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3,4-Bis(4-nitrophenyl)-1,2,5-oxadiazole 2-oxide

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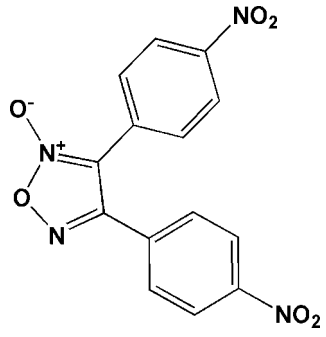
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.060; wR factor = 0.133; data-to-parameter ratio = 16.2.

The title compound, $C_{14}H_8N_4O_6$, a new 1,2,5-oxadiazole *N*-oxide derivative, was formed by dimerization of 4-nitrobenzaldehyde oxime. The compound crystallizes with two independent molecules per asymmetric unit. The *N*-oxide O atom is disordered over two sites in each molecule; site occupancy factors are 0.57/0.43 and 0.5/0.5. The mean planes through the two benzene rings are inclined to the planar 1,2,3-oxadiazole ring by 25.03 (11) and 41.64 (11)° in one molecule, and 22.58 (11) and 42.66 (11)° in the other molecule, the smaller angle being for the ring on the oxide side of the oxadiazole ring in each case. In the crystal structure, the individual molecules form centrosymmetric dimers linked *via* C—H···O hydrogen bonds. The dimers of one molecule are then linked to those of the other molecule *via* C—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For related literature, see: Sillitoe & Harding (1978); Easton *et al.* (1995); Baker *et al.* (2002); Allen (2002); Allen *et al.* (1987); Howe & Shelton (1990); Kerbal *et al.* (1990); Smietana *et al.* (1999).



Experimental

Crystal data

$C_{14}H_8N_4O_6$
M_r = 328.24
Monoclinic, $P2_1/c$
a = 17.3977 (11) Å
b = 7.2813 (4) Å
c = 21.5341 (13) Å
β = 91.667 (5)°
V = 2726.7 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 173 (2) K
0.40 × 0.35 × 0.30 mm

Data collection

Stoe IPDS-2 diffractometer
Absorption correction: none
29047 measured reflections
7339 independent reflections
5558 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.133
S = 1.15
7339 reflections
452 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O5^{i}	0.95	2.59	3.283 (3)	130
C14—H14···O23^{ii}	0.95	2.46	3.244 (3)	139
C25—H25···O25^{iii}	0.95	2.57	3.358 (3)	141
C27—H27···O1A^{iv}	0.95	2.36	3.254 (4)	156
C31—H31···O5^{v}	0.95	2.52	3.261 (3)	135
C34—H34···O4^{ii}	0.95	2.50	3.284 (3)	140

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

Data collection: *X-AREA* (Stoe & Cie, 2006); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2154).

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supplementary materials

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3,4-Bis(4-nitrophenyl)-1,2,5-oxadiazole 2-oxide

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Comment

In the course of our research aimed at the synthesis of new efficient antitubercular agents containing simple pharmacophore sites of the type *X*—C—C—Y we turned our attention to the spiro-isoxazolines which posses a rigid (O=C—C—O) pharmacophore. These compounds display interesting biological properties, such as herbicidal, plant-growth regulatory and antitumor activities (Howe & Shelton, 1990; Smietana *et al.*, 1999). The preparation of the spiro-isoxazolines, in which we are interested, normally involves the reaction of a nitriloxyde [(*E*)-4-nitrobenzaldehyde oxime, (I)] with an isothiochromanone in a solution of hydrogen peroxyde (Kerbal *et al.*, 1990). We have noted many times the formation of a by-product during this reaction. Finally this compound has been isolated and examined crystallographically. It was found to be a new 1,2,5-oxadiazole N-oxide derivative, (II).

The molecular structure of compound (II) is shown in Fig. 1. The compound crystallizes with two independent molecules (1 & 2) per asymmetric unit. The 1,2,5-oxadiazole units are disordered with two alternative positions for the N-oxide O-atom [atom O1a/O1b in molecule 1, and atom O21*a*/O21*b* in molecule 2]. There are some short intramolecular C···O contacts in the two molecules involving the disordered atoms; O1b and neighbouring C-atoms C2 and C3, and atom O21*b* with atom C22. A search of the Cambridge Crystallographic Data Base (Version 1.8, last update May 2007: Allen, 2002) indicates that such short interactions are not unusual. The 1,2,5-oxadiazole ring is planar [to within 0.008 and 0.009 Å, in molecules 1 and 2, respectively] and the bond distances and angles are similar to those in the diphenyl analoque 3,4-Diphenylfurazan N-oxide, (III) [Sillitoe & Harding, 1978)]. They do not indicate the presence of delocalized electron density as in the dichlorophenyl analoque 4,5-bis(2,6-Dichlorophenyl)-1-oxide-2-oxa-1,3-diazole, (IV) [Easton *et al.*, 1995] or a D-mannose-derived furoxan [Baker *et al.*, 2002]. The C?N bonds being significantly shorter than the C—C or O—N bonds. The remainder of the bond distances in (II) are within normal limits (Allen *et al.*, 1987). The best planes through the phenyl rings are inclined to the best plane through the 1,2,5-oxadiazole ring by 25.03 (11) and 41.64 (11)° in molecule 1, and 22.58 (11) and 42.66 (11)° in molecule 2. This is quite different to the situation in (III), where the same dihedral angles are 16.7 and 59.6°, or in (IV), where the same dihedral angles are 63.1 (3) and 65.6 (5)°.

In the crystal structure of (II) the individual molecules are linked to their symmetry related molecule *via* C—H···O hydrogen bonds to form centrosynmetric dimers. These dimers are in turn linked by other C—H···O hydrogen bonds to form a three-dimensional network. Details of the hydrogen bonding are given in Table 1 and Fig. 2.

The formation of compound (II) is similar to that described by Baker *et al.* (2002), who have studied in detail the synthesis and X-ray structure of 3,4-dipyranosyl-1,2,5-oxadiazole 2-oxide. Similarly we found that the reaction of 4-nitrobenzaldehyde oxime with pure NaOCl in $CHCl_3$, but never CH_2Cl_2, gives an almost quantitative yield of (II) (95%), on simply stirring at room temperature for 16 h.

Experimental

The reaction of 4-nitrobenzaldehyde oxime with pure NaOCl, in a 2:1 molar ratio, in $CHCl_3$ (but never CH_2Cl_2) gives an almost quantitative yield of (II) (95%), on stirring at room temperature for 16 h. Yellow block-like crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution of (II).

Refinement

The N-oxide O-atom is disordered over two sites in each molecule (1 & 2); the occupancies were finally fixed at O1a/O1b = 0.57/0.43 and O31*a*/O31*b* = 0.5/0.5. The hydrogen atoms could all be located from difference Fourier maps. They were included in calculated positons and treated as riding atoms with C—H distances = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(parent C-atom).

Figures

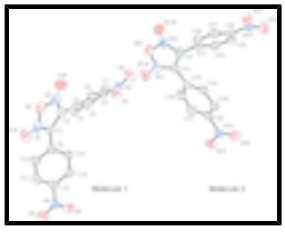


Fig. 1. Molecular structure of the two independent molecules (1 and 2) of compound (II), showing the crystallographic atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. The disordered N-oxide O-atoms, O1B and O21B, bonded to atoms N2 and N22, respectively, are drawn with red and white checkered patterned ellipses. The hydrogen atoms have been omitted for clarity.

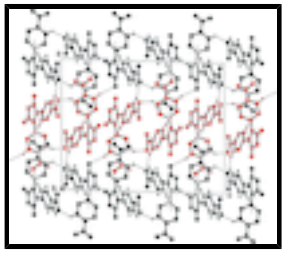


Fig. 2. A view along the *b* axis of the crystal packing of compound (II). The C—H···O contacts are shown as dashed lines. The atoms of Molecule 2 are red [H-atoms not involved in C—H···O contacts have been removed for clarity].

3,4-Bis(4-nitrophenyl)-1,2,5-oxadiazole 2-oxide

Crystal data

$C_{14}H_8N_4O_6$	$F(000)$ = 1344
M_r = 328.24	D_x = 1.599 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 22316 reflections
a = 17.3977 (11) Å	θ = 1.4–29.6°
b = 7.2813 (4) Å	μ = 0.13 mm^{-1}
c = 21.5341 (13) Å	T = 173 K
β = 91.667 (5)°	Block, yellow
V = 2726.7 (3) Å^3	0.40 × 0.35 × 0.30 mm
Z = 8	

Data collection

Stoe IPDS-2 diffractometer	5558 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.054$
graphite	$\theta_{max} = 29.3°$, $\theta_{min} = 1.9°$
Detector resolution: 6.67 pixels mm^{-1}	$h = -23 \rightarrow 23$
φ and ω scans	$k = -9 \rightarrow 9$
29047 measured reflections	$l = -29 \rightarrow 27$
7339 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.060$	H-atom parameters constrained
$wR(F^2) = 0.133$	$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 1.6814P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} = 0.001$
7339 reflections	$\Delta\rho_{max} = 0.22$ e $Å^{-3}$
452 parameters	$\Delta\rho_{min} = -0.17$ e $Å^{-3}$
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0064 (7)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1A	−0.20064 (15)	1.0812 (5)	0.33132 (14)	0.0569 (10)	0.570
O1B	−0.0065 (2)	1.3687 (5)	0.26989 (17)	0.0467 (12)	0.430
O2	−0.11135 (10)	1.2492 (2)	0.29646 (8)	0.0559 (6)	
O3	0.34176 (9)	0.9789 (2)	0.41298 (8)	0.0532 (6)	
O4	0.35733 (9)	1.0989 (3)	0.32312 (9)	0.0613 (6)	
O5	−0.16080 (10)	0.2648 (3)	0.48002 (10)	0.0649 (7)	

O6	−0.03872 (10)	0.2186 (2)	0.47351 (8)	0.0533 (6)	
N1	−0.13345 (11)	1.0918 (3)	0.32958 (9)	0.0482 (6)	
N2	−0.03118 (11)	1.2504 (3)	0.29531 (9)	0.0458 (6)	
N3	0.31689 (10)	1.0437 (2)	0.36429 (9)	0.0408 (5)	
N4	−0.09605 (11)	0.3130 (3)	0.46617 (8)	0.0435 (6)	
C1	−0.07019 (11)	1.0052 (3)	0.34783 (9)	0.0374 (6)	
C2	−0.00587 (12)	1.1062 (3)	0.32676 (9)	0.0376 (6)	
C3	0.07736 (11)	1.0830 (3)	0.33666 (9)	0.0350 (6)	
C4	0.10802 (12)	1.0318 (3)	0.39460 (9)	0.0384 (6)	
C5	0.18658 (11)	1.0173 (3)	0.40352 (10)	0.0378 (6)	
C6	0.23332 (11)	1.0561 (3)	0.35481 (10)	0.0359 (6)	
C7	0.20455 (12)	1.1079 (3)	0.29709 (10)	0.0403 (6)	
C8	0.12612 (12)	1.1209 (3)	0.28824 (10)	0.0403 (6)	
C9	−0.07511 (11)	0.8297 (3)	0.38100 (9)	0.0359 (6)	
C10	−0.14024 (12)	0.7885 (3)	0.41433 (10)	0.0442 (7)	
C11	−0.14705 (12)	0.6194 (3)	0.44269 (11)	0.0449 (7)	
C12	−0.08802 (12)	0.4955 (3)	0.43814 (9)	0.0384 (6)	
C13	−0.02201 (11)	0.5341 (3)	0.40691 (10)	0.0382 (6)	
C14	−0.01649 (11)	0.7014 (3)	0.37723 (9)	0.0373 (6)	
O21A	0.29246 (18)	1.1020 (5)	0.16767 (16)	0.0578 (13)	0.500
O21B	0.49456 (18)	1.3863 (4)	0.22893 (14)	0.0461 (10)	0.500
O22	0.38508 (9)	1.2667 (2)	0.20230 (8)	0.0542 (6)	
O23	0.85144 (9)	1.0823 (3)	0.17637 (9)	0.0620 (7)	
O24	0.82849 (10)	0.9729 (3)	0.08579 (9)	0.0597 (6)	
O25	0.31736 (10)	0.2800 (2)	0.02256 (9)	0.0582 (6)	
O26	0.44007 (10)	0.2367 (2)	0.02397 (8)	0.0537 (6)	
N21	0.36029 (11)	1.1097 (3)	0.17004 (9)	0.0464 (6)	
N22	0.46569 (11)	1.2675 (3)	0.20255 (9)	0.0445 (6)	
N23	0.80723 (10)	1.0366 (2)	0.13458 (9)	0.0420 (6)	
N24	0.38324 (11)	0.3300 (3)	0.03385 (8)	0.0425 (6)	
C21	0.42189 (11)	1.0222 (3)	0.15133 (9)	0.0367 (6)	
C22	0.48800 (11)	1.1233 (3)	0.17116 (9)	0.0366 (6)	
C23	0.57059 (11)	1.0973 (3)	0.16113 (9)	0.0351 (6)	
C24	0.59670 (11)	1.0453 (3)	0.10327 (9)	0.0378 (6)	
C25	0.67416 (12)	1.0243 (3)	0.09420 (10)	0.0384 (6)	
C26	0.72470 (11)	1.0581 (3)	0.14354 (10)	0.0361 (6)	
C27	0.70047 (12)	1.1111 (3)	0.20112 (10)	0.0396 (6)	
C28	0.62323 (12)	1.1303 (3)	0.20988 (9)	0.0391 (6)	
C29	0.41346 (11)	0.8464 (3)	0.11884 (9)	0.0359 (6)	
C30	0.34415 (12)	0.8024 (3)	0.08826 (10)	0.0449 (7)	
C31	0.33437 (12)	0.6338 (3)	0.06037 (11)	0.0452 (7)	
C32	0.39471 (12)	0.5119 (3)	0.06177 (9)	0.0380 (6)	
C33	0.46440 (11)	0.5523 (3)	0.09033 (10)	0.0387 (6)	
C34	0.47300 (11)	0.7196 (3)	0.11986 (10)	0.0375 (6)	
H4	0.07490	1.00690	0.42790	0.0460*	
H7	0.23810	1.13390	0.26420	0.0480*	
H8	0.10510	1.15590	0.24880	0.0480*	
H10	−0.18010	0.87690	0.41760	0.0530*	
H11	−0.19170	0.58940	0.46490	0.0540*	

H13	0.01880	0.44760	0.40580	0.0460*
H14	0.02770	0.72880	0.35410	0.0450*
H5	0.20810	0.98100	0.44270	0.0450*
H24	0.56090	1.02430	0.06990	0.0450*
H25	0.69240	0.98750	0.05500	0.0460*
H27	0.73660	1.13390	0.23410	0.0480*
H28	0.60550	1.16630	0.24940	0.0470*
H30	0.30350	0.88940	0.08670	0.0540*
H31	0.28670	0.60220	0.04050	0.0540*
H33	0.50570	0.46700	0.08970	0.0460*
H34	0.52010	0.74810	0.14110	0.0450*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0354 (14)	0.080 (2)	0.0549 (17)	0.0116 (14)	−0.0039 (12)	0.0144 (15)
O1B	0.054 (2)	0.042 (2)	0.044 (2)	0.0035 (16)	0.0018 (16)	0.0160 (16)
O2	0.0530 (10)	0.0577 (11)	0.0558 (10)	0.0170 (8)	−0.0162 (8)	0.0003 (8)
O3	0.0414 (8)	0.0630 (11)	0.0547 (10)	0.0061 (8)	−0.0064 (7)	0.0046 (8)
O4	0.0396 (9)	0.0799 (13)	0.0647 (11)	−0.0101 (8)	0.0077 (8)	0.0087 (10)
O5	0.0531 (10)	0.0581 (11)	0.0836 (13)	−0.0183 (9)	0.0061 (9)	0.0106 (10)
O6	0.0598 (10)	0.0446 (9)	0.0553 (10)	0.0018 (8)	−0.0015 (8)	0.0031 (7)
N1	0.0411 (10)	0.0575 (12)	0.0454 (10)	0.0076 (9)	−0.0081 (8)	−0.0004 (9)
N2	0.0463 (10)	0.0458 (11)	0.0445 (10)	0.0058 (8)	−0.0100 (8)	0.0011 (9)
N3	0.0367 (9)	0.0356 (9)	0.0501 (10)	−0.0017 (7)	−0.0009 (8)	−0.0050 (8)
N4	0.0467 (10)	0.0430 (10)	0.0406 (9)	−0.0107 (8)	−0.0025 (8)	−0.0041 (8)
C1	0.0344 (10)	0.0432 (11)	0.0342 (10)	0.0044 (8)	−0.0047 (7)	−0.0030 (8)
C2	0.0396 (10)	0.0394 (11)	0.0334 (10)	0.0023 (8)	−0.0049 (8)	−0.0012 (8)
C3	0.0350 (10)	0.0319 (10)	0.0379 (10)	−0.0039 (8)	−0.0036 (8)	0.0014 (8)
C4	0.0388 (10)	0.0399 (11)	0.0365 (10)	−0.0040 (8)	−0.0007 (8)	0.0027 (8)
C5	0.0382 (10)	0.0376 (11)	0.0374 (10)	−0.0017 (8)	−0.0044 (8)	0.0020 (8)
C6	0.0342 (9)	0.0285 (9)	0.0450 (11)	−0.0025 (8)	−0.0009 (8)	−0.0034 (8)
C7	0.0403 (11)	0.0396 (11)	0.0412 (11)	−0.0049 (9)	0.0033 (8)	0.0022 (9)
C8	0.0417 (11)	0.0397 (11)	0.0393 (11)	−0.0041 (9)	−0.0030 (8)	0.0051 (9)
C9	0.0310 (9)	0.0428 (11)	0.0337 (9)	−0.0030 (8)	−0.0030 (7)	−0.0022 (8)
C10	0.0351 (10)	0.0493 (13)	0.0485 (12)	0.0030 (9)	0.0047 (9)	0.0019 (10)
C11	0.0355 (10)	0.0519 (13)	0.0475 (12)	−0.0038 (9)	0.0056 (9)	0.0012 (10)
C12	0.0394 (10)	0.0391 (11)	0.0364 (10)	−0.0069 (8)	−0.0036 (8)	−0.0023 (8)
C13	0.0344 (10)	0.0379 (10)	0.0419 (11)	−0.0026 (8)	−0.0033 (8)	−0.0048 (9)
C14	0.0331 (10)	0.0404 (11)	0.0383 (10)	−0.0041 (8)	0.0005 (8)	−0.0037 (8)
O21A	0.0357 (16)	0.078 (3)	0.060 (2)	0.0089 (16)	0.0076 (14)	−0.0136 (18)
O21B	0.0495 (17)	0.0430 (17)	0.0459 (17)	−0.0024 (14)	0.0008 (13)	−0.0184 (14)
O22	0.0515 (9)	0.0603 (11)	0.0514 (9)	0.0154 (8)	0.0136 (7)	−0.0062 (8)
O23	0.0375 (8)	0.0833 (14)	0.0650 (11)	−0.0078 (9)	−0.0038 (8)	−0.0061 (10)
O24	0.0454 (9)	0.0716 (12)	0.0628 (11)	0.0054 (8)	0.0119 (8)	−0.0120 (9)
O25	0.0519 (10)	0.0559 (11)	0.0669 (11)	−0.0174 (8)	0.0022 (8)	−0.0114 (9)
O26	0.0573 (10)	0.0465 (9)	0.0572 (10)	0.0029 (8)	0.0022 (8)	−0.0064 (8)
N21	0.0406 (10)	0.0574 (12)	0.0414 (10)	0.0057 (9)	0.0070 (7)	−0.0025 (9)

N22	0.0470 (10)	0.0452 (11)	0.0416 (10)	0.0037 (8)	0.0085 (8)	−0.0041 (8)
N23	0.0386 (9)	0.0359 (9)	0.0516 (11)	−0.0028 (7)	0.0045 (8)	0.0039 (8)
N24	0.0481 (10)	0.0408 (10)	0.0386 (9)	−0.0057 (8)	0.0037 (8)	0.0020 (8)
C21	0.0334 (9)	0.0439 (11)	0.0331 (9)	0.0015 (8)	0.0041 (7)	−0.0002 (8)
C22	0.0372 (10)	0.0397 (11)	0.0331 (9)	0.0008 (8)	0.0050 (7)	−0.0004 (8)
C23	0.0371 (10)	0.0309 (10)	0.0375 (10)	−0.0038 (8)	0.0036 (8)	−0.0006 (8)
C24	0.0379 (10)	0.0374 (10)	0.0380 (10)	−0.0030 (8)	0.0015 (8)	−0.0029 (8)
C25	0.0414 (11)	0.0367 (10)	0.0375 (10)	−0.0023 (8)	0.0058 (8)	−0.0039 (8)
C26	0.0371 (10)	0.0280 (9)	0.0433 (11)	−0.0036 (8)	0.0040 (8)	0.0021 (8)
C27	0.0402 (11)	0.0384 (11)	0.0401 (11)	−0.0061 (9)	−0.0022 (8)	−0.0006 (9)
C28	0.0422 (11)	0.0397 (11)	0.0354 (10)	−0.0053 (9)	0.0036 (8)	−0.0030 (8)
C29	0.0319 (9)	0.0415 (11)	0.0343 (9)	−0.0019 (8)	0.0038 (7)	0.0012 (8)
C30	0.0357 (10)	0.0499 (13)	0.0488 (12)	0.0062 (9)	−0.0046 (9)	−0.0042 (10)
C31	0.0338 (10)	0.0525 (13)	0.0488 (12)	−0.0026 (9)	−0.0064 (9)	−0.0063 (10)
C32	0.0395 (10)	0.0400 (11)	0.0346 (10)	−0.0054 (8)	0.0041 (8)	0.0006 (8)
C33	0.0324 (9)	0.0385 (11)	0.0454 (11)	−0.0013 (8)	0.0045 (8)	0.0032 (9)
C34	0.0291 (9)	0.0412 (11)	0.0421 (11)	−0.0049 (8)	0.0005 (8)	0.0026 (9)

Geometric parameters (Å, °)

O1A—N1	1.173 (3)	C10—C11	1.381 (3)
O1B—N2	1.113 (4)	C11—C12	1.373 (3)
O2—N2	1.396 (3)	C12—C13	1.377 (3)
O2—N1	1.409 (3)	C13—C14	1.380 (3)
O3—N3	1.218 (2)	C4—H4	0.9500
O4—N3	1.216 (3)	C5—H5	0.9500
O5—N4	1.225 (3)	C7—H7	0.9500
O6—N4	1.218 (3)	C8—H8	0.9500
O21A—N21	1.181 (4)	C10—H10	0.9500
O21B—N22	1.143 (4)	C11—H11	0.9500
O22—N21	1.399 (3)	C13—H13	0.9500
O22—N22	1.402 (2)	C14—H14	0.9500
O23—N23	1.213 (3)	C21—C22	1.421 (3)
O24—N23	1.216 (3)	C21—C29	1.464 (3)
O25—N24	1.220 (3)	C22—C23	1.471 (3)
O26—N24	1.223 (3)	C23—C24	1.391 (3)
N1—C1	1.318 (3)	C23—C28	1.394 (3)
N2—C2	1.318 (3)	C24—C25	1.376 (3)
N3—C6	1.465 (3)	C25—C26	1.381 (3)
N4—C12	1.468 (3)	C26—C27	1.376 (3)
N21—C21	1.320 (3)	C27—C28	1.370 (3)
N22—C22	1.314 (3)	C29—C30	1.394 (3)
N23—C26	1.463 (3)	C29—C34	1.387 (3)
N24—C32	1.466 (3)	C30—C31	1.375 (3)
C1—C9	1.468 (3)	C31—C32	1.374 (3)
C1—C2	1.424 (3)	C32—C33	1.375 (3)
C2—C3	1.467 (3)	C33—C34	1.380 (3)
C3—C8	1.391 (3)	C24—H24	0.9500
C3—C4	1.393 (3)	C25—H25	0.9500

C4—C5	1.379 (3)	C27—H27	0.9500
C5—C6	1.375 (3)	C28—H28	0.9500
C6—C7	1.379 (3)	C30—H30	0.9500
C7—C8	1.376 (3)	C31—H31	0.9500
C9—C10	1.392 (3)	C33—H33	0.9500
C9—C14	1.387 (3)	C34—H34	0.9500
N1—O2—N2	107.55 (16)	C6—C5—H5	121.00
N21—O22—N22	107.40 (16)	C8—C7—H7	121.00
O1A—N1—O2	110.9 (2)	C6—C7—H7	121.00
O2—N1—C1	107.59 (17)	C7—C8—H8	120.00
O1A—N1—C1	141.5 (3)	C3—C8—H8	120.00
O2—N2—C2	107.76 (18)	C11—C10—H10	120.00
O1B—N2—O2	114.4 (2)	C9—C10—H10	120.00
O1B—N2—C2	137.8 (3)	C10—C11—H11	121.00
O3—N3—O4	123.86 (18)	C12—C11—H11	121.00
O3—N3—C6	118.05 (17)	C14—C13—H13	121.00
O4—N3—C6	118.09 (18)	C12—C13—H13	121.00
O5—N4—C12	117.3 (2)	C13—C14—H14	120.00
O6—N4—C12	118.49 (18)	C9—C14—H14	120.00
O5—N4—O6	124.2 (2)	N21—C21—C22	108.40 (19)
O22—N21—C21	107.74 (17)	N21—C21—C29	119.80 (18)
O21A—N21—O22	110.7 (2)	C22—C21—C29	131.73 (18)
O21A—N21—C21	141.6 (3)	N22—C22—C21	108.67 (18)
O21B—N22—C22	136.8 (2)	N22—C22—C23	118.87 (19)
O22—N22—C22	107.77 (17)	C21—C22—C23	132.43 (19)
O21B—N22—O22	115.5 (2)	C22—C23—C24	120.81 (17)
O23—N23—O24	122.91 (19)	C22—C23—C28	119.36 (18)
O23—N23—C26	118.45 (18)	C24—C23—C28	119.80 (18)
O24—N23—C26	118.64 (18)	C23—C24—C25	120.27 (18)
O25—N24—O26	124.0 (2)	C24—C25—C26	118.37 (19)
O26—N24—C32	118.17 (18)	N23—C26—C25	119.00 (19)
O25—N24—C32	117.85 (18)	N23—C26—C27	118.45 (18)
N1—C1—C9	120.10 (18)	C25—C26—C27	122.55 (19)
C2—C1—C9	131.46 (18)	C26—C27—C28	118.70 (19)
N1—C1—C2	108.35 (19)	C23—C28—C27	120.30 (19)
N2—C2—C3	118.68 (19)	C21—C29—C30	119.99 (18)
C1—C2—C3	132.49 (19)	C21—C29—C34	120.66 (18)
N2—C2—C1	108.73 (18)	C30—C29—C34	119.3 (2)
C4—C3—C8	119.90 (18)	C29—C30—C31	120.4 (2)
C2—C3—C4	120.76 (18)	C30—C31—C32	118.8 (2)
C2—C3—C8	119.28 (18)	N24—C32—C31	118.67 (19)
C3—C4—C5	119.83 (19)	N24—C32—C33	118.98 (19)
C4—C5—C6	118.94 (19)	C31—C32—C33	122.3 (2)
N3—C6—C5	119.18 (19)	C32—C33—C34	118.50 (19)
N3—C6—C7	118.37 (18)	C29—C34—C33	120.61 (18)
C5—C6—C7	122.45 (19)	C23—C24—H24	120.00
C6—C7—C8	118.45 (19)	C25—C24—H24	120.00
C3—C8—C7	120.4 (2)	C24—C25—H25	121.00
C1—C9—C10	119.91 (18)	C26—C25—H25	121.00

C1—C9—C14	120.30 (18)	C26—C27—H27	121.00
C10—C9—C14	119.7 (2)	C28—C27—H27	121.00
C9—C10—C11	120.1 (2)	C23—C28—H28	120.00
C10—C11—C12	118.7 (2)	C27—C28—H28	120.00
C11—C12—C13	122.5 (2)	C29—C30—H30	120.00
N4—C12—C13	118.45 (19)	C31—C30—H30	120.00
N4—C12—C11	119.03 (19)	C30—C31—H31	121.00
C12—C13—C14	118.47 (19)	C32—C31—H31	121.00
C9—C14—C13	120.40 (18)	C32—C33—H33	121.00
C3—C4—H4	120.00	C34—C33—H33	121.00
C5—C4—H4	120.00	C29—C34—H34	120.00
C4—C5—H5	121.00	C33—C34—H34	120.00
N2—O2—N1—O1A	−178.8 (2)	C4—C3—C8—C7	0.1 (3)
N2—O2—N1—C1	0.7 (2)	C2—C3—C4—C5	−177.6 (2)
N1—O2—N2—C2	−1.4 (2)	C3—C4—C5—C6	0.8 (3)
N22—O22—N21—O21A	−179.3 (2)	C4—C5—C6—C7	−0.5 (3)
N22—O22—N21—C21	−0.7 (2)	C4—C5—C6—N3	179.06 (19)
N21—O22—N22—O21B	−178.0 (2)	C5—C6—C7—C8	0.0 (3)
N21—O22—N22—C22	1.5 (2)	N3—C6—C7—C8	−179.54 (19)
O2—N1—C1—C9	−176.89 (17)	C6—C7—C8—C3	0.2 (3)
O1A—N1—C1—C2	179.5 (4)	C14—C9—C10—C11	−1.1 (3)
O1A—N1—C1—C9	2.4 (5)	C1—C9—C14—C13	−178.11 (19)
O2—N1—C1—C2	0.1 (2)	C1—C9—C10—C11	176.3 (2)
O2—N2—C2—C3	−175.41 (17)	C10—C9—C14—C13	−0.7 (3)
O2—N2—C2—C1	1.4 (2)	C9—C10—C11—C12	1.1 (3)
O3—N3—C6—C5	8.3 (3)	C10—C11—C12—N4	−177.74 (19)
O3—N3—C6—C7	−172.13 (19)	C10—C11—C12—C13	0.7 (3)
O4—N3—C6—C5	−171.6 (2)	C11—C12—C13—C14	−2.4 (3)
O4—N3—C6—C7	8.0 (3)	N4—C12—C13—C14	175.99 (18)
O5—N4—C12—C11	16.0 (3)	C12—C13—C14—C9	2.4 (3)
O6—N4—C12—C11	−164.9 (2)	N21—C21—C22—N22	1.4 (2)
O6—N4—C12—C13	16.7 (3)	N21—C21—C22—C23	−176.5 (2)
O5—N4—C12—C13	−162.5 (2)	C29—C21—C22—N22	−175.4 (2)
O22—N21—C21—C22	−0.4 (2)	C29—C21—C22—C23	6.8 (4)
O21A—N21—C21—C22	177.6 (4)	N21—C21—C29—C30	23.0 (3)
O21A—N21—C21—C29	−5.2 (5)	N21—C21—C29—C34	−154.8 (2)
O22—N21—C21—C29	176.84 (17)	C22—C21—C29—C30	−160.6 (2)
O21B—N22—C22—C23	−4.2 (4)	C22—C21—C29—C34	21.6 (3)
O21B—N22—C22—C21	177.6 (3)	N22—C22—C23—C24	−135.9 (2)
O22—N22—C22—C21	−1.7 (2)	N22—C22—C23—C28	42.3 (3)
O22—N22—C22—C23	176.42 (17)	C21—C22—C23—C24	41.7 (3)
O24—N23—C26—C25	−6.9 (3)	C21—C22—C23—C28	−140.1 (2)
O23—N23—C26—C25	173.5 (2)	C22—C23—C24—C25	179.0 (2)
O23—N23—C26—C27	−6.6 (3)	C28—C23—C24—C25	0.8 (3)
O24—N23—C26—C27	173.0 (2)	C22—C23—C28—C27	−178.5 (2)
O25—N24—C32—C33	162.4 (2)	C24—C23—C28—C27	−0.2 (3)
O25—N24—C32—C31	−15.3 (3)	C23—C24—C25—C26	−0.7 (3)
O26—N24—C32—C31	165.6 (2)	C24—C25—C26—N23	−179.92 (19)
O26—N24—C32—C33	−16.7 (3)	C24—C25—C26—C27	0.2 (3)

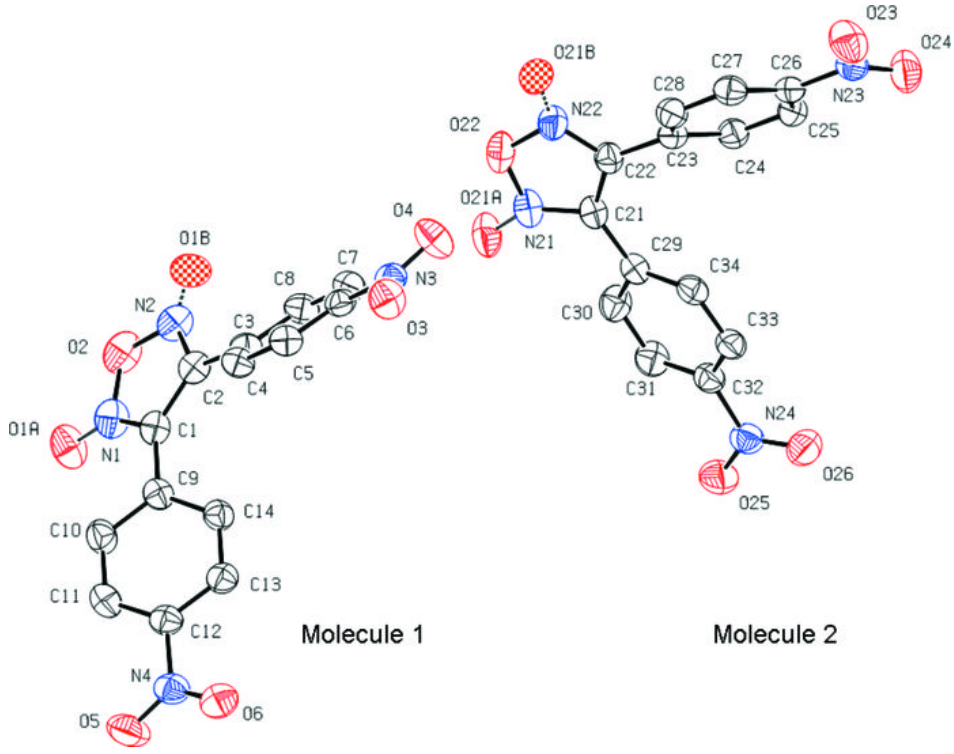
N1—C1—C2—N2	−1.0 (2)	N23—C26—C27—C28	−179.59 (19)
N1—C1—C9—C14	152.1 (2)	C25—C26—C27—C28	0.3 (3)
C2—C1—C9—C10	158.6 (2)	C26—C27—C28—C23	−0.3 (3)
C2—C1—C9—C14	−24.1 (3)	C21—C29—C30—C31	−176.6 (2)
C9—C1—C2—C3	−8.2 (4)	C34—C29—C30—C31	1.2 (3)
N1—C1—C2—C3	175.2 (2)	C21—C29—C34—C33	178.61 (19)
C9—C1—C2—N2	175.6 (2)	C30—C29—C34—C33	0.8 (3)
N1—C1—C9—C10	−25.2 (3)	C29—C30—C31—C32	−1.7 (3)
N2—C2—C3—C4	135.9 (2)	C30—C31—C32—N24	177.95 (19)
N2—C2—C3—C8	−41.2 (3)	C30—C31—C32—C33	0.3 (3)
C1—C2—C3—C4	−40.1 (3)	N24—C32—C33—C34	−175.98 (19)
C1—C2—C3—C8	142.9 (2)	C31—C32—C33—C34	1.7 (3)
C8—C3—C4—C5	−0.6 (3)	C32—C33—C34—C29	−2.2 (3)
C2—C3—C8—C7	177.2 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O5^{i}	0.95	2.59	3.283 (3)	130
C7—H7···O21A	0.95	2.32	3.218 (4)	157
C10—H10···O1A	0.95	2.40	2.955 (4)	117
C14—H14···O23^{ii}	0.95	2.46	3.244 (3)	139
C25—H25···O25^{iii}	0.95	2.57	3.358 (3)	141
C27—H27···O1A^{iv}	0.95	2.36	3.254 (4)	156
C28—H28···O21B	0.95	2.54	2.951 (4)	106
C30—H30···O21A	0.95	2.34	2.929 (4)	119
C31—H31···O5^{v}	0.95	2.52	3.261 (3)	135
C34—H34···O4^{ii}	0.95	2.50	3.284 (3)	140

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

Fig. 1


O23
O24
O21B
C27
C26
C28
N22
N23
O22
C25
C23
C24
C22
O21A
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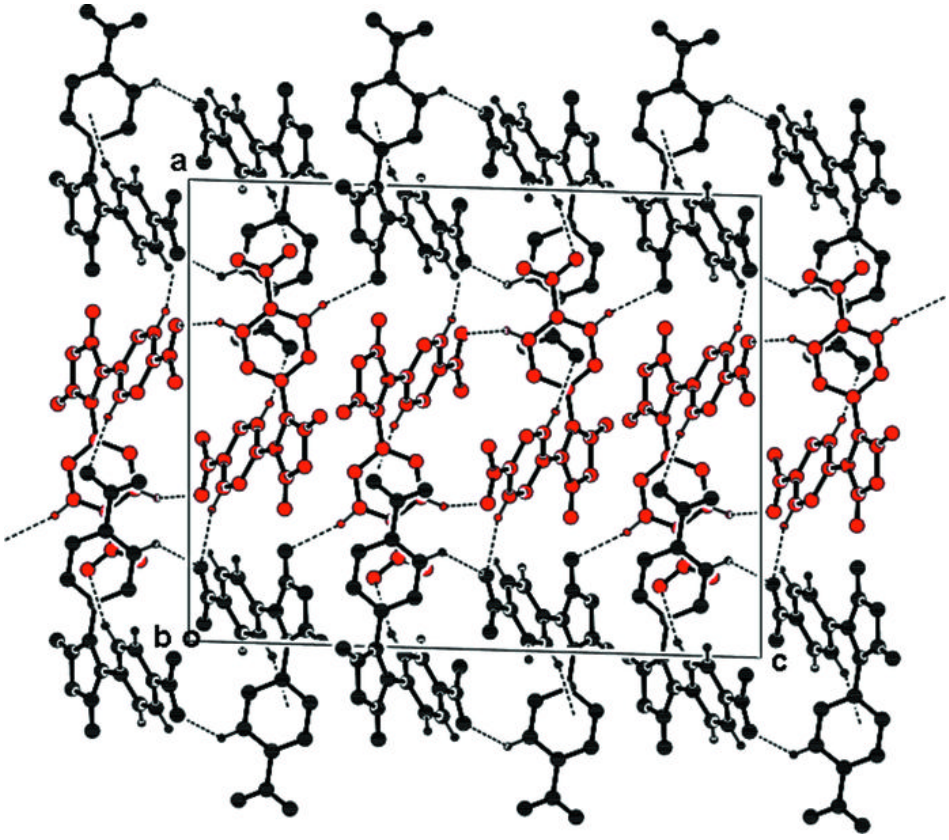
Fig. 2