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5-Nitro-N^2,N^3-bis[(1*R*,2*R*,4*R*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl]isophthalamide dichloromethane hemisolvate

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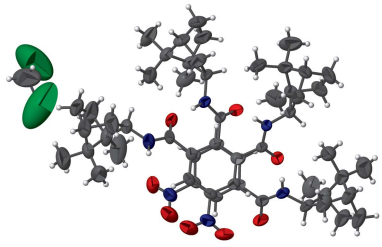
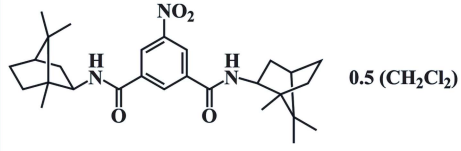
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title hemisolvate, $C_{28}H_{39}N_3O_4{\cdot}0.5CH_2Cl_2$ contains two isophthalamide camphor derivatives and one dichloromethane solvent molecule. In the crystal, the chiral molecules are connected into [100] stacks by N—H···O and C—H···O hydrogen bonds. The stacks are consolidated by C—H···N and C—H···Cl as well as further C—H···O interactions.

3D view



Chemical scheme



Structure description

The determination of the optical purity of chiral compounds by nuclear magnetic resonance spectroscopy (Laaksonen *et al.*, 2015; Li *et al.*, 2016; Pal *et al.*, 2014) with an aid of suitable homochiral additives has gained importance in recent years. The principle of the separation of signals for the enantiomers of the analyte is based on supramolecular interactions with the chiral additive such as hydrogen bonding, C—H···π, π–π interactions, *etc*. We have recently reported a chiral camphor-based Kagan's amide and explored its activity for estimation of enantiomer ratios of a wide variety of functionalized molecules (Kannappan *et al.*, 2015, 2017). As part of our ongoing studies in this area, we now report the synthesis and structure of the title compound (Fig. 1).

The asymmetric unit contains two molecules, *A* (containing C1) and *B* (containing C30), of the chiral isopthalimide compound and one dichloromethane solvent molecule (Fig. 1). In the extended structure, the main molecules are linked by N—H···O hydrogen bonds (Table 1) to generate [100] stacks (Fig. 2) of alternating *A* and *B* molecules, with an approximate rotation of 60° between adjacent molecules in the stack. The structure is

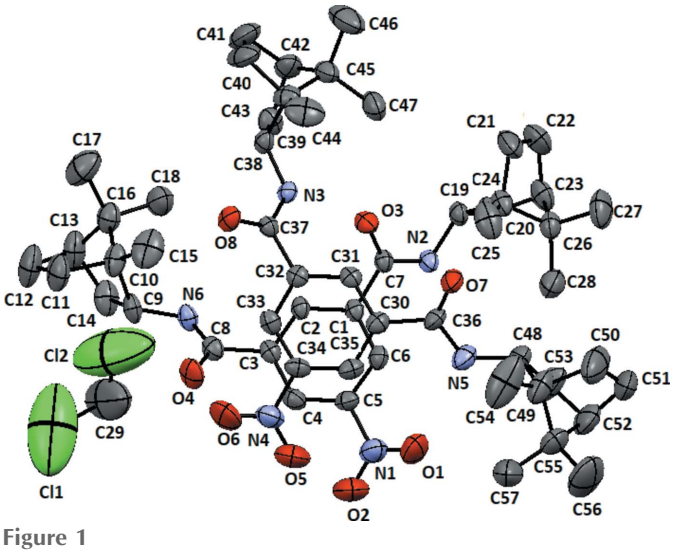


Figure 1
Asymmetric unit of title compound showing displacement ellipsoids drawn at the 40% probability level. Hydrogen atoms are omitted for clarity.

consolidated by C—H···O, C—H···Cl and C—H···N interactions (which also involve the solvent molecules as both donors and acceptors), which consolidate the chains into ribbon-like networks.

Synthesis and crystallization

To a dry 50 ml round-bottom flask containing a solution of (−)-*iso*-bornyl amine (0.25 g, 1.6 mmol) dissolved in dry chloroform (5 ml), was added triethylamine (0.57 ml, 4.1 mmol). The mixture was allowed to cool to 273 K and a solution of *iso*-phthaloyl acid chloride (0.89 g, 3.6 mmol) dissolved in chloroform was added dropwise with a syringe. The reaction mixture was then stirred at room temperature for 4 h. The solvent was evaporated under vacuum and the residue was washed with sodium bicarbonate and extracted with dichloromethane (3 × 50 ml). The organic layer was dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by short column chromatography over silica gel (50% ethyl acetate–petroleum ether) resulting in a white solid. Thin plates were recrystallized from a solvent mixture of dichloromethane/methanol at room temperature.

^{1}H NMR (400 MHz, $CDCl_3$): δ 0.90 (3H, *s*), 0.94 (3H, *s*), 1.04 (3H, *s*), 1.21–1.29 (2H, *m*), 1.35–1.41 (1H, *m*), 1.63–1.79 (3H, *m*), 1.84–1.87 (1H, *m*), 1.96–2.02 (1H, *m*), 4.11–4.16 (1H, *m*), 6.21–6.23 (1H, *d*, *J* = 3.2 Hz), 8.39 (1H, *s*), 8.64 (2H, *s*).

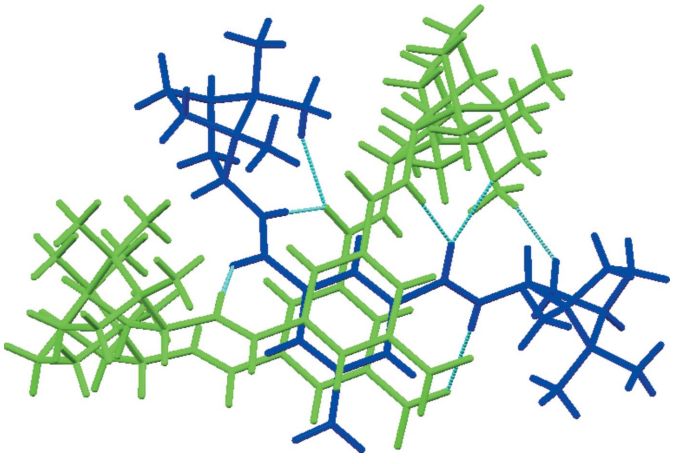


Figure 2
Detail of the extended structure showing [100] stacked layer formation of the C1 (green) and C30 (blue) molecules due to N—H···O and C—H···O hydrogen bonds

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O7	0.86 (1)	2.07 (1)	2.881 (5)	158 (1)
N3—H3···O8	0.86 (1)	2.21 (1)	3.047 (5)	165 (1)
N6—H6A···O4^{i}	0.86 (1)	2.13 (1)	2.970 (4)	165 (1)
C17—H17B···O8	0.96 (2)	2.55 (3)	3.445 (8)	156 (2)
C17—H17B···N3	0.96 (2)	2.55 (1)	3.260 (7)	131 (2)
C24—H24B···Cl1^{ii}	0.97 (1)	2.77 (1)	3.388 (8)	123 (1)
C27—H27B···O7	0.96 (4)	2.56 (4)	3.491 (8)	164 (2)
C27—H27B···N2	0.96 (4)	2.60 (3)	3.218 (8)	122 (3)
C29—H29A···O6^{iii}	0.97 (2)	2.49 (1)	3.229 (14)	133 (1)
C29—H29B···O3^{iv}	0.97 (2)	2.41 (1)	3.046 (14)	123 (1)
C44—H44B···N6	0.96 (3)	2.62 (3)	2.940 (7)	100 (2)
C47—H47B···O4^{i}	0.96 (3)	2.42 (3)	3.331 (8)	158 (1)
C50—H50B···Cl2	0.97 (1)	2.72 (1)	3.617 (9)	154 (1)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_{28}H_{39}N_3O_4{\cdot}CH_2Cl_2$
M_r	1048.17
Crystal system, space group	Triclinic, *P*1
Temperature (K)	293
a, *b*, *c* (Å)	7.2828 (4), 12.6801 (8), 16.9826 (9)
α, β, γ (°)	70.373 (5), 78.541 (5), 78.791 (5)
V (Å^3)	1433.81 (15)
Z	1
Radiation type	Mo *K*α
μ (mm^{-1})	0.17
Crystal size (mm)	0.25 × 0.12 × 0.08
Data collection	
Diffractometer	Agilent Xcalibur, Eos, Gemini
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2017)
T_{min}, T_{max}	0.912, 1.000
No. of measured, independent and observed [$I \geq 2\sigma(I)$] reflections	9570, 8140, 5575
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.684
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.072, 0.208, 1.07
No. of reflections	8140
No. of parameters	671
No. of restraints	7
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.36
Absolute structure	Flack (1983)
Absolute structure parameter	0.1 (3)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2017), *SHELXD* (Sheldrick, 2008), *olex2.refine* (Bourhis *et al.*, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

^{13}C NMR (100 MHz, $CDCl_3$): δ 11.9, 20.2, 20.5, 27.0, 35.9, 38.9, 44.9, 47.3, 49.1, 57.9, 123.9, 130.6, 137.2, 148.3, 163.8.

IR (KBr): ν (cm^{-1}) 3266, 3067, 2953, 1638, 1537, 1457, 1389, 1352, 1114, 979.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Bond length restraints were applied to model the solvent molecule.

Acknowledgements

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full crystallographic data

IUCrData (2018). **3**, x180741 [https://doi.org/10.1107/S2414314618007411]

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Crystal data

$2C_{28}H_{39}N_3O_4{\cdot}CH_2Cl_2$
M_r = 1048.17
Triclinic, $P\overline{1}$
a = 7.2828 (4) Å
b = 12.6801 (8) Å
c = 16.9826 (9) Å
α = 70.373 (5)°
β = 78.541 (5)°
γ = 78.791 (5)°
V = 1433.81 (15) Å^3
Z = 1
$F(000)$ = 1433.81
D_x = 1.214 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1895 reflections
θ = 3.8–24.9°
μ = 0.17 mm^{-1}
T = 293 K
Plate, colourless
0.25 × 0.12 × 0.08 mm

Data collection

Agilent Xcalibur, Eos, Gemini diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1702 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2017)
T_{min} = 0.912, T_{max} = 1.000
9570 measured reflections
8140 independent reflections
5575 reflections with $I \geq 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 29.1°, θ_{min} = 3.4°
h = −9→8
k = −15→16
l = −21→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.208
S = 1.07
8140 reflections
671 parameters
7 restraints
123 constraints
Primary atom site location: dual
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1179P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.1 (3)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C3	−0.0709 (5)	−0.6303 (3)	−0.4855 (3)	0.0378 (9)

O7	0.3557 (4)	−0.5252 (3)	−0.6951 (2)	0.0519 (8)
C37	0.4311 (5)	−0.5267 (3)	−0.6350 (3)	0.0374 (9)
N3	−0.0898 (4)	−0.5098 (3)	−0.3990 (2)	0.0432 (8)
H3	−0.0138 (4)	−0.5626 (3)	−0.3701 (2)	0.0518 (10)*
C31	0.4213 (5)	−0.6375 (4)	−0.4816 (3)	0.0380 (9)
H31	0.3742 (5)	−0.5708 (4)	−0.4686 (3)	0.0456 (11)*
C4	−0.0434 (6)	−0.7339 (4)	−0.4242 (3)	0.0453 (10)
H4	−0.0639 (6)	−0.7397 (4)	−0.3672 (3)	0.0544 (12)*
C30	0.4655 (5)	−0.6342 (3)	−0.5655 (3)	0.0381 (9)
C32	0.4458 (6)	−0.7382 (4)	−0.4163 (3)	0.0445 (10)
C1	0.0193 (5)	−0.7214 (4)	−0.5955 (3)	0.0393 (9)
C2	−0.0385 (5)	−0.6258 (4)	−0.5707 (3)	0.0388 (9)
H2	−0.0567 (5)	−0.5560 (4)	−0.6118 (3)	0.0465 (11)*
C6	0.0454 (6)	−0.8266 (4)	−0.5334 (3)	0.0480 (11)
H6	0.0819 (6)	−0.8929 (4)	−0.5479 (3)	0.0576 (13)*
C33	0.5191 (6)	−0.8373 (4)	−0.4348 (3)	0.0463 (11)
H33	0.5413 (6)	−0.9049 (4)	−0.3920 (3)	0.0556 (13)*
C34	0.5589 (6)	−0.8337 (4)	−0.5190 (3)	0.0461 (10)
O4	−0.2644 (4)	−0.4548 (3)	−0.5058 (2)	0.0537 (8)
N6	0.4826 (4)	−0.4350 (3)	−0.6288 (2)	0.0390 (8)
H6a	0.5362 (4)	−0.4417 (3)	−0.5863 (2)	0.0468 (9)*
O8	0.2376 (5)	−0.6668 (3)	−0.3150 (2)	0.0612 (9)
N2	0.1087 (5)	−0.6359 (3)	−0.7448 (2)	0.0479 (9)
H2a	0.1553 (5)	−0.5897 (3)	−0.7296 (2)	0.0575 (11)*
C8	−0.1496 (5)	−0.5243 (4)	−0.4641 (3)	0.0388 (9)
C5	0.0159 (6)	−0.8289 (4)	−0.4508 (3)	0.0460 (10)
N4	0.6336 (6)	−0.9388 (4)	−0.5380 (3)	0.0668 (12)
C38	0.4524 (6)	−0.3238 (4)	−0.6909 (3)	0.0440 (10)
H38	0.4527 (6)	−0.3341 (4)	−0.7456 (3)	0.0529 (12)*
C7	0.0301 (6)	−0.7189 (4)	−0.6851 (3)	0.0477 (11)
N1	0.0340 (7)	−0.9410 (4)	−0.3838 (3)	0.0726 (13)
O3	−0.0381 (5)	−0.7917 (3)	−0.6991 (2)	0.0686 (10)
C36	0.3776 (6)	−0.7343 (4)	−0.3277 (3)	0.0483 (11)
N5	0.4729 (5)	−0.8057 (4)	−0.2668 (3)	0.0609 (11)
H5	0.5707 (5)	−0.8501 (4)	−0.2800 (3)	0.0731 (14)*
C10	−0.3277 (6)	−0.4052 (4)	−0.3073 (3)	0.0526 (11)
C19	0.1209 (6)	−0.6182 (5)	−0.8350 (3)	0.0537 (12)
H19	0.1230 (6)	−0.6918 (5)	−0.8425 (3)	0.0645 (14)*
C9	−0.1521 (6)	−0.4051 (4)	−0.3766 (3)	0.0455 (10)
H9	−0.1785 (6)	−0.3430 (4)	−0.4278 (3)	0.0546 (12)*
C48	0.4155 (8)	−0.8101 (5)	−0.1790 (3)	0.0683 (15)
H48	0.3501 (8)	−0.7353 (5)	−0.1777 (3)	0.0820 (18)*
O6	0.6104 (7)	−0.9452 (4)	−0.6048 (4)	0.1045 (16)
C39	0.2671 (7)	−0.2463 (4)	−0.6711 (3)	0.0579 (13)
C20	−0.0443 (6)	−0.5350 (5)	−0.8790 (3)	0.0617 (14)
C43	0.6074 (7)	−0.2506 (4)	−0.7011 (3)	0.0624 (14)
H43a	0.6826 (7)	−0.2825 (4)	−0.6553 (3)	0.0748 (16)*
H43b	0.6902 (7)	−0.2435 (4)	−0.7545 (3)	0.0748 (16)*

C16	−0.2411 (7)	−0.4367 (4)	−0.2257 (3)	0.0573 (12)
C13	−0.1110 (8)	−0.3453 (5)	−0.2620 (4)	0.0701 (15)
H13	−0.0326 (8)	−0.3430 (5)	−0.2221 (4)	0.0841 (18)*
O2	0.1482 (8)	−1.0153 (4)	−0.4026 (4)	0.120 (2)
C49	0.2870 (8)	−0.8980 (6)	−0.1239 (4)	0.0806 (19)
C14	0.0015 (7)	−0.3762 (5)	−0.3387 (3)	0.0670 (15)
H14a	0.0977 (7)	−0.4408 (5)	−0.3222 (3)	0.0804 (18)*
H14b	0.0612 (7)	−0.3131 (5)	−0.3785 (3)	0.0804 (18)*
O5	0.7140 (7)	−1.0161 (4)	−0.4861 (4)	0.1091 (17)
O1	−0.0738 (7)	−0.9495 (4)	−0.3167 (3)	0.0989 (15)
C42	0.4952 (9)	−0.1375 (4)	−0.6987 (4)	0.0720 (16)
H42	0.5688 (9)	−0.0864 (4)	−0.6899 (4)	0.0865 (19)*
C23	0.2189 (9)	−0.4713 (6)	−0.9612 (4)	0.0805 (18)
H23	0.3068 (9)	−0.4169 (6)	−0.9929 (4)	0.097 (2)*
C24	0.2968 (7)	−0.5675 (6)	−0.8901 (3)	0.0726 (16)
H24a	0.3625 (7)	−0.5401 (6)	−0.8581 (3)	0.087 (2)*
H24b	0.3834 (7)	−0.6232 (6)	−0.9117 (3)	0.087 (2)*
C55	0.4306 (9)	−0.9997 (5)	−0.0804 (4)	0.0755 (17)
C40	0.2408 (10)	−0.1600 (5)	−0.7582 (5)	0.093 (2)
H40a	0.2552 (10)	−0.1977 (5)	−0.8006 (5)	0.112 (3)*
H40b	0.1168 (10)	−0.1151 (5)	−0.7564 (5)	0.112 (3)*
C45	0.3326 (8)	−0.1729 (5)	−0.6279 (4)	0.0674 (14)
C25	−0.2354 (7)	−0.5368 (7)	−0.8249 (5)	0.098 (2)
H25a	−0.268 (4)	−0.6119 (13)	−0.806 (3)	0.146 (4)*
H25b	−0.231 (2)	−0.514 (5)	−0.777 (2)	0.146 (4)*
H25c	−0.3285 (16)	−0.486 (4)	−0.8575 (12)	0.146 (4)*
C52	0.5194 (11)	−0.9291 (6)	−0.0480 (4)	0.096 (2)
H52	0.6207 (11)	−0.9719 (6)	−0.0149 (4)	0.116 (3)*
C44	0.0985 (7)	−0.3089 (6)	−0.6266 (5)	0.093 (2)
H44a	0.064 (5)	−0.343 (4)	−0.6634 (13)	0.140 (3)*
H44b	0.131 (3)	−0.367 (3)	−0.5761 (19)	0.140 (3)*
H44c	−0.006 (3)	−0.2567 (11)	−0.612 (3)	0.140 (3)*
C11	−0.3923 (10)	−0.2764 (6)	−0.3238 (5)	0.095 (2)
H11a	−0.3969 (10)	−0.2379 (6)	−0.3834 (5)	0.114 (3)*
H11b	−0.5165 (10)	−0.2626 (6)	−0.2924 (5)	0.114 (3)*
C17	−0.1421 (9)	−0.5574 (5)	−0.1954 (4)	0.0795 (17)
H17a	−0.2348 (9)	−0.6084 (6)	−0.173 (3)	0.119 (3)*
H17b	−0.060 (5)	−0.5758 (15)	−0.2420 (7)	0.119 (3)*
H17c	−0.069 (6)	−0.5641 (11)	−0.152 (2)	0.119 (3)*
C15	−0.4780 (7)	−0.4719 (6)	−0.3099 (4)	0.083 (2)
H15a	−0.524 (5)	−0.440 (3)	−0.3634 (13)	0.125 (3)*
H15b	−0.424 (2)	−0.5493 (11)	−0.303 (3)	0.125 (3)*
H15c	−0.581 (3)	−0.468 (4)	−0.265 (2)	0.125 (3)*
C26	0.0388 (8)	−0.4217 (5)	−0.9172 (4)	0.0673 (14)
C21	−0.0349 (9)	−0.5685 (6)	−0.9593 (4)	0.087 (2)
H21a	−0.0267 (9)	−0.6497 (6)	−0.9457 (4)	0.105 (2)*
H21b	−0.1453 (9)	−0.5323 (6)	−0.9871 (4)	0.105 (2)*
C47	0.3889 (9)	−0.2378 (6)	−0.5404 (4)	0.0818 (18)

H47a	0.289 (3)	−0.280 (3)	−0.5059 (11)	0.123 (3)*
H47b	0.503 (4)	−0.289 (3)	−0.5460 (5)	0.123 (3)*
H47c	0.410 (7)	−0.1855 (6)	−0.5144 (13)	0.123 (3)*
C18	−0.3868 (9)	−0.4180 (7)	−0.1495 (4)	0.099 (2)
H18a	−0.462 (6)	−0.346 (2)	−0.1671 (10)	0.149 (4)*
H18b	−0.467 (5)	−0.476 (3)	−0.129 (2)	0.149 (4)*
H18c	−0.3210 (9)	−0.421 (5)	−0.1050 (15)	0.149 (4)*
C22	0.1465 (11)	−0.5258 (7)	−1.0155 (4)	0.104 (3)
H22a	0.1185 (11)	−0.4709 (7)	−1.0688 (4)	0.125 (3)*
H22b	0.2377 (11)	−0.5878 (7)	−1.0265 (4)	0.125 (3)*
C41	0.3960 (11)	−0.0865 (5)	−0.7770 (5)	0.105 (3)
H41a	0.3433 (11)	−0.0081 (5)	−0.7838 (5)	0.125 (3)*
H41b	0.4822 (11)	−0.0911 (5)	−0.8276 (5)	0.125 (3)*
C27	0.0718 (10)	−0.3712 (6)	−0.8505 (4)	0.089 (2)
H27a	−0.0468 (14)	−0.355 (4)	−0.817 (2)	0.134 (3)*
H27b	0.156 (6)	−0.4246 (18)	−0.814 (2)	0.134 (3)*
H27c	0.127 (7)	−0.303 (2)	−0.8786 (4)	0.134 (3)*
C12	−0.2438 (11)	−0.2364 (6)	−0.2932 (5)	0.097 (2)
H12a	−0.2994 (11)	−0.2045 (6)	−0.2478 (5)	0.117 (3)*
H12b	−0.1788 (11)	−0.1806 (6)	−0.3388 (5)	0.117 (3)*
C46	0.1869 (11)	−0.0719 (7)	−0.6189 (6)	0.122 (3)
H46a	0.154 (8)	−0.027 (3)	−0.6734 (9)	0.183 (5)*
H46b	0.076 (4)	−0.0983 (7)	−0.582 (4)	0.183 (5)*
H46c	0.239 (4)	−0.027 (3)	−0.596 (4)	0.183 (5)*
C51	0.3585 (14)	−0.8722 (7)	0.0006 (5)	0.143 (4)
H51a	0.3297 (14)	−0.9203 (7)	0.0582 (5)	0.172 (5)*
H51b	0.3869 (14)	−0.8010 (7)	0.0014 (5)	0.172 (5)*
C28	−0.0819 (11)	−0.3269 (7)	−0.9774 (5)	0.116 (3)
H28a	−0.108 (8)	−0.354 (2)	−1.020 (3)	0.174 (4)*
H28b	−0.199 (4)	−0.304 (4)	−0.9458 (9)	0.174 (4)*
H28c	−0.014 (4)	−0.263 (2)	−1.004 (3)	0.174 (4)*
C53	0.5879 (11)	−0.8391 (6)	−0.1297 (5)	0.110 (3)
H53a	0.7014 (11)	−0.8693 (6)	−0.1604 (5)	0.133 (3)*
H53b	0.6120 (11)	−0.7735 (6)	−0.1183 (5)	0.133 (3)*
C56	0.5597 (14)	−1.0575 (7)	−0.1403 (5)	0.136 (4)
H56a	0.4850 (16)	−1.076 (6)	−0.173 (4)	0.205 (6)*
H56b	0.647 (8)	−1.008 (3)	−0.177 (4)	0.205 (6)*
H56c	0.629 (9)	−1.126 (4)	−0.1084 (6)	0.205 (6)*
C54	0.1448 (10)	−0.9134 (9)	−0.1727 (5)	0.148 (5)
H54a	0.085 (10)	−0.8407 (10)	−0.203 (5)	0.222 (7)*
H54b	0.209 (3)	−0.953 (7)	−0.212 (5)	0.222 (7)*
H54c	0.051 (8)	−0.956 (7)	−0.1339 (8)	0.222 (7)*
C35	0.5289 (6)	−0.7347 (4)	−0.5843 (3)	0.0434 (10)
H35	0.5509 (6)	−0.7354 (4)	−0.6400 (3)	0.0521 (12)*
C57	0.3378 (15)	−1.0945 (7)	−0.0075 (6)	0.160 (5)
H57a	0.4348 (15)	−1.151 (5)	0.019 (4)	0.241 (7)*
H57b	0.259 (12)	−1.0626 (17)	0.034 (3)	0.241 (7)*
H57c	0.262 (12)	−1.129 (6)	−0.0298 (12)	0.241 (7)*

C50	0.2002 (12)	−0.8543 (9)	−0.0482 (5)	0.143 (4)
H50a	0.1500 (12)	−0.7747 (9)	−0.0675 (5)	0.172 (5)*
H50b	0.0990 (12)	−0.8961 (9)	−0.0136 (5)	0.172 (5)*
Cl2	−0.1590 (3)	−0.9594 (3)	0.1272 (2)	0.411 (7)
Cl1	−0.4320 (3)	−0.8155 (3)	0.1924 (3)	0.400 (7)
C29	−0.2136 (16)	−0.8913 (11)	0.1980 (8)	0.273 (12)
H29a	−0.2098 (16)	−0.9451 (11)	0.2541 (8)	0.327 (14)*
H29b	−0.1211 (16)	−0.8409 (11)	0.1884 (8)	0.327 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C3	0.033 (2)	0.049 (2)	0.038 (2)	−0.0048 (17)	−0.0108 (17)	−0.0196 (19)
O7	0.0617 (18)	0.0554 (19)	0.0487 (18)	−0.0157 (15)	−0.0255 (15)	−0.0151 (15)
C37	0.035 (2)	0.042 (2)	0.039 (2)	−0.0037 (16)	−0.0064 (17)	−0.0181 (18)
N3	0.0421 (19)	0.052 (2)	0.0399 (19)	0.0040 (15)	−0.0147 (15)	−0.0207 (17)
C31	0.0269 (19)	0.044 (2)	0.045 (2)	−0.0029 (16)	−0.0063 (16)	−0.0171 (19)
C4	0.049 (2)	0.054 (3)	0.037 (2)	−0.0068 (19)	−0.0133 (19)	−0.015 (2)
C30	0.0282 (19)	0.044 (2)	0.045 (2)	−0.0062 (17)	−0.0096 (17)	−0.0145 (19)
C32	0.037 (2)	0.047 (3)	0.045 (2)	−0.0049 (18)	−0.0073 (18)	−0.009 (2)
C1	0.0301 (19)	0.048 (2)	0.045 (2)	−0.0043 (17)	−0.0059 (17)	−0.022 (2)
C2	0.038 (2)	0.049 (2)	0.035 (2)	−0.0053 (18)	−0.0104 (17)	−0.0180 (18)
C6	0.052 (3)	0.042 (2)	0.058 (3)	−0.0022 (19)	−0.015 (2)	−0.024 (2)
C33	0.040 (2)	0.039 (2)	0.052 (3)	−0.0037 (18)	−0.0091 (19)	−0.004 (2)
C34	0.041 (2)	0.035 (2)	0.061 (3)	−0.0061 (18)	−0.006 (2)	−0.014 (2)
O4	0.0506 (18)	0.0557 (19)	0.062 (2)	0.0124 (14)	−0.0295 (16)	−0.0272 (16)
N6	0.0434 (18)	0.0413 (18)	0.0370 (18)	−0.0046 (14)	−0.0180 (14)	−0.0120 (15)
O8	0.065 (2)	0.067 (2)	0.0429 (18)	0.0176 (17)	−0.0156 (15)	−0.0153 (16)
N2	0.052 (2)	0.058 (2)	0.047 (2)	−0.0166 (18)	−0.0068 (17)	−0.0286 (18)
C8	0.036 (2)	0.049 (2)	0.035 (2)	−0.0047 (18)	−0.0078 (17)	−0.0174 (18)
C5	0.050 (2)	0.035 (2)	0.052 (3)	−0.0025 (18)	−0.019 (2)	−0.008 (2)
N4	0.074 (3)	0.046 (3)	0.078 (3)	−0.002 (2)	−0.003 (3)	−0.023 (3)
C38	0.053 (2)	0.048 (2)	0.036 (2)	−0.0126 (19)	−0.0121 (18)	−0.0133 (19)
C7	0.045 (2)	0.055 (3)	0.053 (3)	−0.002 (2)	−0.016 (2)	−0.026 (2)
N1	0.103 (4)	0.048 (3)	0.068 (3)	−0.009 (3)	−0.030 (3)	−0.011 (2)
O3	0.090 (3)	0.072 (2)	0.066 (2)	−0.031 (2)	−0.0143 (19)	−0.0379 (19)
C36	0.053 (3)	0.047 (2)	0.038 (2)	−0.001 (2)	−0.017 (2)	−0.001 (2)
N5	0.055 (2)	0.066 (3)	0.049 (2)	0.0038 (19)	−0.0124 (19)	−0.005 (2)
C10	0.041 (2)	0.071 (3)	0.055 (3)	0.004 (2)	−0.015 (2)	−0.032 (2)
C19	0.055 (3)	0.075 (3)	0.045 (3)	−0.017 (2)	−0.004 (2)	−0.035 (2)
C9	0.052 (2)	0.053 (3)	0.039 (2)	−0.0063 (19)	−0.0105 (19)	−0.022 (2)
C48	0.096 (4)	0.059 (3)	0.046 (3)	0.002 (3)	−0.026 (3)	−0.009 (2)
O6	0.156 (5)	0.068 (3)	0.106 (4)	−0.017 (3)	−0.014 (3)	−0.051 (3)
C39	0.057 (3)	0.050 (3)	0.068 (3)	0.003 (2)	−0.032 (2)	−0.014 (2)
C20	0.046 (3)	0.100 (4)	0.048 (3)	−0.020 (3)	−0.013 (2)	−0.025 (3)
C43	0.062 (3)	0.067 (3)	0.060 (3)	−0.021 (2)	0.005 (2)	−0.023 (3)
C16	0.052 (3)	0.076 (3)	0.046 (3)	−0.002 (2)	−0.006 (2)	−0.027 (3)
C13	0.078 (4)	0.084 (4)	0.071 (4)	−0.014 (3)	−0.015 (3)	−0.049 (3)

O2	0.171 (5)	0.059 (3)	0.111 (4)	0.029 (3)	−0.039 (4)	−0.017 (3)
C49	0.069 (3)	0.110 (5)	0.047 (3)	−0.012 (3)	−0.007 (3)	−0.004 (3)
C14	0.061 (3)	0.093 (4)	0.065 (3)	−0.032 (3)	−0.001 (2)	−0.041 (3)
O5	0.142 (4)	0.057 (3)	0.115 (4)	0.024 (3)	−0.024 (3)	−0.030 (3)
O1	0.154 (4)	0.075 (3)	0.057 (3)	−0.028 (3)	−0.009 (3)	−0.003 (2)
C42	0.115 (5)	0.043 (3)	0.066 (4)	−0.019 (3)	−0.029 (3)	−0.012 (3)
C23	0.085 (4)	0.122 (5)	0.048 (3)	−0.045 (4)	0.002 (3)	−0.033 (3)
C24	0.046 (3)	0.119 (5)	0.062 (3)	−0.024 (3)	0.004 (2)	−0.038 (3)
C55	0.098 (4)	0.060 (3)	0.056 (3)	−0.014 (3)	−0.011 (3)	0.001 (3)
C40	0.140 (6)	0.050 (3)	0.098 (5)	0.010 (3)	−0.082 (5)	−0.010 (3)
C45	0.085 (4)	0.055 (3)	0.067 (3)	0.021 (3)	−0.033 (3)	−0.031 (3)
C25	0.044 (3)	0.155 (7)	0.090 (5)	−0.013 (3)	−0.017 (3)	−0.030 (5)
C52	0.136 (6)	0.090 (5)	0.061 (4)	−0.005 (4)	−0.053 (4)	−0.004 (4)
C44	0.037 (3)	0.091 (4)	0.147 (7)	0.005 (3)	−0.012 (3)	−0.039 (4)
C11	0.114 (5)	0.096 (5)	0.080 (4)	0.035 (4)	−0.023 (4)	−0.056 (4)
C17	0.089 (4)	0.085 (4)	0.056 (3)	0.002 (3)	−0.014 (3)	−0.016 (3)
C15	0.042 (3)	0.138 (6)	0.091 (4)	−0.013 (3)	−0.004 (3)	−0.065 (4)
C26	0.072 (3)	0.082 (4)	0.056 (3)	−0.018 (3)	−0.017 (3)	−0.024 (3)
C21	0.106 (5)	0.119 (6)	0.057 (4)	−0.039 (4)	−0.023 (3)	−0.035 (4)
C47	0.099 (4)	0.089 (4)	0.070 (4)	0.009 (3)	−0.023 (3)	−0.047 (3)
C18	0.085 (4)	0.154 (7)	0.058 (4)	0.000 (4)	0.005 (3)	−0.048 (4)
C22	0.129 (6)	0.150 (7)	0.047 (3)	−0.058 (5)	−0.012 (4)	−0.027 (4)
C41	0.172 (7)	0.055 (4)	0.082 (5)	−0.010 (4)	−0.063 (5)	0.007 (3)
C27	0.133 (6)	0.073 (4)	0.072 (4)	−0.013 (4)	−0.032 (4)	−0.026 (3)
C12	0.139 (6)	0.078 (4)	0.093 (5)	−0.005 (4)	−0.020 (4)	−0.054 (4)
C46	0.129 (7)	0.104 (6)	0.155 (8)	0.052 (5)	−0.062 (6)	−0.083 (6)
C51	0.256 (12)	0.103 (6)	0.052 (4)	0.054 (7)	−0.052 (6)	−0.027 (4)
C28	0.137 (7)	0.116 (6)	0.095 (6)	−0.026 (5)	−0.059 (5)	−0.003 (5)
C53	0.147 (7)	0.103 (5)	0.087 (5)	−0.052 (5)	−0.065 (5)	0.010 (4)
C56	0.217 (10)	0.085 (5)	0.079 (5)	0.045 (6)	−0.022 (6)	−0.025 (4)
C54	0.101 (6)	0.237 (11)	0.095 (6)	−0.098 (7)	−0.024 (5)	0.010 (7)
C35	0.038 (2)	0.052 (3)	0.047 (2)	−0.0126 (19)	−0.0033 (18)	−0.023 (2)
C57	0.274 (14)	0.112 (7)	0.088 (6)	−0.091 (8)	−0.025 (8)	0.015 (5)
C50	0.146 (7)	0.170 (9)	0.060 (5)	0.049 (7)	0.009 (5)	−0.018 (5)
Cl2	0.201 (5)	0.82 (2)	0.249 (7)	−0.106 (9)	−0.013 (5)	−0.207 (11)
Cl1	0.445 (12)	0.220 (6)	0.561 (16)	−0.012 (7)	−0.333 (12)	−0.022 (8)
C29	0.50 (3)	0.163 (12)	0.243 (19)	0.050 (16)	−0.25 (2)	−0.111 (13)

Geometric parameters (Å, º)

C3—C4	1.383 (6)	C19—C20	1.566 (7)
C3—C2	1.401 (5)	C19—C24	1.539 (7)
C3—C8	1.489 (6)	C9—C14	1.550 (6)
O7—C37	1.245 (5)	C48—C49	1.533 (8)
C37—C30	1.489 (6)	C48—C53	1.563 (9)
C37—N6	1.330 (5)	C39—C40	1.538 (8)
N3—C8	1.342 (5)	C39—C45	1.551 (8)
N3—C9	1.467 (6)	C39—C44	1.522 (8)

C31—C30	1.386 (6)	C20—C25	1.508 (8)
C31—C32	1.388 (6)	C20—C26	1.551 (8)
C4—C5	1.389 (6)	C20—C21	1.543 (8)
C30—C35	1.388 (6)	C43—C42	1.516 (8)
C32—C33	1.379 (6)	C16—C13	1.528 (8)
C32—C36	1.503 (7)	C16—C17	1.522 (8)
C1—C2	1.378 (6)	C16—C18	1.555 (7)
C1—C6	1.401 (6)	C13—C14	1.521 (7)
C1—C7	1.497 (6)	C13—C12	1.521 (9)
C6—C5	1.366 (7)	C49—C55	1.563 (8)
C33—C34	1.388 (7)	C49—C54	1.525 (10)
C34—N4	1.453 (6)	C49—C50	1.543 (10)
C34—C35	1.382 (6)	C42—C45	1.525 (8)
O4—C8	1.228 (5)	C42—C41	1.527 (9)
N6—C38	1.456 (5)	C23—C24	1.511 (9)
O8—C36	1.231 (5)	C23—C26	1.510 (9)
N2—C7	1.323 (6)	C23—C22	1.540 (9)
N2—C19	1.458 (6)	C55—C52	1.489 (10)
C5—N1	1.494 (6)	C55—C56	1.521 (10)
N4—O6	1.209 (7)	C55—C57	1.555 (9)
N4—O5	1.218 (6)	C40—C41	1.522 (10)
C38—C39	1.557 (6)	C45—C47	1.531 (8)
C38—C43	1.545 (6)	C45—C46	1.525 (8)
C7—O3	1.236 (5)	C52—C51	1.504 (11)
N1—O2	1.213 (6)	C52—C53	1.536 (9)
N1—O1	1.234 (7)	C11—C12	1.520 (10)
C36—N5	1.335 (6)	C26—C27	1.550 (9)
N5—C48	1.453 (7)	C26—C28	1.544 (9)
C10—C9	1.556 (6)	C21—C22	1.542 (9)
C10—C16	1.536 (7)	C51—C50	1.489 (13)
C10—C11	1.554 (8)	Cl2—C29	1.651 (9)
C10—C15	1.523 (7)	Cl1—C29	1.693 (9)

C2—C3—C4	119.1 (4)	C44—C39—C38	114.5 (4)
C8—C3—C4	122.2 (4)	C44—C39—C40	114.7 (5)
C8—C3—C2	118.5 (4)	C44—C39—C45	118.6 (5)
C30—C37—O7	119.9 (4)	C25—C20—C19	115.1 (4)
N6—C37—O7	122.9 (4)	C26—C20—C19	102.7 (4)
N6—C37—C30	117.2 (4)	C26—C20—C25	118.7 (5)
C9—N3—C8	121.0 (3)	C21—C20—C19	103.0 (5)
C32—C31—C30	121.6 (4)	C21—C20—C25	114.2 (5)
C5—C4—C3	117.6 (4)	C21—C20—C26	100.9 (4)
C31—C30—C37	121.2 (4)	C42—C43—C38	103.2 (4)
C35—C30—C37	119.8 (4)	C13—C16—C10	93.8 (4)
C35—C30—C31	118.9 (4)	C17—C16—C10	115.8 (4)
C33—C32—C31	119.7 (4)	C17—C16—C13	115.5 (4)
C36—C32—C31	117.3 (4)	C18—C16—C10	113.4 (4)
C36—C32—C33	122.9 (4)	C18—C16—C13	112.2 (5)

C6—C1—C2	118.8 (4)	C18—C16—C17	106.2 (5)
C7—C1—C2	122.2 (4)	C14—C13—C16	101.6 (4)
C7—C1—C6	118.5 (4)	C12—C13—C16	104.3 (5)
C1—C2—C3	122.2 (4)	C12—C13—C14	108.2 (5)
C5—C6—C1	118.1 (4)	C55—C49—C48	102.8 (4)
C34—C33—C32	118.4 (4)	C54—C49—C48	112.1 (5)
N4—C34—C33	118.1 (4)	C54—C49—C55	121.3 (7)
C35—C34—C33	122.3 (4)	C50—C49—C48	103.4 (6)
C35—C34—N4	119.5 (4)	C50—C49—C55	99.5 (5)
C38—N6—C37	123.0 (3)	C50—C49—C54	115.4 (7)
C19—N2—C7	123.9 (4)	C13—C14—C9	102.8 (4)
N3—C8—C3	117.5 (3)	C45—C42—C43	102.1 (4)
O4—C8—C3	119.4 (4)	C41—C42—C43	108.6 (5)
O4—C8—N3	123.2 (4)	C41—C42—C45	102.8 (5)
C6—C5—C4	124.2 (4)	C26—C23—C24	102.9 (4)
N1—C5—C4	117.1 (4)	C22—C23—C24	106.3 (6)
N1—C5—C6	118.6 (4)	C22—C23—C26	103.1 (5)
O6—N4—C34	117.9 (5)	C23—C24—C19	104.3 (4)
O5—N4—C34	118.8 (5)	C52—C55—C49	93.5 (5)
O5—N4—O6	123.3 (5)	C56—C55—C49	114.3 (5)
C39—C38—N6	116.0 (4)	C56—C55—C52	116.3 (7)
C43—C38—N6	112.9 (4)	C57—C55—C49	114.5 (6)
C43—C38—C39	102.7 (4)	C57—C55—C52	112.0 (6)
N2—C7—C1	117.2 (4)	C57—C55—C56	106.3 (6)
O3—C7—C1	118.8 (4)	C41—C40—C39	104.4 (5)
O3—C7—N2	124.0 (4)	C42—C45—C39	93.6 (4)
O2—N1—C5	116.3 (5)	C47—C45—C39	114.4 (5)
O1—N1—C5	116.7 (5)	C47—C45—C42	115.7 (5)
O1—N1—O2	126.9 (5)	C46—C45—C39	113.7 (5)
O8—C36—C32	118.8 (4)	C46—C45—C42	111.9 (6)
N5—C36—C32	117.4 (4)	C46—C45—C47	107.3 (5)
N5—C36—O8	123.7 (4)	C51—C52—C55	105.2 (7)
C48—N5—C36	121.6 (4)	C53—C52—C55	102.2 (5)
C16—C10—C9	103.6 (3)	C53—C52—C51	108.0 (7)
C11—C10—C9	101.2 (4)	C12—C11—C10	104.7 (5)
C11—C10—C16	101.2 (4)	C23—C26—C20	94.5 (5)
C15—C10—C9	114.5 (4)	C27—C26—C20	114.2 (5)
C15—C10—C16	118.8 (5)	C27—C26—C23	113.7 (5)
C15—C10—C11	115.1 (5)	C28—C26—C20	114.4 (5)
C20—C19—N2	115.8 (4)	C28—C26—C23	114.1 (5)
C24—C19—N2	115.0 (4)	C28—C26—C27	106.0 (6)
C24—C19—C20	102.2 (4)	C22—C21—C20	104.1 (5)
C10—C9—N3	116.5 (4)	C21—C22—C23	102.4 (5)
C14—C9—N3	111.7 (4)	C40—C41—C42	102.8 (5)
C14—C9—C10	102.5 (4)	C11—C12—C13	102.2 (5)
C49—C48—N5	117.2 (5)	C50—C51—C52	102.5 (6)
C53—C48—N5	112.3 (5)	C52—C53—C48	101.6 (5)
C53—C48—C49	103.0 (4)	C34—C35—C30	118.9 (4)

C40—C39—C38	102.9 (5)	C51—C50—C49	105.4 (6)
C45—C39—C38	103.1 (4)	Cl1—C29—Cl2	110.8 (6)
C45—C39—C40	100.9 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O7	0.86 (1)	2.07 (1)	2.881 (5)	158 (1)
N3—H3···O8	0.86 (1)	2.21 (1)	3.047 (5)	165 (1)
N6—H6A···O4^{i}	0.86 (1)	2.13 (1)	2.970 (4)	165 (1)
C17—H17B···O8	0.96 (2)	2.55 (3)	3.445 (8)	156 (2)
C17—H17B···N3	0.96 (2)	2.55 (1)	3.260 (7)	131 (2)
C24—H24B···Cl1^{ii}	0.97 (1)	2.77 (1)	3.388 (8)	123 (1)
C27—H27B···O7	0.96 (4)	2.56 (4)	3.491 (8)	164 (2)
C27—H27B···N2	0.96 (4)	2.60 (3)	3.218 (8)	122 (3)
C29—H29A···O6^{iii}	0.97 (2)	2.49 (1)	3.229 (14)	133 (1)
C29—H29B···O3^{iv}	0.97 (2)	2.41 (1)	3.046 (14)	123 (1)
C44—H44B···N6	0.96 (3)	2.62 (3)	2.940 (7)	100 (2)
C47—H47B···O4^{i}	0.96 (3)	2.42 (3)	3.331 (8)	158 (1)
C50—H50B···Cl2	0.97 (1)	2.72 (1)	3.617 (9)	154 (1)

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