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1,7,8,9,10,10-Hexachloro-4-(thiophen-2-ylmethyl)-4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

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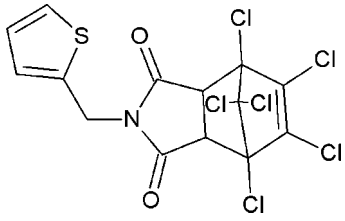
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.075; data-to-parameter ratio = 19.2.

In the title compound, $C_{14}H_7Cl_6NO_2S$, the six-membered ring of the azatricyclo system has a boat conformation whereas the five-membered rings have an envelope conformation. The thiophene ring and the ring of the succinimide moiety enclose a dihedral angle of 67.2 (1)°. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds.

Related literature

For the biological activity of cyclic imides, see: Duarte *et al.* (2006); Nakamura *et al.* (2006); Stefańska *et al.* (2010).



Experimental

Crystal data

$C_{14}H_7Cl_6NO_2S$
M_r = 465.97
Tetragonal, $I4_1/a$
a = 23.8136 (10) Å
c = 12.6240 (9) Å
V = 7158.9 (7) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 1.08 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm **Is this OK?**

Data collection

Xcalibur, Eos diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.978, T_{max} = 0.984
8488 measured reflections
4156 independent reflections
2283 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.075
S = 0.82
4156 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O2^{i}	0.98	2.54	3.064 (3)	113
C6—H6···O2^{i}	0.98	2.51	3.042 (3)	114

Symmetry code: (i) $y-\frac{1}{4}, -x+\frac{7}{4}, z-\frac{1}{4}$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

CRR is grateful to the DST–FIST single-crystal X-ray facility of the Department of Chemistry, Pondicherry University, Pondicherry.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5586).

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supporting information

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1,7,8,9,10,10-Hexachloro-4-(thiophen-2-ylmethyl)-4-azatricyclo-[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

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S1. Comment

Azatricyclo dec-8-ene 3,5 dione derivatives have anti bacterial and anti fungal activities with other important biological activities (Stefańska *et al.*, 2010).

In these structure, the six-membered ring of the norbornene moiety adopts a boat conformation whereas the two five-membered rings adopt envelope conformation.The fusion at atoms C6 and C2 is in *cis* conformation. The planarity around N4 and C3—N4 [1.38 (4) Å] and N4—C5 [1.38 (4) Å] reveals the partial double bond charater to facilitate the electron delocalization from one keto oxygen to other through N4. The crystal structure is stabilized by weak intermolecular C-H···O interactions.

S2. Experimental

1-(thiophen-2-yl)methanamine (1 equiv) and 1,4,5,6,7,7-hexachloro-5-norbornene-2,3-dicarboxylic anhydride (1 equiv) were stirred at room temperature in dry ethyl acetate for 30 min. Ethyl acetate was removed under reduced pressure; the resulting residue was dissolved in toluene. To this reaction mixture was added acetyl chloride (5 equiv) and refluxed for 1 h. The reaction mixture was brought to room temperature and washed with aqueous Na2CO3 and dried over anhydrous Na2SO4. Filtered and concentrated under reduced pressure followed by silica gel column purification afforded the imide, 1,7,8,9,10,10-Hexachloro-4-(thiophen-2-yl-methyl)-4-azatricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione, in 92% yield as colorless solid.

S3. Refinement

The hydrogen atoms were positioned geometrically and refined using a riding model.

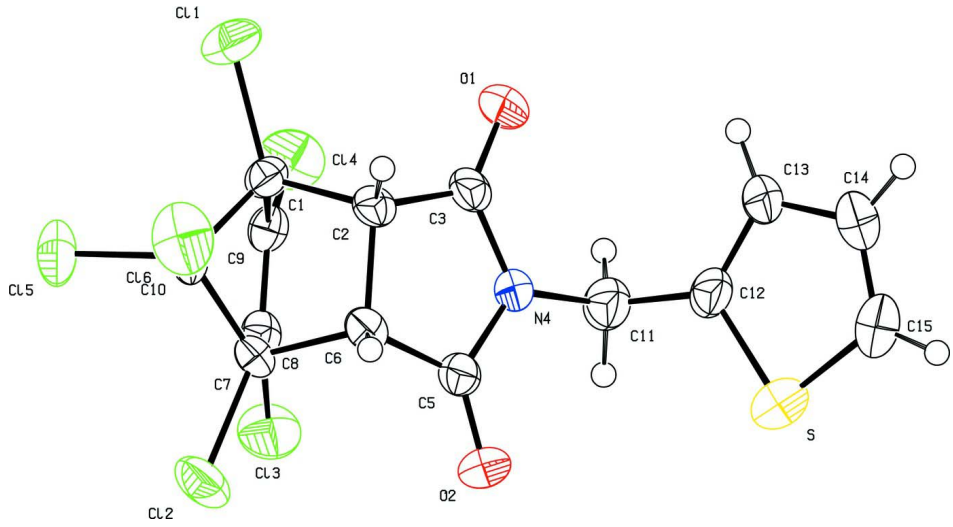

Figure 1

The *ORTEP* diagram of the compound with 30% probability displacement ellipsoids.

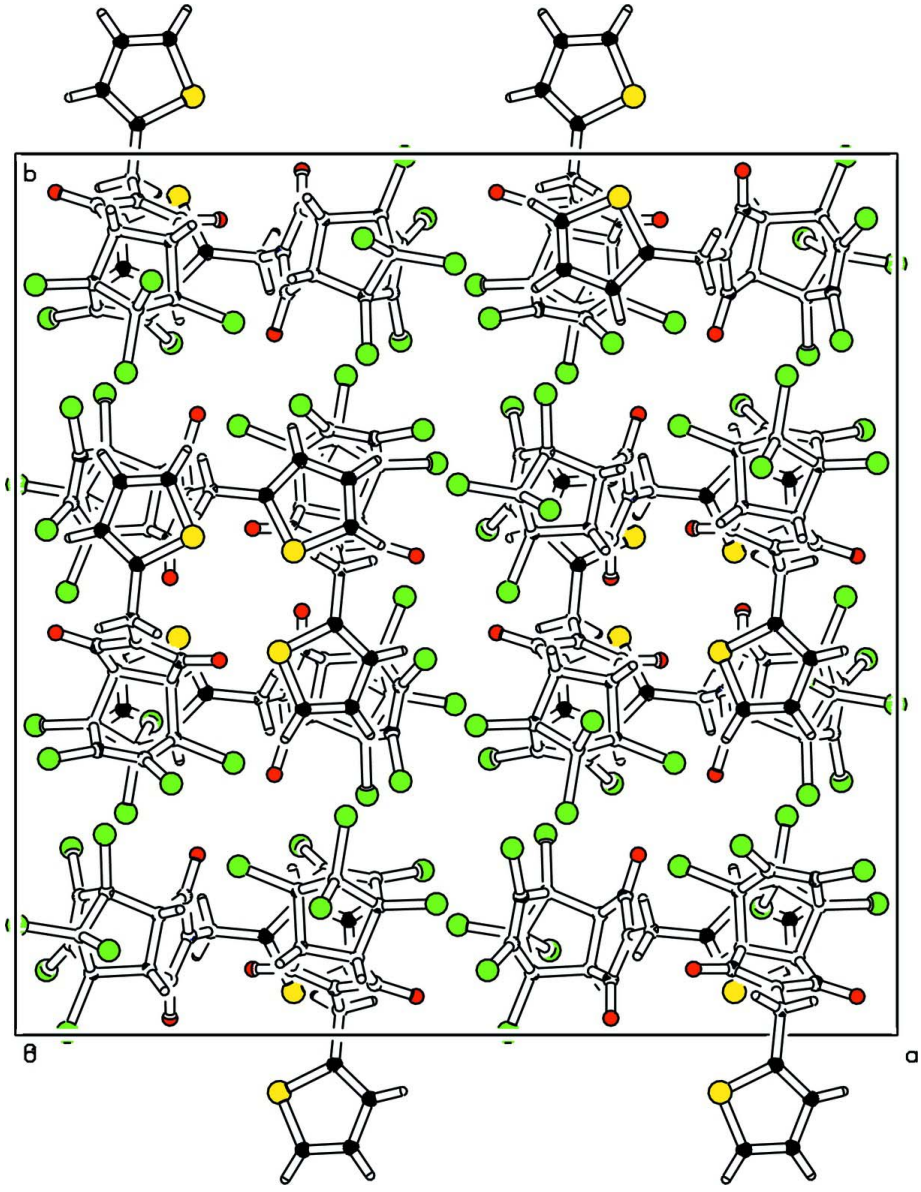

Figure 2

Packing diagram.

1,7,8,9,10,10-Hexachloro-4-(thiophen-2-ylmethyl)-4- azatricyclo[5.2.1.0^{2,6}]dec-8-ene-3,5-dione

Crystal data

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Tetragonal, $I4_1/a$
Hall symbol: -I 4ad
a = 23.8136 (10) Å
c = 12.6240 (9) Å
V = 7158.9 (7) Å^3
Z = 16
$F(000)$ = 3712
D_x = 1.729 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
μ = 1.08 mm^{-1}
T = 293 K
Tetragonal, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 15.9821 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.978, T_{max} = 0.984

8488 measured reflections
4156 independent reflections
2283 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 29.2°, θ_{min} = 3.0°
h = −29→16
k = −28→32
l = −15→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.075
S = 0.82
4156 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.60327 (10)	1.16190 (10)	0.08398 (18)	0.0301 (6)
C2	0.66296 (9)	1.13668 (9)	0.09872 (19)	0.0295 (6)
H2	0.6864	1.1422	0.0358	0.035*
C3	0.69158 (10)	1.15740 (10)	0.1978 (2)	0.0326 (6)
C5	0.67499 (10)	1.06286 (10)	0.2282 (2)	0.0344 (6)
C6	0.65133 (9)	1.07395 (9)	0.11968 (18)	0.0288 (6)
H6	0.6690	1.0500	0.0660	0.035*
C7	0.58607 (9)	1.07054 (9)	0.11446 (18)	0.0276 (5)
C8	0.56310 (9)	1.10361 (10)	0.20743 (17)	0.0291 (6)
C9	0.57323 (9)	1.15729 (10)	0.18980 (18)	0.0285 (6)
C10	0.57402 (9)	1.11314 (11)	0.02405 (18)	0.0342 (6)
C11	0.72133 (10)	1.11804 (11)	0.3724 (2)	0.0432 (7)
H11A	0.7054	1.0897	0.4187	0.052*
H11B	0.7116	1.1546	0.4009	0.052*
C12	0.78385 (11)	1.11200 (10)	0.37121 (19)	0.0391 (7)
C13	0.82275 (11)	1.15206 (11)	0.3454 (2)	0.0485 (8)

H13	0.8140	1.1888	0.3269	0.058*
C14	0.87777 (12)	1.13051 (13)	0.3504 (2)	0.0617 (9)
H14	0.9094	1.1520	0.3355	0.074*
C15	0.88049 (12)	1.07617 (13)	0.3788 (2)	0.0573 (8)
H15	0.9137	1.0559	0.3857	0.069*
N4	0.69675 (8)	1.11223 (8)	0.26664 (16)	0.0321 (5)
O1	0.70632 (7)	1.20435 (7)	0.21805 (15)	0.0486 (5)
O2	0.67518 (8)	1.01900 (7)	0.27599 (16)	0.0550 (5)
S	0.81534 (3)	1.04915 (3)	0.40044 (6)	0.0562 (2)
Cl1	0.60119 (3)	1.22738 (3)	0.02283 (6)	0.0557 (2)
Cl2	0.55865 (3)	1.00296 (3)	0.09833 (6)	0.0508 (2)
Cl3	0.53529 (3)	1.07338 (3)	0.31710 (6)	0.0528 (2)
Cl4	0.56334 (3)	1.21203 (3)	0.27281 (6)	0.0553 (2)
Cl5	0.50195 (3)	1.12482 (3)	0.00103 (6)	0.0524 (2)
Cl6	0.60575 (3)	1.09588 (3)	−0.09771 (5)	0.0593 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0295 (13)	0.0306 (13)	0.0303 (14)	0.0027 (11)	0.0006 (11)	0.0069 (11)
C2	0.0227 (12)	0.0304 (13)	0.0353 (14)	−0.0024 (11)	0.0064 (11)	0.0025 (11)
C3	0.0210 (12)	0.0312 (14)	0.0456 (17)	−0.0027 (12)	0.0021 (12)	−0.0004 (13)
C5	0.0246 (13)	0.0282 (14)	0.0502 (17)	0.0013 (11)	−0.0019 (12)	0.0016 (13)
C6	0.0233 (12)	0.0267 (12)	0.0363 (15)	0.0006 (11)	0.0046 (11)	−0.0074 (11)
C7	0.0259 (12)	0.0271 (13)	0.0300 (14)	−0.0056 (11)	−0.0008 (11)	−0.0045 (11)
C8	0.0221 (12)	0.0384 (14)	0.0269 (14)	−0.0004 (12)	0.0030 (10)	0.0015 (12)
C9	0.0239 (12)	0.0318 (14)	0.0298 (14)	0.0080 (12)	0.0016 (11)	−0.0028 (11)
C10	0.0250 (13)	0.0519 (16)	0.0258 (13)	0.0007 (13)	−0.0012 (11)	−0.0058 (12)
C11	0.0458 (16)	0.0434 (16)	0.0404 (17)	0.0009 (14)	−0.0096 (14)	−0.0005 (13)
C12	0.0431 (16)	0.0383 (15)	0.0358 (16)	0.0025 (14)	−0.0143 (13)	0.0004 (13)
C13	0.0452 (17)	0.0378 (16)	0.062 (2)	−0.0073 (15)	−0.0266 (15)	0.0034 (14)
C14	0.0424 (18)	0.065 (2)	0.078 (2)	−0.0156 (18)	−0.0232 (16)	0.0116 (19)
C15	0.0419 (17)	0.067 (2)	0.064 (2)	0.0061 (17)	−0.0172 (15)	0.0063 (17)
N4	0.0287 (11)	0.0289 (11)	0.0388 (13)	−0.0024 (10)	−0.0077 (10)	0.0019 (10)
O1	0.0490 (11)	0.0291 (10)	0.0678 (14)	−0.0106 (9)	−0.0153 (10)	−0.0003 (9)
O2	0.0545 (12)	0.0307 (10)	0.0798 (16)	−0.0019 (10)	−0.0175 (11)	0.0179 (10)
S	0.0603 (5)	0.0458 (4)	0.0625 (5)	0.0053 (4)	−0.0086 (4)	0.0148 (4)
Cl1	0.0628 (5)	0.0437 (4)	0.0607 (5)	0.0027 (4)	−0.0044 (4)	0.0242 (4)
Cl2	0.0489 (4)	0.0382 (4)	0.0653 (5)	−0.0164 (3)	−0.0050 (4)	−0.0117 (3)
Cl3	0.0537 (4)	0.0620 (5)	0.0427 (4)	−0.0081 (4)	0.0166 (4)	0.0107 (4)
Cl4	0.0606 (5)	0.0437 (4)	0.0617 (5)	0.0095 (4)	0.0148 (4)	−0.0194 (4)
Cl5	0.0325 (3)	0.0753 (5)	0.0494 (4)	−0.0002 (4)	−0.0142 (3)	0.0020 (4)
Cl6	0.0611 (5)	0.0861 (6)	0.0308 (4)	−0.0023 (5)	0.0084 (4)	−0.0126 (4)

Geometric parameters (Å, º)

C1—C9	1.519 (3)	C8—C9	1.320 (3)
C1—C10	1.551 (3)	C8—Cl3	1.695 (2)

C1—C2	1.554 (3)	C9—Cl4	1.689 (2)
C1—Cl1	1.741 (2)	C10—Cl6	1.761 (2)
C2—C3	1.508 (3)	C10—Cl5	1.763 (2)
C2—C6	1.542 (3)	C11—N4	1.464 (3)
C2—H2	0.9800	C11—C12	1.496 (3)
C3—O1	1.199 (3)	C11—H11A	0.9700
C3—N4	1.388 (3)	C11—H11B	0.9700
C5—O2	1.206 (3)	C12—C13	1.369 (3)
C5—N4	1.374 (3)	C12—S	1.714 (3)
C5—C6	1.504 (3)	C13—C14	1.409 (4)
C6—C7	1.558 (3)	C13—H13	0.9300
C6—H6	0.9800	C14—C15	1.344 (4)
C7—C8	1.516 (3)	C14—H14	0.9300
C7—C10	1.554 (3)	C15—S	1.701 (3)
C7—Cl2	1.749 (2)	C15—H15	0.9300

C9—C1—C10	99.40 (18)	C7—C8—Cl3	123.56 (17)
C9—C1—C2	107.31 (18)	C8—C9—C1	107.72 (19)
C10—C1—C2	100.37 (18)	C8—C9—Cl4	128.13 (19)
C9—C1—Cl1	116.18 (16)	C1—C9—Cl4	123.74 (18)
C10—C1—Cl1	116.20 (16)	C1—C10—C7	92.72 (17)
C2—C1—Cl1	115.16 (16)	C1—C10—Cl6	114.06 (17)
C3—C2—C6	104.81 (19)	C7—C10—Cl6	114.17 (17)
C3—C2—C1	112.73 (19)	C1—C10—Cl5	113.56 (17)
C6—C2—C1	103.33 (17)	C7—C10—Cl5	113.82 (16)
C3—C2—H2	111.8	Cl6—C10—Cl5	108.10 (12)
C6—C2—H2	111.8	N4—C11—C12	112.3 (2)
C1—C2—H2	111.8	N4—C11—H11A	109.1
O1—C3—N4	124.3 (2)	C12—C11—H11A	109.1
O1—C3—C2	127.9 (2)	N4—C11—H11B	109.1
N4—C3—C2	107.8 (2)	C12—C11—H11B	109.1
O2—C5—N4	124.2 (2)	H11A—C11—H11B	107.9
O2—C5—C6	127.5 (2)	C13—C12—C11	127.5 (2)
N4—C5—C6	108.2 (2)	C13—C12—S	111.32 (19)
C5—C6—C2	105.01 (19)	C11—C12—S	121.1 (2)
C5—C6—C7	113.76 (19)	C12—C13—C14	111.4 (2)
C2—C6—C7	102.84 (18)	C12—C13—H13	124.3
C5—C6—H6	111.6	C14—C13—H13	124.3
C2—C6—H6	111.6	C15—C14—C13	114.0 (3)
C7—C6—H6	111.6	C15—C14—H14	123.0
C8—C7—C10	99.38 (18)	C13—C14—H14	123.0
C8—C7—C6	107.48 (18)	C14—C15—S	111.3 (2)
C10—C7—C6	100.44 (17)	C14—C15—H15	124.4
C8—C7—Cl2	115.70 (16)	S—C15—H15	124.4
C10—C7—Cl2	116.52 (16)	C5—N4—C3	114.1 (2)
C6—C7—Cl2	115.18 (16)	C5—N4—C11	123.7 (2)
C9—C8—C7	107.86 (19)	C3—N4—C11	122.2 (2)
C9—C8—Cl3	128.35 (19)	C15—S—C12	91.96 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O2^{i}	0.98	2.54	3.064 (3)	113
C6—H6···O2^{i}	0.98	2.51	3.042 (3)	114

Symmetry code: (i) $y-1/4, -x+7/4, z-1/4$.