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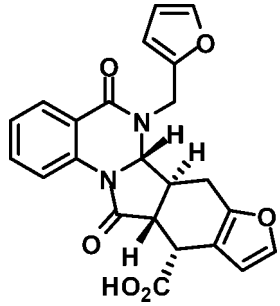
# (6a*S**,6b*S**,11*R**,11a*R**)-6-(2-Furylmethyl)-5,12-dioxo-5,6,6a,6b,7,11,-11a,12-octahydrofuro[3′,2′:5,6]isoindolo[2,1-*a*]quinazoline-11-carboxylic acid



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Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.060; $wR$ factor = 0.160; data-to-parameter ratio = 18.9.

The title compound, $C_{23}H_{18}N_2O_6$, is the product of an intramolecular thermal cycloaddition within 1-malein-2-[(*E*)-2-(2-furyl)vinyl]-4-oxo-3,4-dihydroquinazoline. The molecule comprises a previously unknown fused pentacyclic system containing two five-membered rings (2-pyrrolidinone and furan) and three six-membered rings (benzene, 2,3-dihydro-4-pyrimidinone and dihydrocyclohexane). The central five-membered pyrrolidinone ring has the usual envelope conformation. The six-membered dihydropyrimidinone and dihydrocyclohexane rings adopt a half-boat and a half-chair conformation, respectively. The dihedral angle between the planes of the terminal benzene and furan rings is 45.99 (7)°. In the crystal, O—H···O hydrogen bonds link the molecules into centrosymmetric dimers. Weak C—H···O hydrogen bonds consolidate further the crystal packing, which exhibits $\pi$–$\pi$ interactions, with a short distance of 3.556 (3) Å between the centroids of benzene rings of neighbouring molecules.

## Related literature

For 2-vinylfurans as dienes, see: Kotsuki *et al.* (1981); Keil *et al.* (1990); Kusurkar & Bhosale (1990); Anisimova *et al.* (2006). For the intramolecular Diels–Alder reaction for furan (IMDAF), see: Vogel *et al.* (1999); Zubkov *et al.* (2005, 2009, 2010). For related compounds, see: Chou & Tsai (1992); Chou *et al.* (1997); Sun & Murray (1999); Ohno *et al.* (2005); Patre *et al.* (2007).

## Experimental

### Crystal data

$C_{23}H_{18}N_2O_6$
$M_r$ = 418.39
Monoclinic, $P2_1/n$
$a$ = 8.2364 (5) Å
$b$ = 16.9882 (10) Å
$c$ = 13.1568 (8) Å
$\beta$ = 99.102 (1)°
$V$ = 1817.74 (19) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 100 K
0.30 × 0.20 × 0.18 mm

### Data collection

Bruker SMART 1K CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{min}$ = 0.967, $T_{max}$ = 0.980
21001 measured reflections
5293 independent reflections
4139 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.160
$S$ = 1.00
5293 reflections
280 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H4O···O1$^{i}$ | 0.93 | 1.75 | 2.671 (2) | 174 |
| C2—H2···O3$^{ii}$ | 0.95 | 2.42 | 3.326 (2) | 160 |
| C3—H3···O3$^{iii}$ | 0.95 | 2.56 | 3.384 (2) | 146 |
| C7—H7*B*···O4$^{iv}$ | 0.99 | 2.54 | 3.455 (2) | 155 |
| C11*A*—H11*A*···O1$^{v}$ | 1.00 | 2.38 | 3.325 (2) | 157 |

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: CV5175).

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# supporting information

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# (6a*S**,6b*S**,11*R**,11a*R**)-6-(2-Furylmethyl)-5,12-dioxo-5,6,6a,6b,7,11,11a,12-octahydrofuro[3′,2′:5,6]isoindolo[2,1-*a*]quinazoline-11-carboxylic acid

**Mykola D. Obushak, Yuri I. Horak, Vladimir P. Zaytsev, Ekaterina L. Motorygina, Fedor I. Zubkov and Victor N. Khrustalev**

## S1. Comment

Currently, there are only a few reports concerning the [4 + 2] cycloaddition of 2-vinylfurans with dienophiles (Kotsuki *et al.*, 1981; Keil *et al.*, 1990; Kusurkar & Bhosale, 1990; Anisimova *et al.*, 2006). In the present work, within the scope of our investigations on the intramolecular Diels-Alder reaction of furan (IMDAF) (Vogel *et al.*, 1999; Zubkov *et al.*, 2005, 2009, 2010), we demonstrate the possibility of intramolecular thermal cycloaddition within 1-malein-2-[(*E*)-2-(2-furyl)vinyl]-4-oxo-3,4-dihydroquinazoline. The latter is an intermediate of a reaction of 2-[(*E*)-2-(2-furyl)vinyl]-2,3-dihydroquinazolin-4-one with maleic anhydride (Figure 1). The reaction product contains a previously unknown pentacycle bearing four asymmetrical centers. The main structural fragments of the new pentacycle are quinazoline and furo[2,3-*f*]isoindole (Chou & Tsai, 1992; Chou *et al.*, 1997; Sun & Murray, 1999; Ohno *et al.*, 2005; Patre *et al.*, 2007). The structure of the final product - 6-(2-furylmethyl)-5,12-dioxo-5,6,6a,6 b,7,11,11*a*,12-octahydrofuro[3′,2′:5,6]isoindolo[2,1-*a*]quinazoline-11-carboxylic acid, $C_{23}H_{18}N_2O_6$, (I) was unambiguously established by X-ray diffraction study.

Molecule of (I) comprises a fused pentacyclic system containing two five-membered rings (2-pyrrolidinone and furan) and three six-membered rings (benzene, 2,3-dihydro-4-pyrimidinone and dihydrocyclohexane) (Figure 2). The central five-membered pyrrolidinone ring has usual *envelope* conformation (the C6B carbon atom is out of the plane through the other atoms of the ring by 0.477 (2) Å)), and the central six-membered dihydropyrimidinone and dihydrocyclohexane rings adopt the nonsymmetrical *half-boat* (the N6 nitrogen and C6A carbon atoms are out of the plane through the other atoms of the ring by 0.265 (3) and 0.626 (3) Å, respectively) and nonsymmetrical *half-chair* (the C6B and C11A carbon atoms are out of the plane through the other atoms of the ring by -0.555 (3) and 0.281 (3) Å, respectively) conformations, respectively. The dihedral angle between the planes of the end-cutting benzene and furan rings is 45.99 (7)°.

The furylmethyl ligand and carboxylic acid substituent at the C11 atom arrange from different sides of the main pentacyclic framework. Apparently, such disposition is explained by the fact that, in the crystal, the molecules of (I) form the centrosymmerical dimers through the intermolecular O4—H4O···O1$^{i}$ hydrogen bonding interactions (Table 1). Furthermore, due to the steric reasons within the dimers, the nitrogen N6 atom adopts a trigonal-pyramidal geometry (sum of the bond angles is 357.5°), while the nitrogen N13 atom has a trigonal-planar geometry (sum of the bond angles is 360.0°). Weak intermolecular C—H···O hydrogen bonds consolidate further the crystal packing, which exhibits $\pi$—$\pi$ interactions with the short distance of 3.556 (3) Å between the centroids of benzene rings from the neighbouring molecules [Cg···Cg$^{i}$; symmetry code (i) 1 - *x*, 1 - *y*, -*z*].

The molecule of (I) possesses four asymmetric centers at the C6A, C6B, C11 and C11A carbon atoms and can have potentially numerous diastereomers. The crystal of (I) is racemic and consists of enantiomeric pairs with the following relative configuration of the centers: ***rac***-6a***S****,6 b***S****,11***R****,11*a***R***.

## S2. Experimental

A mixture of the initial 3-(2-furylmethyl)-2-[(*E*)-2-(2-furyl)vinyl]-2,3-dihydroquinazolin-4(1*H*)-one (0.5 g, 1.6 mmol) and maleic anhydride (0.17 g, 1.7 mmol) was refluxed for 8 h in toluene (10 ml). At the end of the reaction the resulting mixture was cooled, and formed brown precipitate was filtered off, washed with benzene(2x10 ml) and ether (2x10 ml). Further crystallization from an ethanol-DMF mixture gives the corresponding acid (0.5 g, 1.2 mmol) as orange prism. Yield is 77%. The single-crystal of the product was obtained by slow crystallization from an ethanol-ethyl acetate mixture. *M*.p. = 499–500 K. IR (KBr), $\nu$/cm$^{-1}$: 1630, 1727 (NCO, $CO_2H$). $^1$H NMR (600 MHz, DMSO-$d_6$, 293 K): $\delta$ = 12.7 (br.s,1*H*, $CO_2H$), 8.24 (d, 1H,H4, $J_{4,3}$ = 7.8), 7.93 (d, 1H, H1, $J_{1,2}$ = 7.8), 7.60 (t, 1H, H2, $J_{1,2}$ = $J_{2,3}$ = 7.8), 7.57 (dd, 1H, H5′, $J_{4',5'}$= 1.8, $J_{3',5'}$ = 0.8), 7.54 (d, 1H, H9, $J_{9,10}$ = 0.9), 7.25 (t, 1H, H3, $J_{2,3}$ = $J_{3,4}$ = 7.8), 6.44 (d, 1H, H10, $J_{9,10}$= 0.9), 6.38 (dd, 1H, H4′, $J_{3',4'}$ = 3.2, $J_{4',5'}$ = 1.8), 6.32 (d, 1H, H3′, $J_{3',4'}$ = 3.2), 5.53 (d, 1H, H6A, $J_{6\,A,6B}$ = 8.2), 5.01 (d, 1H, $NCH_2$, $J_{H14A,H14B}$ = 16.9), 4.73 (d,1*H*, $NCH_2$, $J_{H14A,H14B}$ = 16.9), 3.83 (d, 1H, $H_{11}$, $J_{11,11\,A}$ = 5.0), 3.29 (m, 1H, H6B), 3.16 (dd, 1H, H7A, $J_{7\,A,7B}$ = 15.6, $J_{7\,A,6B}$ = 4.6), 3.04 (dd, 1H, H11A, $J_{11\,A,6B}$ = 12.4, $J_{11,11\,A}$ = 5.0), 2.70 (dd, 1H, H7B, $J_{7\,A,7B}$ = 15.6, $J_{7B,6B}$ = 11.5). Mass spectrum (EI—MS, 70 eV) *m/z*($I_r$, (%)): 418 [$M^+$] (100), 374 (22), 322 (18), 276 (41), 236 (13), 227 (16), 185 (12), 147 (13), 119 (14), 91 (37), 81 (75), 53 (12). Anal. Calcd. for $C_{23}H_{18}N_2O_6$: C, 66.02; H, 4.34; N, 6.70. Found: C, 66.12; H, 4.04; N, 6.83.

## S3. Refinement

The hydroxyl hydrogen atom was localized in the difference-Fourier map and included in the refinement with fixed positional and isotropic displacement parameters [$U_{iso}$(H) = 1.5$U_{eq}$(O)]. The other hydrogen atoms were placed in calculated positions with C–H = 0.95–1.00 Å and refined in the riding model with fixed isotropic displacement parameters [$U_{iso}$(H) = 1.2$U_{eq}$(C)].

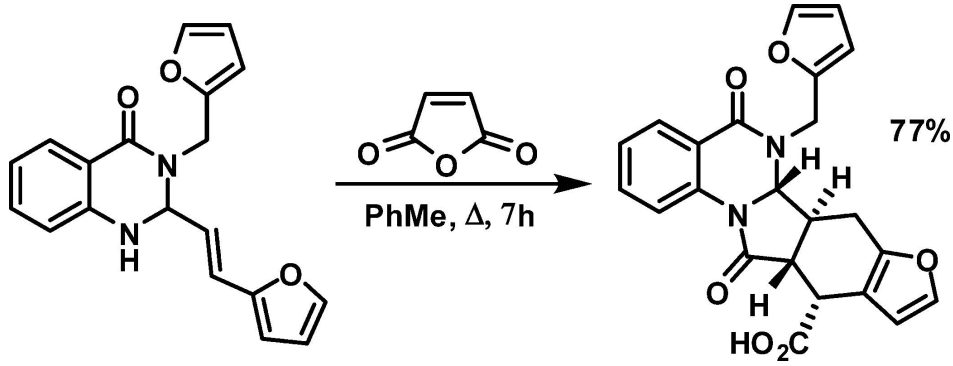


**Figure 1**

Reaction of 2-[(*E*)-2-(2-furyl)vinyl]-2,3-dihydroquinazolin-4-one with maleic anhydride.

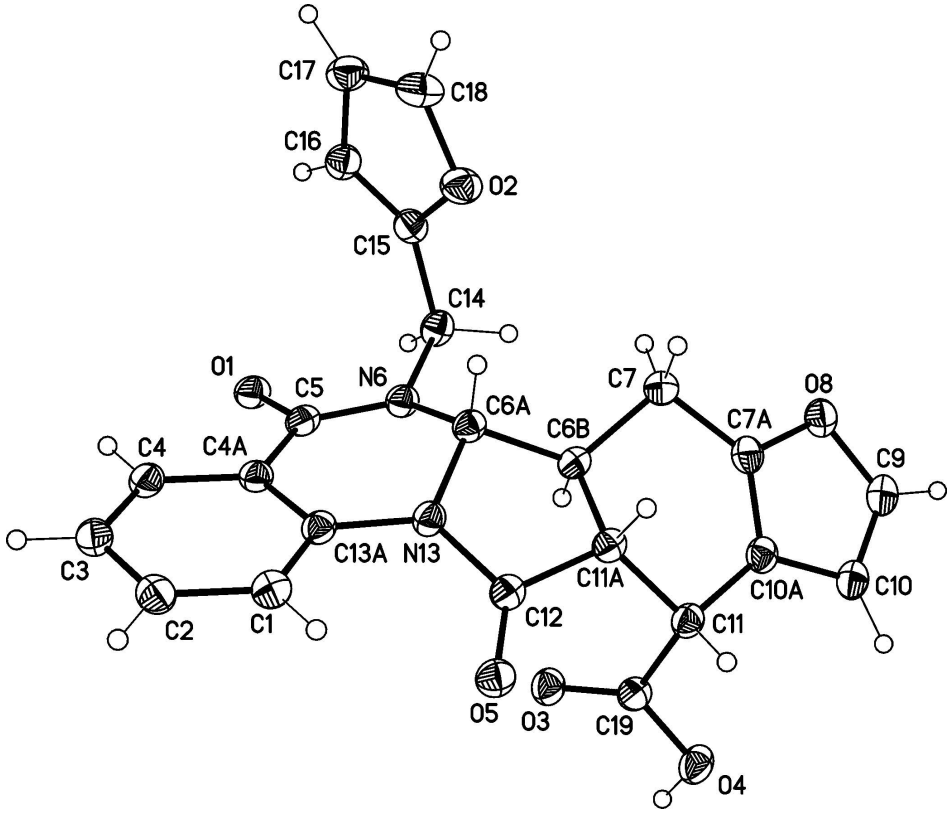


**Figure 2**

Molecular structure of (I). Displacement ellipsoids are shown at the 50% probability level.

**(6a*S**,6b*S**,11*R**,11a*R**)-6-(2-Furylmethyl)-5,12- dioxo-5,6,6a,6b,7,11,11a,12-octahydrofuro[3′,2′:5,6]isoindolo[2,1- *a*]quinazoline-11-carboxylic acid**

*Crystal data*

$C_{23}H_{18}N_2O_6$
$M_r = 418.39$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.2364$ (5) Å
$b = 16.9882$ (10) Å
$c = 13.1568$ (8) Å
$\beta = 99.102$ (1)°
$V = 1817.74$ (19) Å$^3$
$Z = 4$
$F(000) = 872$
$D_x = 1.529$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6904 reflections
$\theta = 2.4$–30.0°
$\mu = 0.11$ mm$^{-1}$
$T = 100$ K
Prism, orange
0.30 × 0.20 × 0.18 mm

*Data collection*

Bruker SMART 1K CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{min} = 0.967$, $T_{max} = 0.980$
21001 measured reflections
5293 independent reflections
4139 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 30.0°$, $\theta_{min} = 2.0°$
$h = -11 \rightarrow 11$
$k = -23 \rightarrow 23$
$l = -18 \rightarrow 18$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.160$
$S = 1.00$
5293 reflections
280 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 1.9P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.45$ e Å$^{-3}$
$\Delta\rho_{min} = -0.34$ e Å$^{-3}$

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.77116 (15) | 0.37826 (7) | 0.24161 (9) | 0.0235 (3) |
| O2 | 0.73217 (17) | 0.53678 (7) | 0.48118 (10) | 0.0286 (3) |
| O3 | 0.47571 (15) | 0.62107 (7) | −0.02374 (9) | 0.0245 (3) |
| O4 | 0.31904 (17) | 0.72270 (7) | −0.08729 (10) | 0.0284 (3) |
| H4O | 0.2827 | 0.6861 | −0.1383 | 0.043* |
| O5 | 0.82503 (15) | 0.69632 (7) | 0.03174 (9) | 0.0237 (3) |
| C1 | 1.0826 (2) | 0.57749 (10) | 0.10046 (13) | 0.0221 (3) |
| H1 | 1.0901 | 0.6300 | 0.0769 | 0.027* |
| C2 | 1.2040 (2) | 0.52240 (10) | 0.08887 (13) | 0.0237 (3) |
| H2 | 1.2942 | 0.5378 | 0.0565 | 0.028* |
| C3 | 1.1958 (2) | 0.44550 (10) | 0.12353 (13) | 0.0248 (3) |
| H3 | 1.2811 | 0.4092 | 0.1165 | 0.030* |
| C4 | 1.0627 (2) | 0.42198 (10) | 0.16832 (13) | 0.0231 (3) |
| H4 | 1.0557 | 0.3693 | 0.1914 | 0.028* |
| C4A | 0.93842 (19) | 0.47584 (9) | 0.17967 (12) | 0.0197 (3) |
| C5 | 0.7966 (2) | 0.44955 (9) | 0.22637 (12) | 0.0203 (3) |
| N6 | 0.69629 (17) | 0.50537 (8) | 0.25528 (10) | 0.0198 (3) |
| C6A | 0.72967 (19) | 0.59004 (9) | 0.24741 (12) | 0.0188 (3) |
| H6A | 0.7925 | 0.6104 | 0.3135 | 0.023* |
| C6B | 0.57091 (19) | 0.63798 (9) | 0.21574 (12) | 0.0192 (3) |
| H6B | 0.4918 | 0.6042 | 0.1694 | 0.023* |
| C7 | 0.4786 (2) | 0.67373 (10) | 0.29657 (13) | 0.0224 (3) |
| H7A | 0.4284 | 0.6322 | 0.3343 | 0.027* |
| H7B | 0.5529 | 0.7060 | 0.3465 | 0.027* |
| C7A | 0.3497 (2) | 0.72356 (9) | 0.23479 (13) | 0.0220 (3) |

| | | | | |
|---|---|---|---|---|
| O8 | 0.20912 (15) | 0.74143 (7) | 0.27305 (10) | 0.0246 (3) |
| C9 | 0.1130 (2) | 0.78350 (10) | 0.19758 (14) | 0.0257 (3) |
| H9 | 0.0071 | 0.8036 | 0.2030 | 0.031* |
| C10 | 0.1885 (2) | 0.79273 (10) | 0.11445 (14) | 0.0240 (3) |
| H10 | 0.1471 | 0.8199 | 0.0526 | 0.029* |
| C10A | 0.3445 (2) | 0.75302 (9) | 0.13854 (13) | 0.0208 (3) |
| C11 | 0.4827 (2) | 0.74087 (9) | 0.07672 (12) | 0.0202 (3) |
| H11 | 0.5173 | 0.7930 | 0.0522 | 0.024* |
| C11A | 0.62601 (19) | 0.70466 (9) | 0.15030 (12) | 0.0189 (3) |
| H11A | 0.6735 | 0.7472 | 0.1986 | 0.023* |
| C12 | 0.76530 (19) | 0.67005 (9) | 0.10328 (12) | 0.0196 (3) |
| N13 | 0.82043 (16) | 0.60449 (8) | 0.16182 (10) | 0.0196 (3) |
| C13A | 0.95024 (19) | 0.55390 (9) | 0.14728 (12) | 0.0193 (3) |
| C14 | 0.5826 (2) | 0.48033 (9) | 0.32540 (13) | 0.0220 (3) |
| H14A | 0.5286 | 0.4304 | 0.3003 | 0.026* |
| H14B | 0.4961 | 0.5207 | 0.3261 | 0.026* |
| C15 | 0.6729 (2) | 0.46908 (10) | 0.43133 (13) | 0.0220 (3) |
| C16 | 0.7223 (2) | 0.40551 (10) | 0.48999 (13) | 0.0244 (3) |
| H16 | 0.6980 | 0.3520 | 0.4731 | 0.029* |
| C17 | 0.8185 (2) | 0.43469 (12) | 0.58253 (14) | 0.0290 (4) |
| H17 | 0.8700 | 0.4044 | 0.6393 | 0.035* |
| C18 | 0.8216 (3) | 0.51364 (12) | 0.57333 (14) | 0.0325 (4) |
| H18 | 0.8777 | 0.5484 | 0.6236 | 0.039* |
| C19 | 0.4283 (2) | 0.68777 (10) | −0.01577 (12) | 0.0212 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0327 (6) | 0.0167 (5) | 0.0209 (6) | −0.0006 (4) | 0.0031 (5) | 0.0008 (4) |
| O2 | 0.0387 (7) | 0.0233 (6) | 0.0229 (6) | −0.0022 (5) | 0.0021 (5) | −0.0022 (5) |
| O3 | 0.0280 (6) | 0.0195 (6) | 0.0251 (6) | 0.0020 (4) | 0.0017 (5) | −0.0018 (4) |
| O4 | 0.0352 (7) | 0.0225 (6) | 0.0241 (6) | 0.0061 (5) | −0.0060 (5) | −0.0028 (5) |
| O5 | 0.0253 (6) | 0.0211 (6) | 0.0250 (6) | −0.0016 (4) | 0.0055 (5) | 0.0028 (4) |
| C1 | 0.0214 (7) | 0.0221 (7) | 0.0222 (7) | −0.0022 (6) | 0.0015 (6) | −0.0004 (6) |
| C2 | 0.0204 (7) | 0.0285 (8) | 0.0217 (8) | −0.0005 (6) | 0.0017 (6) | −0.0034 (6) |
| C3 | 0.0248 (8) | 0.0270 (8) | 0.0217 (7) | 0.0044 (6) | 0.0011 (6) | −0.0023 (6) |
| C4 | 0.0289 (8) | 0.0198 (7) | 0.0199 (7) | 0.0044 (6) | 0.0014 (6) | 0.0005 (6) |
| C4A | 0.0232 (7) | 0.0184 (7) | 0.0169 (7) | 0.0007 (5) | 0.0017 (5) | −0.0006 (5) |
| C5 | 0.0242 (7) | 0.0182 (7) | 0.0174 (7) | 0.0001 (5) | −0.0006 (6) | 0.0011 (5) |
| N6 | 0.0230 (6) | 0.0166 (6) | 0.0198 (6) | −0.0007 (5) | 0.0032 (5) | 0.0011 (5) |
| C6A | 0.0212 (7) | 0.0160 (7) | 0.0188 (7) | −0.0010 (5) | 0.0016 (5) | 0.0008 (5) |
| C6B | 0.0203 (7) | 0.0182 (7) | 0.0188 (7) | 0.0005 (5) | 0.0017 (5) | 0.0022 (5) |
| C7 | 0.0239 (7) | 0.0225 (7) | 0.0212 (7) | 0.0011 (6) | 0.0044 (6) | 0.0011 (6) |
| C7A | 0.0221 (7) | 0.0187 (7) | 0.0251 (8) | 0.0000 (5) | 0.0037 (6) | −0.0031 (6) |
| O8 | 0.0239 (6) | 0.0223 (6) | 0.0284 (6) | 0.0025 (4) | 0.0066 (5) | −0.0004 (5) |
| C9 | 0.0243 (8) | 0.0202 (7) | 0.0321 (9) | 0.0026 (6) | 0.0030 (6) | −0.0017 (6) |
| C10 | 0.0237 (7) | 0.0196 (7) | 0.0279 (8) | 0.0020 (6) | 0.0009 (6) | −0.0016 (6) |
| C10A | 0.0225 (7) | 0.0159 (7) | 0.0235 (8) | −0.0001 (5) | 0.0019 (6) | −0.0020 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11 | 0.0224 (7) | 0.0166 (7) | 0.0210 (7) | 0.0006 (5) | 0.0017 (6) | 0.0007 (5) |
| C11A | 0.0201 (7) | 0.0165 (6) | 0.0195 (7) | −0.0015 (5) | 0.0010 (5) | 0.0002 (5) |
| C12 | 0.0213 (7) | 0.0163 (6) | 0.0203 (7) | −0.0021 (5) | 0.0011 (5) | −0.0009 (5) |
| N13 | 0.0200 (6) | 0.0172 (6) | 0.0216 (6) | 0.0008 (5) | 0.0034 (5) | 0.0019 (5) |
| C13A | 0.0192 (7) | 0.0198 (7) | 0.0181 (7) | 0.0007 (5) | 0.0002 (5) | 0.0001 (5) |
| C14 | 0.0211 (7) | 0.0189 (7) | 0.0264 (8) | −0.0011 (5) | 0.0044 (6) | 0.0032 (6) |
| C15 | 0.0240 (7) | 0.0207 (7) | 0.0221 (7) | −0.0005 (6) | 0.0056 (6) | −0.0005 (6) |
| C16 | 0.0243 (8) | 0.0243 (8) | 0.0252 (8) | 0.0001 (6) | 0.0058 (6) | 0.0032 (6) |
| C17 | 0.0288 (8) | 0.0358 (9) | 0.0229 (8) | 0.0001 (7) | 0.0051 (7) | 0.0046 (7) |
| C18 | 0.0394 (10) | 0.0372 (10) | 0.0201 (8) | −0.0038 (8) | 0.0019 (7) | −0.0021 (7) |
| C19 | 0.0218 (7) | 0.0207 (7) | 0.0209 (7) | 0.0002 (6) | 0.0026 (6) | 0.0008 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C5 | 1.2507 (19) | C7—C7A | 1.494 (2) |
| O2—C18 | 1.373 (2) | C7—H7A | 0.9900 |
| O2—C15 | 1.375 (2) | C7—H7B | 0.9900 |
| O3—C19 | 1.209 (2) | C7A—C10A | 1.356 (2) |
| O4—C19 | 1.334 (2) | C7A—O8 | 1.368 (2) |
| O4—H4O | 0.9287 | O8—C9 | 1.369 (2) |
| O5—C12 | 1.214 (2) | C9—C10 | 1.350 (3) |
| C1—C13A | 1.393 (2) | C9—H9 | 0.9500 |
| C1—C2 | 1.395 (2) | C10—C10A | 1.442 (2) |
| C1—H1 | 0.9500 | C10—H10 | 0.9500 |
| C2—C3 | 1.389 (2) | C10A—C11 | 1.514 (2) |
| C2—H2 | 0.9500 | C11—C19 | 1.524 (2) |
| C3—C4 | 1.383 (2) | C11—C11A | 1.532 (2) |
| C3—H3 | 0.9500 | C11—H11 | 1.0000 |
| C4—C4A | 1.398 (2) | C11A—C12 | 1.506 (2) |
| C4—H4 | 0.9500 | C11A—H11A | 1.0000 |
| C4A—C13A | 1.401 (2) | C12—N13 | 1.389 (2) |
| C4A—C5 | 1.473 (2) | N13—C13A | 1.408 (2) |
| C5—N6 | 1.351 (2) | C14—C15 | 1.485 (2) |
| N6—C6A | 1.471 (2) | C14—H14A | 0.9900 |
| N6—C14 | 1.477 (2) | C14—H14B | 0.9900 |
| C6A—N13 | 1.467 (2) | C15—C16 | 1.352 (2) |
| C6A—C6B | 1.541 (2) | C16—C17 | 1.432 (3) |
| C6A—H6A | 1.0000 | C16—H16 | 0.9500 |
| C6B—C7 | 1.527 (2) | C17—C18 | 1.347 (3) |
| C6B—C11A | 1.534 (2) | C17—H17 | 0.9500 |
| C6B—H6B | 1.0000 | C18—H18 | 0.9500 |
| | | | |
| C18—O2—C15 | 106.38 (14) | C9—C10—C10A | 106.03 (15) |
| C19—O4—H4O | 108.4 | C9—C10—H10 | 127.0 |
| C13A—C1—C2 | 118.66 (15) | C10A—C10—H10 | 127.0 |
| C13A—C1—H1 | 120.7 | C7A—C10A—C10 | 105.85 (15) |
| C2—C1—H1 | 120.7 | C7A—C10A—C11 | 122.43 (15) |
| C3—C2—C1 | 121.48 (16) | C10—C10A—C11 | 131.71 (15) |

| | | | |
|---|---|---|---|
| C3—C2—H2 | 119.3 | C10A—C11—C19 | 111.01 (13) |
| C1—C2—H2 | 119.3 | C10A—C11—C11A | 106.54 (13) |
| C4—C3—C2 | 119.64 (15) | C19—C11—C11A | 111.52 (13) |
| C4—C3—H3 | 120.2 | C10A—C11—H11 | 109.2 |
| C2—C3—H3 | 120.2 | C19—C11—H11 | 109.2 |
| C3—C4—C4A | 119.95 (15) | C11A—C11—H11 | 109.2 |
| C3—C4—H4 | 120.0 | C12—C11A—C11 | 117.25 (13) |
| C4A—C4—H4 | 120.0 | C12—C11A—C6B | 104.73 (12) |
| C4—C4A—C13A | 119.98 (15) | C11—C11A—C6B | 112.60 (13) |
| C4—C4A—C5 | 119.23 (14) | C12—C11A—H11A | 107.3 |
| C13A—C4A—C5 | 120.79 (14) | C11—C11A—H11A | 107.3 |
| O1—C5—N6 | 120.60 (15) | C6B—C11A—H11A | 107.3 |
| O1—C5—C4A | 121.65 (15) | O5—C12—N13 | 126.00 (15) |
| N6—C5—C4A | 117.73 (14) | O5—C12—C11A | 127.11 (14) |
| C5—N6—C6A | 122.48 (14) | N13—C12—C11A | 106.78 (13) |
| C5—N6—C14 | 116.74 (13) | C12—N13—C13A | 127.06 (14) |
| C6A—N6—C14 | 118.00 (13) | C12—N13—C6A | 113.46 (13) |
| N13—C6A—N6 | 109.94 (12) | C13A—N13—C6A | 119.47 (13) |
| N13—C6A—C6B | 102.57 (12) | C1—C13A—C4A | 120.24 (15) |
| N6—C6A—C6B | 112.07 (13) | C1—C13A—N13 | 123.27 (14) |
| N13—C6A—H6A | 110.7 | C4A—C13A—N13 | 116.46 (14) |
| N6—C6A—H6A | 110.7 | N6—C14—C15 | 110.51 (13) |
| C6B—C6A—H6A | 110.7 | N6—C14—H14A | 109.5 |
| C7—C6B—C11A | 108.73 (13) | C15—C14—H14A | 109.5 |
| C7—C6B—C6A | 121.09 (13) | N6—C14—H14B | 109.5 |
| C11A—C6B—C6A | 103.19 (12) | C15—C14—H14B | 109.5 |
| C7—C6B—H6B | 107.7 | H14A—C14—H14B | 108.1 |
| C11A—C6B—H6B | 107.7 | C16—C15—O2 | 110.11 (15) |
| C6A—C6B—H6B | 107.7 | C16—C15—C14 | 134.38 (16) |
| C7A—C7—C6B | 103.64 (13) | O2—C15—C14 | 115.31 (14) |
| C7A—C7—H7A | 111.0 | C15—C16—C17 | 106.55 (16) |
| C6B—C7—H7A | 111.0 | C15—C16—H16 | 126.7 |
| C7A—C7—H7B | 111.0 | C17—C16—H16 | 126.7 |
| C6B—C7—H7B | 111.0 | C18—C17—C16 | 106.50 (16) |
| H7A—C7—H7B | 109.0 | C18—C17—H17 | 126.8 |
| C10A—C7A—O8 | 111.01 (15) | C16—C17—H17 | 126.8 |
| C10A—C7A—C7 | 129.18 (15) | C17—C18—O2 | 110.46 (16) |
| O8—C7A—C7 | 119.72 (15) | C17—C18—H18 | 124.8 |
| C7A—O8—C9 | 105.95 (13) | O2—C18—H18 | 124.8 |
| C10—C9—O8 | 111.16 (15) | O3—C19—O4 | 123.18 (15) |
| C10—C9—H9 | 124.4 | O3—C19—C11 | 124.47 (15) |
| O8—C9—H9 | 124.4 | O4—C19—C11 | 112.35 (14) |

| | | | |
|---|---|---|---|
| C13A—C1—C2—C3 | −0.5 (2) | C19—C11—C11A—C6B | −76.15 (17) |
| C1—C2—C3—C4 | 1.6 (3) | C7—C6B—C11A—C12 | 158.92 (12) |
| C2—C3—C4—C4A | −0.8 (2) | C6A—C6B—C11A—C12 | 29.17 (15) |
| C3—C4—C4A—C13A | −1.1 (2) | C7—C6B—C11A—C11 | −72.59 (16) |
| C3—C4—C4A—C5 | 179.25 (15) | C6A—C6B—C11A—C11 | 157.66 (13) |

| | | | |
|---|---|---|---|
| C4—C4A—C5—O1 | −11.2 (2) | C11—C11A—C12—O5 | 39.4 (2) |
| C13A—C4A—C5—O1 | 169.16 (15) | C6B—C11A—C12—O5 | 165.06 (16) |
| C4—C4A—C5—N6 | 166.94 (14) | C11—C11A—C12—N13 | −144.36 (13) |
| C13A—C4A—C5—N6 | −12.7 (2) | C6B—C11A—C12—N13 | −18.74 (16) |
| O1—C5—N6—C6A | 174.40 (14) | O5—C12—N13—C13A | −3.1 (3) |
| C4A—C5—N6—C6A | −3.8 (2) | C11A—C12—N13—C13A | −179.33 (14) |
| O1—C5—N6—C14 | 13.7 (2) | O5—C12—N13—C6A | 176.28 (15) |
| C4A—C5—N6—C14 | −164.46 (13) | C11A—C12—N13—C6A | 0.03 (17) |
| C5—N6—C6A—N13 | 29.51 (19) | N6—C6A—N13—C12 | 137.81 (13) |
| C14—N6—C6A—N13 | −170.02 (13) | C6B—C6A—N13—C12 | 18.44 (16) |
| C5—N6—C6A—C6B | 142.89 (15) | N6—C6A—N13—C13A | −42.78 (18) |
| C14—N6—C6A—C6B | −56.63 (17) | C6B—C6A—N13—C13A | −162.15 (13) |
| N13—C6A—C6B—C7 | −150.21 (14) | C2—C1—C13A—C4A | −1.4 (2) |
| N6—C6A—C6B—C7 | 91.92 (17) | C2—C1—C13A—N13 | −179.33 (15) |
| N13—C6A—C6B—C11A | −28.44 (15) | C4—C4A—C13A—C1 | 2.2 (2) |
| N6—C6A—C6B—C11A | −146.31 (13) | C5—C4A—C13A—C1 | −178.11 (14) |
| C11A—C6B—C7—C7A | 53.19 (16) | C4—C4A—C13A—N13 | −179.72 (14) |
| C6A—C6B—C7—C7A | 172.26 (13) | C5—C4A—C13A—N13 | −0.1 (2) |
| C6B—C7—C7A—C10A | −20.2 (2) | C12—N13—C13A—C1 | 26.6 (2) |
| C6B—C7—C7A—O8 | 156.02 (14) | C6A—N13—C13A—C1 | −152.68 (15) |
| C10A—C7A—O8—C9 | 0.07 (18) | C12—N13—C13A—C4A | −151.33 (15) |
| C7—C7A—O8—C9 | −176.82 (14) | C6A—N13—C13A—C4A | 29.3 (2) |
| C7A—O8—C9—C10 | −0.28 (19) | C5—N6—C14—C15 | 74.64 (18) |
| O8—C9—C10—C10A | 0.37 (19) | C6A—N6—C14—C15 | −86.95 (16) |
| O8—C7A—C10A—C10 | 0.14 (18) | C18—O2—C15—C16 | 0.29 (19) |
| C7—C7A—C10A—C10 | 176.67 (16) | C18—O2—C15—C14 | −175.39 (15) |
| O8—C7A—C10A—C11 | −178.89 (14) | N6—C14—C15—C16 | −105.4 (2) |
| C7—C7A—C10A—C11 | −2.4 (3) | N6—C14—C15—O2 | 68.88 (18) |
| C9—C10—C10A—C7A | −0.31 (19) | O2—C15—C16—C17 | 0.04 (19) |
| C9—C10—C10A—C11 | 178.60 (16) | C14—C15—C16—C17 | 174.57 (18) |
| C7A—C10A—C11—C19 | 112.13 (17) | C15—C16—C17—C18 | −0.4 (2) |
| C10—C10A—C11—C19 | −66.6 (2) | C16—C17—C18—O2 | 0.6 (2) |
| C7A—C10A—C11—C11A | −9.5 (2) | C15—O2—C18—C17 | −0.5 (2) |
| C10—C10A—C11—C11A | 171.78 (16) | C10A—C11—C19—O3 | −109.14 (18) |
| C10A—C11—C11A—C12 | 166.74 (13) | C11A—C11—C19—O3 | 9.5 (2) |
| C19—C11—C11A—C12 | 45.47 (18) | C10A—C11—C19—O4 | 69.78 (18) |
| C10A—C11—C11A—C6B | 45.13 (17) | C11A—C11—C19—O4 | −171.58 (14) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H4*O*···O1$^{i}$ | 0.93 | 1.75 | 2.671 (2) | 174 |
| C2—H2···O3$^{ii}$ | 0.95 | 2.42 | 3.326 (2) | 160 |
| C3—H3···O3$^{iii}$ | 0.95 | 2.56 | 3.384 (2) | 146 |
| C7—H7*B*···O4$^{iv}$ | 0.99 | 2.54 | 3.455 (2) | 155 |
| C11*A*—H11*A*···O1$^{v}$ | 1.00 | 2.38 | 3.325 (2) | 157 |

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