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1,5-Bis[(2-methoxyethoxy)methyl]-1,5-naphthyridine-4,8(1*H*,5*H*)-dione

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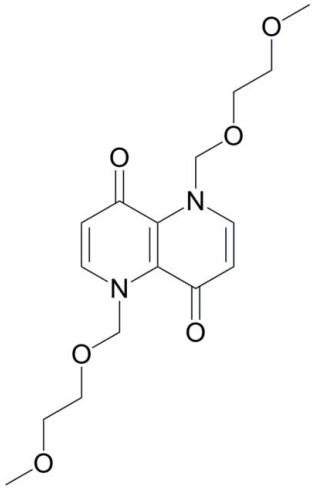
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.140; data-to-parameter ratio = 14.1.

The complete molecule of the title compound, $C_{16}H_{22}N_2O_6$, is generated by crystallographic inversion symmetry. The conformation of the N—C—O—C fragment of the side chain is approximately gauche [torsion angle = −74.84 (17)°]. In the crystal, weak C—H···O interactions link the molecules.

Related literature

The background to the applications of the title compound, see: Shan *et al.* (2005). For the synthesis, see: Toshihiro *et al.* (2002). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{22}N_2O_6$
M_r = 338.36
Monoclinic, $P2_1/n$
a = 7.1610 (14) Å
b = 11.497 (2) Å
c = 10.734 (2) Å
β = 105.45 (3)°
V = 851.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.970, T_{max} = 0.990
3261 measured reflections
1549 independent reflections
1246 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.140
S = 1.01
1549 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 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—H5A···O3^{i}	0.93	2.45	3.264 (2)	147
C6—H6A···O1^{ii}	0.93	2.58	3.397 (2)	147

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6550).

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1,5-Bis[(2-methoxyethoxy)methyl]-1,5-naphthyridine-4,8(1*H*,5*H*)-dione

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S1. Experimental

The title compound was prepared by a method reported in literature (Toshihiro *et al.*, 2002). Colourless blocks were obtained by dissolving it (0.5 g) in methanol (50 ml) and evaporating the solvent slowly at room temperature for about 30 d.

S2. Refinement

H atoms were positioned geometrically and refined as riding groups, with C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.2 for aromatic H, and x = 1.5 for other H.

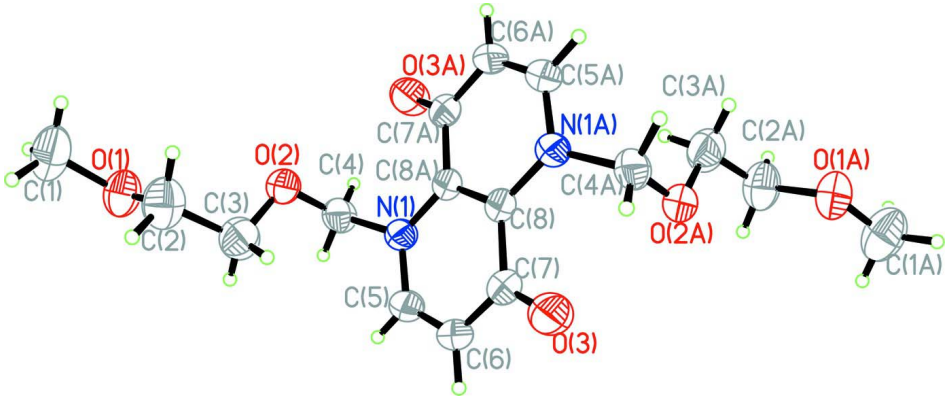

Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

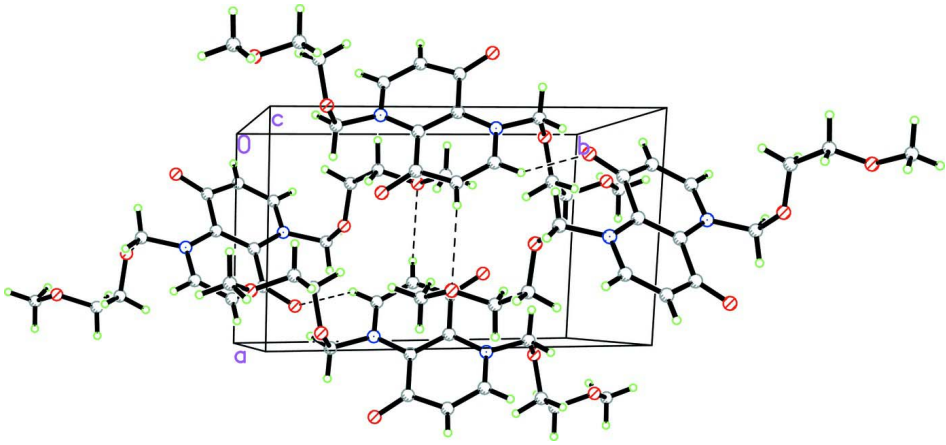

Figure 2

A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{16}H_{22}N_2O_6$
$M_r = 338.36$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.1610$ (14) Å
$b = 11.497$ (2) Å
$c = 10.734$ (2) Å
$\beta = 105.45$ (3)°
$V = 851.8$ (3) Å^3
$Z = 2$
$F(000) = 360$
$D_x = 1.319$ Mg m^{-3}
Melting point: 365 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.970$, $T_{max} = 0.990$
3261 measured reflections
1549 independent reflections
1246 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.7°$
$h = 0 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -12 \rightarrow 12$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.140$
$S = 1.01$
1549 reflections
110 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.30 (2)

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.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.03696 (18)	0.64646 (11)	0.57178 (12)	0.0421 (4)
O1	0.25472 (18)	1.00854 (10)	0.36603 (13)	0.0584 (4)
C1	0.2858 (3)	1.0712 (2)	0.2607 (2)	0.0781 (7)
H1A	0.2389	1.1492	0.2623	0.117*
H1B	0.2177	1.0341	0.1815	0.117*
H1C	0.4219	1.0729	0.2665	0.117*
O2	0.06007 (17)	0.80910 (9)	0.43882 (11)	0.0532 (4)
C5	0.1950 (2)	0.62630 (14)	0.67339 (15)	0.0479 (5)
H5A	0.2437	0.6875	0.7292	0.057*
C2	0.3185 (3)	0.89321 (17)	0.3692 (2)	0.0658 (6)
H2B	0.4581	0.8917	0.3836	0.079*
H2C	0.2603	0.8560	0.2869	0.079*
C3	0.2645 (3)	0.82888 (15)	0.4743 (2)	0.0639 (6)
H3A	0.3325	0.7551	0.4890	0.077*
H3B	0.3014	0.8736	0.5537	0.077*
O3	0.32189 (18)	0.33513 (10)	0.62397 (14)	0.0672 (5)
C4	−0.0118 (2)	0.77036 (13)	0.54011 (17)	0.0494 (5)
H4A	−0.1514	0.7797	0.5165	0.059*
H4B	0.0419	0.8177	0.6160	0.059*
C6	0.2837 (2)	0.52236 (14)	0.69674 (16)	0.0489 (5)
H6A	0.3860	0.5132	0.7704	0.059*
C7	0.2259 (2)	0.42641 (13)	0.61247 (15)	0.0449 (4)
C8	0.0437 (2)	0.44520 (12)	0.50952 (13)	0.0380 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0493 (7)	0.0351 (7)	0.0376 (7)	−0.0011 (5)	0.0041 (5)	−0.0016 (5)
O1	0.0678 (8)	0.0436 (7)	0.0690 (9)	0.0043 (5)	0.0273 (7)	0.0064 (5)
C1	0.0727 (13)	0.0781 (15)	0.0928 (16)	0.0075 (11)	0.0385 (12)	0.0289 (12)
O2	0.0602 (8)	0.0402 (6)	0.0513 (7)	−0.0052 (5)	0.0012 (6)	0.0059 (5)
C5	0.0557 (9)	0.0440 (9)	0.0374 (9)	−0.0068 (7)	0.0009 (7)	−0.0030 (6)
C2	0.0697 (11)	0.0493 (11)	0.0836 (14)	0.0087 (9)	0.0291 (10)	0.0052 (9)

C3	0.0577 (11)	0.0523 (11)	0.0756 (13)	0.0029 (8)	0.0071 (9)	0.0127 (9)
O3	0.0602 (8)	0.0429 (7)	0.0812 (10)	0.0086 (5)	−0.0114 (7)	0.0018 (6)
C4	0.0565 (9)	0.0341 (8)	0.0532 (10)	0.0014 (7)	0.0066 (7)	−0.0044 (7)
C6	0.0490 (9)	0.0464 (9)	0.0413 (9)	−0.0051 (7)	−0.0054 (7)	0.0051 (7)
C7	0.0459 (9)	0.0396 (8)	0.0446 (9)	0.0003 (7)	0.0040 (7)	0.0083 (7)
C8	0.0442 (8)	0.0341 (8)	0.0346 (8)	−0.0035 (6)	0.0086 (6)	0.0034 (6)

Geometric parameters (Å, º)

N1—C5	1.366 (2)	C2—H2B	0.9700
N1—C8^{i}	1.3919 (19)	C2—H2C	0.9700
N1—C4	1.4843 (19)	C3—H3A	0.9700
O1—C2	1.400 (2)	C3—H3B	0.9700
O1—C1	1.407 (2)	O3—C7	1.2426 (18)
C1—H1A	0.9600	C4—H4A	0.9700
C1—H1B	0.9600	C4—H4B	0.9700
C1—H1C	0.9600	C6—C7	1.417 (2)
O2—C4	1.394 (2)	C6—H6A	0.9300
O2—C3	1.429 (2)	C7—C8	1.484 (2)
C5—C6	1.345 (2)	C8—N1^{i}	1.3919 (19)
C5—H5A	0.9300	C8—C8^{i}	1.398 (3)
C2—C3	1.484 (3)		
C5—N1—C8^{i}	119.30 (13)	O2—C3—H3A	109.8
C5—N1—C4	116.10 (13)	C2—C3—H3A	109.8
C8^{i}—N1—C4	123.37 (13)	O2—C3—H3B	109.8
C2—O1—C1	112.60 (16)	C2—C3—H3B	109.8
O1—C1—H1A	109.5	H3A—C3—H3B	108.2
O1—C1—H1B	109.5	O2—C4—N1	111.85 (13)
H1A—C1—H1B	109.5	O2—C4—H4A	109.2
O1—C1—H1C	109.5	N1—C4—H4A	109.2
H1A—C1—H1C	109.5	O2—C4—H4B	109.2
H1B—C1—H1C	109.5	N1—C4—H4B	109.2
C4—O2—C3	114.06 (14)	H4A—C4—H4B	107.9
C6—C5—N1	123.30 (15)	C5—C6—C7	121.98 (14)
C6—C5—H5A	118.3	C5—C6—H6A	119.0
N1—C5—H5A	118.3	C7—C6—H6A	119.0
O1—C2—C3	109.96 (17)	O3—C7—C6	122.30 (14)
O1—C2—H2B	109.7	O3—C7—C8	123.39 (14)
C3—C2—H2B	109.7	C6—C7—C8	114.30 (13)
O1—C2—H2C	109.7	N1^{i}—C8—C8^{i}	119.73 (16)
C3—C2—H2C	109.7	N1^{i}—C8—C7	119.50 (13)
H2B—C2—H2C	108.2	C8^{i}—C8—C7	120.73 (16)
O2—C3—C2	109.45 (16)		
C8^{i}—N1—C5—C6	2.9 (3)	N1—C5—C6—C7	3.7 (3)
C4—N1—C5—C6	−164.78 (16)	C5—C6—C7—O3	170.47 (16)
C1—O1—C2—C3	−174.45 (17)	C5—C6—C7—C8	−8.8 (2)

C4—O2—C3—C2	−167.81 (14)	O3—C7—C8—N1^{i}	6.6 (2)
O1—C2—C3—O2	72.0 (2)	C6—C7—C8—N1^{i}	−174.16 (14)
C3—O2—C4—N1	−74.84 (17)	O3—C7—C8—C8^{i}	−171.19 (18)
C5—N1—C4—O2	97.84 (16)	C6—C7—C8—C8^{i}	8.1 (2)
C8^{i}—N1—C4—O2	−69.31 (19)		

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O3^{ii}	0.93	2.45	3.264 (2)	147
C6—H6*A*···O1^{iii}	0.93	2.58	3.397 (2)	147

Symmetry codes: (ii) −*x*+1/2, *y*+1/2, −*z*+3/2; (iii) *x*+1/2, −*y*+3/2, *z*+1/2.