Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-{3-Cyano-5,5-dimethyl-4-[4-(piperidin-1-yl)buta-1,3-dienyl]-2,5-dihydrofuran-2-ylidene}malononitrile

Graeme J. Gainsford,[a]* M. Delower H. Bhuiyan,[a] Andrew J. Kay[a] and Anthony L. Spek[b]

[a]Industrial Research Limited, PO Box 31-310, Lower Hutt, New Zealand, and [b]Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands
Correspondence e-mail: g.gainsford@irl.cri.nz

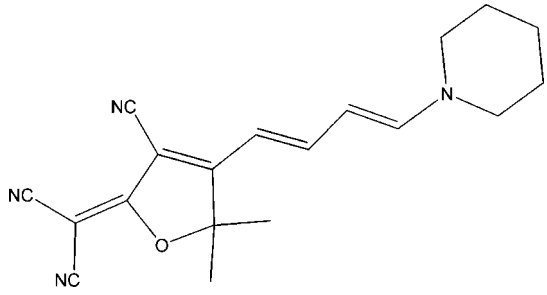
Received 21 December 2007; accepted 7 January 2008

Key indicators: single-crystal X-ray study; T = 119 K; mean σ(C–C) = 0.009 Å; R factor = 0.079; wR factor = 0.203; data-to-parameter ratio = 26.2.

The title compound, $C_{19}H_{20}N_4O$, crystallizes as twinned crystals containing two independent molecules which pack into a three-dimensional matrix *via* several C—H···N(cyano) interactions, with a C···N range of 3.324 (8)–3.568 (8) Å and C—H···N angles in the range 147–166°.

Related literature

For general background, see: Kay *et al.* (2004); Gainsford *et al.* (2007, 2008). For related structures, see: Bock *et al.* (1996); Marder *et al.* (1993); Reck & Dahne (2006); Allen (2002).



Experimental

Crystal data

$C_{19}H_{20}N_4O$
M_r = 320.39
Monoclinic, $P2_1/c$
a = 15.094 (4) Å
b = 18.994 (5) Å
c = 13.332 (4) Å
β = 116.193 (8)°
V = 3429.7 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 119 (2) K
0.35 × 0.25 × 0.09 mm

Data collection

Bruker–Nonius APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.733, T_{max} = 1.0
(expected range = 0.728–0.993)
18712 measured reflections
11128 independent reflections
5766 reflections with $I > 2\sigma(I)$
R_{int} = 0.088

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.203
S = 1.02
11128 reflections
424 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13′—H13′···N2^{i}	0.95	2.49	3.324 (8)	147
C14—H14···N1′	0.95	2.58	3.498 (8)	161
C14′—H14′···N1^{ii}	0.95	2.61	3.484 (8)	153
C16′—H16D···N2^{ii}	0.99	2.55	3.477 (8)	156
C19—H19A···N2^{iii}	0.99	2.60	3.568 (8)	166

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* and *SADABS* (Sheldrick, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

We thank Professor Ward T. Robinson and Dr J. Wikaira of the University of Canterbury for their assistance in data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG3141).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bock, H., Nick, S., Seitz, W., Nather, C. & Bats, J. W. (1996). *Z. Naturforsch. Teil B Chem. Sci.* **51**, 153–171.
Bruker (2005). *APEX2* (Version 2.0-2) and *SAINT* (Version 7.12A). Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gainsford, G. J., Bhuiyan, M. D. H. & Kay, A. J. (2007). *Acta Cryst.* C**63**, o633–o637.
Gainsford, G. J., Bhuiyan, M. D. H. & Kay, A. J. (2008). *Acta Cryst.* C**64**. Submitted.
Kay, A. J., Woolhouse, A. D., Zhao, Y. & Clays, K. (2004). *J. Mater. Chem.* **14**, 1321–1330.
Marder, S. R., Perry, J. W., Tiemann, B. G., Gorman, C. B., Gilmour, S., Biddle, S. L. & Bourhill, G. (1993). *J. Am. Chem. Soc.* **115**, 2524–2526.
Reck, G. & Dahne, L. (2006). Private communication (refcode NEQHAT01). CCDC, Union Road, Cambridge, England.
Sheldrick, G. M. (2003). *SADABS*. Version 2.08. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

 doi:10.1107/S1600536808000548

supporting information

Acta Cryst. (2008). E**64**, o503 [doi:10.1107/S1600536808000548]

2-{3-Cyano-5,5-dimethyl-4-[4-(piperidin-1-yl)buta-1,3-dienyl]-2,5-dihydrofuran-2-ylidene}malononitrile

Graeme J. Gainsford, M. Delower H. Bhuiyan, Andrew J. Kay and Anthony L. Spek

S1. Comment

We have previously reported on the synthesis of a number of high figure of merit chromophores for nonlinear optics (Kay *et al.*, 2004), and the X-ray crystallographic and structural properties of crucial dye precursors used (Gainsford *et al.*, 2007, 2008). As part of our ongoing studies of the structure of these highly polar materials, we report here the crystallographic data of another of the chromophore precursors.

The asymmetric unit contents of the title compound (I) is shown in Figures 1 (unprimed & primed independent molecules hereafter A & B) with hydrogen bond data in Table 1. The structures are almost superimposable with a weighted r.m.s. fit between A & inverted B molecules of 0.61Å (Spek, 2003). The only notable difference in bond lengths concerns those involving atoms C11 & C11′. Minor differences in orientations may be attributed to the packing interactions (Table 1). For example the polyene chains C4, C11—C14 are tilted at 7.1 (5)° & 3.4 (6)° to the "CDFP" 5-membered ring planes (O1, C4—C8) in molecules A & B respectively. Similarly the dicyanomethylene groups (N1,N2, C1—C3) are at 3.5 (4) and 7.7 (4)° to the CDFP ring in A & B, respectively. The piperidin-1-yl rings adopt a chair conformation.

The polyene-C—H···N interactions with adjacent cyano N atoms (C13′, C14, C14′) are commonly observed for these molecules (Gainsford *et al.*, 2008) as is the methylene-C19—H19A interaction with N2′ (entry 5, Table 1). Only once before has it been reported when the adjacent polyene C hydrogen atoms (on C13′ & C14′) are both H bonded to cyano N atoms (NEQHAT01, Reck & Dahne, 2006) from the Cambridge Structural Database (Version 5.29 with November 2007 updates; Allen, 2002). There are few *meta*-*C*(C16′)—*H*···*N*(cyano) interactions reported: two examples exist in the somewhat related compounds YAMXEP (Marder *et al.*, 1993) and ZOSZAI (Bock *et al.*, 1996) with H···N, C—H···N 2.69 & 2.64 Å, 148° & 147°, respectively.

S2. Experimental

To a solution of 5.8 mmole of {4-(4-Acetanilido-*trans*-1,3-butadienyl)-3-cyano-5,5-dimethyl-2(5*H*)-furanylidene} propanedinitrile (compound 11*b*, Kay *et al.*, 2004) in 30 ml of ethanol was added an equimolar quantity of piperidine. The solution was refluxed 1 h, cooled and the product collected by filtration and washed with ethanol. Final crystallization was from ethanol.

S3. Refinement

The measured crystal was a pseudo-merohedral twin. The twin operation was a 2-fold rotation about (1 0 - 2), with twin matrix (-1 0 - 1/0 - 1 0/-0.001 0 1) as indicated by the *PLATON*/TwinRotMat (Spek, 2003) utility that also produced an HKLF5 file for twin refinement with *SHELXL97*. The twin fraction refined to 0.5124 (16). Data was restricted to $2\theta \leq 50°$ (see _refine_special_details for other statistics of the dataset used).

All H atoms bound to carbon were constrained to their expected geometries (C—H 0.98, 0.99 Å). All methyl and tertiary H atoms were refined with U_{iso} 1.5 & 1.2 times respectively that of the U_{eq} of their parent atom. All non-hydrogen atoms were refined with anisotropic thermal parameters.

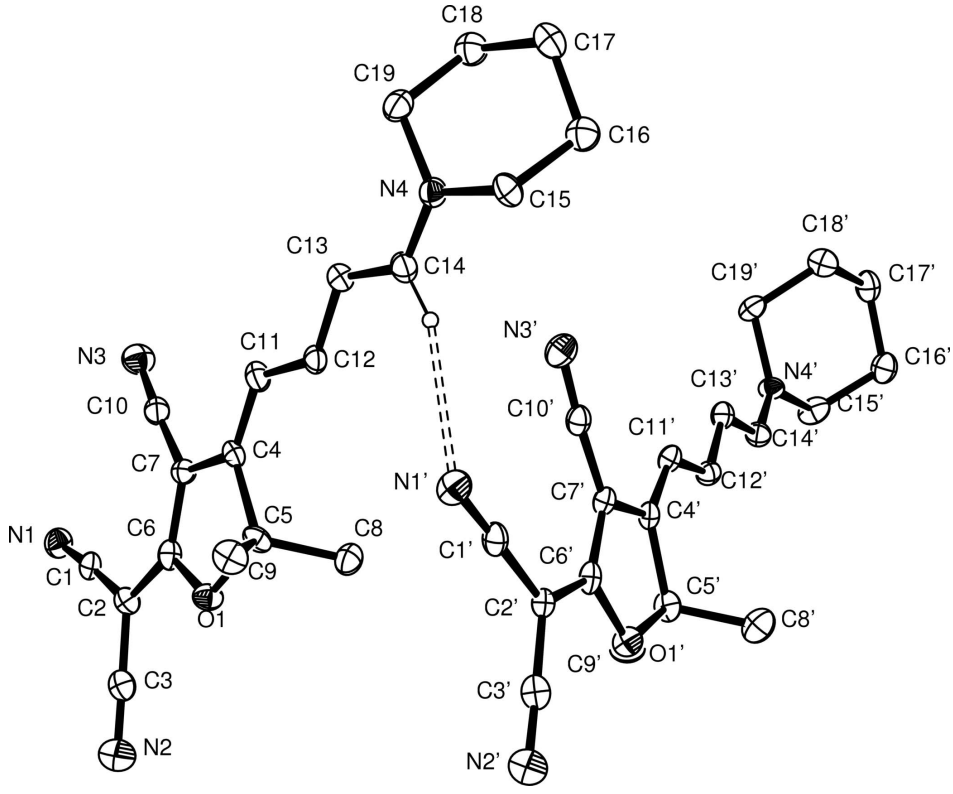


Figure 1

A view of the asymmetic unit; displacement ellipsoids are shown at the 30% probability level. All H atoms except H14 omitted for clarity; dotted line represents a hydrogen bond interaction (Table 1).

2-{3-Cyano-5,5-dimethyl-4-[4-(piperidin-1-yl)buta-1,3-dienyl]- 2,5-dihydrofuran-2-ylidene}malononitrile

Crystal data

$C_{19}H_{20}N_4O$
M_r = 320.39
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 15.094 (4) Å
b = 18.994 (5) Å
c = 13.332 (4) Å
β = 116.193 (8)°
V = 3429.7 (16) Å^3
Z = 8
$F(000)$ = 1360
D_x = 1.241 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4176 reflections
θ = 2.7–30.2°
μ = 0.08 mm^{-1}
T = 119 K
Plate, red
0.35 × 0.25 × 0.09 mm

Data collection

Bruker–Nonius APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
/f and /w scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.733, T_{max} = 1.0
18712 measured reflections

11128 independent reflections
5766 reflections with $I > 2\sigma(I)$
$R_{int} = 0.088$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = -16 \rightarrow 17$
$k = -22 \rightarrow 22$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.203$
$S = 1.02$
11128 reflections
424 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
$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 5.943P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}
Extinction correction: *SHELXL97*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0014 (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. An extinction parameter was refined. Data above 2θ 50° were excluded on the basis of low intensity/error ratio. A total of 477 reflections were either not recorded or affected by the backstop within this θ limit (of 6035 expected). A further 13 reflections (and their Friedel opposites) were omitted from the refinement on the basis of being clearly outliers (with $F_o << F_c$).
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}
O1	0.7393 (3)	0.8303 (2)	0.4515 (3)	0.0343 (10)
N1	0.9567 (4)	0.7590 (3)	0.2970 (5)	0.0422 (15)
N2	0.8344 (4)	0.9570 (3)	0.3440 (4)	0.0489 (15)
N3	0.8724 (4)	0.6091 (3)	0.4162 (5)	0.0473 (15)
N4	0.5250 (3)	0.5399 (2)	0.7252 (4)	0.0326 (12)
C1	0.9063 (4)	0.7849 (3)	0.3282 (5)	0.0356 (16)
C2	0.8470 (4)	0.8229 (3)	0.3707 (5)	0.0321 (14)
C3	0.8397 (4)	0.8966 (4)	0.3552 (5)	0.0362 (15)
C4	0.7276 (4)	0.7135 (3)	0.5029 (5)	0.0289 (15)
C5	0.6857 (4)	0.7869 (3)	0.4989 (5)	0.0311 (15)
C6	0.7962 (4)	0.7888 (3)	0.4209 (5)	0.0305 (14)
C7	0.7908 (4)	0.7191 (3)	0.4492 (5)	0.0283 (14)*
C8	0.5770 (4)	0.7903 (3)	0.4147 (5)	0.0395 (16)
H8A	0.5545	0.8394	0.4055	0.059*
H8B	0.5378	0.7621	0.4420	0.059*
H8C	0.5690	0.7718	0.3426	0.059*

C9	0.7073 (4)	0.8205 (3)	0.6121 (5)	0.0400 (17)
H9A	0.6905	0.8707	0.6015	0.060*
H9B	0.7776	0.8151	0.6630	0.060*
H9C	0.6677	0.7971	0.6440	0.060*
C10	0.8375 (4)	0.6601 (3)	0.4304 (5)	0.0336 (15)
C11	0.7080 (4)	0.6540 (3)	0.5476 (5)	0.0344 (16)
H11	0.7436	0.6126	0.5486	0.041*
C12	0.6398 (4)	0.6493 (3)	0.5920 (4)	0.0297 (14)
H12	0.6046	0.6906	0.5925	0.036*
C13	0.6206 (4)	0.5883 (3)	0.6352 (5)	0.0323 (15)
H13	0.6557	0.5462	0.6387	0.039*
C14	0.5483 (4)	0.5901 (3)	0.6736 (5)	0.0362 (16)
H14	0.5117	0.6327	0.6610	0.043*
C15	0.4434 (4)	0.5482 (3)	0.7556 (5)	0.0389 (16)
H15A	0.4120	0.5948	0.7305	0.047*
H15B	0.4690	0.5458	0.8379	0.047*
C16	0.3684 (4)	0.4911 (3)	0.7020 (5)	0.0438 (17)*
H16A	0.3393	0.4959	0.6197	0.053*
H16B	0.3147	0.4958	0.7250	0.053*
C17	0.4162 (4)	0.4197 (3)	0.7361 (5)	0.0448 (17)
H17A	0.3667	0.3824	0.6982	0.054*
H17B	0.4412	0.4135	0.8178	0.054*
C18	0.5018 (4)	0.4130 (3)	0.7040 (6)	0.0430 (17)*
H18A	0.5355	0.3673	0.7307	0.052*
H18B	0.4756	0.4143	0.6216	0.052*
C19	0.5750 (4)	0.4721 (3)	0.7545 (5)	0.0391 (16)
H19A	0.6084	0.4669	0.8368	0.047*
H19B	0.6259	0.4697	0.7267	0.047*
O1′	0.2394 (3)	0.8340 (2)	0.2535 (3)	0.0340 (10)
N1′	0.4636 (4)	0.7639 (3)	0.6209 (5)	0.0443 (15)
N2′	0.3455 (4)	0.9652 (3)	0.4508 (4)	0.0480 (15)
N3′	0.3582 (4)	0.6149 (3)	0.4407 (4)	0.0443 (15)
N4′	0.0597 (3)	0.5220 (2)	−0.2095 (4)	0.0313 (12)
C1′	0.4113 (4)	0.7928 (3)	0.5417 (5)	0.0347 (15)
C2′	0.3478 (4)	0.8300 (3)	0.4436 (5)	0.0288 (14)
C3′	0.3455 (4)	0.9041 (4)	0.4475 (5)	0.0334 (15)
C4′	0.2259 (4)	0.7152 (3)	0.1981 (5)	0.0269 (14)
C5′	0.1910 (4)	0.7881 (3)	0.1521 (5)	0.0320 (15)
C6′	0.2942 (4)	0.7932 (3)	0.3443 (5)	0.0311 (15)
C7′	0.2857 (4)	0.7224 (3)	0.3153 (5)	0.0295 (15)
C8′	0.0811 (4)	0.7979 (3)	0.1116 (5)	0.0465 (18)
H8′A	0.0653	0.8482	0.1026	0.070*
H8′B	0.0613	0.7778	0.1663	0.070*
H8′C	0.0456	0.7740	0.0396	0.070*
C9′	0.2278 (4)	0.8151 (3)	0.0713 (5)	0.0422 (17)
H9′A	0.2170	0.8660	0.0622	0.063*
H9′B	0.1918	0.7918	−0.0011	0.063*
H9′C	0.2984	0.8050	0.1002	0.063*

C10′	0.3282 (4)	0.6651 (3)	0.3876 (5)	0.0315 (15)
C11′	0.2043 (4)	0.6539 (3)	0.1390 (5)	0.0303 (14)
H11′	0.2299	0.6119	0.1804	0.036*
C12′	0.1479 (4)	0.6464 (3)	0.0229 (5)	0.0296 (14)
H12′	0.1188	0.6872	−0.0203	0.035*
C13′	0.1330 (4)	0.5823 (3)	−0.0312 (5)	0.0318 (15)
H13′	0.1608	0.5407	0.0103	0.038*
C14′	0.0770 (4)	0.5789 (3)	−0.1468 (5)	0.0330 (15)
H14′	0.0486	0.6217	−0.1841	0.040*
C15′	−0.0091 (4)	0.5216 (3)	−0.3290 (5)	0.0425 (18)
H15C	0.0259	0.5050	−0.3723	0.051*
H15D	−0.0324	0.5702	−0.3534	0.051*
C16′	−0.0964 (4)	0.4748 (3)	−0.3530 (5)	0.0440 (17)
H16C	−0.1387	0.4728	−0.4346	0.053*
H16D	−0.1362	0.4944	−0.3172	0.053*
C17′	−0.0618 (4)	0.4010 (3)	−0.3085 (5)	0.0444 (18)
H17C	−0.0300	0.3789	−0.3516	0.053*
H17D	−0.1194	0.3718	−0.3183	0.053*
C18′	0.0114 (4)	0.4035 (3)	−0.1846 (5)	0.0419 (18)
H18C	−0.0224	0.4209	−0.1406	0.050*
H18D	0.0362	0.3554	−0.1583	0.050*
C19′	0.0967 (4)	0.4509 (3)	−0.1666 (5)	0.0374 (15)
H19C	0.1422	0.4535	−0.0858	0.045*
H19D	0.1337	0.4317	−0.2060	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.041 (3)	0.032 (2)	0.036 (3)	0.000 (2)	0.023 (2)	0.003 (2)
N1	0.037 (3)	0.047 (4)	0.047 (4)	0.001 (3)	0.023 (3)	0.002 (3)
N2	0.066 (4)	0.047 (4)	0.037 (4)	−0.002 (3)	0.026 (3)	−0.004 (3)
N3	0.051 (4)	0.047 (4)	0.053 (4)	0.002 (3)	0.031 (3)	0.005 (3)
N4	0.028 (3)	0.036 (3)	0.034 (3)	0.000 (3)	0.014 (2)	0.002 (3)
C1	0.024 (4)	0.039 (4)	0.035 (4)	−0.001 (3)	0.005 (3)	0.002 (3)
C2	0.033 (3)	0.034 (4)	0.032 (4)	−0.004 (3)	0.016 (3)	−0.002 (3)
C3	0.036 (4)	0.044 (4)	0.028 (4)	−0.006 (4)	0.014 (3)	−0.004 (3)
C4	0.024 (3)	0.030 (4)	0.030 (4)	−0.008 (3)	0.010 (3)	−0.004 (3)
C5	0.037 (4)	0.029 (4)	0.035 (4)	−0.007 (3)	0.023 (3)	−0.002 (3)
C6	0.025 (3)	0.043 (4)	0.024 (3)	−0.001 (3)	0.012 (3)	−0.001 (3)
C8	0.033 (4)	0.047 (4)	0.040 (4)	0.005 (3)	0.017 (3)	0.005 (3)
C9	0.053 (4)	0.044 (4)	0.027 (4)	−0.004 (3)	0.021 (3)	−0.010 (3)
C10	0.030 (4)	0.042 (4)	0.027 (3)	−0.003 (3)	0.011 (3)	0.008 (3)
C11	0.028 (3)	0.041 (4)	0.034 (4)	−0.006 (3)	0.014 (3)	0.000 (3)
C12	0.023 (3)	0.035 (4)	0.027 (3)	−0.004 (3)	0.007 (3)	0.000 (3)
C13	0.030 (4)	0.031 (4)	0.035 (4)	−0.003 (3)	0.013 (3)	−0.001 (3)
C14	0.029 (4)	0.039 (4)	0.036 (4)	−0.006 (3)	0.010 (3)	0.002 (3)
C15	0.036 (4)	0.043 (4)	0.045 (4)	−0.007 (3)	0.024 (3)	0.001 (3)
C17	0.043 (4)	0.046 (4)	0.047 (4)	−0.013 (4)	0.020 (3)	−0.005 (4)

C19	0.034 (4)	0.047 (4)	0.039 (4)	0.008 (3)	0.018 (3)	0.010 (3)
O1′	0.038 (3)	0.037 (3)	0.022 (2)	0.001 (2)	0.0086 (19)	−0.005 (2)
N1′	0.045 (4)	0.045 (4)	0.034 (4)	0.003 (3)	0.010 (3)	−0.007 (3)
N2′	0.060 (4)	0.049 (4)	0.037 (4)	−0.004 (3)	0.023 (3)	−0.004 (3)
N3′	0.042 (3)	0.051 (4)	0.035 (3)	0.006 (3)	0.012 (3)	0.003 (3)
N4′	0.035 (3)	0.024 (3)	0.028 (3)	−0.001 (2)	0.008 (2)	−0.003 (2)
C1′	0.027 (4)	0.043 (4)	0.029 (4)	−0.004 (3)	0.007 (3)	−0.008 (3)
C2′	0.022 (3)	0.034 (4)	0.028 (3)	−0.001 (3)	0.009 (3)	−0.001 (3)
C3′	0.035 (4)	0.047 (4)	0.020 (3)	−0.004 (4)	0.014 (3)	−0.006 (3)
C4′	0.020 (3)	0.033 (4)	0.027 (3)	−0.001 (3)	0.009 (2)	−0.005 (3)
C5′	0.031 (4)	0.043 (4)	0.015 (3)	−0.005 (3)	0.004 (2)	−0.006 (3)
C6′	0.020 (3)	0.046 (4)	0.024 (3)	−0.001 (3)	0.008 (3)	−0.003 (3)
C7′	0.021 (3)	0.035 (4)	0.028 (4)	0.002 (3)	0.007 (3)	0.000 (3)
C8′	0.046 (4)	0.055 (4)	0.034 (4)	0.009 (4)	0.013 (3)	−0.001 (3)
C9′	0.059 (4)	0.039 (4)	0.030 (4)	0.002 (3)	0.020 (3)	0.006 (3)
C10′	0.026 (4)	0.037 (4)	0.030 (4)	−0.001 (3)	0.010 (3)	−0.006 (3)
C11′	0.024 (3)	0.036 (4)	0.026 (3)	0.004 (3)	0.007 (3)	0.000 (3)
C12′	0.031 (3)	0.028 (3)	0.031 (4)	0.003 (3)	0.015 (3)	0.000 (3)
C13′	0.026 (3)	0.038 (4)	0.028 (4)	−0.001 (3)	0.009 (3)	−0.002 (3)
C14′	0.034 (4)	0.028 (4)	0.036 (4)	−0.002 (3)	0.015 (3)	−0.006 (3)
C15′	0.051 (4)	0.045 (4)	0.021 (3)	0.008 (4)	0.007 (3)	0.000 (3)
C16′	0.036 (4)	0.054 (5)	0.035 (4)	0.000 (4)	0.009 (3)	−0.004 (3)
C17′	0.034 (4)	0.052 (5)	0.043 (4)	−0.007 (4)	0.014 (3)	−0.010 (3)
C18′	0.050 (4)	0.041 (4)	0.034 (4)	−0.005 (3)	0.017 (3)	−0.005 (3)
C19′	0.037 (4)	0.033 (4)	0.037 (4)	0.010 (3)	0.012 (3)	0.000 (3)

Geometric parameters (Å, º)

O1—C6	1.355 (6)	O1′—C6′	1.365 (6)
O1—C5	1.478 (6)	O1′—C5′	1.500 (6)
N1—C1	1.128 (7)	N1′—C1′	1.141 (7)
N2—C3	1.156 (7)	N2′—C3′	1.161 (7)
N3—C10	1.157 (7)	N3′—C10′	1.154 (7)
N4—C14	1.310 (7)	N4′—C14′	1.320 (6)
N4—C19	1.458 (6)	N4′—C15′	1.468 (6)
N4—C15	1.466 (7)	N4′—C19′	1.477 (6)
C1—C2	1.446 (8)	C1′—C2′	1.422 (8)
C2—C6	1.381 (8)	C2′—C6′	1.396 (7)
C2—C3	1.412 (8)	C2′—C3′	1.410 (8)
C4—C11	1.370 (7)	C4′—C11′	1.362 (7)
C4—C7	1.426 (8)	C4′—C7′	1.425 (7)
C4—C5	1.523 (8)	C4′—C5′	1.511 (7)
C5—C8	1.527 (7)	C5′—C9′	1.503 (8)
C5—C9	1.534 (8)	C5′—C8′	1.512 (7)
C6—C7	1.390 (7)	C6′—C7′	1.390 (7)
C7—C10	1.404 (8)	C7′—C10′	1.407 (8)
C8—H8A	0.9800	C8′—H8′A	0.9800
C8—H8B	0.9800	C8′—H8′B	0.9800

C8—H8C	0.9800	C8′—H8′C	0.9800
C9—H9A	0.9800	C9′—H9′A	0.9800
C9—H9B	0.9800	C9′—H9′B	0.9800
C9—H9C	0.9800	C9′—H9′C	0.9800
C11—C12	1.398 (7)	C11′—C12′	1.405 (8)
C11—H11	0.9500	C11′—H11′	0.9500
C12—C13	1.380 (7)	C12′—C13′	1.382 (7)
C12—H12	0.9500	C12′—H12′	0.9500
C13—C14	1.396 (8)	C13′—C14′	1.395 (7)
C13—H13	0.9500	C13′—H13′	0.9500
C14—H14	0.9500	C14′—H14′	0.9500
C15—C16	1.501 (7)	C15′—C16′	1.503 (8)
C15—H15A	0.9900	C15′—H15C	0.9900
C15—H15B	0.9900	C15′—H15D	0.9900
C16—C17	1.509 (7)	C16′—C17′	1.522 (8)
C16—H16A	0.9900	C16′—H16C	0.9900
C16—H16B	0.9900	C16′—H16D	0.9900
C17—C18	1.536 (8)	C17′—C18′	1.530 (7)
C17—H17A	0.9900	C17′—H17C	0.9900
C17—H17B	0.9900	C17′—H17D	0.9900
C18—C19	1.508 (7)	C18′—C19′	1.501 (7)
C18—H18A	0.9900	C18′—H18C	0.9900
C18—H18B	0.9900	C18′—H18D	0.9900
C19—H19A	0.9900	C19′—H19C	0.9900
C19—H19B	0.9900	C19′—H19D	0.9900

C6—O1—C5	110.2 (4)	C6′—O1′—C5′	109.4 (4)
C14—N4—C19	123.9 (5)	C14′—N4′—C15′	122.6 (5)
C14—N4—C15	121.3 (5)	C14′—N4′—C19′	124.6 (5)
C19—N4—C15	114.8 (5)	C15′—N4′—C19′	112.4 (4)
N1—C1—C2	175.4 (7)	N1′—C1′—C2′	178.8 (7)
C6—C2—C3	120.7 (6)	C6′—C2′—C3′	121.4 (5)
C6—C2—C1	121.9 (5)	C6′—C2′—C1′	119.8 (5)
C3—C2—C1	117.4 (6)	C3′—C2′—C1′	118.7 (5)
N2—C3—C2	179.1 (8)	N2′—C3′—C2′	178.7 (7)
C11—C4—C7	126.9 (6)	C11′—C4′—C7′	126.2 (5)
C11—C4—C5	127.2 (5)	C11′—C4′—C5′	126.6 (5)
C7—C4—C5	105.9 (5)	C7′—C4′—C5′	107.2 (5)
O1—C5—C4	103.2 (4)	O1′—C5′—C9′	105.1 (4)
O1—C5—C8	106.0 (5)	O1′—C5′—C4′	102.9 (4)
C4—C5—C8	111.1 (5)	C9′—C5′—C4′	115.5 (5)
O1—C5—C9	105.7 (4)	O1′—C5′—C8′	105.8 (5)
C4—C5—C9	116.2 (5)	C9′—C5′—C8′	113.3 (5)
C8—C5—C9	113.5 (5)	C4′—C5′—C8′	112.8 (5)
O1—C6—C2	115.8 (5)	O1′—C6′—C7′	110.9 (5)
O1—C6—C7	110.5 (5)	O1′—C6′—C2′	115.4 (5)
C2—C6—C7	133.7 (6)	C7′—C6′—C2′	133.7 (6)
C6—C7—C10	128.1 (5)	C6′—C7′—C10′	126.9 (5)

C6—C7—C4	109.9 (5)	C6′—C7′—C4′	109.5 (5)
C10—C7—C4	122.0 (5)	C10′—C7′—C4′	123.6 (5)
C5—C8—H8A	109.5	C5′—C8′—H8′A	109.5
C5—C8—H8B	109.5	C5′—C8′—H8′B	109.5
H8A—C8—H8B	109.5	H8′A—C8′—H8′B	109.5
C5—C8—H8C	109.5	C5′—C8′—H8′C	109.5
H8A—C8—H8C	109.5	H8′A—C8′—H8′C	109.5
H8B—C8—H8C	109.5	H8′B—C8′—H8′C	109.5
C5—C9—H9A	109.5	C5′—C9′—H9′A	109.5
C5—C9—H9B	109.5	C5′—C9′—H9′B	109.5
H9A—C9—H9B	109.5	H9′A—C9′—H9′B	109.5
C5—C9—H9C	109.5	C5′—C9′—H9′C	109.5
H9A—C9—H9C	109.5	H9′A—C9′—H9′C	109.5
H9B—C9—H9C	109.5	H9′B—C9′—H9′C	109.5
N3—C10—C7	176.2 (6)	N3′—C10′—C7′	174.9 (6)
C4—C11—C12	125.3 (6)	C4′—C11′—C12′	126.7 (6)
C4—C11—H11	117.3	C4′—C11′—H11′	116.7
C12—C11—H11	117.3	C12′—C11′—H11′	116.7
C13—C12—C11	124.1 (6)	C13′—C12′—C11′	122.9 (6)
C13—C12—H12	118.0	C13′—C12′—H12′	118.5
C11—C12—H12	118.0	C11′—C12′—H12′	118.5
C12—C13—C14	118.4 (6)	C12′—C13′—C14′	119.8 (6)
C12—C13—H13	120.8	C12′—C13′—H13′	120.1
C14—C13—H13	120.8	C14′—C13′—H13′	120.1
N4—C14—C13	127.5 (6)	N4′—C14′—C13′	126.3 (6)
N4—C14—H14	116.3	N4′—C14′—H14′	116.9
C13—C14—H14	116.3	C13′—C14′—H14′	116.9
N4—C15—C16	109.8 (5)	N4′—C15′—C16′	111.6 (5)
N4—C15—H15A	109.7	N4′—C15′—H15C	109.3
C16—C15—H15A	109.7	C16′—C15′—H15C	109.3
N4—C15—H15B	109.7	N4′—C15′—H15D	109.3
C16—C15—H15B	109.7	C16′—C15′—H15D	109.3
H15A—C15—H15B	108.2	H15C—C15′—H15D	108.0
C15—C16—C17	110.3 (5)	C15′—C16′—C17′	110.2 (5)
C15—C16—H16A	109.6	C15′—C16′—H16C	109.6
C17—C16—H16A	109.6	C17′—C16′—H16C	109.6
C15—C16—H16B	109.6	C15′—C16′—H16D	109.6
C17—C16—H16B	109.6	C17′—C16′—H16D	109.6
H16A—C16—H16B	108.1	H16C—C16′—H16D	108.1
C16—C17—C18	109.8 (5)	C16′—C17′—C18′	110.6 (5)
C16—C17—H17A	109.7	C16′—C17′—H17C	109.5
C18—C17—H17A	109.7	C18′—C17′—H17C	109.5
C16—C17—H17B	109.7	C16′—C17′—H17D	109.5
C18—C17—H17B	109.7	C18′—C17′—H17D	109.5
H17A—C17—H17B	108.2	H17C—C17′—H17D	108.1
C19—C18—C17	110.8 (5)	C19′—C18′—C17′	110.6 (5)
C19—C18—H18A	109.5	C19′—C18′—H18C	109.5
C17—C18—H18A	109.5	C17′—C18′—H18C	109.5

C19—C18—H18B	109.5	C19′—C18′—H18D	109.5
C17—C18—H18B	109.5	C17′—C18′—H18D	109.5
H18A—C18—H18B	108.1	H18C—C18′—H18D	108.1
N4—C19—C18	110.3 (5)	N4′—C19′—C18′	109.6 (4)
N4—C19—H19A	109.6	N4′—C19′—H19C	109.8
C18—C19—H19A	109.6	C18′—C19′—H19C	109.8
N4—C19—H19B	109.6	N4′—C19′—H19D	109.8
C18—C19—H19B	109.6	C18′—C19′—H19D	109.8
H19A—C19—H19B	108.1	H19C—C19′—H19D	108.2

C6—O1—C5—C4	5.6 (5)	C6′—O1′—C5′—C9′	−121.2 (5)
C6—O1—C5—C8	−111.2 (5)	C6′—O1′—C5′—C4′	0.1 (6)
C6—O1—C5—C9	128.1 (5)	C6′—O1′—C5′—C8′	118.7 (5)
C11—C4—C5—O1	174.9 (5)	C11′—C4′—C5′—O1′	−178.6 (5)
C7—C4—C5—O1	−5.7 (6)	C7′—C4′—C5′—O1′	1.6 (6)
C11—C4—C5—C8	−71.9 (7)	C11′—C4′—C5′—C9′	−64.7 (8)
C7—C4—C5—C8	107.4 (5)	C7′—C4′—C5′—C9′	115.5 (5)
C11—C4—C5—C9	59.8 (7)	C11′—C4′—C5′—C8′	67.9 (8)
C7—C4—C5—C9	−120.8 (5)	C7′—C4′—C5′—C8′	−112.0 (5)
C5—O1—C6—C2	177.3 (5)	C5′—O1′—C6′—C7′	−1.7 (7)
C5—O1—C6—C7	−3.3 (6)	C5′—O1′—C6′—C2′	176.7 (5)
C3—C2—C6—O1	0.9 (8)	C3′—C2′—C6′—O1′	−0.9 (8)
C1—C2—C6—O1	−176.6 (5)	C1′—C2′—C6′—O1′	−176.6 (5)
C3—C2—C6—C7	−178.2 (6)	C3′—C2′—C6′—C7′	177.0 (7)
C1—C2—C6—C7	4.3 (11)	C1′—C2′—C6′—C7′	1.3 (11)
O1—C6—C7—C10	179.1 (5)	O1′—C6′—C7′—C10′	−177.2 (5)
C2—C6—C7—C10	−1.7 (11)	C2′—C6′—C7′—C10′	4.8 (11)
O1—C6—C7—C4	−0.6 (7)	O1′—C6′—C7′—C4′	2.8 (7)
C2—C6—C7—C4	178.5 (6)	C2′—C6′—C7′—C4′	−175.2 (6)
C11—C4—C7—C6	−176.5 (5)	C11′—C4′—C7′—C6′	177.5 (6)
C5—C4—C7—C6	4.1 (7)	C5′—C4′—C7′—C6′	−2.7 (6)
C11—C4—C7—C10	3.8 (10)	C11′—C4′—C7′—C10′	−2.5 (10)
C5—C4—C7—C10	−175.6 (5)	C5′—C4′—C7′—C10′	177.3 (5)
C7—C4—C11—C12	−174.8 (6)	C7′—C4′—C11′—C12′	−177.9 (6)
C5—C4—C11—C12	4.4 (10)	C5′—C4′—C11′—C12′	2.3 (10)
C4—C11—C12—C13	179.1 (5)	C4′—C11′—C12′—C13′	177.1 (6)
C11—C12—C13—C14	−177.9 (6)	C11′—C12′—C13′—C14′	−179.4 (5)
C19—N4—C14—C13	1.9 (10)	C15′—N4′—C14′—C13′	173.0 (6)
C15—N4—C14—C13	−176.8 (6)	C19′—N4′—C14′—C13′	0.6 (10)
C12—C13—C14—N4	−174.1 (5)	C12′—C13′—C14′—N4′	177.6 (5)
C14—N4—C15—C16	121.5 (6)	C14′—N4′—C15′—C16′	−115.0 (6)
C19—N4—C15—C16	−57.3 (6)	C19′—N4′—C15′—C16′	58.3 (7)
N4—C15—C16—C17	57.2 (7)	N4′—C15′—C16′—C17′	−55.0 (7)
C15—C16—C17—C18	−57.4 (7)	C15′—C16′—C17′—C18′	53.9 (7)
C16—C17—C18—C19	55.7 (7)	C16′—C17′—C18′—C19′	−55.8 (7)
C14—N4—C19—C18	−123.2 (6)	C14′—N4′—C19′—C18′	114.3 (6)
C15—N4—C19—C18	55.5 (7)	C15′—N4′—C19′—C18′	−58.9 (7)
C17—C18—C19—N4	−53.5 (7)	C17′—C18′—C19′—N4′	57.2 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13′—H13′···N2^{i}	0.95	2.49	3.324 (8)	147
C14—H14···N1′	0.95	2.58	3.498 (8)	161
C14′—H14′···N1^{ii}	0.95	2.61	3.484 (8)	153
C16′—H16*D*···N2^{ii}	0.99	2.55	3.477 (8)	156
C19—H19*A*···N2′^{iii}	0.99	2.60	3.568 (8)	166

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