (Yap *et al.*, 2005).

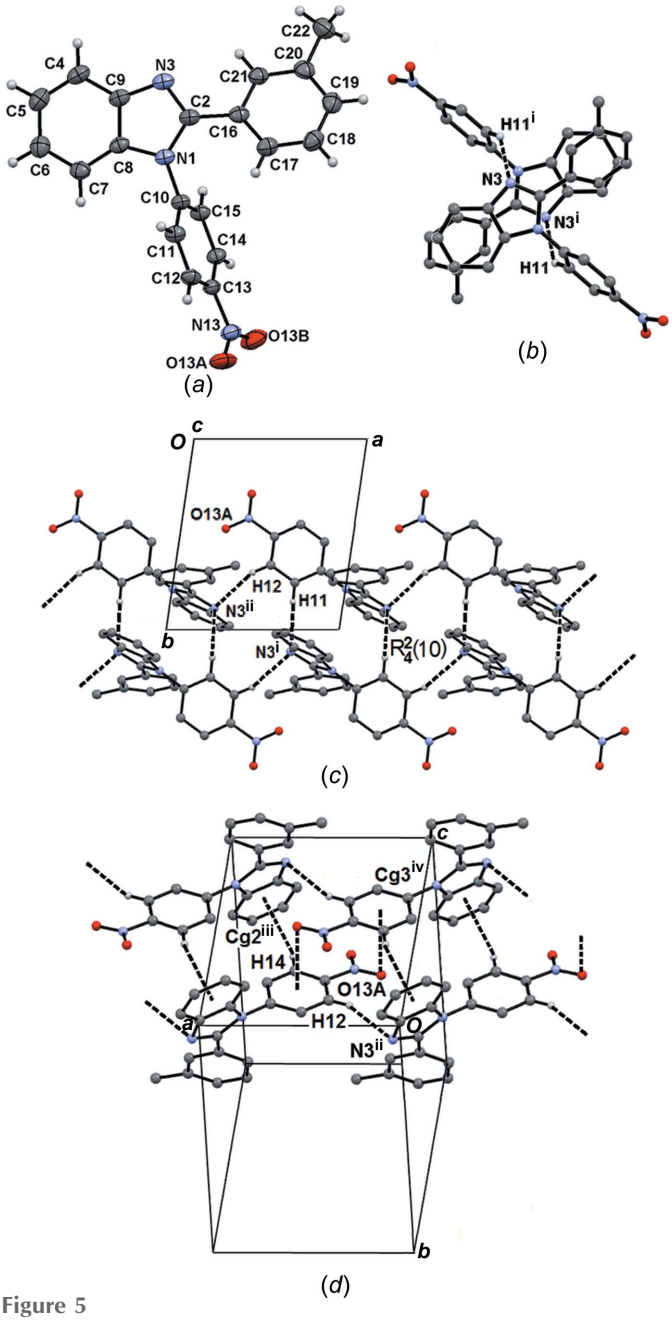


Figure 5
(*a*) The molecular structure of compound (III), with displacement ellipsoids drawn at the 30% probability level. The supramolecular structure as (*b*) zero-dimensional, (*c*) one-dimensional, and (*d*) two- and three-dimensional.

The crystal structure of compound (II) is an example of a compound with many symmetry-independent molecules in the asymmetric unit. This phenomenon has been extensively analysed elsewhere (Bernstein *et al.*, 2008). The introduction of a C2-Ph ring in compound (II) to the already present N-nitroBz ring in (I) is expected to increase the rotational barrier of the latter, reducing the possibilities of conformational isomers. Nevertheless, the effect is the opposite and contrasts with similar structures lacking the nitro group, such as 1-phenyl-2-*p*-tolyl-1*H*-benzimidazole (Mohandas *et al.*, 2013) and 1,2-diphenyl-1*H*-benzimidazole (Rosepriya *et al.*, 2012), or those containing a nitro group, 6-ethyl-1-(4-nitrophenyl)-2-phenyl-1*H*-benzimidazole (Kumar & Punniyamurthy, 2012), but having steric constraints. All of them only have one molecule in the asymmetric unit.

Table 6
Hydrogen-bond geometry (Å, °) for (III).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···N3^{i}	0.93	2.66	3.431 (2)	141
C12—H12···N3^{ii}	0.93	2.47	3.348 (2)	157

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

3.3. Molecular and supramolecular structure of compound (III)

Compound (III) crystallizes in the triclinic space group $P\overline{1}$, with one molecule in the asymmetric unit (Fig. 5*a*). Both N-nitroBz and C2-MeBz rings are twisted from the mean Bzm plane, the angles between the planes being 67.74 (4) (Bzm and N-nitroBz), 28.21 (5) (Bzm and C2-MeBz) and 64.77 (5)° (N-nitroBz and C2-MeBz), *i.e.* more twisted than in compound (II). The NO_2 group is almost in the plane of the N-nitroBz ring [C14—C13—N13—O13*B* = 6.9 (2)°].

Molecules of (III) are self-assembled in pairs through C11—H11···N3^{i} interactions in the form of an $R_2^2(12)$ ring (Fig. 5*b*). Infinite tapes propagating along the *a*-axis direction are developed by C12—H12···N3^{ii} soft hydrogen bonds, forming an $R_4^2(10)$ ring motif (Fig. 5*c*). Finally, the two- and three-dimensional structures are arranged through C14—H14···*Cg*2^{iii} and N13—O13*A*···*Cg*3^{iv} dispersive interactions [O13*A*···*Cg*3^{iv} = 3.254 (2) Å and N13···O13*A*···*Cg*3 = 94.12 (11)°; *Cg*2 is the centroid of the C4–C9 ring and *Cg*3 is the centroid of the C10–C15 ring; symmetry codes: (iii) $-x, -y+1, -z$; (iv) $-x+1, -y+1, -z+2$] (Fig. 5*d*). The geometrical parameters and symmetry codes of the hydrogen bonding for compound (III) are listed in Table 6.

3.4. Calculated molecular structures of compounds (I)–(III)

Ab initio theoretical density functional theory (DFT) calculations at the B3LYP/6-31G(d,p) level of theory were performed to support the experimental findings. The calculated geometric parameters are in agreement with the experimental ones. In general, the differences between the geometrical parameters in the experimental and optimized geometries are in most cases 0.01 Å (bond lengths) and 0.5° (bond angles), but large differences are observed for torsion angles that might be attributed to the gas-phase calculations without considering the crystal-packing forces. Additionally, the greater differences in favour of the N-nitroBz ring might be attributed to the presence of the nitro group, which is involved in intermolecular interactions.

The nitro group retrieves electronic density from the benzene ring, so the Csp^2—H hydrogens bear a significant positive charge, particularly H12 and H14, which are both in *ortho* positions with respect to the nitro group. The calculated MKS charges are listed in Table 7. These H atoms lead to the

Table 7
Selected MKS charges calculated at the B3LYP/6-31G(d,p) level of theory for compounds (I)–(III).

	MKS charge				MKS charge		
Atom	(I)	(II)	(III)	Atom	(I)	(II)	(III)
N1	−0.183	−0.324	−0.409	H4	0.183	0.172	0.164
C2	0.277	0.473	0.531	H6	0.142	0.133	0.133
N3	−0.597	−0.621	−0.541	H7	0.146	0.147	0.142
C10	0.185	0.303	0.359	H12	0.156	0.161	0.165
N13	0.661	0.648	0.659	H14	0.163	0.171	0.177
O13*A*	−0.394	−0.392	−0.396	H11	0.133	0.148	0.155
O13*B*	−0.394	−0.389	−0.393	H15	0.137	0.128	0.131

Table 8
Experimental and theoretically calculated torsion angles (°) in compounds (I)–(III).

	Molecule	(I)	(II)	(III)
C8—N1—C10—C11	Calculated	−39.43	58.60	58.72
C8—N1—C10—C11	*A*	−32.47	−59.16	72.23 (19)
C28—N21—C30—C31	*B*	38.81	59.18	
C48—N41—C50—C51	*C*		63.54	
C68—N61—C70—C71	*D*		60.94	
N1—C2—C16—C17	Calculated		33.73	33.31
N1—C2—C16—C17	*A*		−29.19	29.5 (2)
N21—C22—C36—C37	*B*		29.69	
N41—C42—C56—C57	*C*		29.99	
N61—C62—C76—C77	*D*		32.26	

formation of the hydrogen-bonding network in compound (I). The *n*–π^* donor–acceptor interactions ONO···Csp^2 and N_{py}···NO_2 observed in (I) and (II), respectively, are charge assisted. In both molecules, the N atom of the nitro group bears the most positive charge, followed by the C atom of the NCN fragment in the heterocyclic ring. In contrast, the pyridine-like N atom (N_{py}) of the heterocycle bears the most negative charge, followed by the O atoms of the nitro group. The C2-Ph substitution in compound (II) has the effect of increasing the absolute value of the charges in the NCN fragment with the concomitant diminution of the dipolar moment [2.32 Debye in (I) to 1.84 Debye in (II)].

The theoretical energy profiles of compounds (I)–(III) were also calculated to estimate the energy involved in the interconversion between the N-nitroBz and C2-Ph rotamers. The experimental and theoretical torsion-angle values are listed in Table 8. The calculated most-stable rotamer of compound (I) is similar to that adopted by molecule (I*A*) in the crystal lattice, where the N-nitroBz ring is twisted from the mean Bz plane, with a C8—N1—C10—C11 torsion angle of −39.43 (calculated) *versus* −32.47° (experimental). The maximum energy values of 3.27 or 2.10 kcal mol^{-1} were found when the N-nitroBz ring is coplanar or perpendicular to the benzimidazole heterocycle, respectively. Because of symmetry reasons and the small cost in energy, the calculated rotamer with a C8—N1—C10—C11 torsion angle of 39.43° is equally probable (see Fig. S1 in the supporting information). The rotational barriers of compounds (I)–(III) are listed in Table S1 in the supporting information.

In the case of compound (II), the calculated C8—N1—C10—C11 and N1—C2—C16—C17 torsion angles of 58.60 and 33.73°, respectively, are in close correspondence with the mean absolute value of the four molecules (II*A*)–(II*D*) found in the asymmetric unit (60.7±2.1 and 30.3±1.4°, respectively). The N1—C2—C16—C17 torsion angle was fixed at 33.73° to calculate the rotational barrier of the N-nitroBz ring. A maximum peak of energy 8.95 kcal mol^{-1} was found when the N-nitroBz ring is in the same plane as the Bz heterocycle and of only 1.60 kcal mol^{-1} when perpendicular (Fig. 6). Thus, the steric effect of the C2-Ph ring increases by 2.7-fold the energy required to rotate the N-nitroBz ring. The rotational barrier of the C2-Ph ring was calculated by fixing the C8—N1—C10—C11 torsion angle at 58.60°. Two energy maxima were found when the C2-Ph ring is perpendicular to or coplanar with the benzimidazole plane, with values of 3.60 and 0.99 kcal mol^{-1}, respectively (see Fig. S2 in the supporting information). Therefore, the rotational barrier of the C2-Ph ring is just 40% of the energy required to rotate the N-nitroBz ring.

The calculated C8—N1—C10—C11 torsion angle of 58.72° contrasts with the experimental value of 72.23 (19)° for compound (III). This difference could be explained because of C11—H11···N3 hydrogen bonding to form the already described self-paired structure (*vide supra*). However, the energy profile and the maximum peak of energy 9.20 kcal mol^{-1} were found to be very similar to those exhibited by compound (II), in agreement with a negligible steric effect from the methyl group (see Figs. S3 and S4 in the supporting information).

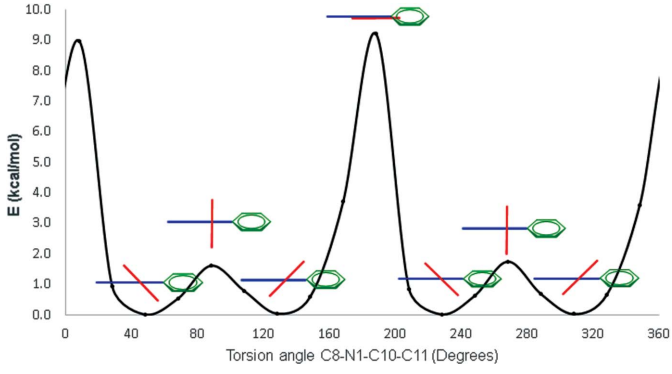


Figure 6
Theoretical rotation profile of the C8—N1—C10—C11 torsion angle in compound (II). The Bzm heterocycle in shown in blue, the N-nitroBz ring in red and the C2-Ph ring in green.

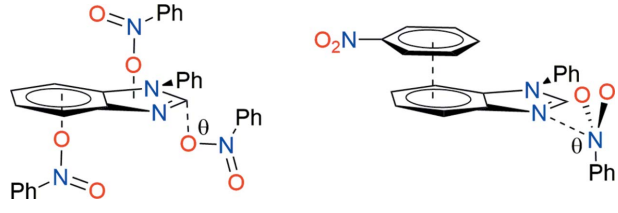


Figure 7
Pictorial representations of several interactions of the nitro group in the supramolecular architecture of compounds (I)–(III), showing nitro–π^* (left), and π–π^* and N···NO_2 (right). The θ angles are around 90°

In summary, compounds (I)–(III) are nonplanar molecules having two strong hydrogen-bonding acceptors groups, *i.e.* an electron-withdrawing nitro group at one end and an electron-donating amino group at the other end. However, the structures lack strong hydrogen-bond donors. Thus, the crystal networks are developed by dispersive soft interactions, with the participation of the nitro group, namely Csp^2—H···ONO, π_{NO_2}–π and n–π^* (n = O and N_{py}; π^* = Csp^2 and N_{NO_2}) interactions (Fig. 7).

Theoretical calculations confirmed that the presence of the C2-Bz ring increases the rotational barrier of the N-nitroBz ring, thus fewer conformers are expected. However, compound (II) is a high Z'-structure where equi-energetic conformers co-exist in the crystal network. Calculations also supported the fact that orthogonal ONO···Csp^2 and N_{py}···N_{NO_2} interactions are assisted by electrostatic attraction. The study of these molecules bearing the benzimidazole pharmacophore and nitroarenes would address the issues related to the steric and geometrical preferences for the occurrence of molecular aggregation through nitro-group interactions with important pharmacological protein targets and the development of new materials. In this regard, a family of nitroarene–benzimidazole compounds are under investigation as COX inhibitors by our research group.

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supporting information

Acta Cryst. (2018). C**74**, 428-436 [https://doi.org/10.1107/S2053229618003406]

Involvement of conformational isomerism in the complexity of the crystal network of 1-(4-nitrophenyl)-1*H*-1,3-benzimidazole derivatives driven by C—H···*A* (*A* = NO_2, N_{py} and π) and orthogonal $N_{py}\cdots NO_2$ and $ONO\cdots Csp^2$ interactions

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Computing details

For all structures, data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *WinGX* (Farrugia, 2012).

1-(4-Nitrophenyl)-1*H*-1,3-benzodiazole (I)

Crystal data

$C_{13}H_9N_3O_2$
M_r = 239.23
Monoclinic, *C*2/*c*
a = 25.074 (3) Å
b = 7.1422 (8) Å
c = 24.283 (3) Å
β = 96.599 (2)°
V = 4319.9 (9) Å^3
Z = 16
F(000) = 1984
D_x = 1.471 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 600 reflections
θ = 20–25°
μ = 0.10 mm^{-1}
T = 293 K
Block, colorless
0.30 × 0.28 × 0.24 mm

Data collection

Bruker APEXII area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
19923 measured reflections
3811 independent reflections
3303 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 25.0°, θ_{min} = 1.6°
h = −29→29
k = −8→8
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.153
S = 1.20
3811 reflections
325 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 7.491P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C2	0.25381 (12)	0.5904 (4)	0.40442 (12)	0.0388 (7)
H2	0.2385	0.6234	0.4362	0.047*
C4	0.35364 (11)	0.5709 (4)	0.31521 (13)	0.0424 (8)
H4	0.3846	0.6341	0.3294	0.051*
C5	0.35045 (12)	0.4820 (5)	0.26494 (13)	0.0449 (8)
H5	0.3793	0.4879	0.2441	0.054*
C6	0.30440 (12)	0.3825 (4)	0.24458 (13)	0.0425 (7)
H6	0.3038	0.3210	0.2108	0.051*
C7	0.25996 (11)	0.3720 (4)	0.27264 (11)	0.0361 (7)
H7	0.2296	0.3048	0.2588	0.043*
C8	0.26273 (10)	0.4672 (4)	0.32285 (11)	0.0312 (6)
C9	0.30881 (11)	0.5638 (4)	0.34450 (11)	0.0344 (7)
C10	0.17268 (11)	0.4276 (4)	0.36043 (11)	0.0321 (6)
C11	0.13949 (11)	0.4267 (4)	0.31083 (12)	0.0395 (7)
H11	0.1531	0.4597	0.2781	0.047*
C12	0.08636 (11)	0.3770 (4)	0.30986 (12)	0.0405 (7)
H12	0.0639	0.3746	0.2766	0.049*
C13	0.06694 (11)	0.3311 (4)	0.35868 (12)	0.0340 (6)
C14	0.09945 (12)	0.3323 (4)	0.40834 (12)	0.0408 (7)
H14	0.0856	0.3018	0.4411	0.049*
C15	0.15252 (11)	0.3792 (4)	0.40897 (12)	0.0389 (7)
H15	0.1750	0.3783	0.4422	0.047*
N1	0.22712 (9)	0.4857 (3)	0.36256 (9)	0.0328 (5)
N3	0.30201 (10)	0.6395 (4)	0.39623 (10)	0.0411 (6)
N13	0.01039 (10)	0.2810 (4)	0.35820 (11)	0.0437 (6)
O13A	−0.01830 (9)	0.2855 (4)	0.31412 (10)	0.0647 (7)
O13B	−0.00616 (9)	0.2363 (4)	0.40131 (11)	0.0710 (8)
C22	0.49595 (12)	0.3051 (5)	0.32353 (13)	0.0446 (8)
H22	0.4717	0.3061	0.2915	0.054*
C24	0.61964 (12)	0.3622 (5)	0.40363 (16)	0.0517 (9)
H24	0.6463	0.4021	0.3827	0.062*
C25	0.63054 (13)	0.3330 (5)	0.45930 (16)	0.0576 (10)
H25	0.6651	0.3549	0.4763	0.069*
C26	0.59116 (13)	0.2711 (5)	0.49148 (14)	0.0526 (9)
H26	0.6001	0.2507	0.5292	0.063*
C27	0.53921 (12)	0.2400 (5)	0.46773 (13)	0.0458 (8)

H27	0.5127	0.1985	0.4887	0.055*
C28	0.52828 (11)	0.2731 (4)	0.41152 (12)	0.0356 (7)
C29	0.56765 (11)	0.3307 (4)	0.37923 (13)	0.0392 (7)
C30	0.42893 (11)	0.2110 (4)	0.38619 (11)	0.0330 (6)
C31	0.40956 (11)	0.2813 (4)	0.43321 (11)	0.0366 (7)
H31	0.4311	0.3587	0.4573	0.044*
C32	0.35848 (11)	0.2365 (4)	0.44432 (11)	0.0365 (7)
H32	0.3454	0.2802	0.4762	0.044*
C33	0.32713 (11)	0.1251 (4)	0.40702 (11)	0.0335 (6)
C34	0.34509 (11)	0.0603 (4)	0.35931 (12)	0.0375 (7)
H34	0.3227	−0.0101	0.3341	0.045*
C35	0.39664 (11)	0.1007 (4)	0.34931 (12)	0.0383 (7)
H35	0.4098	0.0540	0.3178	0.046*
N21	0.48178 (9)	0.2565 (3)	0.37450 (9)	0.0375 (6)
N23	0.54622 (10)	0.3496 (4)	0.32391 (11)	0.0489 (7)
N33	0.27291 (10)	0.0751 (4)	0.41822 (11)	0.0416 (6)
O33A	0.26246 (9)	0.0800 (5)	0.46603 (10)	0.0746 (9)
O33B	0.24054 (8)	0.0308 (3)	0.37950 (9)	0.0539 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0420 (17)	0.0412 (18)	0.0339 (16)	−0.0029 (14)	0.0070 (13)	−0.0037 (13)
C4	0.0285 (15)	0.0397 (17)	0.059 (2)	−0.0026 (13)	0.0042 (14)	0.0102 (15)
C5	0.0383 (17)	0.0483 (19)	0.0512 (19)	0.0080 (15)	0.0182 (14)	0.0133 (16)
C6	0.0487 (19)	0.0416 (18)	0.0388 (17)	0.0085 (14)	0.0124 (14)	0.0031 (14)
C7	0.0371 (16)	0.0346 (16)	0.0362 (16)	−0.0029 (12)	0.0020 (13)	0.0011 (13)
C8	0.0308 (14)	0.0290 (15)	0.0334 (15)	−0.0015 (11)	0.0019 (11)	0.0064 (12)
C9	0.0340 (15)	0.0297 (15)	0.0383 (16)	0.0002 (12)	−0.0011 (12)	0.0043 (13)
C10	0.0333 (15)	0.0316 (15)	0.0312 (15)	−0.0015 (12)	0.0022 (12)	−0.0039 (12)
C11	0.0368 (16)	0.0513 (19)	0.0312 (15)	−0.0038 (14)	0.0081 (12)	0.0016 (14)
C12	0.0330 (16)	0.0540 (19)	0.0337 (16)	−0.0010 (14)	−0.0002 (12)	−0.0032 (14)
C13	0.0292 (14)	0.0336 (15)	0.0397 (16)	−0.0010 (12)	0.0058 (12)	−0.0021 (13)
C14	0.0415 (17)	0.0501 (19)	0.0322 (16)	−0.0033 (14)	0.0102 (13)	0.0051 (14)
C15	0.0357 (16)	0.0497 (19)	0.0302 (15)	−0.0041 (13)	−0.0004 (12)	0.0043 (13)
N1	0.0310 (12)	0.0344 (13)	0.0327 (12)	−0.0044 (10)	0.0026 (10)	−0.0015 (10)
N3	0.0367 (14)	0.0428 (15)	0.0424 (15)	−0.0048 (11)	−0.0010 (11)	−0.0052 (12)
N13	0.0332 (14)	0.0487 (16)	0.0497 (17)	−0.0005 (12)	0.0064 (13)	−0.0010 (13)
O13A	0.0339 (12)	0.097 (2)	0.0604 (16)	−0.0077 (13)	−0.0052 (11)	0.0016 (14)
O13B	0.0463 (14)	0.109 (2)	0.0609 (16)	−0.0161 (14)	0.0191 (12)	0.0133 (15)
C22	0.0383 (17)	0.060 (2)	0.0366 (17)	−0.0005 (15)	0.0088 (13)	0.0045 (15)
C24	0.0297 (16)	0.0456 (19)	0.082 (3)	−0.0073 (14)	0.0165 (17)	−0.0087 (18)
C25	0.0301 (17)	0.062 (2)	0.079 (3)	−0.0013 (16)	−0.0046 (17)	−0.026 (2)
C26	0.0449 (19)	0.061 (2)	0.049 (2)	0.0065 (17)	−0.0059 (16)	−0.0154 (17)
C27	0.0376 (17)	0.055 (2)	0.0441 (18)	−0.0010 (15)	0.0039 (14)	−0.0048 (15)
C28	0.0286 (14)	0.0364 (16)	0.0418 (17)	−0.0034 (12)	0.0039 (12)	−0.0062 (13)
C29	0.0316 (16)	0.0351 (16)	0.0532 (19)	−0.0025 (12)	0.0149 (14)	−0.0047 (14)
C30	0.0306 (15)	0.0357 (16)	0.0331 (15)	−0.0030 (12)	0.0052 (12)	0.0030 (12)

C31	0.0327 (15)	0.0429 (17)	0.0337 (15)	−0.0050 (13)	0.0017 (12)	−0.0037 (13)
C32	0.0349 (15)	0.0453 (17)	0.0297 (15)	−0.0010 (13)	0.0049 (12)	−0.0011 (13)
C33	0.0280 (14)	0.0345 (16)	0.0373 (16)	−0.0009 (12)	0.0011 (12)	0.0068 (13)
C34	0.0329 (15)	0.0392 (17)	0.0386 (16)	−0.0063 (13)	−0.0029 (12)	−0.0054 (13)
C35	0.0363 (16)	0.0427 (17)	0.0366 (16)	−0.0016 (13)	0.0076 (13)	−0.0064 (13)
N21	0.0299 (13)	0.0473 (15)	0.0362 (13)	−0.0063 (11)	0.0073 (10)	0.0000 (11)
N23	0.0405 (15)	0.0574 (18)	0.0514 (17)	−0.0017 (13)	0.0171 (13)	0.0096 (14)
N33	0.0329 (14)	0.0453 (15)	0.0469 (16)	−0.0030 (12)	0.0056 (12)	0.0040 (12)
O33A	0.0472 (15)	0.128 (3)	0.0517 (15)	−0.0226 (15)	0.0197 (12)	−0.0028 (16)
O33B	0.0329 (12)	0.0669 (16)	0.0599 (15)	−0.0102 (11)	−0.0028 (11)	−0.0040 (12)

Geometric parameters (Å, º)

C2—N3	1.295 (4)	C22—N23	1.299 (4)
C2—N1	1.373 (4)	C22—N21	1.371 (4)
C2—H2	0.9300	C22—H22	0.9300
C4—C5	1.370 (4)	C24—C25	1.364 (5)
C4—C9	1.398 (4)	C24—C29	1.387 (4)
C4—H4	0.9300	C24—H24	0.9300
C5—C6	1.397 (4)	C25—C26	1.400 (5)
C5—H5	0.9300	C25—H25	0.9300
C6—C7	1.373 (4)	C26—C27	1.381 (4)
C6—H6	0.9300	C26—H26	0.9300
C7—C8	1.391 (4)	C27—C28	1.381 (4)
C7—H7	0.9300	C27—H27	0.9300
C8—N1	1.394 (3)	C28—C29	1.391 (4)
C8—C9	1.396 (4)	C28—N21	1.393 (4)
C9—N3	1.396 (4)	C29—N23	1.395 (4)
C10—C15	1.379 (4)	C30—C35	1.383 (4)
C10—C11	1.383 (4)	C30—C31	1.385 (4)
C10—N1	1.422 (3)	C30—N21	1.424 (3)
C11—C12	1.376 (4)	C31—C32	1.377 (4)
C11—H11	0.9300	C31—H31	0.9300
C12—C13	1.372 (4)	C32—C33	1.381 (4)
C12—H12	0.9300	C32—H32	0.9300
C13—C14	1.376 (4)	C33—C34	1.371 (4)
C13—N13	1.461 (4)	C33—N33	1.461 (4)
C14—C15	1.371 (4)	C34—C35	1.373 (4)
C14—H14	0.9300	C34—H34	0.9300
C15—H15	0.9300	C35—H35	0.9300
N13—O13B	1.212 (3)	N33—O33B	1.211 (3)
N13—O13A	1.220 (3)	N33—O33A	1.220 (3)
N3—C2—N1	114.7 (3)	N23—C22—N21	114.0 (3)
N3—C2—H2	122.6	N23—C22—H22	123.0
N1—C2—H2	122.6	N21—C22—H22	123.0
C5—C4—C9	117.9 (3)	C25—C24—C29	118.0 (3)
C5—C4—H4	121.0	C25—C24—H24	121.0

C9—C4—H4	121.0	C29—C24—H24	121.0
C4—C5—C6	120.8 (3)	C24—C25—C26	121.9 (3)
C4—C5—H5	119.6	C24—C25—H25	119.1
C6—C5—H5	119.6	C26—C25—H25	119.1
C7—C6—C5	122.5 (3)	C27—C26—C25	120.6 (3)
C7—C6—H6	118.8	C27—C26—H26	119.7
C5—C6—H6	118.8	C25—C26—H26	119.7
C6—C7—C8	116.5 (3)	C26—C27—C28	117.1 (3)
C6—C7—H7	121.7	C26—C27—H27	121.4
C8—C7—H7	121.7	C28—C27—H27	121.4
C7—C8—N1	132.9 (2)	C27—C28—C29	122.3 (3)
C7—C8—C9	121.9 (3)	C27—C28—N21	132.8 (3)
N1—C8—C9	105.1 (2)	C29—C28—N21	104.9 (2)
C8—C9—N3	110.4 (2)	C24—C29—C28	120.0 (3)
C8—C9—C4	120.4 (3)	C24—C29—N23	129.4 (3)
N3—C9—C4	129.2 (3)	C28—C29—N23	110.6 (3)
C15—C10—C11	120.0 (3)	C35—C30—C31	120.4 (3)
C15—C10—N1	119.1 (2)	C35—C30—N21	119.3 (2)
C11—C10—N1	120.8 (2)	C31—C30—N21	120.3 (2)
C12—C11—C10	120.0 (3)	C32—C31—C30	120.0 (3)
C12—C11—H11	120.0	C32—C31—H31	120.0
C10—C11—H11	120.0	C30—C31—H31	120.0
C13—C12—C11	119.0 (3)	C31—C32—C33	118.5 (3)
C13—C12—H12	120.5	C31—C32—H32	120.7
C11—C12—H12	120.5	C33—C32—H32	120.7
C12—C13—C14	121.5 (3)	C34—C33—C32	122.1 (3)
C12—C13—N13	119.5 (3)	C34—C33—N33	118.5 (3)
C14—C13—N13	119.0 (3)	C32—C33—N33	119.4 (3)
C15—C14—C13	119.2 (3)	C33—C34—C35	119.1 (3)
C15—C14—H14	120.4	C33—C34—H34	120.5
C13—C14—H14	120.4	C35—C34—H34	120.5
C14—C15—C10	120.2 (3)	C34—C35—C30	119.9 (3)
C14—C15—H15	119.9	C34—C35—H35	120.1
C10—C15—H15	119.9	C30—C35—H35	120.1
C2—N1—C8	105.5 (2)	C22—N21—C28	106.1 (2)
C2—N1—C10	124.8 (2)	C22—N21—C30	125.5 (2)
C8—N1—C10	129.5 (2)	C28—N21—C30	128.3 (2)
C2—N3—C9	104.2 (2)	C22—N23—C29	104.4 (2)
O13B—N13—O13A	122.6 (3)	O33B—N33—O33A	123.2 (3)
O13B—N13—C13	119.1 (3)	O33B—N33—C33	118.3 (2)
O13A—N13—C13	118.3 (3)	O33A—N33—C33	118.5 (3)

C9—C4—C5—C6	−2.0 (4)	C29—C24—C25—C26	0.7 (5)
C4—C5—C6—C7	1.6 (5)	C24—C25—C26—C27	−1.0 (5)
C5—C6—C7—C8	0.4 (4)	C25—C26—C27—C28	−0.2 (5)
C6—C7—C8—N1	−178.5 (3)	C26—C27—C28—C29	1.6 (5)
C6—C7—C8—C9	−1.9 (4)	C26—C27—C28—N21	179.7 (3)
C7—C8—C9—N3	−177.3 (3)	C25—C24—C29—C28	0.7 (5)

N1—C8—C9—N3	0.2 (3)	C25—C24—C29—N23	−180.0 (3)
C7—C8—C9—C4	1.4 (4)	C27—C28—C29—C24	−2.0 (5)
N1—C8—C9—C4	178.9 (2)	N21—C28—C29—C24	179.5 (3)
C5—C4—C9—C8	0.5 (4)	C27—C28—C29—N23	178.6 (3)
C5—C4—C9—N3	179.0 (3)	N21—C28—C29—N23	0.1 (3)
C15—C10—C11—C12	−0.2 (5)	C35—C30—C31—C32	2.1 (4)
N1—C10—C11—C12	−177.5 (3)	N21—C30—C31—C32	−179.9 (3)
C10—C11—C12—C13	0.8 (5)	C30—C31—C32—C33	−1.6 (4)
C11—C12—C13—C14	−0.5 (5)	C31—C32—C33—C34	−0.8 (4)
C11—C12—C13—N13	179.0 (3)	C31—C32—C33—N33	179.7 (3)
C12—C13—C14—C15	−0.5 (5)	C32—C33—C34—C35	2.7 (4)
N13—C13—C14—C15	180.0 (3)	N33—C33—C34—C35	−177.7 (3)
C13—C14—C15—C10	1.1 (5)	C33—C34—C35—C30	−2.3 (4)
C11—C10—C15—C14	−0.8 (5)	C31—C30—C35—C34	−0.1 (4)
N1—C10—C15—C14	176.5 (3)	N21—C30—C35—C34	−178.2 (3)
N3—C2—N1—C8	−0.4 (3)	N23—C22—N21—C28	0.4 (4)
N3—C2—N1—C10	−175.2 (3)	N23—C22—N21—C30	176.7 (3)
C7—C8—N1—C2	177.2 (3)	C27—C28—N21—C22	−178.5 (3)
C9—C8—N1—C2	0.1 (3)	C29—C28—N21—C22	−0.3 (3)
C7—C8—N1—C10	−8.4 (5)	C27—C28—N21—C30	5.3 (5)
C9—C8—N1—C10	174.6 (3)	C29—C28—N21—C30	−176.4 (3)
C15—C10—N1—C2	−36.3 (4)	C35—C30—N21—C22	41.4 (4)
C11—C10—N1—C2	141.1 (3)	C31—C30—N21—C22	−136.7 (3)
C15—C10—N1—C8	150.2 (3)	C35—C30—N21—C28	−143.1 (3)
C11—C10—N1—C8	−32.5 (4)	C31—C30—N21—C28	38.8 (4)
N1—C2—N3—C9	0.5 (3)	N21—C22—N23—C29	−0.3 (4)
C8—C9—N3—C2	−0.4 (3)	C24—C29—N23—C22	−179.3 (3)
C4—C9—N3—C2	−179.0 (3)	C28—C29—N23—C22	0.1 (4)
C12—C13—N13—O13B	178.3 (3)	C34—C33—N33—O33B	−22.4 (4)
C14—C13—N13—O13B	−2.2 (4)	C32—C33—N33—O33B	157.1 (3)
C12—C13—N13—O13A	−1.5 (4)	C34—C33—N33—O33A	157.6 (3)
C14—C13—N13—O13A	178.0 (3)	C32—C33—N33—O33A	−22.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O13*A*^{i}	0.93	2.45	3.350 (4)	164
C15—H15···O33*A*^{ii}	0.93	2.59	3.515 (4)	173
C24—H24···O33*B*^{iii}	0.93	2.55	3.375 (4)	149
C35—H35···O13*A*^{iv}	0.93	2.64	3.281 (4)	127

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

1-(4-Nitrophenyl)–2-phenyl-1*H*-1,3-benzodiazole (II)

Crystal data

$C_{19}H_{13}N_3O_2$
M_r = 315.32
Triclinic, $P\overline{1}$

a = 10.2685 (7) Å
b = 15.1411 (10) Å
c = 19.4521 (14) Å

α = 91.886 (1)°
β = 95.725 (1)°
γ = 90.118 (1)°
V = 3007.6 (4) Å^3
Z = 8
F(000) = 1312
D_x = 1.393 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 600 reflections
θ = 20–25°
μ = 0.09 mm^{-1}
T = 100 K
Block, colorless
0.38 × 0.34 × 0.32 mm

Data collection

Bruker APEXII area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
16980 measured reflections
10449 independent reflections

8103 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.0°, θ_{min} = 1.4°
h = −9→12
k = −14→18
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.120
S = 1.05
10449 reflections
865 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.5375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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.

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

	x	y	z	U_{iso}*/U_{eq}
C2	0.1515 (2)	1.07279 (14)	0.36289 (11)	0.0175 (5)
C4	−0.1750 (2)	1.03061 (15)	0.39161 (12)	0.0208 (5)
H4	−0.2322	1.0767	0.3761	0.025*
C5	−0.2219 (2)	0.95717 (15)	0.42144 (12)	0.0233 (6)
H5	−0.3127	0.9528	0.4265	0.028*
C6	−0.1387 (2)	0.88896 (15)	0.44450 (12)	0.0236 (5)
H6	−0.1737	0.8401	0.4661	0.028*
C7	−0.0065 (2)	0.89132 (15)	0.43651 (11)	0.0205 (5)
H7	0.0501	0.8444	0.4509	0.025*
C8	0.0395 (2)	0.96505 (14)	0.40655 (11)	0.0174 (5)
C9	−0.0420 (2)	1.03526 (14)	0.38496 (11)	0.0179 (5)
C10	0.2716 (2)	0.92931 (14)	0.38947 (11)	0.0165 (5)
C11	0.3151 (2)	0.88616 (14)	0.44931 (11)	0.0189 (5)
H11	0.2733	0.8957	0.4903	0.023*
C12	0.4198 (2)	0.82938 (14)	0.44862 (11)	0.0177 (5)
H12	0.4507	0.7990	0.4889	0.021*

C13	0.4790 (2)	0.81746 (14)	0.38844 (11)	0.0167 (5)
C14	0.4346 (2)	0.85825 (14)	0.32788 (11)	0.0185 (5)
H14	0.4755	0.8477	0.2867	0.022*
C15	0.3291 (2)	0.91468 (14)	0.32888 (12)	0.0184 (5)
H15	0.2963	0.9433	0.2880	0.022*
C16	0.2621 (2)	1.12559 (14)	0.34408 (11)	0.0177 (5)
C17	0.3883 (2)	1.11962 (14)	0.37722 (12)	0.0201 (5)
H17	0.4062	1.0785	0.4127	0.024*
C18	0.4880 (2)	1.17298 (15)	0.35899 (12)	0.0225 (5)
H18	0.5737	1.1683	0.3820	0.027*
C19	0.4630 (2)	1.23322 (15)	0.30731 (12)	0.0239 (5)
H19	0.5318	1.2692	0.2944	0.029*
C20	0.3382 (2)	1.24090 (15)	0.27461 (12)	0.0242 (6)
H20	0.3209	1.2829	0.2396	0.029*
C21	0.2384 (2)	1.18783 (15)	0.29252 (12)	0.0217 (5)
H21	0.1528	1.1935	0.2697	0.026*
C22	0.2973 (2)	0.57288 (14)	0.36167 (11)	0.0169 (5)
C24	0.6346 (2)	0.53140 (15)	0.39070 (12)	0.0215 (5)
H24	0.6858	0.5777	0.3752	0.026*
C25	0.6931 (2)	0.45797 (15)	0.42078 (12)	0.0242 (6)
H25	0.7858	0.4537	0.4258	0.029*
C26	0.6179 (2)	0.38991 (15)	0.44392 (12)	0.0234 (6)
H26	0.6610	0.3411	0.4655	0.028*
C27	0.4833 (2)	0.39180 (15)	0.43632 (12)	0.0207 (5)
H27	0.4322	0.3451	0.4514	0.025*
C28	0.4260 (2)	0.46550 (14)	0.40549 (11)	0.0169 (5)
C29	0.4991 (2)	0.53540 (14)	0.38393 (11)	0.0177 (5)
C30	0.1871 (2)	0.43012 (14)	0.38899 (11)	0.0171 (5)
C31	0.1658 (2)	0.38662 (14)	0.44859 (11)	0.0192 (5)
H31	0.2235	0.3961	0.4895	0.023*
C32	0.0607 (2)	0.32964 (14)	0.44849 (11)	0.0183 (5)
H32	0.0450	0.2995	0.4889	0.022*
C33	−0.0211 (2)	0.31758 (14)	0.38797 (11)	0.0167 (5)
C34	0.0004 (2)	0.35821 (14)	0.32740 (11)	0.0181 (5)
H34	−0.0568	0.3479	0.2864	0.022*
C35	0.1068 (2)	0.41403 (14)	0.32790 (11)	0.0179 (5)
H35	0.1253	0.4414	0.2867	0.021*
C36	0.1793 (2)	0.62558 (14)	0.34271 (11)	0.0169 (5)
C37	0.0668 (2)	0.61938 (15)	0.37676 (12)	0.0210 (5)
H37	0.0630	0.5786	0.4126	0.025*
C38	−0.0397 (2)	0.67267 (15)	0.35837 (12)	0.0223 (5)
H38	−0.1166	0.6678	0.3814	0.027*
C39	−0.0351 (2)	0.73325 (15)	0.30653 (12)	0.0241 (6)
H39	−0.1087	0.7692	0.2938	0.029*
C40	0.0771 (2)	0.74088 (16)	0.27373 (12)	0.0263 (6)
H40	0.0813	0.7828	0.2387	0.032*
C41	0.1836 (2)	0.68775 (15)	0.29154 (12)	0.0220 (5)
H41	0.2606	0.6936	0.2687	0.026*

C42	0.6046 (2)	0.32149 (14)	0.13331 (11)	0.0180 (5)
C44	0.2747 (2)	0.26202 (16)	0.08789 (12)	0.0248 (6)
H44	0.2161	0.3071	0.0998	0.030*
C45	0.2294 (2)	0.18343 (17)	0.05630 (12)	0.0280 (6)
H45	0.1378	0.1747	0.0459	0.034*
C46	0.3148 (2)	0.11655 (16)	0.03923 (12)	0.0269 (6)
H46	0.2798	0.0633	0.0175	0.032*
C47	0.4486 (2)	0.12550 (15)	0.05296 (12)	0.0232 (5)
H47	0.5065	0.0796	0.0417	0.028*
C48	0.4943 (2)	0.20493 (15)	0.08407 (11)	0.0190 (5)
C49	0.4102 (2)	0.27280 (15)	0.10166 (11)	0.0207 (5)
C50	0.7330 (2)	0.18147 (14)	0.11465 (11)	0.0181 (5)
C51	0.7744 (2)	0.13377 (15)	0.05877 (12)	0.0209 (5)
H51	0.7295	0.1388	0.0140	0.025*
C52	0.8816 (2)	0.07892 (15)	0.06895 (12)	0.0219 (5)
H52	0.9118	0.0461	0.0313	0.026*
C53	0.9439 (2)	0.07272 (14)	0.13498 (12)	0.0202 (5)
C54	0.9039 (2)	0.11920 (15)	0.19113 (12)	0.0210 (5)
H54	0.9484	0.1135	0.2360	0.025*
C55	0.7967 (2)	0.17468 (14)	0.18037 (11)	0.0197 (5)
H55	0.7673	0.2079	0.2181	0.024*
C56	0.7150 (2)	0.38079 (14)	0.15714 (11)	0.0191 (5)
C57	0.8333 (2)	0.37789 (15)	0.12712 (12)	0.0212 (5)
H57	0.8467	0.3337	0.0927	0.025*
C58	0.9308 (2)	0.43909 (15)	0.14735 (12)	0.0234 (5)
H58	1.0111	0.4368	0.1270	0.028*
C59	0.9117 (2)	0.50382 (15)	0.19727 (12)	0.0236 (6)
H59	0.9783	0.5465	0.2105	0.028*
C60	0.7959 (2)	0.50634 (15)	0.22780 (12)	0.0246 (6)
H60	0.7834	0.5504	0.2624	0.030*
C61	0.6984 (2)	0.44524 (15)	0.20822 (12)	0.0225 (5)
H61	0.6193	0.4471	0.2297	0.027*
C62	0.2428 (2)	0.17767 (14)	0.86536 (11)	0.0181 (5)
C64	−0.0708 (2)	0.22821 (15)	0.91233 (12)	0.0223 (5)
H64	−0.1309	0.1807	0.9019	0.027*
C65	−0.1090 (2)	0.30663 (15)	0.94241 (12)	0.0242 (6)
H65	−0.1970	0.3132	0.9528	0.029*
C66	−0.0207 (2)	0.37656 (15)	0.95792 (12)	0.0228 (5)
H66	−0.0501	0.4294	0.9789	0.027*
C67	0.1076 (2)	0.37108 (14)	0.94363 (11)	0.0203 (5)
H67	0.1672	0.4188	0.9540	0.024*
C68	0.1456 (2)	0.29211 (15)	0.91321 (11)	0.0178 (5)
C69	0.0588 (2)	0.22124 (14)	0.89785 (11)	0.0195 (5)
C70	0.3739 (2)	0.31956 (14)	0.88461 (11)	0.0179 (5)
C71	0.4341 (2)	0.36686 (15)	0.94198 (12)	0.0203 (5)
H71	0.4024	0.3619	0.9859	0.024*
C72	0.5399 (2)	0.42075 (15)	0.93455 (12)	0.0213 (5)
H72	0.5830	0.4522	0.9734	0.026*

C73	0.5819 (2)	0.42816 (14)	0.86987 (12)	0.0200 (5)
C74	0.5231 (2)	0.38242 (14)	0.81240 (12)	0.0196 (5)
H74	0.5539	0.3887	0.7684	0.024*
C75	0.4184 (2)	0.32725 (14)	0.82019 (11)	0.0187 (5)
H75	0.3772	0.2947	0.7814	0.022*
C76	0.3455 (2)	0.11983 (14)	0.84147 (12)	0.0200 (5)
C77	0.4752 (2)	0.12432 (15)	0.87140 (12)	0.0223 (5)
H77	0.5012	0.1694	0.9051	0.027*
C78	0.5657 (2)	0.06312 (15)	0.85184 (12)	0.0245 (6)
H78	0.6538	0.0667	0.8721	0.029*
C79	0.5289 (2)	−0.00323 (15)	0.80300 (12)	0.0252 (6)
H79	0.5908	−0.0459	0.7905	0.030*
C80	0.4010 (2)	−0.00682 (16)	0.77255 (12)	0.0262 (6)
H80	0.3757	−0.0515	0.7384	0.031*
C81	0.3104 (2)	0.05402 (15)	0.79144 (12)	0.0231 (5)
H81	0.2231	0.0510	0.7701	0.028*
N1	0.16391 (17)	0.98928 (12)	0.39070 (9)	0.0179 (4)
N3	0.03091 (17)	1.10186 (12)	0.35902 (9)	0.0186 (4)
N13	0.59586 (18)	0.76198 (11)	0.38945 (9)	0.0173 (4)
N21	0.29532 (17)	0.48948 (12)	0.38976 (9)	0.0177 (4)
N23	0.41651 (18)	0.60195 (12)	0.35743 (9)	0.0183 (4)
N33	−0.13782 (18)	0.26153 (12)	0.38922 (9)	0.0178 (4)
N41	0.61997 (18)	0.23668 (12)	0.10492 (9)	0.0190 (4)
N43	0.48177 (18)	0.34492 (12)	0.13137 (10)	0.0210 (4)
N53	1.05829 (19)	0.01485 (12)	0.14548 (11)	0.0236 (5)
N61	0.26408 (18)	0.26361 (12)	0.89163 (9)	0.0182 (4)
N63	0.12216 (18)	0.15069 (12)	0.86829 (10)	0.0206 (4)
N73	0.69384 (19)	0.48662 (13)	0.86188 (11)	0.0240 (5)
O13A	0.63947 (15)	0.73120 (10)	0.44486 (8)	0.0216 (4)
O13B	0.64664 (15)	0.74983 (10)	0.33559 (8)	0.0239 (4)
O33A	−0.20837 (15)	0.24875 (10)	0.33517 (8)	0.0244 (4)
O33B	−0.16081 (15)	0.23189 (10)	0.44480 (8)	0.0216 (4)
O53A	1.10452 (15)	−0.01608 (10)	0.09414 (9)	0.0276 (4)
O53B	1.10234 (17)	0.00072 (11)	0.20495 (9)	0.0356 (5)
O73A	0.75194 (16)	0.52034 (10)	0.91447 (9)	0.0291 (4)
O73B	0.72322 (18)	0.49830 (12)	0.80348 (9)	0.0377 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0177 (13)	0.0155 (12)	0.0193 (12)	0.0006 (10)	0.0013 (9)	0.0019 (9)
C4	0.0156 (13)	0.0195 (13)	0.0266 (13)	0.0026 (10)	0.0005 (10)	−0.0058 (10)
C5	0.0147 (13)	0.0215 (13)	0.0339 (14)	−0.0041 (10)	0.0052 (10)	−0.0064 (11)
C6	0.0247 (14)	0.0178 (13)	0.0287 (13)	−0.0053 (11)	0.0054 (11)	−0.0001 (10)
C7	0.0205 (13)	0.0164 (12)	0.0245 (13)	−0.0007 (10)	0.0026 (10)	0.0002 (10)
C8	0.0138 (12)	0.0194 (12)	0.0191 (12)	0.0003 (10)	0.0025 (9)	−0.0013 (10)
C9	0.0177 (13)	0.0160 (12)	0.0195 (12)	−0.0001 (10)	0.0009 (9)	−0.0039 (9)
C10	0.0135 (12)	0.0127 (11)	0.0232 (12)	−0.0021 (9)	0.0020 (9)	−0.0004 (9)

C11	0.0176 (13)	0.0191 (12)	0.0206 (12)	0.0005 (10)	0.0059 (10)	−0.0008 (10)
C12	0.0183 (13)	0.0163 (12)	0.0186 (12)	−0.0001 (10)	0.0012 (9)	0.0028 (9)
C13	0.0127 (12)	0.0123 (11)	0.0250 (12)	−0.0017 (9)	0.0014 (9)	0.0004 (9)
C14	0.0184 (13)	0.0177 (12)	0.0198 (12)	−0.0020 (10)	0.0045 (10)	0.0002 (10)
C15	0.0179 (13)	0.0152 (12)	0.0220 (12)	0.0000 (10)	0.0001 (10)	0.0035 (9)
C16	0.0191 (13)	0.0141 (12)	0.0203 (12)	0.0014 (10)	0.0044 (10)	−0.0026 (9)
C17	0.0217 (13)	0.0154 (12)	0.0229 (12)	0.0033 (10)	0.0004 (10)	0.0008 (10)
C18	0.0171 (13)	0.0222 (13)	0.0278 (13)	0.0007 (10)	0.0020 (10)	−0.0040 (11)
C19	0.0227 (14)	0.0215 (13)	0.0286 (13)	−0.0048 (11)	0.0097 (11)	−0.0023 (11)
C20	0.0291 (15)	0.0212 (13)	0.0230 (13)	−0.0013 (11)	0.0044 (11)	0.0050 (10)
C21	0.0174 (13)	0.0224 (13)	0.0249 (13)	0.0012 (10)	0.0000 (10)	0.0017 (10)
C22	0.0187 (13)	0.0165 (12)	0.0155 (11)	−0.0007 (10)	0.0020 (9)	0.0003 (9)
C24	0.0181 (13)	0.0187 (13)	0.0277 (13)	−0.0019 (10)	0.0043 (10)	−0.0033 (10)
C25	0.0151 (13)	0.0236 (14)	0.0331 (14)	0.0024 (11)	0.0009 (10)	−0.0065 (11)
C26	0.0220 (14)	0.0203 (13)	0.0274 (13)	0.0076 (11)	0.0004 (10)	−0.0005 (10)
C27	0.0208 (13)	0.0159 (12)	0.0255 (13)	−0.0001 (10)	0.0034 (10)	0.0006 (10)
C28	0.0132 (12)	0.0162 (12)	0.0210 (12)	−0.0009 (9)	0.0018 (9)	−0.0034 (9)
C29	0.0173 (13)	0.0169 (12)	0.0187 (12)	0.0010 (10)	0.0016 (9)	−0.0035 (9)
C30	0.0149 (12)	0.0131 (12)	0.0236 (12)	0.0015 (9)	0.0030 (9)	0.0013 (9)
C31	0.0174 (13)	0.0178 (12)	0.0215 (12)	−0.0007 (10)	−0.0020 (10)	0.0002 (10)
C32	0.0189 (13)	0.0168 (12)	0.0199 (12)	0.0018 (10)	0.0032 (9)	0.0049 (9)
C33	0.0163 (12)	0.0109 (11)	0.0233 (12)	0.0016 (9)	0.0046 (9)	0.0009 (9)
C34	0.0175 (13)	0.0165 (12)	0.0199 (12)	0.0019 (10)	−0.0002 (10)	−0.0007 (10)
C35	0.0179 (13)	0.0159 (12)	0.0205 (12)	0.0010 (10)	0.0031 (9)	0.0049 (9)
C36	0.0168 (12)	0.0137 (12)	0.0194 (12)	−0.0024 (10)	−0.0011 (9)	−0.0022 (9)
C37	0.0237 (14)	0.0172 (12)	0.0222 (12)	−0.0044 (10)	0.0034 (10)	0.0018 (10)
C38	0.0163 (13)	0.0206 (13)	0.0296 (13)	−0.0018 (10)	0.0027 (10)	−0.0051 (11)
C39	0.0196 (14)	0.0225 (13)	0.0290 (13)	0.0046 (10)	−0.0037 (10)	−0.0001 (11)
C40	0.0286 (15)	0.0241 (14)	0.0259 (13)	0.0041 (11)	0.0004 (11)	0.0043 (11)
C41	0.0189 (13)	0.0244 (13)	0.0229 (13)	−0.0018 (11)	0.0028 (10)	0.0016 (10)
C42	0.0169 (13)	0.0169 (12)	0.0202 (12)	0.0001 (10)	0.0017 (9)	0.0020 (10)
C44	0.0194 (13)	0.0282 (14)	0.0276 (13)	−0.0003 (11)	0.0022 (10)	0.0131 (11)
C45	0.0204 (14)	0.0343 (15)	0.0288 (14)	−0.0101 (12)	−0.0045 (11)	0.0130 (12)
C46	0.0266 (15)	0.0266 (14)	0.0265 (13)	−0.0091 (12)	−0.0034 (11)	0.0064 (11)
C47	0.0257 (14)	0.0207 (13)	0.0229 (13)	−0.0014 (11)	0.0007 (10)	0.0036 (10)
C48	0.0178 (13)	0.0194 (12)	0.0197 (12)	−0.0019 (10)	−0.0007 (9)	0.0055 (10)
C49	0.0214 (13)	0.0203 (13)	0.0207 (12)	−0.0014 (10)	0.0006 (10)	0.0074 (10)
C50	0.0147 (12)	0.0153 (12)	0.0245 (12)	−0.0035 (10)	0.0018 (10)	0.0023 (10)
C51	0.0189 (13)	0.0225 (13)	0.0205 (12)	−0.0029 (10)	−0.0012 (10)	0.0001 (10)
C52	0.0206 (13)	0.0190 (13)	0.0263 (13)	−0.0031 (10)	0.0049 (10)	−0.0060 (10)
C53	0.0171 (13)	0.0148 (12)	0.0289 (13)	−0.0005 (10)	0.0032 (10)	0.0029 (10)
C54	0.0188 (13)	0.0202 (13)	0.0236 (13)	−0.0039 (10)	−0.0008 (10)	0.0026 (10)
C55	0.0210 (13)	0.0167 (12)	0.0221 (12)	−0.0041 (10)	0.0059 (10)	−0.0014 (10)
C56	0.0188 (13)	0.0162 (12)	0.0220 (12)	0.0015 (10)	−0.0019 (10)	0.0059 (10)
C57	0.0225 (13)	0.0161 (12)	0.0252 (13)	0.0006 (10)	0.0030 (10)	0.0030 (10)
C58	0.0192 (13)	0.0252 (13)	0.0260 (13)	−0.0009 (11)	0.0005 (10)	0.0084 (11)
C59	0.0223 (14)	0.0194 (13)	0.0274 (13)	−0.0043 (10)	−0.0083 (10)	0.0078 (10)
C60	0.0274 (15)	0.0175 (13)	0.0277 (13)	0.0034 (11)	−0.0029 (11)	−0.0018 (10)

C61	0.0195 (13)	0.0196 (13)	0.0282 (13)	0.0050 (10)	0.0004 (10)	0.0009 (10)
C62	0.0189 (13)	0.0166 (12)	0.0190 (12)	−0.0003 (10)	0.0018 (9)	0.0019 (9)
C64	0.0189 (13)	0.0208 (13)	0.0274 (13)	−0.0008 (10)	0.0027 (10)	0.0024 (10)
C65	0.0177 (13)	0.0277 (14)	0.0283 (13)	0.0037 (11)	0.0070 (10)	0.0053 (11)
C66	0.0248 (14)	0.0178 (13)	0.0267 (13)	0.0062 (11)	0.0060 (10)	0.0023 (10)
C67	0.0243 (14)	0.0152 (12)	0.0213 (12)	−0.0005 (10)	0.0016 (10)	0.0018 (10)
C68	0.0148 (12)	0.0206 (13)	0.0185 (12)	0.0023 (10)	0.0027 (9)	0.0040 (10)
C69	0.0211 (13)	0.0173 (12)	0.0201 (12)	0.0029 (10)	0.0021 (10)	0.0021 (10)
C70	0.0152 (12)	0.0130 (12)	0.0260 (13)	0.0026 (9)	0.0046 (10)	0.0030 (10)
C71	0.0204 (13)	0.0207 (13)	0.0206 (12)	0.0023 (10)	0.0050 (10)	0.0028 (10)
C72	0.0219 (13)	0.0174 (12)	0.0242 (13)	−0.0016 (10)	0.0017 (10)	−0.0033 (10)
C73	0.0173 (13)	0.0144 (12)	0.0290 (13)	0.0020 (10)	0.0063 (10)	0.0024 (10)
C74	0.0215 (13)	0.0174 (12)	0.0212 (12)	0.0038 (10)	0.0069 (10)	0.0023 (10)
C75	0.0201 (13)	0.0150 (12)	0.0206 (12)	0.0007 (10)	0.0006 (10)	−0.0002 (9)
C76	0.0215 (13)	0.0153 (12)	0.0247 (13)	0.0003 (10)	0.0082 (10)	0.0045 (10)
C77	0.0243 (14)	0.0191 (13)	0.0238 (13)	−0.0011 (11)	0.0034 (10)	0.0051 (10)
C78	0.0202 (13)	0.0251 (14)	0.0293 (14)	0.0034 (11)	0.0057 (10)	0.0093 (11)
C79	0.0267 (15)	0.0187 (13)	0.0329 (14)	0.0073 (11)	0.0142 (11)	0.0075 (11)
C80	0.0322 (15)	0.0199 (13)	0.0282 (14)	−0.0005 (11)	0.0119 (11)	−0.0017 (11)
C81	0.0210 (13)	0.0237 (13)	0.0255 (13)	−0.0012 (11)	0.0063 (10)	0.0022 (10)
N1	0.0166 (11)	0.0161 (10)	0.0213 (10)	0.0021 (8)	0.0031 (8)	0.0032 (8)
N3	0.0145 (11)	0.0182 (10)	0.0235 (10)	0.0011 (8)	0.0031 (8)	0.0024 (8)
N13	0.0172 (11)	0.0133 (10)	0.0212 (11)	−0.0023 (8)	0.0017 (8)	0.0000 (8)
N21	0.0155 (10)	0.0143 (10)	0.0230 (10)	−0.0011 (8)	0.0005 (8)	0.0022 (8)
N23	0.0164 (11)	0.0164 (10)	0.0224 (10)	−0.0015 (8)	0.0030 (8)	0.0015 (8)
N33	0.0166 (11)	0.0156 (10)	0.0217 (11)	0.0029 (8)	0.0037 (8)	0.0029 (8)
N41	0.0161 (11)	0.0178 (10)	0.0231 (10)	−0.0002 (8)	0.0010 (8)	0.0017 (8)
N43	0.0172 (11)	0.0197 (11)	0.0256 (11)	−0.0001 (9)	−0.0003 (8)	0.0029 (8)
N53	0.0202 (11)	0.0176 (11)	0.0324 (12)	−0.0028 (9)	−0.0003 (9)	−0.0012 (9)
N61	0.0187 (11)	0.0138 (10)	0.0227 (10)	−0.0024 (8)	0.0043 (8)	0.0010 (8)
N63	0.0191 (11)	0.0181 (11)	0.0251 (11)	0.0000 (9)	0.0044 (8)	0.0000 (8)
N73	0.0181 (11)	0.0190 (11)	0.0360 (13)	0.0016 (9)	0.0083 (9)	−0.0008 (9)
O13A	0.0225 (9)	0.0182 (9)	0.0235 (9)	0.0019 (7)	−0.0023 (7)	0.0058 (7)
O13B	0.0225 (9)	0.0266 (9)	0.0234 (9)	0.0057 (7)	0.0064 (7)	0.0009 (7)
O33A	0.0195 (9)	0.0286 (10)	0.0241 (9)	−0.0044 (7)	−0.0027 (7)	0.0007 (7)
O33B	0.0212 (9)	0.0210 (9)	0.0238 (9)	−0.0006 (7)	0.0052 (7)	0.0075 (7)
O53A	0.0221 (10)	0.0240 (9)	0.0379 (11)	0.0028 (8)	0.0092 (8)	−0.0012 (8)
O53B	0.0344 (11)	0.0357 (11)	0.0334 (11)	0.0115 (9)	−0.0116 (8)	−0.0034 (8)
O73A	0.0214 (10)	0.0243 (10)	0.0404 (11)	−0.0026 (8)	−0.0025 (8)	−0.0007 (8)
O73B	0.0390 (12)	0.0377 (11)	0.0399 (11)	−0.0127 (9)	0.0215 (9)	−0.0012 (9)

Geometric parameters (Å, º)

C2—N3	1.311 (3)	C44—C49	1.398 (3)
C2—N1	1.393 (3)	C44—H44	0.9500
C2—C16	1.470 (3)	C45—C46	1.395 (3)
C4—C5	1.378 (3)	C45—H45	0.9500
C4—C9	1.387 (3)	C46—C47	1.378 (3)

C4—H4	0.9500	C46—H46	0.9500
C5—C6	1.398 (3)	C47—C48	1.388 (3)
C5—H5	0.9500	C47—H47	0.9500
C6—C7	1.382 (3)	C48—N41	1.393 (3)
C6—H6	0.9500	C48—C49	1.399 (3)
C7—C8	1.380 (3)	C49—N43	1.392 (3)
C7—H7	0.9500	C50—C55	1.384 (3)
C8—N1	1.395 (3)	C50—C51	1.388 (3)
C8—C9	1.403 (3)	C50—N41	1.433 (3)
C9—N3	1.392 (3)	C51—C52	1.382 (3)
C10—C15	1.382 (3)	C51—H51	0.9500
C10—C11	1.389 (3)	C52—C53	1.383 (3)
C10—N1	1.434 (3)	C52—H52	0.9500
C11—C12	1.379 (3)	C53—C54	1.377 (3)
C11—H11	0.9500	C53—N53	1.468 (3)
C12—C13	1.378 (3)	C54—C55	1.389 (3)
C12—H12	0.9500	C54—H54	0.9500
C13—C14	1.385 (3)	C55—H55	0.9500
C13—N13	1.466 (3)	C56—C61	1.393 (3)
C14—C15	1.382 (3)	C56—C57	1.400 (3)
C14—H14	0.9500	C57—C58	1.381 (3)
C15—H15	0.9500	C57—H57	0.9500
C16—C17	1.394 (3)	C58—C59	1.385 (3)
C16—C21	1.403 (3)	C58—H58	0.9500
C17—C18	1.383 (3)	C59—C60	1.381 (3)
C17—H17	0.9500	C59—H59	0.9500
C18—C19	1.385 (3)	C60—C61	1.379 (3)
C18—H18	0.9500	C60—H60	0.9500
C19—C20	1.380 (3)	C61—H61	0.9500
C19—H19	0.9500	C62—N63	1.311 (3)
C20—C21	1.380 (3)	C62—N61	1.391 (3)
C20—H20	0.9500	C62—C76	1.473 (3)
C21—H21	0.9500	C64—C65	1.381 (3)
C22—N23	1.311 (3)	C64—C69	1.391 (3)
C22—N21	1.393 (3)	C64—H64	0.9500
C22—C36	1.475 (3)	C65—C66	1.398 (3)
C24—C25	1.384 (3)	C65—H65	0.9500
C24—C29	1.386 (3)	C66—C67	1.375 (3)
C24—H24	0.9500	C66—H66	0.9500
C25—C26	1.399 (3)	C67—C68	1.390 (3)
C25—H25	0.9500	C67—H67	0.9500
C26—C27	1.376 (3)	C68—N61	1.391 (3)
C26—H26	0.9500	C68—C69	1.398 (3)
C27—C28	1.388 (3)	C69—N63	1.390 (3)
C27—H27	0.9500	C70—C75	1.385 (3)
C28—C29	1.396 (3)	C70—C71	1.396 (3)
C28—N21	1.397 (3)	C70—N61	1.429 (3)
C29—N23	1.396 (3)	C71—C72	1.379 (3)

C30—C31	1.388 (3)	C71—H71	0.9500
C30—C35	1.391 (3)	C72—C73	1.378 (3)
C30—N21	1.426 (3)	C72—H72	0.9500
C31—C32	1.380 (3)	C73—C74	1.381 (3)
C31—H31	0.9500	C73—N73	1.473 (3)
C32—C33	1.383 (3)	C74—C75	1.383 (3)
C32—H32	0.9500	C74—H74	0.9500
C33—C34	1.384 (3)	C75—H75	0.9500
C33—N33	1.470 (3)	C76—C81	1.391 (3)
C34—C35	1.379 (3)	C76—C77	1.400 (3)
C34—H34	0.9500	C77—C78	1.385 (3)
C35—H35	0.9500	C77—H77	0.9500
C36—C37	1.393 (3)	C78—C79	1.385 (3)
C36—C41	1.396 (3)	C78—H78	0.9500
C37—C38	1.385 (3)	C79—C80	1.386 (3)
C37—H37	0.9500	C79—H79	0.9500
C38—C39	1.389 (3)	C80—C81	1.378 (3)
C38—H38	0.9500	C80—H80	0.9500
C39—C40	1.378 (3)	C81—H81	0.9500
C39—H39	0.9500	N13—O13B	1.225 (2)
C40—C41	1.381 (3)	N13—O13A	1.232 (2)
C40—H40	0.9500	N33—O33A	1.225 (2)
C41—H41	0.9500	N33—O33B	1.228 (2)
C42—N43	1.309 (3)	N53—O53B	1.225 (2)
C42—N41	1.396 (3)	N53—O53A	1.228 (2)
C42—C56	1.473 (3)	N73—O73B	1.222 (3)
C44—C45	1.379 (3)	N73—O73A	1.227 (2)

N3—C2—N1	112.59 (19)	C48—C47—H47	121.7
N3—C2—C16	123.2 (2)	C47—C48—N41	132.4 (2)
N1—C2—C16	124.1 (2)	C47—C48—C49	122.4 (2)
C5—C4—C9	118.0 (2)	N41—C48—C49	105.21 (19)
C5—C4—H4	121.0	N43—C49—C44	129.6 (2)
C9—C4—H4	121.0	N43—C49—C48	110.4 (2)
C4—C5—C6	121.5 (2)	C44—C49—C48	120.0 (2)
C4—C5—H5	119.3	C55—C50—C51	121.1 (2)
C6—C5—H5	119.3	C55—C50—N41	119.1 (2)
C7—C6—C5	121.2 (2)	C51—C50—N41	119.75 (19)
C7—C6—H6	119.4	C52—C51—C50	119.4 (2)
C5—C6—H6	119.4	C52—C51—H51	120.3
C8—C7—C6	116.8 (2)	C50—C51—H51	120.3
C8—C7—H7	121.6	C51—C52—C53	118.8 (2)
C6—C7—H7	121.6	C51—C52—H52	120.6
C7—C8—N1	132.3 (2)	C53—C52—H52	120.6
C7—C8—C9	122.7 (2)	C54—C53—C52	122.7 (2)
N1—C8—C9	105.03 (19)	C54—C53—N53	118.7 (2)
C4—C9—N3	129.9 (2)	C52—C53—N53	118.6 (2)
C4—C9—C8	119.7 (2)	C53—C54—C55	118.2 (2)

N3—C9—C8	110.35 (19)	C53—C54—H54	120.9
C15—C10—C11	121.2 (2)	C55—C54—H54	120.9
C15—C10—N1	119.65 (19)	C50—C55—C54	119.9 (2)
C11—C10—N1	119.13 (19)	C50—C55—H55	120.1
C12—C11—C10	119.3 (2)	C54—C55—H55	120.1
C12—C11—H11	120.3	C61—C56—C57	118.9 (2)
C10—C11—H11	120.3	C61—C56—C42	118.9 (2)
C13—C12—C11	118.9 (2)	C57—C56—C42	122.0 (2)
C13—C12—H12	120.6	C58—C57—C56	120.2 (2)
C11—C12—H12	120.6	C58—C57—H57	119.9
C12—C13—C14	122.5 (2)	C56—C57—H57	119.9
C12—C13—N13	118.56 (19)	C57—C58—C59	120.2 (2)
C14—C13—N13	118.94 (19)	C57—C58—H58	119.9
C15—C14—C13	118.3 (2)	C59—C58—H58	119.9
C15—C14—H14	120.9	C60—C59—C58	120.0 (2)
C13—C14—H14	120.9	C60—C59—H59	120.0
C10—C15—C14	119.8 (2)	C58—C59—H59	120.0
C10—C15—H15	120.1	C61—C60—C59	120.3 (2)
C14—C15—H15	120.1	C61—C60—H60	119.9
C17—C16—C21	118.3 (2)	C59—C60—H60	119.9
C17—C16—C2	123.1 (2)	C60—C61—C56	120.5 (2)
C21—C16—C2	118.6 (2)	C60—C61—H61	119.8
C18—C17—C16	120.7 (2)	C56—C61—H61	119.8
C18—C17—H17	119.6	N63—C62—N61	112.70 (19)
C16—C17—H17	119.6	N63—C62—C76	122.8 (2)
C17—C18—C19	120.1 (2)	N61—C62—C76	124.4 (2)
C17—C18—H18	120.0	C65—C64—C69	117.6 (2)
C19—C18—H18	120.0	C65—C64—H64	121.2
C20—C19—C18	120.0 (2)	C69—C64—H64	121.2
C20—C19—H19	120.0	C64—C65—C66	121.3 (2)
C18—C19—H19	120.0	C64—C65—H65	119.4
C19—C20—C21	120.2 (2)	C66—C65—H65	119.4
C19—C20—H20	119.9	C67—C66—C65	121.9 (2)
C21—C20—H20	119.9	C67—C66—H66	119.0
C20—C21—C16	120.7 (2)	C65—C66—H66	119.0
C20—C21—H21	119.7	C66—C67—C68	116.6 (2)
C16—C21—H21	119.7	C66—C67—H67	121.7
N23—C22—N21	112.57 (19)	C68—C67—H67	121.7
N23—C22—C36	123.2 (2)	C67—C68—N61	132.4 (2)
N21—C22—C36	124.09 (19)	C67—C68—C69	122.3 (2)
C25—C24—C29	118.0 (2)	N61—C68—C69	105.30 (19)
C25—C24—H24	121.0	N63—C69—C64	129.3 (2)
C29—C24—H24	121.0	N63—C69—C68	110.4 (2)
C24—C25—C26	121.1 (2)	C64—C69—C68	120.3 (2)
C24—C25—H25	119.5	C75—C70—C71	120.5 (2)
C26—C25—H25	119.5	C75—C70—N61	119.3 (2)
C27—C26—C25	121.8 (2)	C71—C70—N61	120.2 (2)
C27—C26—H26	119.1	C72—C71—C70	119.7 (2)

C25—C26—H26	119.1	C72—C71—H71	120.1
C26—C27—C28	116.5 (2)	C70—C71—H71	120.1
C26—C27—H27	121.8	C73—C72—C71	118.9 (2)
C28—C27—H27	121.8	C73—C72—H72	120.6
C27—C28—C29	122.7 (2)	C71—C72—H72	120.6
C27—C28—N21	132.1 (2)	C72—C73—C74	122.3 (2)
C29—C28—N21	105.15 (18)	C72—C73—N73	118.8 (2)
C24—C29—C28	119.9 (2)	C74—C73—N73	118.9 (2)
C24—C29—N23	129.6 (2)	C73—C74—C75	118.7 (2)
C28—C29—N23	110.46 (19)	C73—C74—H74	120.7
C31—C30—C35	120.7 (2)	C75—C74—H74	120.7
C31—C30—N21	119.61 (19)	C74—C75—C70	119.9 (2)
C35—C30—N21	119.68 (19)	C74—C75—H75	120.1
C32—C31—C30	120.0 (2)	C70—C75—H75	120.1
C32—C31—H31	120.0	C81—C76—C77	118.8 (2)
C30—C31—H31	120.0	C81—C76—C62	118.9 (2)
C31—C32—C33	118.3 (2)	C77—C76—C62	122.1 (2)
C31—C32—H32	120.9	C78—C77—C76	120.1 (2)
C33—C32—H32	120.9	C78—C77—H77	120.0
C32—C33—C34	122.7 (2)	C76—C77—H77	120.0
C32—C33—N33	118.18 (19)	C77—C78—C79	120.5 (2)
C34—C33—N33	119.07 (19)	C77—C78—H78	119.7
C35—C34—C33	118.4 (2)	C79—C78—H78	119.7
C35—C34—H34	120.8	C78—C79—C80	119.4 (2)
C33—C34—H34	120.8	C78—C79—H79	120.3
C34—C35—C30	119.8 (2)	C80—C79—H79	120.3
C34—C35—H35	120.1	C81—C80—C79	120.5 (2)
C30—C35—H35	120.1	C81—C80—H80	119.8
C37—C36—C41	118.8 (2)	C79—C80—H80	119.8
C37—C36—C22	122.4 (2)	C80—C81—C76	120.7 (2)
C41—C36—C22	118.6 (2)	C80—C81—H81	119.7
C38—C37—C36	120.0 (2)	C76—C81—H81	119.7
C38—C37—H37	120.0	C2—N1—C8	106.42 (18)
C36—C37—H37	120.0	C2—N1—C10	128.21 (18)
C37—C38—C39	120.6 (2)	C8—N1—C10	123.93 (18)
C37—C38—H38	119.7	C2—N3—C9	105.57 (18)
C39—C38—H38	119.7	O13B—N13—O13A	123.29 (19)
C40—C39—C38	119.6 (2)	O13B—N13—C13	118.52 (18)
C40—C39—H39	120.2	O13A—N13—C13	118.18 (18)
C38—C39—H39	120.2	C22—N21—C28	106.35 (17)
C39—C40—C41	120.2 (2)	C22—N21—C30	127.98 (18)
C39—C40—H40	119.9	C28—N21—C30	124.28 (18)
C41—C40—H40	119.9	C22—N23—C29	105.44 (18)
C40—C41—C36	120.8 (2)	O33A—N33—O33B	123.69 (19)
C40—C41—H41	119.6	O33A—N33—C33	118.24 (18)
C36—C41—H41	119.6	O33B—N33—C33	118.05 (18)
N43—C42—N41	112.55 (19)	C48—N41—C42	106.24 (18)
N43—C42—C56	123.7 (2)	C48—N41—C50	123.81 (18)

N41—C42—C56	123.5 (2)	C42—N41—C50	127.17 (18)
C45—C44—C49	117.5 (2)	C42—N43—C49	105.58 (19)
C45—C44—H44	121.2	O53B—N53—O53A	123.9 (2)
C49—C44—H44	121.2	O53B—N53—C53	118.07 (19)
C44—C45—C46	121.5 (2)	O53A—N53—C53	118.04 (19)
C44—C45—H45	119.2	C62—N61—C68	106.21 (18)
C46—C45—H45	119.2	C62—N61—C70	127.61 (19)
C47—C46—C45	121.8 (2)	C68—N61—C70	124.86 (18)
C47—C46—H46	119.1	C62—N63—C69	105.42 (19)
C45—C46—H46	119.1	O73B—N73—O73A	124.2 (2)
C46—C47—C48	116.6 (2)	O73B—N73—C73	118.1 (2)
C46—C47—H47	121.7	O73A—N73—C73	117.7 (2)

C9—C4—C5—C6	0.0 (3)	C67—C68—C69—N63	−179.60 (19)
C4—C5—C6—C7	−1.8 (4)	N61—C68—C69—N63	0.6 (2)
C5—C6—C7—C8	1.7 (3)	C67—C68—C69—C64	0.6 (3)
C6—C7—C8—N1	−179.9 (2)	N61—C68—C69—C64	−179.3 (2)
C6—C7—C8—C9	0.2 (3)	C75—C70—C71—C72	−0.8 (3)
C5—C4—C9—N3	−178.0 (2)	N61—C70—C71—C72	−179.9 (2)
C5—C4—C9—C8	1.9 (3)	C70—C71—C72—C73	1.5 (3)
C7—C8—C9—C4	−2.0 (3)	C71—C72—C73—C74	−1.1 (3)
N1—C8—C9—C4	178.05 (19)	C71—C72—C73—N73	179.3 (2)
C7—C8—C9—N3	177.9 (2)	C72—C73—C74—C75	0.0 (3)
N1—C8—C9—N3	−2.1 (2)	N73—C73—C74—C75	179.66 (19)
C15—C10—C11—C12	1.6 (3)	C73—C74—C75—C70	0.7 (3)
N1—C10—C11—C12	−179.10 (19)	C71—C70—C75—C74	−0.3 (3)
C10—C11—C12—C13	0.4 (3)	N61—C70—C75—C74	178.81 (19)
C11—C12—C13—C14	−2.1 (3)	N63—C62—C76—C81	31.8 (3)
C11—C12—C13—N13	175.69 (18)	N61—C62—C76—C81	−153.0 (2)
C12—C13—C14—C15	1.7 (3)	N63—C62—C76—C77	−143.0 (2)
N13—C13—C14—C15	−176.07 (18)	N61—C62—C76—C77	32.3 (3)
C11—C10—C15—C14	−2.0 (3)	C81—C76—C77—C78	−0.9 (3)
N1—C10—C15—C14	178.72 (19)	C62—C76—C77—C78	173.8 (2)
C13—C14—C15—C10	0.3 (3)	C76—C77—C78—C79	−0.4 (3)
N3—C2—C16—C17	146.6 (2)	C77—C78—C79—C80	1.4 (4)
N1—C2—C16—C17	−29.2 (3)	C78—C79—C80—C81	−1.2 (4)
N3—C2—C16—C21	−30.4 (3)	C79—C80—C81—C76	−0.2 (4)
N1—C2—C16—C21	153.8 (2)	C77—C76—C81—C80	1.2 (3)
C21—C16—C17—C18	−0.8 (3)	C62—C76—C81—C80	−173.7 (2)
C2—C16—C17—C18	−177.8 (2)	N3—C2—N1—C8	−1.0 (2)
C16—C17—C18—C19	0.0 (3)	C16—C2—N1—C8	175.2 (2)
C17—C18—C19—C20	0.9 (3)	N3—C2—N1—C10	165.51 (19)
C18—C19—C20—C21	−0.9 (4)	C16—C2—N1—C10	−18.3 (3)
C19—C20—C21—C16	0.1 (4)	C7—C8—N1—C2	−178.1 (2)
C17—C16—C21—C20	0.8 (3)	C9—C8—N1—C2	1.8 (2)
C2—C16—C21—C20	177.9 (2)	C7—C8—N1—C10	14.6 (4)
C29—C24—C25—C26	−0.2 (3)	C9—C8—N1—C10	−165.42 (19)
C24—C25—C26—C27	1.6 (4)	C15—C10—N1—C2	−44.9 (3)

C25—C26—C27—C28	−1.0 (3)	C11—C10—N1—C2	135.9 (2)
C26—C27—C28—C29	−1.0 (3)	C15—C10—N1—C8	119.5 (2)
C26—C27—C28—N21	−180.0 (2)	C11—C10—N1—C8	−59.8 (3)
C25—C24—C29—C28	−1.7 (3)	N1—C2—N3—C9	−0.3 (2)
C25—C24—C29—N23	178.3 (2)	C16—C2—N3—C9	−176.5 (2)
C27—C28—C29—C24	2.4 (3)	C4—C9—N3—C2	−178.7 (2)
N21—C28—C29—C24	−178.39 (19)	C8—C9—N3—C2	1.5 (2)
C27—C28—C29—N23	−177.6 (2)	C12—C13—N13—O13B	178.40 (19)
N21—C28—C29—N23	1.6 (2)	C14—C13—N13—O13B	−3.7 (3)
C35—C30—C31—C32	−2.8 (3)	C12—C13—N13—O13A	−2.9 (3)
N21—C30—C31—C32	179.5 (2)	C14—C13—N13—O13A	175.00 (19)
C30—C31—C32—C33	0.0 (3)	N23—C22—N21—C28	1.1 (2)
C31—C32—C33—C34	1.9 (3)	C36—C22—N21—C28	−175.25 (19)
C31—C32—C33—N33	−175.98 (19)	N23—C22—N21—C30	−165.7 (2)
C32—C33—C34—C35	−1.0 (3)	C36—C22—N21—C30	17.9 (3)
N33—C33—C34—C35	176.89 (19)	C27—C28—N21—C22	177.5 (2)
C33—C34—C35—C30	−1.9 (3)	C29—C28—N21—C22	−1.6 (2)
C31—C30—C35—C34	3.8 (3)	C27—C28—N21—C30	−15.1 (4)
N21—C30—C35—C34	−178.5 (2)	C29—C28—N21—C30	165.81 (19)
N23—C22—C36—C37	−146.3 (2)	C31—C30—N21—C22	−136.1 (2)
N21—C22—C36—C37	29.7 (3)	C35—C30—N21—C22	46.1 (3)
N23—C22—C36—C41	29.7 (3)	C31—C30—N21—C28	59.2 (3)
N21—C22—C36—C41	−154.3 (2)	C35—C30—N21—C28	−118.5 (2)
C41—C36—C37—C38	1.7 (3)	N21—C22—N23—C29	−0.1 (2)
C22—C36—C37—C38	177.8 (2)	C36—C22—N23—C29	176.31 (19)
C36—C37—C38—C39	−0.7 (3)	C24—C29—N23—C22	179.1 (2)
C37—C38—C39—C40	−0.7 (3)	C28—C29—N23—C22	−1.0 (2)
C38—C39—C40—C41	1.0 (3)	C32—C33—N33—O33A	−177.96 (19)
C39—C40—C41—C36	0.1 (3)	C34—C33—N33—O33A	4.1 (3)
C37—C36—C41—C40	−1.5 (3)	C32—C33—N33—O33B	3.8 (3)
C22—C36—C41—C40	−177.7 (2)	C34—C33—N33—O33B	−174.19 (19)
C49—C44—C45—C46	0.6 (3)	C47—C48—N41—C42	−179.9 (2)
C44—C45—C46—C47	0.0 (4)	C49—C48—N41—C42	−0.3 (2)
C45—C46—C47—C48	−0.6 (3)	C47—C48—N41—C50	−17.6 (4)
C46—C47—C48—N41	−179.9 (2)	C49—C48—N41—C50	161.93 (19)
C46—C47—C48—C49	0.7 (3)	N43—C42—N41—C48	−0.5 (2)
C45—C44—C49—N43	178.6 (2)	C56—C42—N41—C48	−175.4 (2)
C45—C44—C49—C48	−0.6 (3)	N43—C42—N41—C50	−162.0 (2)
C47—C48—C49—N43	−179.4 (2)	C56—C42—N41—C50	23.1 (3)
N41—C48—C49—N43	1.0 (2)	C55—C50—N41—C48	−114.5 (2)
C47—C48—C49—C44	−0.1 (3)	C51—C50—N41—C48	63.5 (3)
N41—C48—C49—C44	−179.7 (2)	C55—C50—N41—C42	43.9 (3)
C55—C50—C51—C52	−0.3 (3)	C51—C50—N41—C42	−138.0 (2)
N41—C50—C51—C52	−178.4 (2)	N41—C42—N43—C49	1.1 (2)
C50—C51—C52—C53	0.5 (3)	C56—C42—N43—C49	176.0 (2)
C51—C52—C53—C54	−0.2 (3)	C44—C49—N43—C42	179.5 (2)
C51—C52—C53—N53	−179.6 (2)	C48—C49—N43—C42	−1.3 (2)
C52—C53—C54—C55	−0.3 (3)	C54—C53—N53—O53B	10.0 (3)

N53—C53—C54—C55	179.12 (19)	C52—C53—N53—O53B	−170.6 (2)
C51—C50—C55—C54	−0.1 (3)	C54—C53—N53—O53A	−169.6 (2)
N41—C50—C55—C54	177.90 (19)	C52—C53—N53—O53A	9.8 (3)
C53—C54—C55—C50	0.4 (3)	N63—C62—N61—C68	0.4 (2)
N43—C42—C56—C61	31.6 (3)	C76—C62—N61—C68	−175.2 (2)
N41—C42—C56—C61	−154.0 (2)	N63—C62—N61—C70	−166.8 (2)
N43—C42—C56—C57	−144.4 (2)	C76—C62—N61—C70	17.5 (3)
N41—C42—C56—C57	30.0 (3)	C67—C68—N61—C62	179.6 (2)
C61—C56—C57—C58	−1.0 (3)	C69—C68—N61—C62	−0.6 (2)
C42—C56—C57—C58	175.0 (2)	C67—C68—N61—C70	−12.7 (4)
C56—C57—C58—C59	−0.2 (3)	C69—C68—N61—C70	167.1 (2)
C57—C58—C59—C60	1.1 (3)	C75—C70—N61—C62	46.9 (3)
C58—C59—C60—C61	−0.8 (3)	C71—C70—N61—C62	−134.0 (2)
C59—C60—C61—C56	−0.5 (3)	C75—C70—N61—C68	−118.1 (2)
C57—C56—C61—C60	1.4 (3)	C71—C70—N61—C68	61.0 (3)
C42—C56—C61—C60	−174.8 (2)	N61—C62—N63—C69	−0.1 (2)
C69—C64—C65—C66	−0.1 (3)	C76—C62—N63—C69	175.7 (2)
C64—C65—C66—C67	0.5 (4)	C64—C69—N63—C62	179.5 (2)
C65—C66—C67—C68	−0.3 (3)	C68—C69—N63—C62	−0.3 (2)
C66—C67—C68—N61	179.6 (2)	C72—C73—N73—O73B	−174.0 (2)
C66—C67—C68—C69	−0.2 (3)	C74—C73—N73—O73B	6.3 (3)
C65—C64—C69—N63	179.8 (2)	C72—C73—N73—O73A	6.1 (3)
C65—C64—C69—C68	−0.4 (3)	C74—C73—N73—O73A	−173.55 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O13*A*^{i}	0.95	2.52	3.298 (3)	139
C7—H7···O33*B*^{ii}	0.95	2.54	3.357 (3)	145
C15—H15···O53*B*^{iii}	0.95	2.61	3.468 (3)	151
C26—H26···O33*B*^{iv}	0.95	2.52	3.302 (3)	140
C27—H27···O13*A*^{v}	0.95	2.53	3.355 (3)	145
C35—H35···O73*B*^{v}	0.95	2.64	3.527 (3)	156
C44—H44···O73*A*^{v}	0.95	2.66	3.309 (3)	126
C52—H52···O53*A*^{vi}	0.95	2.46	3.299 (3)	148
C54—H54···N3^{vii}	0.95	2.47	3.412 (3)	171
C55—H55···O33*A*^{iv}	0.95	2.33	3.184 (3)	149
C64—H64···O53*A*^{viii}	0.95	2.51	3.227 (3)	132
C74—H74···N23^{v}	0.95	2.50	3.437 (3)	167
C75—H75···O13*B*^{v}	0.95	2.34	3.217 (3)	153

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

2-(3-Methylphenyl)-1-(4-nitrophenyl)-1*H*-1,3-benzodiazole (III)

Crystal data

$C_{20}H_{15}N_3O_2$
M_r = 329.35

Triclinic, $P\overline{1}$
a = 8.186 (4) Å

b = 9.806 (4) Å
c = 11.264 (5) Å
α = 112.825 (7)°
β = 98.468 (7)°
γ = 94.276 (7)°
V = 815.6 (6) Å^3
Z = 2
F(000) = 344

D_x = 1.341 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 600 reflections
θ = 20–25°
μ = 0.09 mm^{-1}
T = 273 K
Block, yellow
0.40 × 0.30 × 0.25 mm

Data collection

Bruker APEXII area detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
9543 measured reflections
3787 independent reflections

3177 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 28.3°, θ_{min} = 2.0°
h = −10→10
k = −12→13
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.132
S = 1.04
3787 reflections
228 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0639P)^2 + 0.1338P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.015 (4)

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.

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

	x	y	z	U_{iso}*/U_{eq}
C2	1.11569 (16)	0.81089 (14)	0.92065 (14)	0.0462 (3)
C4	1.36041 (19)	0.97893 (17)	1.23845 (17)	0.0607 (4)
H4	1.4673	1.0193	1.2394	0.073*
C5	1.3122 (2)	0.98585 (18)	1.35173 (17)	0.0664 (4)
H5	1.3879	1.0322	1.4308	0.080*
C6	1.1528 (2)	0.92531 (18)	1.35185 (16)	0.0649 (4)
H6	1.1248	0.9326	1.4309	0.078*
C7	1.0360 (2)	0.85496 (16)	1.23786 (15)	0.0573 (4)
H7	0.9297	0.8137	1.2376	0.069*
C8	1.08493 (16)	0.84889 (14)	1.12389 (14)	0.0479 (3)
C9	1.24396 (16)	0.90932 (14)	1.12153 (15)	0.0489 (3)
C10	0.84671 (15)	0.69016 (14)	0.94718 (13)	0.0440 (3)
C11	0.70368 (17)	0.75156 (15)	0.97650 (14)	0.0498 (3)

H11	0.7098	0.8534	1.0266	0.060*
C12	0.55165 (16)	0.66014 (16)	0.93071 (14)	0.0505 (3)
H12	0.4539	0.6990	0.9500	0.061*
C13	0.54769 (15)	0.51089 (16)	0.85613 (13)	0.0470 (3)
C14	0.68956 (17)	0.44715 (15)	0.82913 (14)	0.0490 (3)
H14	0.6832	0.3450	0.7804	0.059*
C15	0.84127 (16)	0.53875 (15)	0.87623 (14)	0.0478 (3)
H15	0.9393	0.4988	0.8603	0.057*
C16	1.08101 (17)	0.76249 (15)	0.77786 (14)	0.0488 (3)
C17	0.92362 (19)	0.74599 (17)	0.70399 (16)	0.0567 (4)
H17	0.8316	0.7633	0.7446	0.068*
C18	0.9041 (2)	0.70399 (18)	0.57068 (17)	0.0655 (4)
H18	0.7987	0.6941	0.5216	0.079*
C19	1.0384 (2)	0.67648 (19)	0.50893 (17)	0.0668 (4)
H19	1.0225	0.6460	0.4184	0.080*
C20	1.1972 (2)	0.69374 (18)	0.58030 (16)	0.0619 (4)
C21	1.21581 (19)	0.73705 (16)	0.71352 (15)	0.0547 (4)
H21	1.3220	0.7499	0.7625	0.066*
C22	1.3455 (3)	0.6623 (3)	0.5141 (2)	0.0863 (6)
H22A	1.4421	0.6702	0.5774	0.129*
H22B	1.3654	0.7336	0.4768	0.129*
H22C	1.3234	0.5632	0.4458	0.129*
N1	1.00386 (13)	0.78549 (12)	0.99329 (11)	0.0470 (3)
N3	1.25900 (13)	0.88564 (13)	0.99495 (12)	0.0507 (3)
N13	0.38467 (15)	0.41564 (17)	0.80090 (14)	0.0605 (3)
O13A	0.26128 (13)	0.46980 (16)	0.83535 (15)	0.0814 (4)
O13B	0.37989 (17)	0.28725 (17)	0.72329 (15)	0.0950 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0385 (6)	0.0393 (6)	0.0630 (8)	0.0068 (5)	0.0110 (6)	0.0223 (6)
C4	0.0458 (8)	0.0504 (8)	0.0757 (11)	0.0017 (6)	−0.0019 (7)	0.0204 (7)
C5	0.0671 (10)	0.0557 (9)	0.0642 (10)	0.0054 (7)	−0.0074 (8)	0.0188 (7)
C6	0.0769 (11)	0.0547 (9)	0.0605 (9)	0.0077 (8)	0.0090 (8)	0.0224 (7)
C7	0.0572 (8)	0.0489 (8)	0.0636 (9)	0.0024 (6)	0.0131 (7)	0.0210 (7)
C8	0.0422 (7)	0.0382 (6)	0.0592 (8)	0.0042 (5)	0.0058 (6)	0.0171 (6)
C9	0.0413 (7)	0.0384 (6)	0.0638 (9)	0.0063 (5)	0.0070 (6)	0.0182 (6)
C10	0.0347 (6)	0.0438 (6)	0.0540 (7)	0.0026 (5)	0.0069 (5)	0.0215 (6)
C11	0.0440 (7)	0.0440 (7)	0.0631 (8)	0.0095 (5)	0.0119 (6)	0.0223 (6)
C12	0.0355 (6)	0.0592 (8)	0.0650 (9)	0.0130 (6)	0.0125 (6)	0.0317 (7)
C13	0.0356 (6)	0.0556 (8)	0.0533 (7)	−0.0006 (5)	0.0033 (5)	0.0291 (6)
C14	0.0459 (7)	0.0429 (7)	0.0553 (8)	0.0021 (5)	0.0073 (6)	0.0186 (6)
C15	0.0377 (6)	0.0458 (7)	0.0606 (8)	0.0087 (5)	0.0107 (6)	0.0213 (6)
C16	0.0469 (7)	0.0416 (7)	0.0618 (8)	0.0045 (5)	0.0094 (6)	0.0256 (6)
C17	0.0500 (8)	0.0539 (8)	0.0708 (10)	0.0091 (6)	0.0080 (7)	0.0312 (7)
C18	0.0638 (10)	0.0626 (9)	0.0708 (10)	0.0045 (7)	−0.0031 (8)	0.0340 (8)
C19	0.0816 (12)	0.0593 (9)	0.0597 (9)	−0.0021 (8)	0.0066 (8)	0.0290 (8)

C20	0.0672 (10)	0.0558 (8)	0.0664 (10)	−0.0001 (7)	0.0177 (8)	0.0285 (7)
C21	0.0492 (8)	0.0546 (8)	0.0640 (9)	0.0023 (6)	0.0104 (6)	0.0289 (7)
C22	0.0834 (13)	0.0968 (14)	0.0758 (12)	0.0001 (11)	0.0316 (10)	0.0283 (11)
N1	0.0379 (6)	0.0437 (6)	0.0572 (7)	0.0014 (4)	0.0080 (5)	0.0192 (5)
N3	0.0387 (6)	0.0440 (6)	0.0681 (8)	0.0037 (4)	0.0093 (5)	0.0218 (5)
N13	0.0411 (7)	0.0732 (9)	0.0696 (8)	−0.0062 (6)	0.0000 (6)	0.0379 (7)
O13A	0.0366 (6)	0.0975 (9)	0.1198 (11)	0.0018 (6)	0.0092 (6)	0.0576 (8)
O13B	0.0664 (8)	0.0791 (9)	0.1033 (10)	−0.0214 (7)	0.0018 (7)	0.0093 (8)

Geometric parameters (Å, º)

C2—N3	1.3117 (17)	C13—C14	1.377 (2)
C2—N1	1.3835 (18)	C13—N13	1.4693 (18)
C2—C16	1.465 (2)	C14—C15	1.3786 (19)
C4—C5	1.369 (3)	C14—H14	0.9300
C4—C9	1.395 (2)	C15—H15	0.9300
C4—H4	0.9300	C16—C17	1.388 (2)
C5—C6	1.393 (2)	C16—C21	1.393 (2)
C5—H5	0.9300	C17—C18	1.375 (2)
C6—C7	1.376 (2)	C17—H17	0.9300
C6—H6	0.9300	C18—C19	1.374 (3)
C7—C8	1.381 (2)	C18—H18	0.9300
C7—H7	0.9300	C19—C20	1.385 (2)
C8—N1	1.3900 (19)	C19—H19	0.9300
C8—C9	1.3978 (19)	C20—C21	1.373 (2)
C9—N3	1.379 (2)	C20—C22	1.507 (2)
C10—C15	1.3798 (19)	C21—H21	0.9300
C10—C11	1.3809 (19)	C22—H22A	0.9600
C10—N1	1.4302 (16)	C22—H22B	0.9600
C11—C12	1.3799 (19)	C22—H22C	0.9600
C11—H11	0.9300	N13—O13B	1.2163 (19)
C12—C13	1.371 (2)	N13—O13A	1.2200 (18)
C12—H12	0.9300		

N3—C2—N1	112.01 (13)	C15—C14—H14	120.9
N3—C2—C16	122.97 (12)	C14—C15—C10	119.57 (12)
N1—C2—C16	125.02 (12)	C14—C15—H15	120.2
C5—C4—C9	117.82 (15)	C10—C15—H15	120.2
C5—C4—H4	121.1	C17—C16—C21	118.31 (15)
C9—C4—H4	121.1	C17—C16—C2	124.02 (13)
C4—C5—C6	121.79 (15)	C21—C16—C2	117.63 (13)
C4—C5—H5	119.1	C18—C17—C16	119.81 (15)
C6—C5—H5	119.1	C18—C17—H17	120.1
C7—C6—C5	121.63 (16)	C16—C17—H17	120.1
C7—C6—H6	119.2	C19—C18—C17	120.86 (16)
C5—C6—H6	119.2	C19—C18—H18	119.6
C6—C7—C8	116.36 (15)	C17—C18—H18	119.6
C6—C7—H7	121.8	C18—C19—C20	120.60 (16)

C8—C7—H7	121.8	C18—C19—H19	119.7
C7—C8—N1	132.32 (13)	C20—C19—H19	119.7
C7—C8—C9	122.97 (13)	C21—C20—C19	118.17 (16)
N1—C8—C9	104.68 (12)	C21—C20—C22	120.49 (16)
N3—C9—C4	130.01 (14)	C19—C20—C22	121.32 (17)
N3—C9—C8	110.56 (12)	C20—C21—C16	122.23 (15)
C4—C9—C8	119.42 (15)	C20—C21—H21	118.9
C15—C10—C11	121.37 (12)	C16—C21—H21	118.9
C15—C10—N1	119.47 (11)	C20—C22—H22A	109.5
C11—C10—N1	119.15 (12)	C20—C22—H22B	109.5
C12—C11—C10	119.26 (13)	H22A—C22—H22B	109.5
C12—C11—H11	120.4	C20—C22—H22C	109.5
C10—C11—H11	120.4	H22A—C22—H22C	109.5
C13—C12—C11	118.62 (12)	H22B—C22—H22C	109.5
C13—C12—H12	120.7	C2—N1—C8	106.90 (11)
C11—C12—H12	120.7	C2—N1—C10	128.40 (12)
C12—C13—C14	122.83 (12)	C8—N1—C10	123.76 (11)
C12—C13—N13	118.44 (12)	C2—N3—C9	105.84 (11)
C14—C13—N13	118.72 (13)	O13B—N13—O13A	123.54 (14)
C13—C14—C15	118.27 (13)	O13B—N13—C13	118.33 (14)
C13—C14—H14	120.9	O13A—N13—C13	118.14 (14)

C9—C4—C5—C6	0.3 (2)	C17—C18—C19—C20	−1.4 (2)
C4—C5—C6—C7	0.2 (3)	C18—C19—C20—C21	0.8 (2)
C5—C6—C7—C8	−0.5 (2)	C18—C19—C20—C22	179.27 (16)
C6—C7—C8—N1	178.35 (14)	C19—C20—C21—C16	0.6 (2)
C6—C7—C8—C9	0.3 (2)	C22—C20—C21—C16	−177.90 (15)
C5—C4—C9—N3	−179.33 (14)	C17—C16—C21—C20	−1.3 (2)
C5—C4—C9—C8	−0.5 (2)	C2—C16—C21—C20	−178.96 (13)
C7—C8—C9—N3	179.25 (13)	N3—C2—N1—C8	−0.48 (15)
N1—C8—C9—N3	0.74 (14)	C16—C2—N1—C8	−179.98 (12)
C7—C8—C9—C4	0.2 (2)	N3—C2—N1—C10	−169.61 (12)
N1—C8—C9—C4	−178.30 (12)	C16—C2—N1—C10	10.9 (2)
C15—C10—C11—C12	−1.8 (2)	C7—C8—N1—C2	−178.48 (15)
N1—C10—C11—C12	179.18 (12)	C9—C8—N1—C2	−0.18 (14)
C10—C11—C12—C13	−0.7 (2)	C7—C8—N1—C10	−8.7 (2)
C11—C12—C13—C14	2.6 (2)	C9—C8—N1—C10	169.58 (11)
C11—C12—C13—N13	−176.34 (12)	C15—C10—N1—C2	60.70 (19)
C12—C13—C14—C15	−1.9 (2)	C11—C10—N1—C2	−120.30 (15)
N13—C13—C14—C15	176.99 (12)	C15—C10—N1—C8	−106.76 (15)
C13—C14—C15—C10	−0.6 (2)	C11—C10—N1—C8	72.24 (17)
C11—C10—C15—C14	2.5 (2)	N1—C2—N3—C9	0.92 (14)
N1—C10—C15—C14	−178.52 (12)	C16—C2—N3—C9	−179.57 (12)
N3—C2—C16—C17	−149.93 (14)	C4—C9—N3—C2	177.87 (14)
N1—C2—C16—C17	29.5 (2)	C8—C9—N3—C2	−1.04 (14)
N3—C2—C16—C21	27.58 (19)	C12—C13—N13—O13B	172.07 (15)
N1—C2—C16—C21	−152.98 (13)	C14—C13—N13—O13B	−6.9 (2)
C21—C16—C17—C18	0.7 (2)	C12—C13—N13—O13A	−7.9 (2)

C2—C16—C17—C18	178.16 (13)	C14—C13—N13—O13A	173.19 (13)
C16—C17—C18—C19	0.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···N3^{i}	0.93	2.66	3.431 (2)	141
C12—H12···N3^{ii}	0.93	2.47	3.348 (2)	157

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