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2-Methyl-3′-(4-methylphenyl)-4′-(2-nitrophenyl)-4′*H*-spiro[chroman-3,5′-isoxazol]-4-one

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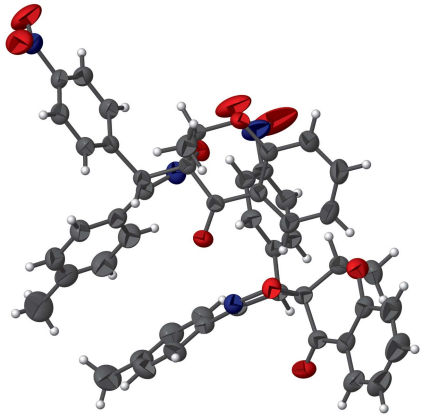
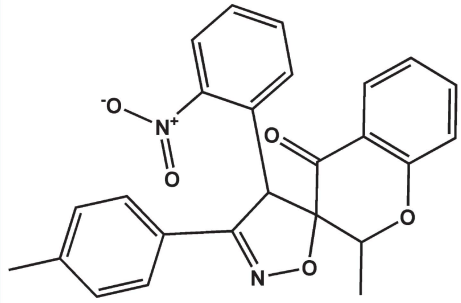
The title compound, $C_{25}H_{20}N_2O_5$, crystallizes with two molecules (*A* and *B*) in the asymmetric unit with similar conformations. The five-membered rings are both in envelope conformations with the spiro C atom as the flap. The six-membered heterocycles display half-chair conformations. The mean plane through the isoxazole ring is nearly perpendicular to those through the spiro-chroman system and the 4-nitrophenyl moieties, as indicated by the dihedral angles of 81.42 (9) and 87.58 (8)°, respectively, between them in molecule *A*. Equivalent data for molecule *B* are 75.58 (9) and 84.15 (8)°, respectively. The *p*-tolyl plane makes a dihedral angles of 24.10 (9) and 28.78 (8)° with the isoxazole ring in molecules *A* and *B*, respectively. In the crystal, molecules are linked by C—H···O and C—H···N hydrogen bonds and C—H···π interaction, forming a three-dimensional network.

3D view



Chemical scheme



Structure description

Spiroisoxazolines have various biological properties such as herbicidal (De Amici *et al.*, 1990)and plant-growth regulatory activities (Howe & Shelton, 1990) and have applications as antitumor agents (Smietana *et al.*, 1999) and anti-HIV agents (Liu *et al.*, 1997). In this work we have studied the regio- and stereoselective synthesis of spiroisoxazoline 2-methyl-4′-(nitrophenyl)-3′-(*p*-tolyl)-4′*H*-spiro[chroman-3,5′-isoxazol]-4-one obtained by the 1,3-dipolar cycloaddition (Mahfoud *et al.*, 2015, Boughaleb *et al.*, 2011) of (*E*)-2-methyl-3-(4-nitrobenzylidene)chroman-4-one and 4-tolylbenzonitrile oxide. This concerted reaction affords a single regio-isomer which is the *trans*-spiroisoxazoline

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

*Cg*1 and *Cg*2 are the centroids of the C44–C49 and C11–C16 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1^{i}	0.98	2.40	3.3466 (17)	162
C35—H35···O6^{ii}	0.98	2.25	3.2072 (17)	164
C16—H16···N3^{i}	0.93	2.56	3.3091 (19)	138
C23—H23···*Cg*1	0.93	2.95	3.581 (2)	127
C30—H30···*Cg*2^{iii}	0.93	3.00	3.758 (2)	140

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y+2, -z$; (iii) $x, y, z-1$.

(Bakhouch *et al.*, 2014). The structure of the product was confirmed by the present X-ray study.

The asymmetric unit of the title compound is formed by two molecules (*A* and *B*) with almost the same conformation, as shown in Fig. 1. The most important difference between them lies in the orientation of the phenyl rings, as can be seen in the overlay plot (Fig. 2). The isoxazole ring belonging to each molecule is linked to a spiro-chroman system with additional 3′-*p*-tolyl and 4-nitrophenyl ring substituents. The five-membered rings (N1/O3/C9/C10/C18) and (N3/O8/C34/C35/C43) display envelope conformations on C9 and C43, as indicated by the total puckering amplitudes $Q2 = 0.3093$ (15) and 0.3143 (15) Å and spherical polar angles $\varphi2 = 138.2$ (3) and 138.4 (3)°, respectively. Moreover, the six-membered heterocycles adopt half-chair conformations with the following puckering parameters: $Q = 0.4758$ (17) Å, $\theta = 129.2$ (2)°, $\varphi2 = 97.0$ (2)° for molecule *A* and $Q = 0.4409$ (17) Å, $\theta = 50.5$ (2)°, $\varphi2 = 270.8$ (3)°, for *B*. The dihedral angles between the mean plane through the isoxazole rings and the *p*-tolyl planes are 24.10 (9) and 28.78 (8)°, in molecules *A* and *B*, respectively. The mean plane through the isoxazole ring is nearly perpendicular to those through the spiro-chroman system and the 4-nitrophenyl moieties, as indicated by the dihedral angles of 81.42 (9) and 87.58 (8)°, respectively, between them in molecule *A* [75.58 (9) and 84.15 (8)°, respectively in *B*].

In the crystal, the molecules are linked by C10—H10···O1, C35—H35···O6 and C16—H16···N3 hydrogen bonds, in addition to C23—H23···π(*Cg*1) and C30—H30···π(*Cg*2) interactions, forming a three-dimensional network as shown in Fig. 3 and Table 1.

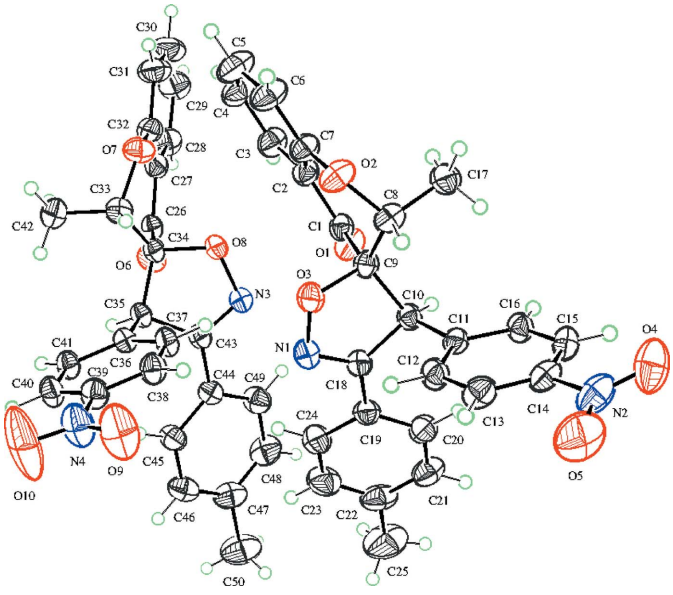


Figure 1
Plot of the molecules of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small circles.

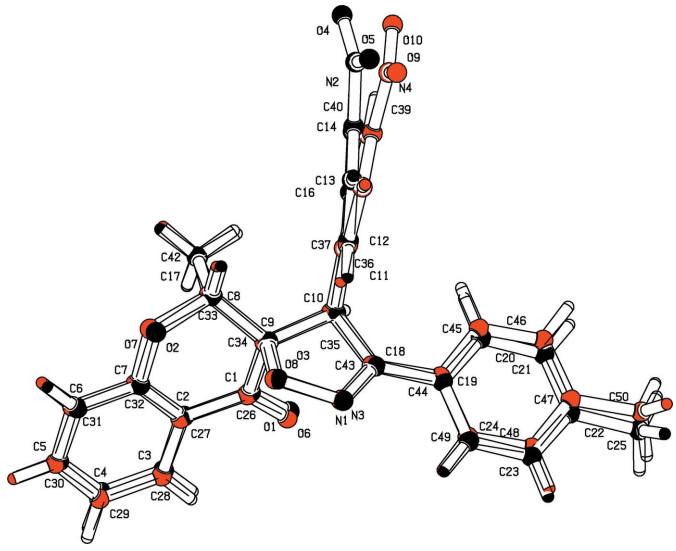


Figure 2
Overlay plot of molecule *B* (red) on molecule *A* (black).

Synthesis and crystallization

In a 100 ml flask, 2 mmol of (*E*)-2-methyl-3-(4-nitrobenzylidene) chroman-4-one and 2.4 mmol of *p*-tolylnitrioxide were dissolved in 20 ml of chloroform. The mixture was cooled to 273 K under magnetic stirring in an ice bath. Then, 15 ml of bleach (NaOCl) at 18° was added in small doses without

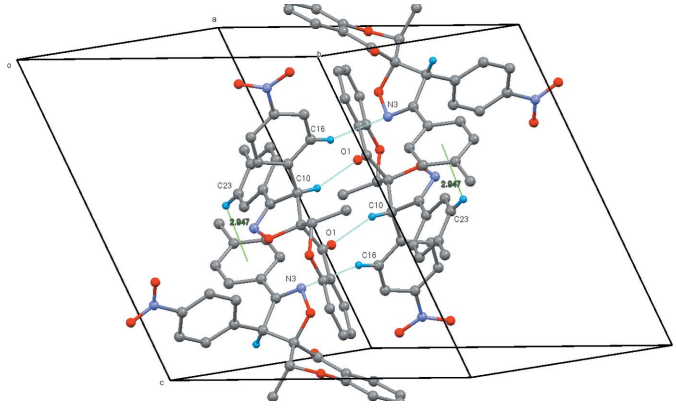


Figure 3
Crystal packing for the title compound showing molecules linked by hydrogen bonds (dashed blue lines) and C—H···π interaction (green lines).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{25}H_{20}N_2O_5$
M_r	428.43
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	11.5219 (6), 14.2321 (7), 14.7033 (7)
α, β, γ (°)	72.201 (2), 73.469 (2), 73.647 (2)
V (Å^3)	2150.18 (19)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.36 × 0.28 × 0.25
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.639, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	86936, 10255, 7363
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.122, 1.02
No. of reflections	10255
No. of parameters	581
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.26

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

exceeding 278 K. The mixture was left under magnetic stirring for 16 h at room temperature, then washed with water until the pH was neutral and dried on sodium sulfate. The solvent was removed under reduced pressure to leave an oily residue. The precipitated compound was then recrystallized from ethanol solution as colourless blocks.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{25}H_{20}N_2O_5$
M_r = 428.43
Triclinic, $P\overline{1}$
a = 11.5219 (6) Å
b = 14.2321 (7) Å
c = 14.7033 (7) Å
α = 72.201 (2)°
β = 73.469 (2)°
γ = 73.647 (2)°
V = 2150.18 (19) Å^3
Z = 4
$F(000)$ = 896
D_x = 1.323 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10255 reflections
θ = 2.1–27.9°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.36 × 0.28 × 0.25 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.639, T_{max} = 0.747
86936 measured reflections
10255 independent reflections
7363 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.9°, θ_{min} = 2.1°
h = −15→15
k = −18→18
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.122
S = 1.02
10255 reflections
581 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.6603P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.71087 (13)	0.49608 (11)	0.37433 (10)	0.0365 (3)
C2	0.79239 (13)	0.45907 (11)	0.29001 (10)	0.0388 (3)
C3	0.75481 (16)	0.48309 (12)	0.20169 (11)	0.0495 (4)
H3	0.676591	0.522713	0.196050	0.059*
C4	0.83235 (19)	0.44868 (15)	0.12350 (13)	0.0623 (5)
H4	0.806249	0.463973	0.065331	0.075*
C5	0.9494 (2)	0.39122 (16)	0.13101 (14)	0.0678 (5)
H5	1.002242	0.369206	0.077203	0.081*
C6	0.98877 (17)	0.36613 (15)	0.21703 (13)	0.0600 (5)
H6	1.067477	0.326919	0.221550	0.072*
C7	0.91024 (14)	0.39975 (12)	0.29698 (11)	0.0428 (3)
C8	0.86434 (14)	0.37965 (12)	0.47069 (11)	0.0421 (3)
H8	0.914902	0.373053	0.517865	0.051*
C9	0.77534 (12)	0.48180 (11)	0.45741 (10)	0.0357 (3)
C10	0.68255 (12)	0.51028 (10)	0.54800 (10)	0.0343 (3)
H10	0.602563	0.494634	0.554534	0.041*
C11	0.72823 (12)	0.46262 (10)	0.64259 (10)	0.0346 (3)
C12	0.82793 (14)	0.48833 (12)	0.65780 (11)	0.0443 (4)
H12	0.862619	0.540155	0.612152	0.053*
C13	0.87597 (15)	0.43763 (14)	0.74007 (12)	0.0493 (4)
H13	0.942495	0.455001	0.750203	0.059*
C14	0.82365 (15)	0.36116 (13)	0.80655 (11)	0.0460 (4)
C15	0.72337 (15)	0.33473 (12)	0.79497 (11)	0.0466 (4)
H15	0.689002	0.283001	0.841108	0.056*
C16	0.67506 (14)	0.38710 (11)	0.71294 (11)	0.0405 (3)
H16	0.606059	0.371505	0.704844	0.049*
C17	0.79860 (17)	0.29277 (12)	0.50363 (13)	0.0533 (4)
H17A	0.752555	0.298441	0.456400	0.080*
H17B	0.742974	0.294719	0.565910	0.080*
H17C	0.858576	0.230032	0.509483	0.080*
C18	0.67423 (14)	0.62358 (11)	0.51415 (10)	0.0392 (3)
C19	0.56998 (15)	0.69988 (11)	0.54889 (11)	0.0418 (3)
C20	0.48044 (16)	0.67386 (13)	0.63232 (12)	0.0504 (4)
H20	0.488054	0.607106	0.668633	0.060*
C21	0.37977 (18)	0.74655 (15)	0.66194 (14)	0.0599 (5)
H21	0.320723	0.727894	0.718208	0.072*
C22	0.3655 (2)	0.84620 (14)	0.60948 (15)	0.0637 (5)
C23	0.4534 (2)	0.87148 (14)	0.52541 (16)	0.0673 (5)
H23	0.444183	0.938028	0.488495	0.081*
C24	0.55390 (19)	0.80070 (13)	0.49508 (13)	0.0566 (4)
H24	0.611932	0.819838	0.438259	0.068*
C25	0.2545 (3)	0.9249 (2)	0.6427 (2)	0.1065 (10)
H25A	0.213014	0.959960	0.589852	0.160*
H25B	0.281923	0.972199	0.662035	0.160*
H25C	0.198459	0.892328	0.697207	0.160*

C26	0.52270 (13)	0.81733 (11)	0.02227 (10)	0.0380 (3)
C27	0.53883 (15)	0.73810 (11)	−0.02741 (11)	0.0425 (3)
C28	0.44133 (18)	0.72885 (14)	−0.06096 (12)	0.0541 (4)
H28	0.366042	0.775090	−0.054226	0.065*
C29	0.4568 (2)	0.65138 (17)	−0.10395 (15)	0.0704 (6)
H29	0.391315	0.644228	−0.124695	0.084*
C30	0.5697 (2)	0.58434 (17)	−0.11614 (16)	0.0773 (6)
H30	0.579713	0.532603	−0.145751	0.093*
C31	0.6673 (2)	0.59255 (14)	−0.08550 (14)	0.0642 (5)
H31	0.743292	0.547740	−0.095499	0.077*
C32	0.65154 (16)	0.66868 (12)	−0.03921 (11)	0.0466 (4)
C33	0.74921 (14)	0.76072 (12)	0.01846 (11)	0.0425 (3)
H33	0.811056	0.738537	0.058444	0.051*
C34	0.62391 (13)	0.80089 (10)	0.07759 (10)	0.0353 (3)
C35	0.61372 (13)	0.89159 (10)	0.11657 (10)	0.0346 (3)
H35	0.586305	0.955024	0.070535	0.041*
C36	0.72907 (13)	0.89408 (10)	0.14622 (10)	0.0350 (3)
C37	0.76963 (14)	0.82115 (11)	0.22526 (11)	0.0406 (3)
H37	0.727948	0.769001	0.258268	0.049*
C38	0.87077 (14)	0.82544 (12)	0.25505 (11)	0.0437 (3)
H38	0.897624	0.777030	0.307961	0.052*
C39	0.93090 (14)	0.90306 (12)	0.20459 (11)	0.0440 (3)
C40	0.89462 (15)	0.97603 (12)	0.12563 (12)	0.0458 (4)
H40	0.937658	1.027306	0.092445	0.055*
C41	0.79271 (14)	0.97113 (11)	0.09698 (11)	0.0406 (3)
H41	0.766366	1.019984	0.044101	0.049*
C42	0.78724 (17)	0.83952 (14)	−0.07436 (13)	0.0570 (4)
H42A	0.867909	0.812801	−0.109343	0.085*
H42B	0.789421	0.898954	−0.057477	0.085*
H42C	0.728397	0.856544	−0.114837	0.085*
C43	0.51140 (13)	0.87094 (10)	0.20643 (10)	0.0356 (3)
C44	0.43061 (13)	0.94700 (11)	0.25823 (11)	0.0389 (3)
C45	0.46515 (16)	1.03638 (12)	0.24885 (13)	0.0490 (4)
H45	0.541165	1.048068	0.209429	0.059*
C46	0.38805 (18)	1.10767 (13)	0.29729 (14)	0.0572 (4)
H46	0.413516	1.166340	0.290698	0.069*
C47	0.27383 (17)	1.09373 (14)	0.35538 (14)	0.0581 (5)
C48	0.23854 (17)	1.00576 (16)	0.36250 (15)	0.0634 (5)
H48	0.161258	0.995375	0.400164	0.076*
C49	0.31503 (15)	0.93341 (14)	0.31522 (13)	0.0521 (4)
H49	0.289006	0.875070	0.321568	0.063*
C50	0.1902 (2)	1.17146 (19)	0.40908 (19)	0.0893 (7)
H50A	0.204511	1.154431	0.473957	0.134*
H50B	0.105262	1.172562	0.412870	0.134*
H50C	0.207563	1.236831	0.374590	0.134*
N1	0.76748 (13)	0.64877 (10)	0.44746 (10)	0.0468 (3)
N2	0.87729 (15)	0.30507 (15)	0.89270 (11)	0.0638 (4)
N3	0.50112 (11)	0.77822 (9)	0.23520 (9)	0.0402 (3)

N4	1.03716 (16)	0.90838 (14)	0.23749 (13)	0.0704 (5)
O1	0.60250 (9)	0.53733 (9)	0.37823 (8)	0.0499 (3)
O2	0.95481 (9)	0.37380 (9)	0.37996 (8)	0.0501 (3)
O3	0.84776 (9)	0.56013 (8)	0.42149 (8)	0.0463 (3)
O4	0.85062 (15)	0.22382 (14)	0.93883 (11)	0.0900 (5)
O5	0.94819 (19)	0.34191 (16)	0.91234 (13)	0.1043 (6)
O6	0.43257 (10)	0.88679 (8)	0.02632 (9)	0.0499 (3)
O7	0.74976 (11)	0.66992 (8)	−0.00674 (9)	0.0509 (3)
O8	0.58807 (10)	0.72448 (7)	0.16663 (7)	0.0404 (2)
O9	1.06326 (16)	0.84887 (14)	0.31079 (13)	0.1002 (6)
O10	1.0951 (2)	0.97103 (19)	0.18936 (18)	0.1604 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0335 (7)	0.0356 (7)	0.0364 (7)	−0.0062 (6)	−0.0071 (6)	−0.0045 (6)
C2	0.0390 (7)	0.0379 (7)	0.0358 (7)	−0.0058 (6)	−0.0073 (6)	−0.0066 (6)
C3	0.0542 (9)	0.0471 (9)	0.0401 (8)	0.0006 (7)	−0.0155 (7)	−0.0061 (7)
C4	0.0780 (13)	0.0640 (11)	0.0383 (9)	0.0036 (10)	−0.0189 (9)	−0.0144 (8)
C5	0.0712 (13)	0.0766 (13)	0.0453 (10)	0.0061 (10)	−0.0070 (9)	−0.0259 (9)
C6	0.0494 (10)	0.0700 (12)	0.0533 (10)	0.0074 (8)	−0.0082 (8)	−0.0260 (9)
C7	0.0395 (8)	0.0469 (9)	0.0400 (8)	−0.0043 (6)	−0.0086 (6)	−0.0126 (7)
C8	0.0369 (7)	0.0488 (9)	0.0384 (8)	0.0009 (6)	−0.0120 (6)	−0.0131 (7)
C9	0.0317 (7)	0.0386 (7)	0.0356 (7)	−0.0092 (6)	−0.0056 (6)	−0.0075 (6)
C10	0.0313 (6)	0.0357 (7)	0.0354 (7)	−0.0100 (5)	−0.0048 (5)	−0.0075 (6)
C11	0.0330 (7)	0.0369 (7)	0.0335 (7)	−0.0092 (6)	−0.0035 (5)	−0.0101 (6)
C12	0.0438 (8)	0.0511 (9)	0.0417 (8)	−0.0224 (7)	−0.0058 (7)	−0.0085 (7)
C13	0.0432 (8)	0.0671 (11)	0.0462 (9)	−0.0174 (8)	−0.0111 (7)	−0.0195 (8)
C14	0.0445 (8)	0.0567 (10)	0.0336 (8)	−0.0023 (7)	−0.0088 (6)	−0.0141 (7)
C15	0.0490 (9)	0.0481 (9)	0.0367 (8)	−0.0149 (7)	−0.0041 (7)	−0.0027 (7)
C16	0.0376 (7)	0.0451 (8)	0.0394 (8)	−0.0161 (6)	−0.0054 (6)	−0.0071 (6)
C17	0.0674 (11)	0.0400 (9)	0.0477 (9)	−0.0034 (8)	−0.0174 (8)	−0.0067 (7)
C18	0.0452 (8)	0.0370 (7)	0.0352 (7)	−0.0119 (6)	−0.0078 (6)	−0.0066 (6)
C19	0.0506 (9)	0.0374 (8)	0.0393 (8)	−0.0070 (7)	−0.0137 (7)	−0.0104 (6)
C20	0.0561 (10)	0.0443 (9)	0.0448 (9)	−0.0032 (7)	−0.0102 (7)	−0.0103 (7)
C21	0.0608 (11)	0.0646 (12)	0.0488 (10)	0.0025 (9)	−0.0099 (8)	−0.0227 (9)
C22	0.0754 (13)	0.0543 (11)	0.0639 (12)	0.0133 (9)	−0.0297 (10)	−0.0292 (9)
C23	0.0902 (15)	0.0396 (9)	0.0699 (13)	0.0016 (9)	−0.0311 (12)	−0.0119 (9)
C24	0.0722 (12)	0.0413 (9)	0.0519 (10)	−0.0091 (8)	−0.0155 (9)	−0.0062 (8)
C25	0.112 (2)	0.0837 (17)	0.106 (2)	0.0373 (15)	−0.0270 (17)	−0.0469 (16)
C26	0.0407 (8)	0.0341 (7)	0.0365 (7)	−0.0111 (6)	−0.0106 (6)	−0.0003 (6)
C27	0.0535 (9)	0.0424 (8)	0.0336 (7)	−0.0158 (7)	−0.0121 (7)	−0.0045 (6)
C28	0.0623 (11)	0.0620 (11)	0.0444 (9)	−0.0198 (9)	−0.0188 (8)	−0.0094 (8)
C29	0.0932 (16)	0.0805 (14)	0.0586 (11)	−0.0307 (12)	−0.0320 (11)	−0.0214 (10)
C30	0.1142 (19)	0.0693 (13)	0.0666 (13)	−0.0174 (13)	−0.0323 (13)	−0.0326 (11)
C31	0.0874 (14)	0.0557 (11)	0.0537 (11)	−0.0046 (10)	−0.0203 (10)	−0.0245 (9)
C32	0.0604 (10)	0.0428 (8)	0.0359 (8)	−0.0109 (7)	−0.0120 (7)	−0.0075 (7)
C33	0.0407 (8)	0.0419 (8)	0.0450 (8)	−0.0058 (6)	−0.0115 (7)	−0.0114 (7)

C34	0.0386 (7)	0.0301 (7)	0.0350 (7)	−0.0084 (6)	−0.0101 (6)	−0.0019 (6)
C35	0.0360 (7)	0.0295 (7)	0.0372 (7)	−0.0088 (5)	−0.0114 (6)	−0.0020 (6)
C36	0.0345 (7)	0.0338 (7)	0.0365 (7)	−0.0086 (6)	−0.0078 (6)	−0.0070 (6)
C37	0.0426 (8)	0.0365 (7)	0.0415 (8)	−0.0141 (6)	−0.0127 (6)	0.0007 (6)
C38	0.0440 (8)	0.0444 (8)	0.0400 (8)	−0.0104 (7)	−0.0142 (7)	−0.0007 (7)
C39	0.0383 (8)	0.0520 (9)	0.0453 (8)	−0.0153 (7)	−0.0130 (7)	−0.0082 (7)
C40	0.0462 (8)	0.0464 (9)	0.0460 (9)	−0.0226 (7)	−0.0094 (7)	−0.0021 (7)
C41	0.0434 (8)	0.0373 (8)	0.0388 (8)	−0.0122 (6)	−0.0118 (6)	0.0001 (6)
C42	0.0566 (10)	0.0591 (11)	0.0499 (10)	−0.0184 (8)	0.0016 (8)	−0.0124 (8)
C43	0.0342 (7)	0.0353 (7)	0.0397 (7)	−0.0105 (6)	−0.0124 (6)	−0.0054 (6)
C44	0.0370 (7)	0.0392 (8)	0.0425 (8)	−0.0069 (6)	−0.0137 (6)	−0.0095 (6)
C45	0.0486 (9)	0.0418 (8)	0.0582 (10)	−0.0117 (7)	−0.0094 (8)	−0.0148 (7)
C46	0.0646 (11)	0.0438 (9)	0.0684 (12)	−0.0050 (8)	−0.0210 (9)	−0.0208 (8)
C47	0.0550 (10)	0.0566 (11)	0.0634 (11)	0.0103 (8)	−0.0245 (9)	−0.0250 (9)
C48	0.0395 (9)	0.0773 (13)	0.0705 (12)	−0.0049 (9)	−0.0051 (8)	−0.0274 (10)
C49	0.0412 (8)	0.0539 (10)	0.0641 (11)	−0.0128 (7)	−0.0089 (8)	−0.0185 (8)
C50	0.0798 (15)	0.0837 (16)	0.1007 (18)	0.0227 (12)	−0.0250 (13)	−0.0491 (14)
N1	0.0513 (8)	0.0419 (7)	0.0457 (7)	−0.0175 (6)	−0.0037 (6)	−0.0079 (6)
N2	0.0560 (9)	0.0849 (12)	0.0437 (8)	0.0007 (8)	−0.0156 (7)	−0.0160 (8)
N3	0.0422 (7)	0.0383 (7)	0.0400 (7)	−0.0119 (5)	−0.0073 (5)	−0.0079 (5)
N4	0.0658 (10)	0.0810 (12)	0.0736 (11)	−0.0375 (9)	−0.0357 (9)	0.0068 (9)
O1	0.0344 (5)	0.0627 (7)	0.0452 (6)	0.0009 (5)	−0.0107 (5)	−0.0114 (5)
O2	0.0335 (5)	0.0671 (7)	0.0467 (6)	0.0048 (5)	−0.0110 (5)	−0.0217 (6)
O3	0.0390 (6)	0.0497 (6)	0.0490 (6)	−0.0186 (5)	0.0026 (5)	−0.0132 (5)
O4	0.0839 (11)	0.0959 (12)	0.0638 (9)	−0.0095 (9)	−0.0254 (8)	0.0176 (9)
O5	0.1184 (14)	0.1341 (16)	0.0819 (11)	−0.0263 (12)	−0.0629 (11)	−0.0194 (11)
O6	0.0460 (6)	0.0428 (6)	0.0627 (7)	−0.0033 (5)	−0.0236 (5)	−0.0102 (5)
O7	0.0531 (7)	0.0428 (6)	0.0575 (7)	−0.0001 (5)	−0.0162 (5)	−0.0187 (5)
O8	0.0507 (6)	0.0296 (5)	0.0380 (5)	−0.0095 (4)	−0.0095 (5)	−0.0035 (4)
O9	0.0981 (12)	0.1161 (13)	0.0996 (12)	−0.0536 (11)	−0.0689 (10)	0.0269 (10)
O10	0.161 (2)	0.188 (2)	0.1607 (19)	−0.1428 (19)	−0.1137 (17)	0.0853 (17)

Geometric parameters (Å, º)

C1—O1	1.2141 (17)	C27—C32	1.395 (2)
C1—C2	1.468 (2)	C27—C28	1.400 (2)
C1—C9	1.538 (2)	C28—C29	1.378 (3)
C2—C7	1.396 (2)	C28—H28	0.9300
C2—C3	1.399 (2)	C29—C30	1.382 (3)
C3—C4	1.369 (2)	C29—H29	0.9300
C3—H3	0.9300	C30—C31	1.369 (3)
C4—C5	1.381 (3)	C30—H30	0.9300
C4—H4	0.9300	C31—C32	1.393 (2)
C5—C6	1.376 (3)	C31—H31	0.9300
C5—H5	0.9300	C32—O7	1.354 (2)
C6—C7	1.387 (2)	C33—O7	1.4471 (18)
C6—H6	0.9300	C33—C34	1.514 (2)
C7—O2	1.3649 (18)	C33—C42	1.521 (2)

C8—O2	1.4495 (18)	C33—H33	0.9754
C8—C17	1.513 (2)	C34—O8	1.4636 (16)
C8—C9	1.518 (2)	C34—C35	1.5288 (19)
C8—H8	0.9943	C35—C43	1.514 (2)
C9—O3	1.4598 (17)	C35—C36	1.5243 (19)
C9—C10	1.5353 (19)	C35—H35	0.9800
C10—C18	1.5187 (19)	C36—C41	1.3891 (19)
C10—C11	1.5199 (19)	C36—C37	1.395 (2)
C10—H10	0.9800	C37—C38	1.379 (2)
C11—C16	1.3895 (19)	C37—H37	0.9300
C11—C12	1.392 (2)	C38—C39	1.374 (2)
C12—C13	1.383 (2)	C38—H38	0.9300
C12—H12	0.9300	C39—C40	1.377 (2)
C13—C14	1.374 (2)	C39—N4	1.466 (2)
C13—H13	0.9300	C40—C41	1.381 (2)
C14—C15	1.379 (2)	C40—H40	0.9300
C14—N2	1.474 (2)	C41—H41	0.9300
C15—C16	1.385 (2)	C42—H42A	0.9600
C15—H15	0.9300	C42—H42B	0.9600
C16—H16	0.9300	C42—H42C	0.9600
C17—H17A	0.9600	C43—N3	1.2855 (18)
C17—H17B	0.9600	C43—C44	1.467 (2)
C17—H17C	0.9600	C44—C49	1.387 (2)
C18—N1	1.2833 (19)	C44—C45	1.394 (2)
C18—C19	1.462 (2)	C45—C46	1.378 (2)
C19—C20	1.387 (2)	C45—H45	0.9300
C19—C24	1.400 (2)	C46—C47	1.380 (3)
C20—C21	1.385 (2)	C46—H46	0.9300
C20—H20	0.9300	C47—C48	1.388 (3)
C21—C22	1.380 (3)	C47—C50	1.509 (3)
C21—H21	0.9300	C48—C49	1.378 (3)
C22—C23	1.380 (3)	C48—H48	0.9300
C22—C25	1.516 (3)	C49—H49	0.9300
C23—C24	1.371 (3)	C50—H50A	0.9600
C23—H23	0.9300	C50—H50B	0.9600
C24—H24	0.9300	C50—H50C	0.9600
C25—H25A	0.9600	N1—O3	1.4245 (17)
C25—H25B	0.9600	N2—O5	1.222 (2)
C25—H25C	0.9600	N2—O4	1.222 (2)
C26—O6	1.2174 (17)	N3—O8	1.4389 (16)
C26—C27	1.466 (2)	N4—O10	1.193 (2)
C26—C34	1.532 (2)	N4—O9	1.205 (2)
O1—C1—C2	123.70 (13)	C29—C28—C27	120.12 (19)
O1—C1—C9	121.97 (13)	C29—C28—H28	119.9
C2—C1—C9	114.30 (12)	C27—C28—H28	119.9
C7—C2—C3	118.94 (14)	C28—C29—C30	119.80 (19)
C7—C2—C1	119.87 (13)	C28—C29—H29	120.1

C3—C2—C1	121.19 (13)	C30—C29—H29	120.1
C4—C3—C2	120.46 (16)	C31—C30—C29	121.33 (18)
C4—C3—H3	119.8	C31—C30—H30	119.3
C2—C3—H3	119.8	C29—C30—H30	119.3
C3—C4—C5	119.97 (17)	C30—C31—C32	119.34 (19)
C3—C4—H4	120.0	C30—C31—H31	120.3
C5—C4—H4	120.0	C32—C31—H31	120.3
C6—C5—C4	120.87 (17)	O7—C32—C31	116.44 (16)
C6—C5—H5	119.6	O7—C32—C27	123.28 (14)
C4—C5—H5	119.6	C31—C32—C27	120.28 (17)
C5—C6—C7	119.52 (16)	O7—C33—C34	111.37 (12)
C5—C6—H6	120.2	O7—C33—C42	109.51 (13)
C7—C6—H6	120.2	C34—C33—C42	111.25 (13)
O2—C7—C6	117.00 (14)	O7—C33—H33	103.8
O2—C7—C2	122.76 (13)	C34—C33—H33	109.9
C6—C7—C2	120.23 (15)	C42—C33—H33	110.9
O2—C8—C17	110.12 (13)	O8—C34—C33	110.83 (11)
O2—C8—C9	109.52 (12)	O8—C34—C35	103.02 (11)
C17—C8—C9	112.49 (13)	C33—C34—C35	116.73 (12)
O2—C8—H8	103.9	O8—C34—C26	102.43 (10)
C17—C8—H8	110.1	C33—C34—C26	110.00 (12)
C9—C8—H8	110.3	C35—C34—C26	112.65 (11)
O3—C9—C8	108.26 (11)	C43—C35—C36	110.13 (11)
O3—C9—C10	103.38 (11)	C43—C35—C34	98.22 (11)
C8—C9—C10	118.42 (12)	C36—C35—C34	115.46 (11)
O3—C9—C1	104.48 (11)	C43—C35—H35	110.8
C8—C9—C1	109.41 (12)	C36—C35—H35	110.8
C10—C9—C1	111.76 (11)	C34—C35—H35	110.8
C18—C10—C11	113.03 (11)	C41—C36—C37	119.08 (13)
C18—C10—C9	97.56 (11)	C41—C36—C35	120.38 (12)
C11—C10—C9	114.07 (11)	C37—C36—C35	120.50 (12)
C18—C10—H10	110.5	C38—C37—C36	120.79 (13)
C11—C10—H10	110.5	C38—C37—H37	119.6
C9—C10—H10	110.5	C36—C37—H37	119.6
C16—C11—C12	118.93 (13)	C39—C38—C37	118.32 (14)
C16—C11—C10	119.63 (12)	C39—C38—H38	120.8
C12—C11—C10	121.31 (12)	C37—C38—H38	120.8
C13—C12—C11	120.70 (14)	C38—C39—C40	122.75 (14)
C13—C12—H12	119.7	C38—C39—N4	118.18 (14)
C11—C12—H12	119.7	C40—C39—N4	119.06 (14)
C14—C13—C12	118.80 (14)	C39—C40—C41	118.30 (14)
C14—C13—H13	120.6	C39—C40—H40	120.9
C12—C13—H13	120.6	C41—C40—H40	120.9
C13—C14—C15	122.21 (14)	C40—C41—C36	120.76 (14)
C13—C14—N2	118.79 (15)	C40—C41—H41	119.6
C15—C14—N2	119.01 (16)	C36—C41—H41	119.6
C14—C15—C16	118.38 (14)	C33—C42—H42A	109.5
C14—C15—H15	120.8	C33—C42—H42B	109.5

C16—C15—H15	120.8	H42A—C42—H42B	109.5
C15—C16—C11	120.93 (14)	C33—C42—H42C	109.5
C15—C16—H16	119.5	H42A—C42—H42C	109.5
C11—C16—H16	119.5	H42B—C42—H42C	109.5
C8—C17—H17A	109.5	N3—C43—C44	121.72 (13)
C8—C17—H17B	109.5	N3—C43—C35	113.51 (13)
H17A—C17—H17B	109.5	C44—C43—C35	124.77 (12)
C8—C17—H17C	109.5	C49—C44—C45	118.06 (15)
H17A—C17—H17C	109.5	C49—C44—C43	120.57 (14)
H17B—C17—H17C	109.5	C45—C44—C43	121.32 (14)
N1—C18—C19	121.23 (13)	C46—C45—C44	120.82 (16)
N1—C18—C10	113.52 (13)	C46—C45—H45	119.6
C19—C18—C10	125.20 (13)	C44—C45—H45	119.6
C20—C19—C24	118.06 (15)	C45—C46—C47	121.32 (17)
C20—C19—C18	121.30 (14)	C45—C46—H46	119.3
C24—C19—C18	120.53 (15)	C47—C46—H46	119.3
C21—C20—C19	120.48 (16)	C46—C47—C48	117.63 (16)
C21—C20—H20	119.8	C46—C47—C50	121.2 (2)
C19—C20—H20	119.8	C48—C47—C50	121.2 (2)
C22—C21—C20	121.21 (18)	C49—C48—C47	121.71 (17)
C22—C21—H21	119.4	C49—C48—H48	119.1
C20—C21—H21	119.4	C47—C48—H48	119.1
C23—C22—C21	118.18 (17)	C48—C49—C44	120.43 (17)
C23—C22—C25	121.2 (2)	C48—C49—H49	119.8
C21—C22—C25	120.6 (2)	C44—C49—H49	119.8
C24—C23—C22	121.48 (18)	C47—C50—H50A	109.5
C24—C23—H23	119.3	C47—C50—H50B	109.5
C22—C23—H23	119.3	H50A—C50—H50B	109.5
C23—C24—C19	120.58 (18)	C47—C50—H50C	109.5
C23—C24—H24	119.7	H50A—C50—H50C	109.5
C19—C24—H24	119.7	H50B—C50—H50C	109.5
C22—C25—H25A	109.5	C18—N1—O3	108.80 (12)
C22—C25—H25B	109.5	O5—N2—O4	123.94 (18)
H25A—C25—H25B	109.5	O5—N2—C14	117.70 (18)
C22—C25—H25C	109.5	O4—N2—C14	118.34 (17)
H25A—C25—H25C	109.5	C43—N3—O8	108.27 (11)
H25B—C25—H25C	109.5	O10—N4—O9	122.85 (17)
O6—C26—C27	123.78 (14)	O10—N4—C39	118.15 (16)
O6—C26—C34	121.71 (13)	O9—N4—C39	118.99 (15)
C27—C26—C34	114.29 (12)	C7—O2—C8	116.92 (11)
C32—C27—C28	119.08 (15)	N1—O3—C9	106.63 (10)
C32—C27—C26	119.90 (14)	C32—O7—C33	117.78 (12)
C28—C27—C26	120.99 (15)	N3—O8—C34	106.52 (9)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C44–C49 and C11–C16 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1^{i}	0.98	2.40	3.3466 (17)	162
C35—H35···O6^{ii}	0.98	2.25	3.2072 (17)	164
C16—H16···N3^{i}	0.93	2.56	3.3091 (19)	138
C23—H23···*Cg*1	0.93	2.95	3.581 (2)	127
C30—H30···*Cg*2^{iii}	0.93	3.00	3.758 (2)	140

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