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7-Nitro-1,2,3a,4-tetrahydrobenzo[*b*]-[1,3]oxazolo[3,2-*d*]oxazine: a new heterocycle

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.076
wR factor = 0.095
Data-to-parameter ratio = 10.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{10}H_{10}N_2O_4$, crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit. The five-membered ring of the oxazole moiety adopts a half-chair conformation in molecule *A* and an envelope conformation in molecule *B*. The oxazine ring adopts a sofa conformation in both molecules.

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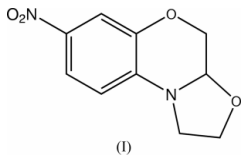
Comment

The oxazole moiety is frequently found in biologically active molecules and natural products. During the development of facile synthetic methods for compounds having this moiety, we synthesized 7-nitro-1,2,3a,4-tetrahydrobenzo-[*b*][1,3]oxazolo[3,2-*d*]oxazine, (I), for the first time.



(I)

The molecular structure of (I) is shown in Fig. 1. The title compound crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit. All the bond lengths and angles have normal values (Allen *et al.*, 1987). The five-membered ring adopts a half-chair conformation in molecule *A* and an envelope conformation in molecule *B*. The oxazine ring adopts a sofa conformation in both molecules..

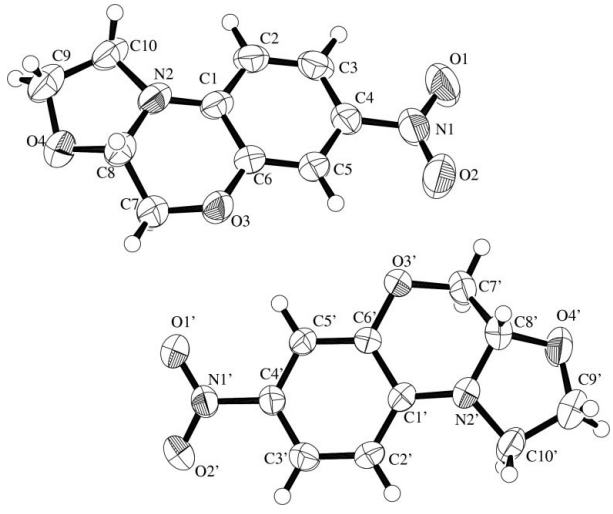


Figure 1
A perspective view of the asymmetric unit of the title compound, with displacement ellipsoids drawn at the 50% probability level.

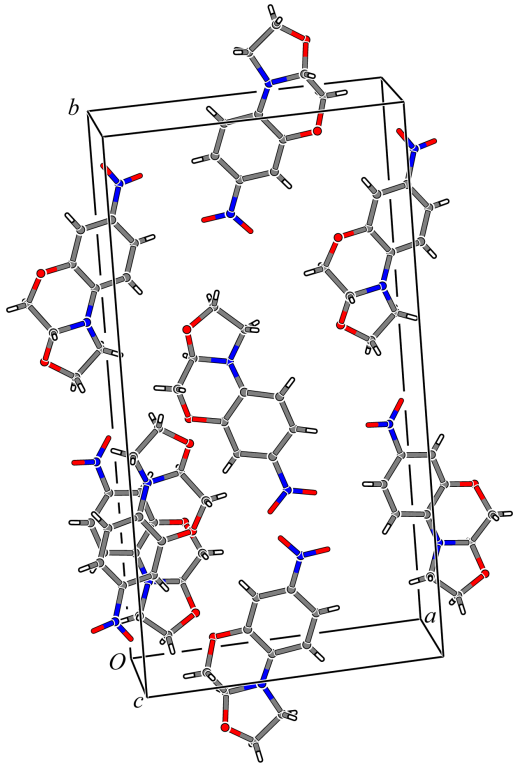


Figure 2
Packing diagram, viewed down the *c* axis.

Experimental

The title compound was prepared by nitration (using fuming nitric acid) of tricyclic oxazolooxazine, *i.e.* 1,2,3a,4-tetrahydrobenzo-[*b*][1,3]oxazolo[3,2-*d*] oxazine, which was synthesized according to the method of Lohray *et al.* (2000). Crystals suitable for X-ray diffraction analysis were grown from a mixture of *n*-butanol and chloroform (ratio 9:1).

Crystal data

$C_{10}H_{10}N_2O_4$
M_r = 222.20
Monoclinic, $P2_1/c$
a = 12.164 (2) Å
b = 22.079 (4) Å
c = 7.373 (2) Å
β = 100.86 (2)°
V = 1944.6 (6) Å^3
Z = 8
D_x = 1.518 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 25.8–29.5°
μ = 1.02 mm^{-1}
T = 298.2 K
Block, yellow
0.45 × 0.30 × 0.20 mm

Data collection

Rigaku AFC-7*S* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North *et al.*, 1968)
T_{min} = 0.586, T_{max} = 0.816
4615 measured reflections
3769 independent reflections
3111 reflections with $I > 0.5\sigma(I)$
R_{int} = 0.082
θ_{max} = 70.0°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 26$
$l = -8 \rightarrow 3$
3 standard reflections
every 150 reflections
intensity decay: *ca* 1.0%

Refinement

Refinement on F
R = 0.076
wR = 0.095
S = 1.98
3111 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o) + 0.00016|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1*A*—N1*A*	1.221 (4)	O4*B*—C8*B*	1.406 (3)
O2*A*—N1*A*	1.228 (4)	O4*B*—C9*B*	1.413 (4)
O3*A*—C6*A*	1.383 (3)	N1*A*—C4*A*	1.446 (4)
O3*A*—C7*A*	1.426 (3)	N2*A*—C8*A*	1.446 (4)
O4*A*—C8*A*	1.399 (3)	N2*A*—C1*A*	1.354 (3)
O4*A*—C9*A*	1.433 (4)	N2*A*—C10*A*	1.455 (3)
O1*B*—N1*B*	1.231 (3)	N1*B*—C4*B*	1.438 (3)
O2*B*—N1*B*	1.231 (3)	N2*B*—C1*B*	1.371 (3)
O3*B*—C6*B*	1.371 (3)	N2*B*—C8*B*	1.453 (3)
O3*B*—C7*B*	1.432 (3)	N2*B*—C10*B*	1.458 (3)
C6*A*—O3*A*—C7*A*	112.74 (19)	O3*A*—C6*A*—C1*A*	119.9 (2)
C8*A*—O4*A*—C9*A*	104.8 (2)	O3*A*—C6*A*—C5*A*	118.2 (2)
C6*B*—O3*B*—C7*B*	113.23 (18)	O3*A*—C7*A*—C8*A*	110.0 (2)
C8*B*—O4*B*—C9*B*	105.7 (2)	O4*A*—C8*A*—N2*A*	105.5 (2)
O1*A*—N1*A*—C4*A*	118.9 (2)	O4*A*—C8*A*—C7*A*	110.9 (3)
O1*A*—N1*A*—O2*A*	122.8 (3)	N2*A*—C8*A*—C7*A*	110.5 (2)
O2*A*—N1*A*—C4*A*	118.3 (3)	O4*A*—C9*A*—C10*A*	104.8 (2)
C8*A*—N2*A*—C10*A*	109.3 (2)	N2*A*—C10*A*—C9*A*	101.5 (3)
C1*A*—N2*A*—C10*A*	126.7 (2)	N2*B*—C1*B*—C2*B*	122.5 (2)
C1*A*—N2*A*—C8*A*	121.0 (2)	N2*B*—C1*B*—C6*B*	118.3 (2)
O2*B*—N1*B*—C4*B*	118.65 (19)	N1*B*—C4*B*—C3*B*	119.5 (2)
O1*B*—N1*B*—O2*B*	122.1 (2)	N1*B*—C4*B*—C5*B*	118.28 (19)
O1*B*—N1*B*—C4*B*	119.2 (2)	O3*B*—C6*B*—C1*B*	120.96 (18)
C1*B*—N2*B*—C10*B*	124.4 (2)	O3*B*—C6*B*—C5*B*	118.56 (19)
C8*B*—N2*B*—C10*B*	110.2 (2)	O3*B*—C7*B*—C8*B*	109.7 (2)
C1*B*—N2*B*—C8*B*	120.67 (19)	O4*B*—C8*B*—N2*B*	105.2 (2)
N2*A*—C1*A*—C2*A*	123.2 (2)	O4*B*—C8*B*—C7*B*	112.0 (2)
N2*A*—C1*A*—C6*A*	119.1 (2)	N2*B*—C8*B*—C7*B*	110.5 (2)
N1*A*—C4*A*—C5*A*	118.4 (2)	O4*B*—C9*B*—C10*B*	107.2 (2)
N1*A*—C4*A*—C3*A*	119.6 (2)	N2*B*—C10*B*—C9*B*	100.7 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3*A*—H2*A*···O2*B*^{i}	0.95	2.42	3.200 (3)	140
C5*B*—H3*B*···O3*A*	0.95	2.59	3.517 (3)	165
C7*A*—H5*A*···O1*B*	0.95	2.54	3.222 (4)	129
C8*A*—H6*A*···O2*B*^{ii}	0.95	2.57	3.325 (4)	136

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

H atoms were included in calculated positions and refined using a riding model, with C—H = 0.95 Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *TEXSAN*; molecular graphics: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.

Lohray, B. B., Bhushan, V., Reddy, A. S. & Rao, V. V. (2000). *Indian J. Chem. Sect. B*, **39**, 297–299.
Molecular Structure Corporation (1994). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA. (Present address: MSC, 9009 New Trails Drive, The Woodlands, TX 77381, USA.)
Molecular Structure Corporation (1995). *TEXSAN*. Version 1.7-2. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA. (Present address: MSC, 9009 New Trails Drive, The Woodlands, TX 77381, USA.)
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.