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3-Morpholino-8,8-dicyanoheptafulvene

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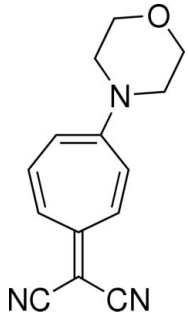
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.063; wR factor = 0.163; data-to-parameter ratio = 16.3.

There are two crystallographically independent molecules, located in general positions, in the asymmetric unit of the title compound, $C_{14}H_{13}N_3O$, which is also known as 4-morpholino-2,4,6-cycloheptatriene-Δ1,α-malononitrile. The bond lengths in the conjugated system indicate a highly polar electronic structure induced by the mesomeric effects of the dialkylamino and dicyanomethylene groups.

Related literature

The related structure of 1-(8,8-dicyanoheptafulven-3-yl)aza-15-crown-5 ether is described by Kubo *et al.* (1997). For related literature, see: Shimanouchi *et al.* (1966).



Experimental

Crystal data

$C_{14}H_{13}N_3O$
M_r = 239.28
Monoclinic, $P2_1/c$
a = 17.211 (6) Å
b = 9.616 (3) Å
c = 15.654 (6) Å
β = 111.7161 (15)°
V = 2406.8 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 123.1 K
0.12 × 0.10 × 0.10 mm

Data collection

Rigaku Saturn CCD diffractometer
Absorption correction: none
18824 measured reflections
5323 independent reflections
4062 reflections with $I > 2\sigma(I)$
R_{int} = 0.070

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.163
S = 1.18
5323 reflections
326 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C9	1.149 (2)	C6—C7	1.374 (2)
N2—C10	1.146 (2)	C8—C9	1.419 (2)
N3—C4	1.374 (2)	C8—C10	1.428 (2)
N4—C23	1.152 (3)	C15—C16	1.439 (2)
N5—C24	1.151 (2)	C15—C21	1.418 (2)
N6—C18	1.384 (2)	C15—C22	1.404 (2)
C1—C2	1.442 (2)	C16—C17	1.353 (2)
C1—C7	1.410 (2)	C17—C18	1.443 (2)
C1—C8	1.404 (2)	C18—C19	1.379 (2)
C2—C3	1.359 (2)	C19—C20	1.404 (2)
C3—C4	1.443 (2)	C20—C21	1.367 (2)
C4—C5	1.385 (2)	C22—C23	1.419 (2)
C5—C6	1.404 (2)	C22—C24	1.421 (2)
C1···C20^{i}	3.305 (3)	C3···C16	3.355 (3)
C12—O1—C13—C14	59.8 (2)	C25—N6—C28—C27	53.0 (2)
C13—O1—C12—C11	−61.1 (2)	C28—N6—C25—C26	−54.8 (2)
C26—O2—C27—C28	59.1 (2)	N3—C11—C12—O1	58.1 (2)
C27—O2—C26—C25	−60.2 (2)	O1—C13—C14—N3	−55.0 (2)
C4—N3—C11—C12	156.52 (19)	N6—C25—C26—O2	58.0 (2)
C11—N3—C14—C13	51.1 (2)	O2—C27—C28—N6	−55.5 (2)
C14—N3—C11—C12	−53.1 (2)		

Symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear* (Rigaku, 1999); data reduction: *CrystalStructure* (Rigaku/MSC, 2006); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2002); program(s) used to refine structure: *SHELXL97* (Sheldrick 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *Mercury* (Version 1.3; Bruno *et al.*, 2002); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2034).

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supplementary materials

Acta Cryst. (2007). E**63**, o2694 [**doi:10.1107/S1600536807020156**]

3-Morpholino-8,8-dicyanoheptafulvene

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Comment

The asymmetric units of compound (I) consists of two crystallographically independent molecules (Figure 1). The 8,8-dicyanoheptafulvene ring of (I) is nearly planar. The mean deviation of each atom from the plane are 0.074 (2) Å and 0.050 (2) Å for both independent molecules. The morpholin ring is in a chair conformation. The dihedral angles between the 8,8-dicyanoheptafulvene planes (defined by C1—C10/N1/N2 and C15—C24/N4/N5) and the morpholin rings (defined by C11—C14/N3/O1 and C25—C28/N6/O2) amount to 19.2 (1)° and 31.1 (1)° in both independent molecules.

The bond length in the seven-membered ring of (I) are different from those in 8,8-dicyanoheptafulvene itself (Shimanouchi *et al.*, 1966) but are comparable to those in 1-(8,8-dicyanoheptafulven-3-yl)aza-15-crown-5 ether (Kubo *et al.*, 1997) (Table 1). The bond lengths in the conjugated system indicate a highly polar electronic structure induced by the mesomeric effects of the dialkylamino and dicyano groups.

Experimental

A pyridine solution (5 ml) of 3-bromo-8,8-dicyanoheptafulvene (0.028 g, 0.12 mmol) and morpholin (0.106 g, 1.2 mmol) was refluxed for 3 hr. The solvent was evaporated *in vacuo* to leave a reidue, which was chromatographed in chloroform on a silica-gel column. On slow evaporation of the solvent, I was obtained in 93% yield.. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.38 (dt, 1H, J = 12.8, 1.1 Hz), 6.89–7.01 (m, 3H), 6.16 (dt, 1H, 7.7, 2.9 Hz), 3.85 (t, 4H, J = 5.1 Hz), 3.41 (t, 4H, t, J = 5.1 Hz); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 160.7, 158.1, 139.5, 135.2, 132.4, 124.7, 116.6, 116.3, 114.5, 66.1 (2 C), 62.8, 48.5 (2 C).

Refinement

The H atoms were positioned with idealized geometry and were refined isotropic (U_{iso}(H) = 1.2U_{eq}(C)) using a riding model with C—H = 0.95 Å for aromatic H atoms and C—H = 0.99 Å for methylene H atoms.

Figures

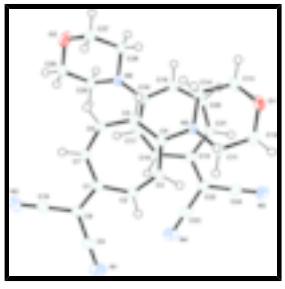


Fig. 1. Crystal structure of I with labelling and displacement ellipsoids drawn at the 50% probability level.

3-Morpholino-8,8-dicyanoheptafulvene

Crystal data

$C_{14}H_{13}N_3O$	F_{000} = 1008.00
M_r = 239.28	D_x = 1.321 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: -P 2ybc	Cell parameters from 5427 reflections
a = 17.211 (6) Å	θ = 3.0–27.5º
b = 9.616 (3) Å	μ = 0.09 mm^{-1}
c = 15.654 (6) Å	T = 123.1 K
β = 111.7161 (15)º	Prism, red
V = 2406.8 (14) Å^3	0.12 × 0.10 × 0.10 mm
Z = 8	

Data collection

Rigaku Saturn CCD diffractometer	4062 reflections with $I > 2\sigma(I)$
Detector resolution: 7.31 pixels mm^{-1}	R_{int} = 0.070
ω scans	θ_{max} = 27.5º
Absorption correction: none	h = −20→22
18824 measured reflections	k = −12→12
5323 independent reflections	l = −20→18

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.063	$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.9287P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.163	$(\Delta/\sigma)_{max} < 0.001$
S = 1.18	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
5323 reflections	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
326 parameters	Extinction correction: none

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement was performed using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (S) are based on F^2. *R*-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating *R*-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}

O1	−0.03816 (9)	−0.00758 (16)	0.61156 (11)	0.0330 (3)
O2	0.52996 (10)	−0.09502 (18)	0.89199 (11)	0.0378 (4)
N1	0.26256 (15)	0.8091 (2)	0.91253 (16)	0.0455 (5)
N2	0.47909 (12)	0.5782 (2)	1.09258 (13)	0.0319 (4)
N3	0.10298 (10)	0.14313 (17)	0.73007 (11)	0.0211 (3)
N4	0.20624 (16)	0.7272 (2)	0.62645 (16)	0.0495 (6)
N5	0.02318 (13)	0.4923 (2)	0.40170 (14)	0.0353 (4)
N6	0.39795 (10)	0.05910 (17)	0.76250 (11)	0.0228 (3)
C1	0.30062 (12)	0.4501 (2)	0.91321 (13)	0.0197 (4)
C2	0.22176 (12)	0.4605 (2)	0.83688 (14)	0.0218 (4)
C3	0.16690 (12)	0.3629 (2)	0.78627 (14)	0.0220 (4)
C4	0.16957 (12)	0.2131 (2)	0.79220 (13)	0.0193 (4)
C5	0.23624 (12)	0.1395 (2)	0.85317 (13)	0.0202 (4)
C6	0.31148 (12)	0.1900 (2)	0.91806 (14)	0.0217 (4)
C7	0.34082 (12)	0.3221 (2)	0.94496 (14)	0.0226 (4)
C8	0.33810 (13)	0.5743 (2)	0.95590 (14)	0.0230 (4)
C9	0.29686 (14)	0.7044 (2)	0.93199 (16)	0.0298 (4)
C10	0.41643 (13)	0.5761 (2)	1.03156 (14)	0.0245 (4)
C11	0.01675 (12)	0.1943 (2)	0.70682 (14)	0.0240 (4)
C12	−0.03851 (14)	0.1407 (2)	0.61387 (16)	0.0319 (5)
C13	0.04469 (13)	−0.0549 (2)	0.62929 (15)	0.0281 (4)
C14	0.10594 (12)	−0.0083 (2)	0.72198 (14)	0.0238 (4)
C15	0.20456 (12)	0.3697 (2)	0.57679 (13)	0.0211 (4)
C16	0.28229 (12)	0.3782 (2)	0.65449 (14)	0.0224 (4)
C17	0.33733 (12)	0.2803 (2)	0.70308 (14)	0.0227 (4)
C18	0.33545 (12)	0.1308 (2)	0.69460 (13)	0.0195 (4)
C19	0.27353 (12)	0.0583 (2)	0.62729 (13)	0.0214 (4)
C20	0.20300 (12)	0.1103 (2)	0.55582 (14)	0.0222 (4)
C21	0.17225 (12)	0.2419 (2)	0.53275 (14)	0.0216 (4)
C22	0.15946 (13)	0.4928 (2)	0.54439 (14)	0.0252 (4)
C23	0.18555 (15)	0.6228 (2)	0.58869 (17)	0.0333 (5)
C24	0.08411 (14)	0.4934 (2)	0.46566 (15)	0.0263 (4)
C25	0.48537 (12)	0.1050 (2)	0.79207 (15)	0.0271 (4)
C26	0.53368 (14)	0.0530 (2)	0.88780 (16)	0.0346 (5)
C27	0.44536 (14)	−0.1381 (2)	0.86626 (16)	0.0316 (5)
C28	0.39156 (13)	−0.0915 (2)	0.76986 (14)	0.0247 (4)
H1	0.2049	0.5533	0.8184	0.026*
H2	0.1187	0.3993	0.7391	0.026*
H3	0.2302	0.0412	0.8508	0.024*
H4	0.3496	0.1188	0.9496	0.026*
H5	0.3953	0.3279	0.9910	0.027*
H6	0.0165	0.2972	0.7064	0.029*
H7	−0.0050	0.1625	0.7539	0.029*
H8	−0.0964	0.1742	0.5994	0.038*
H9	−0.0187	0.1775	0.5665	0.038*
H10	0.0633	−0.0193	0.5807	0.034*
H11	0.0447	−0.1577	0.6265	0.034*
H12	0.0918	−0.0529	0.7714	0.029*
H13	0.1632	−0.0374	0.7291	0.029*

H14	0.2979	0.4704	0.6757	0.027*
H15	0.3853	0.3160	0.7509	0.027*
H16	0.2794	−0.0401	0.6296	0.026*
H17	0.1702	0.0405	0.5156	0.027*
H18	0.1223	0.2486	0.4799	0.026*
H19	0.4877	0.2078	0.7911	0.033*
H20	0.5107	0.0686	0.7492	0.033*
H21	0.5928	0.0827	0.9069	0.042*
H22	0.5103	0.0943	0.9310	0.042*
H23	0.4222	−0.0993	0.9104	0.038*
H24	0.4434	−0.2407	0.8697	0.038*
H25	0.4104	−0.1381	0.7245	0.030*
H26	0.3326	−0.1178	0.7563	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0216 (7)	0.0363 (9)	0.0343 (8)	−0.0061 (6)	0.0024 (6)	−0.0094 (6)
O2	0.0241 (8)	0.0440 (9)	0.0378 (9)	0.0055 (7)	0.0027 (7)	0.0088 (7)
N1	0.0508 (13)	0.0223 (10)	0.0578 (14)	0.0010 (9)	0.0133 (11)	0.0023 (9)
N2	0.0301 (10)	0.0355 (10)	0.0277 (10)	−0.0078 (8)	0.0078 (8)	−0.0061 (8)
N3	0.0162 (7)	0.0202 (8)	0.0228 (8)	0.0005 (6)	0.0026 (6)	−0.0033 (6)
N4	0.0549 (14)	0.0277 (11)	0.0544 (14)	0.0018 (10)	0.0069 (11)	−0.0046 (10)
N5	0.0348 (11)	0.0361 (11)	0.0312 (11)	0.0086 (8)	0.0078 (9)	0.0042 (8)
N6	0.0176 (8)	0.0248 (9)	0.0230 (8)	0.0001 (6)	0.0040 (6)	−0.0012 (6)
C1	0.0232 (9)	0.0190 (9)	0.0192 (9)	−0.0016 (7)	0.0103 (8)	−0.0002 (7)
C2	0.0217 (9)	0.0173 (9)	0.0252 (10)	0.0038 (7)	0.0073 (8)	0.0032 (7)
C3	0.0202 (9)	0.0203 (10)	0.0232 (10)	0.0037 (7)	0.0052 (8)	0.0016 (7)
C4	0.0198 (9)	0.0219 (9)	0.0162 (9)	−0.0015 (7)	0.0066 (7)	−0.0018 (7)
C5	0.0207 (9)	0.0173 (9)	0.0206 (9)	0.0025 (7)	0.0054 (8)	0.0009 (7)
C6	0.0191 (9)	0.0192 (9)	0.0239 (10)	0.0043 (7)	0.0043 (8)	0.0011 (7)
C7	0.0174 (9)	0.0225 (10)	0.0240 (10)	0.0003 (7)	0.0029 (8)	−0.0011 (7)
C8	0.0245 (10)	0.0198 (9)	0.0250 (10)	−0.0026 (7)	0.0095 (8)	0.0015 (8)
C9	0.0315 (11)	0.0214 (10)	0.0326 (12)	−0.0069 (8)	0.0073 (9)	−0.0013 (9)
C10	0.0288 (11)	0.0217 (10)	0.0255 (10)	−0.0049 (8)	0.0131 (9)	−0.0035 (8)
C11	0.0184 (9)	0.0278 (10)	0.0226 (10)	0.0026 (8)	0.0038 (8)	0.0002 (8)
C12	0.0232 (10)	0.0340 (12)	0.0320 (12)	0.0005 (9)	0.0024 (9)	−0.0017 (9)
C13	0.0236 (10)	0.0294 (11)	0.0285 (11)	−0.0044 (8)	0.0063 (9)	−0.0077 (9)
C14	0.0216 (10)	0.0195 (9)	0.0277 (11)	−0.0021 (7)	0.0062 (8)	−0.0031 (8)
C15	0.0230 (10)	0.0221 (10)	0.0197 (10)	−0.0003 (7)	0.0095 (8)	0.0029 (7)
C16	0.0210 (9)	0.0218 (9)	0.0230 (10)	−0.0033 (7)	0.0064 (8)	−0.0014 (7)
C17	0.0199 (9)	0.0250 (10)	0.0203 (10)	−0.0056 (7)	0.0040 (8)	−0.0043 (7)
C18	0.0178 (9)	0.0228 (10)	0.0193 (9)	−0.0006 (7)	0.0083 (7)	−0.0007 (7)
C19	0.0219 (9)	0.0210 (9)	0.0203 (10)	−0.0011 (7)	0.0068 (8)	−0.0020 (7)
C20	0.0210 (9)	0.0230 (10)	0.0209 (10)	−0.0039 (7)	0.0057 (8)	−0.0029 (8)
C21	0.0209 (9)	0.0259 (10)	0.0168 (9)	−0.0006 (7)	0.0055 (7)	0.0007 (7)
C22	0.0272 (10)	0.0230 (10)	0.0239 (11)	0.0007 (8)	0.0078 (9)	0.0019 (8)
C23	0.0347 (12)	0.0246 (11)	0.0363 (13)	0.0051 (9)	0.0079 (10)	0.0033 (9)

C24	0.0326 (11)	0.0245 (10)	0.0243 (11)	0.0055 (8)	0.0134 (9)	0.0028 (8)
C25	0.0185 (9)	0.0309 (11)	0.0294 (11)	−0.0019 (8)	0.0058 (8)	−0.0029 (9)
C26	0.0241 (11)	0.0415 (13)	0.0289 (12)	−0.0008 (9)	−0.0012 (9)	0.0011 (10)
C27	0.0257 (11)	0.0348 (12)	0.0309 (12)	0.0032 (9)	0.0063 (9)	0.0066 (9)
C28	0.0218 (10)	0.0248 (10)	0.0249 (10)	0.0023 (8)	0.0058 (8)	0.0028 (8)

Geometric parameters (Å, °)

O1—C12	1.427 (2)	C19—C20	1.404 (2)
O1—C13	1.423 (2)	C20—C21	1.367 (2)
O2—C26	1.427 (2)	C22—C23	1.419 (2)
O2—C27	1.421 (2)	C22—C24	1.421 (2)
N1—C9	1.149 (2)	C25—C26	1.505 (2)
N2—C10	1.146 (2)	C27—C28	1.517 (2)
N3—C4	1.374 (2)	C2—H1	0.950
N3—C11	1.476 (2)	C3—H2	0.950
N3—C14	1.464 (2)	C5—H3	0.950
N4—C23	1.152 (3)	C6—H4	0.950
N5—C24	1.151 (2)	C7—H5	0.950
N6—C18	1.384 (2)	C11—H6	0.990
N6—C25	1.469 (2)	C11—H7	0.990
N6—C28	1.460 (2)	C12—H8	0.990
C1—C2	1.442 (2)	C12—H9	0.990
C1—C7	1.410 (2)	C13—H10	0.990
C1—C8	1.404 (2)	C13—H11	0.990
C2—C3	1.359 (2)	C14—H12	0.990
C3—C4	1.443 (2)	C14—H13	0.990
C4—C5	1.385 (2)	C16—H14	0.950
C5—C6	1.404 (2)	C17—H15	0.950
C6—C7	1.374 (2)	C19—H16	0.950
C8—C9	1.419 (2)	C20—H17	0.950
C8—C10	1.428 (2)	C21—H18	0.950
C11—C12	1.504 (2)	C25—H19	0.990
C13—C14	1.512 (2)	C25—H20	0.990
C15—C16	1.439 (2)	C26—H21	0.990
C15—C21	1.418 (2)	C26—H22	0.990
C15—C22	1.404 (2)	C27—H23	0.990
C16—C17	1.353 (2)	C27—H24	0.990
C17—C18	1.443 (2)	C28—H25	0.990
C18—C19	1.379 (2)	C28—H26	0.990
O1···C2^{i}	3.552 (2)	H2···C15	3.391
O1···C3^{i}	3.412 (3)	H2···C16	3.533
O1···C20^{ii}	3.223 (2)	H2···C22	3.489
O1···C21^{ii}	3.413 (2)	H2···H7^{xi}	3.226
O2···C6^{iii}	3.334 (2)	H2···H12^{xi}	3.595
O2···C7^{iii}	3.468 (2)	H3···N1^{xii}	2.4144
O2···C16^{iv}	3.575 (3)	H3···C9^{xii}	3.513

O2···C17^{iv}	3.374 (3)	H3···H23	3.366
N1···C5^{v}	3.294 (2)	H3···H26	3.091
N1···C23^{vi}	3.535 (4)	H4···O2^{iii}	2.5876
N1···C27^{v}	3.517 (3)	H4···N1^{xii}	3.288
N2···N2^{vii}	3.568 (3)	H4···N6	3.3768
N2···C7^{vii}	3.496 (3)	H4···C26^{iii}	3.076
N2···C10^{vii}	3.435 (3)	H4···C27	3.480
N2···C25^{vii}	3.478 (2)	H4···H21^{iii}	2.852
N2···C25^{viii}	3.552 (3)	H4···H22	2.895
N2···C26^{vii}	3.573 (3)	H4···H22^{iii}	3.190
N4···C13^{v}	3.495 (3)	H4···H23	2.627
N4···C19^{v}	3.386 (2)	H5···O2^{iii}	2.8773
N4···C28^{v}	3.599 (2)	H5···N2^{vii}	3.048
N5···N5^{ix}	3.457 (3)	H5···C10^{vii}	3.510
N5···C11^{ix}	3.402 (2)	H5···C27^{iii}	3.363
N5···C11^{x}	3.505 (3)	H5···H22	3.348
N5···C12^{ix}	3.554 (3)	H5···H24^{iii}	2.947
N5···C14^{x}	3.596 (3)	H6···O1^{xi}	3.3162
N5···C22^{ix}	3.546 (3)	H6···N5^{ix}	2.563
N5···C24^{ix}	3.252 (3)	H6···C13^{xi}	3.428
N6···C6	3.523 (3)	H6···C14^{xi}	3.308
C1···C19^{viii}	3.551 (3)	H6···C24^{ix}	3.2948
C1···C20^{viii}	3.305 (3)	H6···H7^{xi}	3.585
C2···O1^{xi}	3.552 (2)	H6···H11^{xi}	3.190
C2···C16	3.473 (3)	H6···H12^{xi}	2.481
C3···O1^{xi}	3.412 (3)	H7···N5^{viii}	2.641
C3···C15	3.571 (3)	H7···C13^{xi}	3.482
C3···C16	3.355 (3)	H7···C24^{viii}	3.439
C5···N1^{xii}	3.294 (2)	H7···H1^{i}	3.368
C5···C18	3.496 (3)	H7···H2^{i}	3.226
C5···C21^{viii}	3.565 (3)	H7···H6^{i}	3.585
C6···O2^{iii}	3.334 (2)	H7···H11^{xi}	2.812
C6···N6	3.523 (3)	H7···H12^{xi}	3.073
C6···C21^{viii}	3.547 (3)	H7···H18^{viii}	3.509
C7···O2^{iii}	3.468 (2)	H8···N1^{i}	3.083
C7···N2^{vii}	3.496 (3)	H8···N4^{ix}	3.468
C7···C20^{viii}	3.480 (3)	H8···N5^{ix}	3.448
C8···C19^{viii}	3.502 (3)	H8···C2^{i}	3.386
C9···C27^{v}	3.439 (3)	H8···C9^{i}	3.313
C10···N2^{vii}	3.435 (3)	H8···C23^{ix}	3.390
C10···C19^{viii}	3.563 (3)	H8···C24^{ix}	3.385

C11···N5^{ix}	3.402 (2)	H8···H1^{i}	2.877
C11···N5^{viii}	3.505 (3)	H8···H10^{ii}	3.416
C12···N5^{ix}	3.554 (3)	H8···H12^{xi}	3.297
C13···N4^{xii}	3.495 (3)	H8···H17^{ii}	2.725
C14···N5^{viii}	3.596 (3)	H9···N5^{ix}	3.219
C15···C3	3.571 (3)	H9···C13^{ii}	3.157
C16···O2^{xiii}	3.575 (3)	H9···C21	3.569
C16···C2	3.473 (3)	H9···C23^{ix}	3.560
C16···C3	3.355 (3)	H9···C24^{ix}	3.334
C17···O2^{xiii}	3.374 (3)	H9···H10^{ii}	2.627
C18···C5	3.496 (3)	H9···H11^{ii}	2.892
C19···N4^{xii}	3.386 (2)	H9···H17^{ii}	3.220
C19···C1^{x}	3.551 (3)	H9···H18	3.259
C19···C8^{x}	3.502 (3)	H10···O1^{ii}	2.8887
C19···C10^{x}	3.563 (3)	H10···N4^{xii}	3.347
C20···O1^{ii}	3.223 (2)	H10···C12^{ii}	3.139
C20···C1^{x}	3.305 (3)	H10···C13^{ii}	3.201
C20···C7^{x}	3.480 (3)	H10···C19	3.496
C21···O1^{ii}	3.413 (2)	H10···C20	2.859
C21···C5^{x}	3.565 (3)	H10···C21	3.380
C21···C6^{x}	3.547 (3)	H10···H8^{ii}	3.416
C22···N5^{ix}	3.546 (3)	H10···H9^{ii}	2.627
C23···N1^{xiv}	3.535 (4)	H10···H10^{ii}	2.682
C24···N5^{ix}	3.252 (3)	H10···H11^{ii}	3.523
C24···C24^{ix}	3.444 (3)	H10···H16	3.513
C25···N2^{vii}	3.478 (2)	H10···H17	2.478
C25···N2^{x}	3.552 (3)	H10···H18	3.368
C26···N2^{vii}	3.573 (3)	H11···N4^{xii}	2.993
C27···N1^{xii}	3.517 (3)	H11···N5^{ii}	3.395
C27···C9^{xii}	3.439 (3)	H11···C11^{i}	3.463
C28···N4^{xii}	3.599 (2)	H11···C23^{xii}	3.427
O1···H1^{i}	3.4804	H11···H6^{i}	3.190
O1···H2^{i}	3.2537	H11···H7^{i}	2.812
O1···H6^{i}	3.3162	H11···H9^{ii}	2.892
O1···H10^{ii}	2.8887	H11···H10^{ii}	3.523
O1···H17^{ii}	2.4252	H11···H18^{ii}	2.873
O1···H18^{ii}	2.8250	H12···N1^{xii}	3.237
O2···H4^{iii}	2.5876	H12···N5^{viii}	2.771
O2···H5^{iii}	2.8773	H12···C11^{i}	3.161
O2···H14^{iv}	3.5519	H12···C24^{viii}	3.145
O2···H15^{iv}	3.2048	H12···H2^{i}	3.595

O2···H19^{iv}	3.355	H12···H6^{i}	2.481
O2···H22^{iii}	3.094	H12···H7^{i}	3.073
O2···H23^{iii}	3.443	H12···H8^{i}	3.297
N1···H3^{v}	2.4144	H13···N1^{xii}	3.117
N1···H4^{v}	3.288	H13···N4^{xii}	3.023
N1···H8^{xi}	3.083	H13···C18	3.589
N1···H12^{v}	3.237	H13···C19	3.039
N1···H13^{v}	3.117	H13···C20	3.350
N1···H21^{vii}	3.172	H13···H16	2.955
N1···H23^{v}	2.897	H13···H17	3.471
N1···H24^{v}	3.451	H13···H26	2.891
N1···H26^{v}	3.180	H14···O2^{xiii}	3.5519
N2···H5^{vii}	3.048	H14···C2	3.246
N2···H15^{vii}	2.8729	H14···C3	3.470
N2···H19^{vii}	2.666	H14···C26^{xiii}	3.486
N2···H20^{viii}	2.705	H14···H1	3.291
N2···H21^{vii}	3.488	H14···H20^{xiii}	3.202
N2···H22^{vii}	3.183	H14···H21^{xiii}	2.860
N2···H24^{iii}	3.475	H15···O2^{xiii}	3.2048
N2···H25^{viii}	2.793	H15···N2^{vii}	2.8729
N4···H1	3.446	H15···C6	3.521
N4···H8^{ix}	3.468	H15···C7	3.398
N4···H10^{v}	3.347	H15···H20^{xiii}	3.017
N4···H11^{v}	2.993	H15···H25^{xiii}	3.420
N4···H13^{v}	3.023	H16···N4^{xii}	2.560
N4···H16^{v}	2.560	H16···C8^{x}	3.249
N4···H17^{v}	3.417	H16···C9^{x}	3.584
N4···H25^{v}	3.521	H16···C10^{x}	3.274
N4···H26^{v}	2.795	H16···C23^{xii}	3.572
N5···H2^{ix}	2.8151	H16···H10	3.513
N5···H6^{ix}	2.563	H16···H13	2.955
N5···H7^{x}	2.641	H17···O1^{ii}	2.4252
N5···H8^{ix}	3.448	H17···N4^{xii}	3.417
N5···H9^{ix}	3.219	H17···C1^{x}	3.206
N5···H11^{ii}	3.395	H17···C2^{x}	3.233
N5···H12^{x}	2.771	H17···C8^{x}	3.527
N6···H4	3.3768	H17···C12^{ii}	2.981
C1···H17^{viii}	3.206	H17···C13	3.394
C2···H8^{xi}	3.386	H17···C13^{ii}	3.5550
C2···H14	3.246	H17···H1^{x}	3.473
C2···H17^{viii}	3.233	H17···H8^{ii}	2.725

C3···H14	3.470	H17···H9^{ii}	3.220
C3···H18^{viii}	3.556	H17···H10	2.478
C4···H18^{viii}	3.346	H17···H13	3.471
C5···H18^{viii}	3.437	H18···O1^{ii}	2.8250
C6···H15	3.521	H18···C3^{x}	3.556
C6···H22	3.476	H18···C4^{x}	3.346
C6···H23	3.399	H18···C5^{x}	3.437
C7···H15	3.398	H18···C13^{ii}	3.319
C8···H16^{viii}	3.249	H18···H7^{x}	3.509
C8···H17^{viii}	3.527	H18···H9	3.259
C8···H24^{v}	3.174	H18···H10	3.368
C9···H3^{v}	3.513	H18···H11^{ii}	2.873
C9···H8^{xi}	3.313	H19···O2^{xiii}	3.355
C9···H16^{viii}	3.584	H19···N2^{vii}	2.666
C9···H21^{vii}	3.258	H19···C10^{vii}	3.370
C9···H23^{v}	2.978	H19···C27^{xiii}	3.419
C9···H24^{v}	3.067	H19···C28^{xiii}	3.228
C9···H26^{v}	3.481	H19···H20^{xiii}	3.528
C10···H5^{vii}	3.510	H19···H24^{xiii}	3.193
C10···H16^{viii}	3.274	H19···H25^{xiii}	2.377
C10···H19^{vii}	3.370	H20···N2^{x}	2.705
C10···H20^{viii}	3.477	H20···C10^{x}	3.477
C10···H21^{vii}	3.439	H20···C27^{xiii}	3.578
C10···H22^{vii}	3.381	H20···H14^{iv}	3.202
C10···H24^{v}	3.257	H20···H15^{iv}	3.017
C10···H25^{viii}	3.118	H20···H19^{iv}	3.528
C11···H11^{xi}	3.463	H20···H24^{xiii}	2.922
C11···H12^{xi}	3.161	H20···H25^{xiii}	3.091
C12···H1^{i}	3.505	H21···N1^{vii}	3.172
C12···H10^{ii}	3.139	H21···N2^{vii}	3.488
C12···H17^{ii}	2.981	H21···C9^{vii}	3.258
C13···H6^{i}	3.428	H21···C10^{vii}	3.439
C13···H7^{i}	3.482	H21···C16^{iv}	3.308
C13···H9^{ii}	3.157	H21···H4^{iii}	2.852
C13···H10^{ii}	3.201	H21···H14^{iv}	2.860
C13···H17	3.394	H21···H23^{iii}	2.968
C13···H17^{ii}	3.5550	H21···H25^{xiii}	3.371
C13···H18^{ii}	3.319	H22···O2^{iii}	3.094
C14···H6^{i}	3.308	H22···N2^{vii}	3.183
C15···H2	3.391	H22···C6	3.476
C16···H2	3.533	H22···C10^{vii}	3.381

C16···H21^{xiii}	3.308	H22···C26^{iii}	3.494
C18···H13	3.589	H22···C27^{iii}	3.006
C19···H10	3.496	H22···H4	2.895
C19···H13	3.039	H22···H4^{iii}	3.190
C20···H10	2.859	H22···H5	3.348
C20···H13	3.350	H22···H22^{iii}	2.940
C21···H9	3.569	H22···H23^{iii}	2.319
C21···H10	3.380	H22···H24^{iii}	3.242
C22···H2	3.489	H23···O2^{iii}	3.443
C23···H1	3.550	H23···N1^{xii}	2.897
C23···H8^{ix}	3.390	H23···C6	3.399
C23···H9^{ix}	3.560	H23···C9^{xii}	2.978
C23···H11^{v}	3.427	H23···C26^{iii}	2.996
C23···H16^{v}	3.572	H23···H3	3.366
C24···H6^{ix}	3.2948	H23···H4	2.627
C24···H7^{x}	3.439	H23···H21^{iii}	2.968
C24···H8^{ix}	3.385	H23···H22^{iii}	2.319
C24···H9^{ix}	3.334	H24···N1^{xii}	3.451
C24···H12^{x}	3.145	H24···N2^{iii}	3.475
C25···H24^{xiii}	3.523	H24···C8^{xii}	3.174
C25···H25^{xiii}	3.120	H24···C9^{xii}	3.067
C26···H4^{iii}	3.076	H24···C10^{xii}	3.257
C26···H14^{iv}	3.486	H24···C25^{iv}	3.523
C26···H22^{iii}	3.494	H24···H5^{iii}	2.947
C26···H23^{iii}	2.996	H24···H19^{iv}	3.193
C27···H4	3.480	H24···H20^{iv}	2.922
C27···H5^{iii}	3.363	H24···H22^{iii}	3.242
C27···H19^{iv}	3.419	H25···N2^{x}	2.793
C27···H20^{iv}	3.578	H25···N4^{xii}	3.521
C27···H22^{iii}	3.006	H25···C10^{x}	3.118
C28···H19^{iv}	3.228	H25···C25^{iv}	3.120
H1···O1^{xi}	3.4804	H25···H15^{iv}	3.420
H1···N4	3.446	H25···H19^{iv}	2.377
H1···C12^{xi}	3.505	H25···H20^{iv}	3.091
H1···C23	3.550	H25···H21^{iv}	3.371
H1···H7^{xi}	3.368	H26···N1^{xii}	3.180
H1···H8^{xi}	2.877	H26···N4^{xii}	2.795
H1···H14	3.291	H26···C9^{xii}	3.481
H1···H17^{viii}	3.473	H26···H3	3.091
H2···O1^{xi}	3.2537	H26···H13	2.891
H2···N5^{ix}	2.8151		

C12—O1—C13	109.14 (16)	C5—C6—H4	113.68
C26—O2—C27	109.64 (17)	C7—C6—H4	113.68
C4—N3—C11	120.73 (17)	C1—C7—H5	115.75
C4—N3—C14	120.10 (14)	C6—C7—H5	115.75
C11—N3—C14	111.97 (15)	N3—C11—H6	109.64
C18—N6—C25	120.45 (17)	N3—C11—H7	109.66
C18—N6—C28	119.65 (14)	C12—C11—H6	109.65
C25—N6—C28	111.81 (15)	C12—C11—H7	109.65
C2—C1—C7	122.77 (16)	H6—C11—H7	108.2
C2—C1—C8	117.47 (16)	O1—C12—H8	109.43
C7—C1—C8	119.75 (15)	O1—C12—H9	109.4
C1—C2—C3	132.31 (17)	C11—C12—H8	109.4
C2—C3—C4	130.79 (16)	C11—C12—H9	109.42
N3—C4—C3	116.41 (15)	H8—C12—H9	108.02
N3—C4—C5	119.97 (17)	O1—C13—H10	109.05
C3—C4—C5	123.55 (16)	O1—C13—H11	109.06
C4—C5—C6	129.01 (17)	C14—C13—H10	109.06
C5—C6—C7	132.64 (17)	C14—C13—H11	109.06
C1—C7—C6	128.49 (16)	H10—C13—H11	107.8
C1—C8—C9	121.87 (16)	N3—C14—H12	109.65
C1—C8—C10	122.11 (17)	N3—C14—H13	109.64
C9—C8—C10	115.78 (17)	C13—C14—H12	109.65
N1—C9—C8	179.2 (2)	C13—C14—H13	109.6
N2—C10—C8	179.5 (2)	H12—C14—H13	108.16
N3—C11—C12	110.04 (18)	C15—C16—H14	113.81
O1—C12—C11	111.08 (16)	C17—C16—H14	113.81
O1—C13—C14	112.66 (19)	C16—C17—H15	114.55
N3—C14—C13	110.07 (15)	C18—C17—H15	114.56
C16—C15—C21	122.33 (16)	C18—C19—H16	115.67
C16—C15—C22	118.29 (16)	C20—C19—H16	115.69
C21—C15—C22	119.37 (15)	C19—C20—H17	113.71
C15—C16—C17	132.38 (18)	C21—C20—H17	113.71
C16—C17—C18	130.89 (16)	C15—C21—H18	115.49
N6—C18—C17	116.16 (14)	C20—C21—H18	115.49
N6—C18—C19	119.75 (17)	N6—C25—H19	109.7
C17—C18—C19	123.98 (16)	N6—C25—H20	109.7
C18—C19—C20	128.64 (18)	C26—C25—H19	109.7
C19—C20—C21	132.58 (17)	C26—C25—H20	109.7
C15—C21—C20	129.02 (16)	H19—C25—H20	108.2
C15—C22—C23	122.25 (16)	O2—C26—H21	109.4
C15—C22—C24	121.44 (17)	O2—C26—H22	109.4
C23—C22—C24	116.30 (18)	C25—C26—H21	109.4
N4—C23—C22	178.3 (3)	C25—C26—H22	109.4
N5—C24—C22	179.3 (2)	H21—C26—H22	108.0
N6—C25—C26	109.75 (19)	O2—C27—H23	109.1
O2—C26—C25	111.08 (17)	O2—C27—H24	109.1
O2—C27—C28	112.3 (2)	C28—C27—H23	109.1
N6—C28—C27	109.60 (15)	C28—C27—H24	109.1
C1—C2—H1	113.85	H23—C27—H24	107.9

C3—C2—H1	113.84	N6—C28—H25	109.8
C2—C3—H2	114.61	N6—C28—H26	109.8
C4—C3—H2	114.60	C27—C28—H25	109.7
C4—C5—H3	115.50	C27—C28—H26	109.7
C6—C5—H3	115.49	H25—C28—H26	108.2
C12—O1—C13—C14	59.8 (2)	C8—C1—C7—C6	173.9 (2)
C13—O1—C12—C11	−61.1 (2)	C1—C2—C3—C4	−0.1 (3)
C26—O2—C27—C28	59.1 (2)	C2—C3—C4—N3	179.8 (2)
C27—O2—C26—C25	−60.2 (2)	C2—C3—C4—C5	−3.3 (4)
C4—N3—C11—C12	156.52 (19)	N3—C4—C5—C6	176.3 (2)
C11—N3—C4—C3	−41.0 (3)	C3—C4—C5—C6	−0.6 (4)
C11—N3—C4—C5	141.9 (2)	C4—C5—C6—C7	3.9 (4)
C4—N3—C14—C13	−158.4 (2)	C5—C6—C7—C1	0.5 (4)
C14—N3—C4—C3	171.0 (2)	N3—C11—C12—O1	58.1 (2)
C14—N3—C4—C5	−6.0 (3)	O1—C13—C14—N3	−55.0 (2)
C11—N3—C14—C13	51.1 (2)	C16—C15—C21—C20	3.7 (4)
C14—N3—C11—C12	−53.1 (2)	C21—C15—C16—C17	−2.6 (4)
C18—N6—C25—C26	156.65 (19)	C16—C15—C22—C23	−3.6 (3)
C25—N6—C18—C17	−46.0 (2)	C16—C15—C22—C24	177.3 (2)
C25—N6—C18—C19	137.7 (2)	C22—C15—C16—C17	177.0 (2)
C18—N6—C28—C27	−158.2 (2)	C21—C15—C22—C23	176.0 (2)
C28—N6—C18—C17	167.8 (2)	C21—C15—C22—C24	−3.1 (3)
C28—N6—C18—C19	−8.5 (3)	C22—C15—C21—C20	−175.9 (2)
C25—N6—C28—C27	53.0 (2)	C15—C16—C17—C18	−1.9 (4)
C28—N6—C25—C26	−54.8 (2)	C16—C17—C18—N6	−173.0 (2)
C2—C1—C7—C6	−6.9 (4)	C16—C17—C18—C19	3.1 (4)
C7—C1—C2—C3	6.7 (4)	N6—C18—C19—C20	176.5 (2)
C2—C1—C8—C9	6.4 (3)	C17—C18—C19—C20	0.5 (4)
C2—C1—C8—C10	−179.5 (2)	C18—C19—C20—C21	−2.9 (4)
C8—C1—C2—C3	−174.0 (2)	C19—C20—C21—C15	−0.0 (4)
C7—C1—C8—C9	−174.3 (2)	N6—C25—C26—O2	58.0 (2)
C7—C1—C8—C10	−0.2 (3)	O2—C27—C28—N6	−55.5 (2)

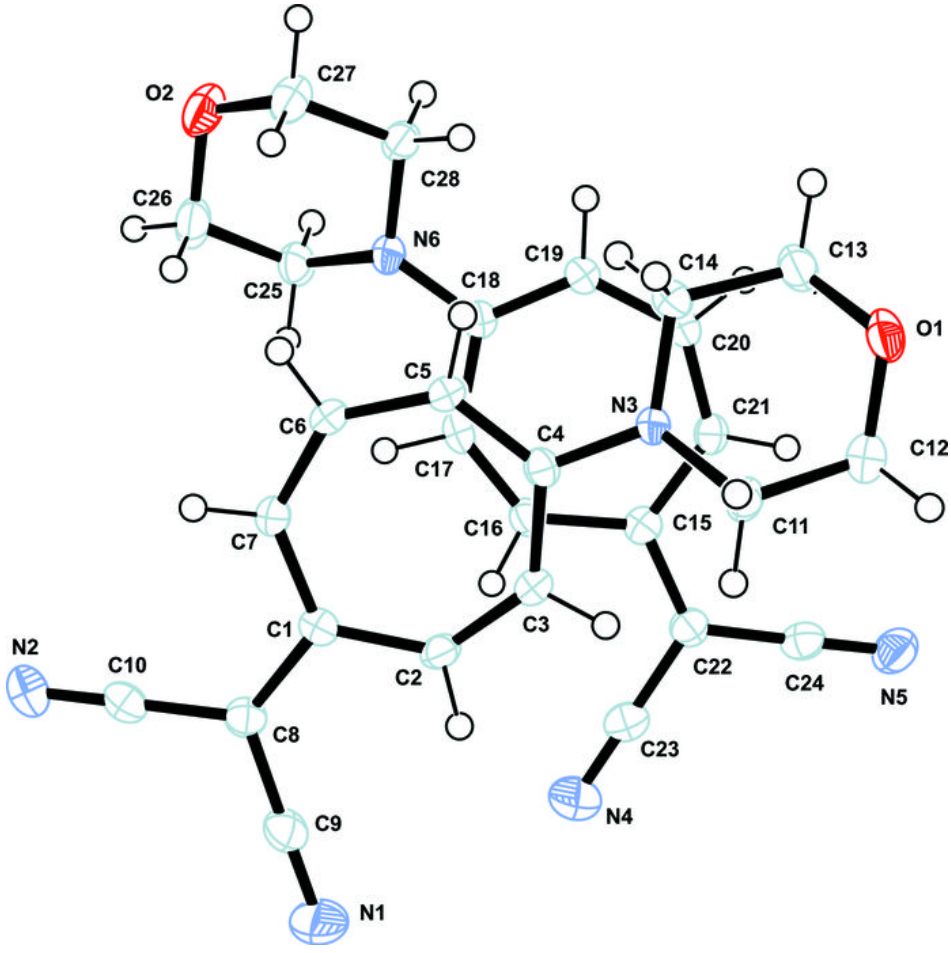
Symmetry codes: (i) −*x*, *y*−1/2, −*z*+3/2; (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*, −*z*+2; (iv) −*x*+1, *y*−1/2, −*z*+3/2; (v) *x*, *y*+1, *z*; (vi) *x*, −*y*+3/2, *z*+1/2; (vii) −*x*+1, −*y*+1, −*z*+2; (viii) *x*, −*y*+1/2, *z*+1/2; (ix) −*x*, −*y*+1, −*z*+1; (x) *x*, −*y*+1/2, *z*−1/2; (xi) −*x*, *y*+1/2, −*z*+3/2; (xii) *x*, *y*−1, *z*; (xiii) −*x*+1, *y*+1/2, −*z*+3/2; (xiv) *x*, −*y*+3/2, *z*−1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H3···N1^{xii}	0.95	2.41	3.294 (2)	154
C6—H4···O2^{iii}	0.95	2.59	3.334 (2)	136
C11—H6···N5^{ix}	0.99	2.56	3.402 (2)	143
C19—H16···N4^{xii}	0.95	2.56	3.386 (2)	146
C20—H17···O1^{ii}	0.95	2.43	3.223 (2)	142

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

Fig. 1


O2
C27
C28
C26
N6
C25
C18
C19
C14
C13
O1
C20
C5
C6
N3
C21
C12
C4
C17
C7
C16
C15
C11
C1
C3
N2
C10
C2
C22
C24
N5
C8
C23
C9
N4
N1