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Ethyl 2-(propionylamino)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

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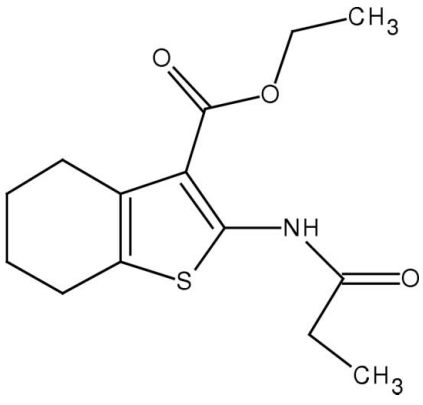
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.034; wR factor = 0.094; data-to-parameter ratio = 19.4.

Geometric parameters of the title compound, $C_{14}H_{19}NO_3S$, are in the usual ranges. In one of the two molecules in the asymmetric unit two methylene groups of the cyclohexene ring are disordered over two positions with site occupation factors of 0.612 (5) and 0.388 (5). Taking only the major occupied sites into account, the two molecules show essentially the same geometric parameters (r.m.s. deviation for all non-H atoms 0.084 Å). There is an intramolecular N—H···O hydrogen bond. The molecules crystallize in planes parallel to (220).

Related literature

For related structures, see: Harrison *et al.* (2006); Vasu *et al.* (2004*a*,*b*).

For related literature, see also: Campaigne *et al.* (1970); Ramanathan & Namboothiri (1978); Cannito *et al.* (1990); Anilkumar *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{19}NO_3S$
M_r = 281.37
Triclinic, $P\overline{1}$
a = 8.5746 (4) Å
b = 11.2010 (6) Å
c = 15.8831 (8) Å
α = 78.888 (5)°
β = 75.363 (4)°
γ = 74.078 (4)°
V = 1406.81 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 173 (2) K
0.43 × 0.37 × 0.32 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan *MULABS* (Spek, 2003; Blessing, 1995)
T_{min} = 0.906, T_{max} = 0.929
43668 measured reflections
7185 independent reflections
6368 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.06
7185 reflections
370 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21···O31	0.882 (19)	1.971 (18)	2.6872 (13)	137.3 (15)
N21A—H21A···O31A	0.91 (2)	1.960 (19)	2.6859 (14)	135.6 (16)

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2012).

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supplementary materials

Acta Cryst. (2007). E**63**, o2949 [**doi:10.1107/S1600536807024282**]

Ethyl 2-(propionylamino)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

H. S. Yathirajan, S. Bindya, B. K. Sarojini, B. Narayana and M. Bolte

Comment

Thiophene derivatives are known to exhibit an array of biological effects, including analgesic and anti–inflammatory activities. The title compound was prepared by the reaction of a mixture of ethyl 2–amino–4,5,6,7–tetrahydro–1–benzothiophene–3–carboxylate, propionic anhydride and zinc dust.

Geometric parameters of the title compound, $C_{14}H_{19}NO_3S$, (Figs. 1 and 2) are in the usual ranges. In one of the two molecules in the asymmetric unit two methylene groups (C7A, C8A and C7', C8') of the cyclohexene ring are disordered over two positions with site occupation factors of 0.612 (5) and 0.388 (5). Taking only the major occupied sites into account the two molecules show essentially the same geometric parameters (r.m.s. deviation for all non H atoms 0.084 Å). The molecular conformation is stabilized by an N—H···O intramolecular hydrogen bond. The molecules crystallize in planes parallel to the (220) plane (Fig. 3).

Experimental

Ethyl 2–amino–4,5,6,7–tetrahydro–1–benzothiophene–3–carboxylate (3.5 g, 0.0155 mol), propionic anhydride (10.5 ml) and zinc dust (0.883 g, 0.015 mol) were refluxed for 2 hr (see scheme_2).

The reaction mixture was then cooled to room temperature and the precipitated product was filtered. The crude product was dissolved in methanol (35 ml) and filtered over hyflo. The filtrate was slowly cooled to room temperature and filtered to collect the solid. The product was obtained as colourless crystals with a yield of 58.1%. *X*–ray quality crystals were obtained from acetone by slow evaporation (m.p.: 352–354 K). Analysis for $C_{14}H_{19}NO_3S$; Found (Calculated): C: 59.58 (59.76); H: 6.68 (6.81); N: 4.83 (4.98); S: 11.32% (11.40%). IR (KBr): 3436 & 3244 (–NH–), 2931 & 2873(–CH–), 1666 & 1546 (–C=O) and 1250 cm^{-1} (C—O).

Refinement

H atoms were found in a difference map, but those bonded to C were refined using a riding model with C_{methyl}—H = 0.98Å or $C_{methylene}$—H = 0.99Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$. H atoms bonded to N were freely refined. In one of the two molecules in the asymmetric unit two methylene groups of the cyclohexene ring are disordered over two positions with site occupation factors of 0.612 (5) and 0.388 (5).

Figures

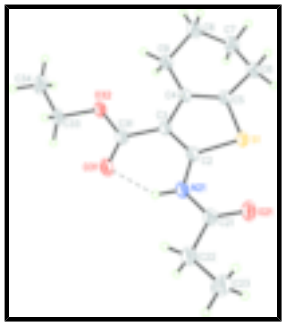


Fig. 1. Perspective view of molecule one (major fragment) the title compound with the atom numbering scheme; displacement ellipsoids are at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius. The intramolecular hydrogen bond is shown as a dashed line.

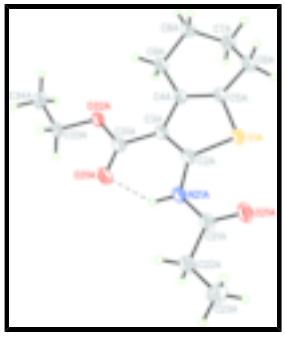


Fig. 2. Perspective view of molecule two the title compound with the atom numbering-scheme; displacement ellipsoids are at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius. The intramolecular hydrogen bond is shown as a dashed line.

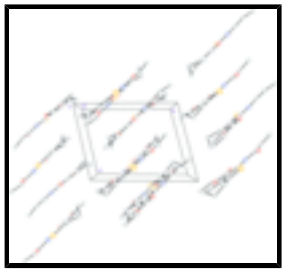


Fig. 3. Packing diagram of the title compound with view onto the *ab* plane. H atoms are omitted for clarity.

Fig. 4. Reaction scheme.

Ethyl 2-(propionylamino)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

Crystal data

$C_{14}H_{19}NO_3S$	$Z = 4$
$M_r = 281.37$	$F_{000} = 600$
Triclinic, $P\overline{1}$	$D_x = 1.329$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 353 K
$a = 8.5746$ (4) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.2010$ (6) Å	Cell parameters from 42668 reflections
$c = 15.8831$ (8) Å	$\theta = 3.6$–27.8º
$\alpha = 78.888$ (5)º	$\mu = 0.23$ mm^{-1}
$\beta = 75.363$ (4)º	$T = 173$ (2) K

γ = 74.078 (4)°	Block, colourless
V = 1406.81 (13) Å^3	0.43 × 0.37 × 0.32 mm

Data collection

STOE IPDS II two-circle diffractometer	7185 independent reflections
Radiation source: fine–focus sealed tube	6368 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.039
T = 173(2) K	θ_{max} = 28.7°
ω scans	θ_{min} = 3.5°
Absorption correction: multi-scan MULABS (Spek, 2003; Blessing, 1995)	h = −11→11
T_{min} = 0.906, T_{max} = 0.929	k = −14→15
43668 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.094	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.3923P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
7185 reflections	$\Delta\rho_{max}$ = 0.51 e Å^{-3}
370 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
S1	0.15558 (4)	0.56091 (3)	0.126108 (17)	0.02536 (8)	
C2	0.16267 (14)	0.56701 (11)	0.23282 (7)	0.0243 (2)	

C3	0.06318 (14)	0.67684 (10)	0.26405 (7)	0.0230 (2)	
C4	−0.01899 (14)	0.75970 (10)	0.19772 (7)	0.0222 (2)	
C5	0.01834 (14)	0.70767 (11)	0.12156 (7)	0.0237 (2)	
C6	−0.04590 (16)	0.76676 (11)	0.03980 (8)	0.0288 (2)	
H6A	−0.0846	0.7051	0.0182	0.035*	
H6B	0.0444	0.7929	−0.0066	0.035*	
C7	−0.18829 (18)	0.87996 (13)	0.05944 (9)	0.0363 (3)	
H7A	−0.2099	0.9312	0.0035	0.044*	
H7B	−0.2898	0.8514	0.0907	0.044*	
C8	−0.1504 (2)	0.95968 (13)	0.11503 (9)	0.0398 (3)	
H8A	−0.2419	1.0357	0.1225	0.048*	
H8B	−0.0475	0.9867	0.0843	0.048*	
C9	−0.12879 (16)	0.88911 (11)	0.20526 (8)	0.0277 (2)	
H9A	−0.0793	0.9361	0.2340	0.033*	
H9B	−0.2387	0.8830	0.2424	0.033*	
N21	0.25877 (13)	0.47144 (10)	0.28096 (7)	0.0291 (2)	
H21	0.245 (2)	0.4852 (16)	0.3355 (12)	0.043 (5)*	
C21	0.36469 (16)	0.36579 (12)	0.25001 (8)	0.0291 (2)	
O21	0.38178 (13)	0.34543 (10)	0.17491 (6)	0.0405 (2)	
C22	0.45589 (18)	0.27957 (13)	0.31725 (9)	0.0371 (3)	
H22A	0.3753	0.2436	0.3647	0.045*	
H22B	0.5059	0.3291	0.3438	0.045*	
C23	0.5913 (2)	0.17375 (15)	0.27818 (11)	0.0476 (4)	
H23D	0.6462	0.1210	0.3242	0.071*	
H23E	0.6729	0.2087	0.2320	0.071*	
H23F	0.5423	0.1231	0.2530	0.071*	
O31	0.11664 (13)	0.61778 (9)	0.40865 (6)	0.0360 (2)	
C31	0.04853 (15)	0.69525 (11)	0.35531 (7)	0.0253 (2)	
O32	−0.04810 (11)	0.80677 (8)	0.37599 (5)	0.02900 (19)	
C33	−0.06552 (17)	0.82922 (12)	0.46596 (8)	0.0312 (3)	
H33A	0.0440	0.8272	0.4768	0.037*	
H33B	−0.1118	0.7638	0.5083	0.037*	
C34	−0.1811 (2)	0.95633 (14)	0.47668 (10)	0.0449 (4)	
H34A	−0.1950	0.9745	0.5364	0.067*	
H34B	−0.2891	0.9570	0.4662	0.067*	
H34C	−0.1341	1.0202	0.4344	0.067*	
S1A	0.85238 (4)	0.40867 (3)	0.378490 (18)	0.02823 (8)	
C2A	0.84650 (15)	0.40766 (11)	0.27082 (7)	0.0248 (2)	
C3A	0.73074 (14)	0.50898 (11)	0.24008 (7)	0.0240 (2)	
C4A	0.64458 (14)	0.59055 (11)	0.30668 (7)	0.0234 (2)	
C5A	0.69911 (15)	0.54767 (11)	0.38350 (7)	0.0248 (2)	
C6A	0.64157 (17)	0.61157 (12)	0.46500 (8)	0.0307 (3)	
H6C	0.7379	0.6080	0.4899	0.037*	0.612 (5)
H6D	0.5645	0.5684	0.5097	0.037*	0.612 (5)
H6E	0.7252	0.6547	0.4693	0.037*	0.388 (5)
H6F	0.6265	0.5490	0.5178	0.037*	0.388 (5)
C7A	0.5534 (3)	0.7485 (2)	0.44090 (15)	0.0315 (6)	0.612 (5)
H7A1	0.4989	0.7883	0.4948	0.038*	0.612 (5)
H7A2	0.6346	0.7957	0.4045	0.038*	0.612 (5)

C8A	0.4241 (3)	0.7499 (3)	0.38978 (16)	0.0310 (6)	0.612 (5)
H8A1	0.3517	0.8353	0.3829	0.037*	0.612 (5)
H8A2	0.3538	0.6921	0.4227	0.037*	0.612 (5)
C7'	0.4708 (6)	0.7100 (4)	0.4588 (2)	0.0325 (10)	0.388 (5)
H7'1	0.3827	0.6656	0.4645	0.039*	0.388 (5)
H7'2	0.4381	0.7622	0.5069	0.039*	0.388 (5)
C8'	0.4914 (7)	0.7921 (4)	0.3708 (2)	0.0353 (11)	0.388 (5)
H8'1	0.5908	0.8260	0.3607	0.042*	0.388 (5)
H8'2	0.3931	0.8633	0.3695	0.042*	0.388 (5)
C9A	0.51108 (16)	0.70908 (12)	0.29782 (8)	0.0314 (3)	
H9C	0.4284	0.6950	0.2695	0.038*	0.612 (5)
H9D	0.5606	0.7764	0.2602	0.038*	0.612 (5)
H9E	0.4044	0.6875	0.3026	0.038*	0.388 (5)
H9F	0.5394	0.7572	0.2392	0.038*	0.388 (5)
N21A	0.94887 (13)	0.31478 (10)	0.22147 (7)	0.0278 (2)	
H21A	0.936 (2)	0.3277 (17)	0.1651 (13)	0.048 (5)*	
C21A	1.06336 (16)	0.21330 (11)	0.25016 (8)	0.0293 (2)	
O21A	1.08301 (14)	0.19337 (9)	0.32565 (6)	0.0418 (2)	
C22A	1.16166 (19)	0.13158 (12)	0.17983 (9)	0.0365 (3)	
H22C	1.2386	0.1768	0.1369	0.044*	
H22D	1.0843	0.1167	0.1483	0.044*	
C23A	1.2609 (2)	0.00611 (14)	0.21596 (11)	0.0470 (4)	
H23A	1.3219	−0.0423	0.1676	0.071*	
H23B	1.1854	−0.0403	0.2574	0.071*	
H23C	1.3396	0.0200	0.2462	0.071*	
C31A	0.71044 (15)	0.52428 (11)	0.14876 (8)	0.0267 (2)	
O31A	0.78895 (13)	0.45064 (9)	0.09632 (6)	0.0388 (2)	
O32A	0.59646 (11)	0.62749 (8)	0.12746 (5)	0.02721 (18)	
C33A	0.56826 (16)	0.64382 (12)	0.03856 (8)	0.0303 (3)	
H33C	0.5240	0.5745	0.0312	0.036*	
H33D	0.6738	0.6437	−0.0050	0.036*	
C34A	0.44487 (17)	0.76753 (13)	0.02499 (9)	0.0357 (3)	
H34D	0.4237	0.7808	−0.0343	0.054*	
H34E	0.4900	0.8355	0.0323	0.054*	
H34F	0.3408	0.7665	0.0682	0.054*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.03213 (15)	0.02470 (14)	0.01692 (12)	−0.00072 (11)	−0.00530 (10)	−0.00588 (10)
C2	0.0282 (5)	0.0246 (5)	0.0180 (5)	−0.0015 (4)	−0.0059 (4)	−0.0036 (4)
C3	0.0270 (5)	0.0239 (5)	0.0165 (5)	−0.0018 (4)	−0.0054 (4)	−0.0042 (4)
C4	0.0257 (5)	0.0227 (5)	0.0175 (5)	−0.0043 (4)	−0.0051 (4)	−0.0025 (4)
C5	0.0275 (5)	0.0241 (5)	0.0185 (5)	−0.0038 (4)	−0.0059 (4)	−0.0026 (4)
C6	0.0373 (6)	0.0283 (6)	0.0205 (5)	−0.0033 (5)	−0.0107 (5)	−0.0029 (4)
C7	0.0429 (7)	0.0373 (7)	0.0265 (6)	0.0027 (6)	−0.0164 (5)	−0.0051 (5)
C8	0.0572 (9)	0.0300 (6)	0.0275 (6)	0.0044 (6)	−0.0166 (6)	−0.0038 (5)
C9	0.0335 (6)	0.0248 (5)	0.0217 (5)	0.0008 (5)	−0.0082 (4)	−0.0038 (4)

N21	0.0362 (5)	0.0276 (5)	0.0183 (4)	0.0052 (4)	−0.0082 (4)	−0.0062 (4)
C21	0.0322 (6)	0.0283 (6)	0.0223 (5)	0.0023 (5)	−0.0058 (4)	−0.0062 (4)
O21	0.0515 (6)	0.0386 (5)	0.0240 (4)	0.0091 (4)	−0.0101 (4)	−0.0123 (4)
C22	0.0426 (7)	0.0338 (7)	0.0266 (6)	0.0095 (5)	−0.0111 (5)	−0.0062 (5)
C23	0.0478 (8)	0.0380 (7)	0.0508 (9)	0.0141 (6)	−0.0188 (7)	−0.0152 (7)
O31	0.0490 (5)	0.0327 (5)	0.0213 (4)	0.0082 (4)	−0.0149 (4)	−0.0076 (3)
C31	0.0296 (5)	0.0255 (5)	0.0190 (5)	−0.0007 (4)	−0.0062 (4)	−0.0056 (4)
O32	0.0388 (5)	0.0273 (4)	0.0178 (4)	0.0035 (3)	−0.0093 (3)	−0.0078 (3)
C33	0.0423 (7)	0.0305 (6)	0.0189 (5)	0.0019 (5)	−0.0095 (5)	−0.0101 (4)
C34	0.0609 (9)	0.0360 (7)	0.0327 (7)	0.0115 (7)	−0.0171 (7)	−0.0167 (6)
S1A	0.03550 (16)	0.02789 (15)	0.01688 (13)	0.00213 (11)	−0.00784 (11)	−0.00349 (10)
C2A	0.0297 (5)	0.0246 (5)	0.0181 (5)	−0.0025 (4)	−0.0051 (4)	−0.0037 (4)
C3A	0.0274 (5)	0.0251 (5)	0.0176 (5)	−0.0015 (4)	−0.0054 (4)	−0.0046 (4)
C4A	0.0258 (5)	0.0254 (5)	0.0185 (5)	−0.0036 (4)	−0.0045 (4)	−0.0053 (4)
C5A	0.0283 (5)	0.0261 (5)	0.0178 (5)	−0.0031 (4)	−0.0039 (4)	−0.0044 (4)
C6A	0.0394 (6)	0.0306 (6)	0.0188 (5)	0.0002 (5)	−0.0073 (5)	−0.0063 (4)
C7A	0.0403 (14)	0.0289 (11)	0.0244 (10)	−0.0014 (10)	−0.0091 (9)	−0.0087 (8)
C8A	0.0316 (12)	0.0318 (12)	0.0255 (10)	0.0024 (9)	−0.0056 (9)	−0.0094 (9)
C7'	0.042 (2)	0.0312 (18)	0.0179 (14)	−0.0002 (16)	−0.0025 (13)	−0.0055 (12)
C8'	0.050 (3)	0.0279 (18)	0.0217 (16)	0.0022 (17)	−0.0059 (16)	−0.0068 (13)
C9A	0.0357 (6)	0.0326 (6)	0.0225 (5)	0.0051 (5)	−0.0098 (5)	−0.0095 (5)
N21A	0.0354 (5)	0.0255 (5)	0.0187 (4)	0.0023 (4)	−0.0076 (4)	−0.0052 (4)
C21A	0.0382 (6)	0.0242 (5)	0.0229 (5)	−0.0009 (5)	−0.0087 (5)	−0.0036 (4)
O21A	0.0579 (6)	0.0366 (5)	0.0238 (4)	0.0084 (4)	−0.0157 (4)	−0.0059 (4)
C22A	0.0498 (8)	0.0273 (6)	0.0272 (6)	0.0070 (5)	−0.0129 (5)	−0.0087 (5)
C23A	0.0562 (9)	0.0312 (7)	0.0501 (9)	0.0127 (6)	−0.0241 (7)	−0.0133 (6)
C31A	0.0307 (6)	0.0278 (6)	0.0207 (5)	−0.0013 (4)	−0.0082 (4)	−0.0054 (4)
O31A	0.0484 (6)	0.0381 (5)	0.0242 (4)	0.0118 (4)	−0.0145 (4)	−0.0134 (4)
O32A	0.0331 (4)	0.0282 (4)	0.0191 (4)	0.0016 (3)	−0.0102 (3)	−0.0067 (3)
C33A	0.0360 (6)	0.0338 (6)	0.0211 (5)	0.0016 (5)	−0.0136 (5)	−0.0082 (5)
C34A	0.0378 (7)	0.0366 (7)	0.0323 (6)	0.0022 (5)	−0.0173 (5)	−0.0065 (5)

Geometric parameters (Å, °)

S1—C2	1.7263 (11)	C4A—C5A	1.3702 (15)
S1—C5	1.7401 (12)	C4A—C9A	1.5061 (16)
C2—N21	1.3888 (15)	C5A—C6A	1.5073 (16)
C2—C3	1.3903 (15)	C6A—C7A	1.534 (2)
C3—C4	1.4517 (15)	C6A—C7'	1.586 (4)
C3—C31	1.4725 (15)	C6A—H6C	0.9900
C4—C5	1.3707 (15)	C6A—H6D	0.9900
C4—C9	1.5044 (15)	C6A—H6E	0.9900
C5—C6	1.5027 (15)	C6A—H6F	0.9900
C6—C7	1.5187 (18)	C7A—C8A	1.526 (4)
C6—H6A	0.9900	C7A—H7A1	0.9900
C6—H6B	0.9900	C7A—H7A2	0.9900
C7—C8	1.513 (2)	C8A—C9A	1.555 (2)
C7—H7A	0.9900	C8A—H8A1	0.9900
C7—H7B	0.9900	C8A—H8A2	0.9900

C8—C9	1.5265 (17)	C7'—C8'	1.516 (6)
C8—H8A	0.9900	C7'—H7'1	0.9900
C8—H8B	0.9900	C7'—H7'2	0.9900
C9—H9A	0.9900	C8'—C9A	1.571 (4)
C9—H9B	0.9900	C8'—H8'1	0.9900
N21—C21	1.3696 (15)	C8'—H8'2	0.9900
N21—H21	0.882 (19)	C9A—H9C	0.9900
C21—O21	1.2226 (15)	C9A—H9D	0.9900
C21—C22	1.5150 (17)	C9A—H9E	0.9900
C22—C23	1.5174 (18)	C9A—H9F	0.9900
C22—H22A	0.9900	N21A—C21A	1.3703 (15)
C22—H22B	0.9900	N21A—H21A	0.91 (2)
C23—H23D	0.9800	C21A—O21A	1.2223 (15)
C23—H23E	0.9800	C21A—C22A	1.5136 (17)
C23—H23F	0.9800	C22A—C23A	1.5194 (18)
O31—C31	1.2249 (14)	C22A—H22C	0.9900
C31—O32	1.3444 (14)	C22A—H22D	0.9900
O32—C33	1.4621 (13)	C23A—H23A	0.9800
C33—C34	1.5073 (18)	C23A—H23B	0.9800
C33—H33A	0.9900	C23A—H23C	0.9800
C33—H33B	0.9900	C31A—O31A	1.2265 (15)
C34—H34A	0.9800	C31A—O32A	1.3426 (14)
C34—H34B	0.9800	O32A—C33A	1.4609 (13)
C34—H34C	0.9800	C33A—C34A	1.5105 (17)
S1A—C2A	1.7259 (11)	C33A—H33C	0.9900
S1A—C5A	1.7406 (12)	C33A—H33D	0.9900
C2A—C3A	1.3874 (15)	C34A—H34D	0.9800
C2A—N21A	1.3902 (15)	C34A—H34E	0.9800
C3A—C4A	1.4514 (15)	C34A—H34F	0.9800
C3A—C31A	1.4761 (15)		
C2—S1—C5	90.87 (5)	C5A—C6A—C7A	108.91 (11)
N21—C2—C3	124.53 (10)	C5A—C6A—C7'	108.06 (15)
N21—C2—S1	122.72 (9)	C5A—C6A—H6C	109.9
C3—C2—S1	112.75 (8)	C7A—C6A—H6C	109.9
C2—C3—C4	111.59 (10)	C7'—C6A—H6C	134.9
C2—C3—C31	120.03 (10)	C5A—C6A—H6D	109.9
C4—C3—C31	128.36 (10)	C7A—C6A—H6D	109.9
C5—C4—C3	111.66 (10)	H6C—C6A—H6D	108.3
C5—C4—C9	121.17 (10)	C5A—C6A—H6E	110.1
C3—C4—C9	127.15 (10)	C7'—C6A—H6E	110.1
C4—C5—C6	125.97 (10)	C5A—C6A—H6F	110.1
C4—C5—S1	113.11 (8)	C7'—C6A—H6F	110.1
C6—C5—S1	120.92 (8)	H6E—C6A—H6F	108.4
C5—C6—C7	109.80 (10)	C8A—C7A—C6A	108.0 (2)
C5—C6—H6A	109.7	C8A—C7A—H7A1	110.1
C7—C6—H6A	109.7	C6A—C7A—H7A1	110.1
C5—C6—H6B	109.7	C8A—C7A—H7A2	110.1
C7—C6—H6B	109.7	C6A—C7A—H7A2	110.1
H6A—C6—H6B	108.2	H7A1—C7A—H7A2	108.4

C8—C7—C6	111.64 (11)	C7A—C8A—C9A	110.2 (2)
C8—C7—H7A	109.3	C7A—C8A—H8A1	109.6
C6—C7—H7A	109.3	C9A—C8A—H8A1	109.6
C8—C7—H7B	109.3	C7A—C8A—H8A2	109.6
C6—C7—H7B	109.3	C9A—C8A—H8A2	109.6
H7A—C7—H7B	108.0	H8A1—C8A—H8A2	108.1
C7—C8—C9	112.01 (12)	C8'—C7'—C6A	108.8 (3)
C7—C8—H8A	109.2	C8'—C7'—H7'1	109.9
C9—C8—H8A	109.2	C6A—C7'—H7'1	109.9
C7—C8—H8B	109.2	C8'—C7'—H7'2	109.9
C9—C8—H8B	109.2	C6A—C7'—H7'2	109.9
H8A—C8—H8B	107.9	H7'1—C7'—H7'2	108.3
C4—C9—C8	111.02 (10)	C7'—C8'—C9A	107.7 (4)
C4—C9—H9A	109.4	C7'—C8'—H8'1	110.2
C8—C9—H9A	109.4	C9A—C8'—H8'1	110.2
C4—C9—H9B	109.4	C7'—C8'—H8'2	110.2
C8—C9—H9B	109.4	C9A—C8'—H8'2	110.2
H9A—C9—H9B	108.0	H8'1—C8'—H8'2	108.5
C21—N21—C2	125.52 (10)	C4A—C9A—C8A	110.12 (12)
C21—N21—H21	121.9 (11)	C4A—C9A—C8'	111.15 (17)
C2—N21—H21	112.6 (11)	C4A—C9A—H9C	109.6
O21—C21—N21	121.98 (11)	C8A—C9A—H9C	109.6
O21—C21—C22	124.46 (11)	C8'—C9A—H9C	131.3
N21—C21—C22	113.57 (10)	C4A—C9A—H9D	109.6
C21—C22—C23	112.79 (11)	C8A—C9A—H9D	109.6
C21—C22—H22A	109.0	H9C—C9A—H9D	108.2
C23—C22—H22A	109.0	C4A—C9A—H9E	109.4
C21—C22—H22B	109.0	C8'—C9A—H9E	109.4
C23—C22—H22B	109.0	C4A—C9A—H9F	109.4
H22A—C22—H22B	107.8	C8'—C9A—H9F	109.4
C22—C23—H23D	109.5	H9E—C9A—H9F	108.0
C22—C23—H23E	109.5	C21A—N21A—C2A	126.02 (10)
H23D—C23—H23E	109.5	C21A—N21A—H21A	120.6 (12)
C22—C23—H23F	109.5	C2A—N21A—H21A	113.4 (12)
H23D—C23—H23F	109.5	O21A—C21A—N21A	122.00 (11)
H23E—C23—H23F	109.5	O21A—C21A—C22A	124.30 (11)
O31—C31—O32	121.97 (10)	N21A—C21A—C22A	113.70 (10)
O31—C31—C3	124.32 (11)	C21A—C22A—C23A	113.24 (11)
O32—C31—C3	113.70 (10)	C21A—C22A—H22C	108.9
C31—O32—C33	115.53 (9)	C23A—C22A—H22C	108.9
O32—C33—C34	107.40 (10)	C21A—C22A—H22D	108.9
O32—C33—H33A	110.2	C23A—C22A—H22D	108.9
C34—C33—H33A	110.2	H22C—C22A—H22D	107.7
O32—C33—H33B	110.2	C22A—C23A—H23A	109.5
C34—C33—H33B	110.2	C22A—C23A—H23B	109.5
H33A—C33—H33B	108.5	H23A—C23A—H23B	109.5
C33—C34—H34A	109.5	C22A—C23A—H23C	109.5
C33—C34—H34B	109.5	H23A—C23A—H23C	109.5
H34A—C34—H34B	109.5	H23B—C23A—H23C	109.5

C33—C34—H34C	109.5	O31A—C31A—O32A	122.25 (10)
H34A—C34—H34C	109.5	O31A—C31A—C3A	124.05 (11)
H34B—C34—H34C	109.5	O32A—C31A—C3A	113.70 (10)
C2A—S1A—C5A	90.98 (5)	C31A—O32A—C33A	115.44 (9)
C3A—C2A—N21A	124.70 (10)	O32A—C33A—C34A	107.80 (10)
C3A—C2A—S1A	112.64 (8)	O32A—C33A—H33C	110.1
N21A—C2A—S1A	122.66 (9)	C34A—C33A—H33C	110.1
C2A—C3A—C4A	111.75 (10)	O32A—C33A—H33D	110.1
C2A—C3A—C31A	120.08 (10)	C34A—C33A—H33D	110.1
C4A—C3A—C31A	128.15 (10)	H33C—C33A—H33D	108.5
C5A—C4A—C3A	111.69 (10)	C33A—C34A—H34D	109.5
C5A—C4A—C9A	121.16 (10)	C33A—C34A—H34E	109.5
C3A—C4A—C9A	127.15 (10)	H34D—C34A—H34E	109.5
C4A—C5A—C6A	125.90 (11)	C33A—C34A—H34F	109.5
C4A—C5A—S1A	112.94 (8)	H34D—C34A—H34F	109.5
C6A—C5A—S1A	121.13 (9)	H34E—C34A—H34F	109.5
C5—S1—C2—N21	−179.53 (11)	C2A—C3A—C4A—C5A	0.19 (15)
C5—S1—C2—C3	0.31 (10)	C31A—C3A—C4A—C5A	−178.48 (12)
N21—C2—C3—C4	178.68 (11)	C2A—C3A—C4A—C9A	−179.95 (12)
S1—C2—C3—C4	−1.15 (13)	C31A—C3A—C4A—C9A	1.4 (2)
N21—C2—C3—C31	−2.91 (19)	C3A—C4A—C5A—C6A	177.54 (12)
S1—C2—C3—C31	177.26 (9)	C9A—C4A—C5A—C6A	−2.33 (19)
C2—C3—C4—C5	1.64 (14)	C3A—C4A—C5A—S1A	−0.62 (13)
C31—C3—C4—C5	−176.59 (12)	C9A—C4A—C5A—S1A	179.51 (9)
C2—C3—C4—C9	−176.55 (11)	C2A—S1A—C5A—C4A	0.68 (10)
C31—C3—C4—C9	5.2 (2)	C2A—S1A—C5A—C6A	−177.58 (11)
C3—C4—C5—C6	179.49 (11)	C4A—C5A—C6A—C7A	−16.7 (2)
C9—C4—C5—C6	−2.19 (19)	S1A—C5A—C6A—C7A	161.29 (15)
C3—C4—C5—S1	−1.43 (13)	C4A—C5A—C6A—C7'	18.1 (3)
C9—C4—C5—S1	176.89 (9)	S1A—C5A—C6A—C7'	−163.9 (2)
C2—S1—C5—C4	0.67 (10)	C5A—C6A—C7A—C8A	50.6 (3)
C2—S1—C5—C6	179.80 (10)	C7'—C6A—C7A—C8A	−43.6 (3)
C4—C5—C6—C7	−12.61 (17)	C6A—C7A—C8A—C9A	−69.6 (3)
S1—C5—C6—C7	168.38 (9)	C5A—C6A—C7'—C8'	−51.3 (4)
C5—C6—C7—C8	43.83 (16)	C7A—C6A—C7'—C8'	45.8 (3)
C6—C7—C8—C9	−63.31 (16)	C6A—C7'—C8'—C9A	69.7 (5)
C5—C4—C9—C8	−14.42 (17)	C5A—C4A—C9A—C8A	−13.5 (2)
C3—C4—C9—C8	163.61 (12)	C3A—C4A—C9A—C8A	166.65 (17)
C7—C8—C9—C4	46.10 (16)	C5A—C4A—C9A—C8'	18.6 (3)
C3—C2—N21—C21	−176.11 (12)	C3A—C4A—C9A—C8'	−161.3 (3)
S1—C2—N21—C21	3.70 (19)	C7A—C8A—C9A—C4A	48.8 (3)
C2—N21—C21—O21	−1.2 (2)	C7A—C8A—C9A—C8'	−48.9 (3)
C2—N21—C21—C22	178.67 (12)	C7'—C8'—C9A—C4A	−51.7 (4)
O21—C21—C22—C23	8.7 (2)	C7'—C8'—C9A—C8A	42.1 (3)
N21—C21—C22—C23	−171.16 (13)	C3A—C2A—N21A—C21A	−179.51 (12)
C2—C3—C31—O31	−1.6 (2)	S1A—C2A—N21A—C21A	0.99 (18)
C4—C3—C31—O31	176.48 (12)	C2A—N21A—C21A—O21A	2.0 (2)
C2—C3—C31—O32	178.38 (11)	C2A—N21A—C21A—C22A	−177.37 (12)
C4—C3—C31—O32	−3.51 (18)	O21A—C21A—C22A—C23A	13.0 (2)

O31—C31—O32—C33	0.26 (18)	N21A—C21A—C22A—C23A	−167.74 (13)
C3—C31—O32—C33	−179.74 (10)	C2A—C3A—C31A—O31A	1.3 (2)
C31—O32—C33—C34	−178.46 (12)	C4A—C3A—C31A—O31A	179.83 (13)
C5A—S1A—C2A—C3A	−0.56 (10)	C2A—C3A—C31A—O32A	−178.83 (11)
C5A—S1A—C2A—N21A	178.99 (11)	C4A—C3A—C31A—O32A	−0.25 (18)
N21A—C2A—C3A—C4A	−179.22 (11)	O31A—C31A—O32A—C33A	2.02 (18)
S1A—C2A—C3A—C4A	0.32 (13)	C3A—C31A—O32A—C33A	−177.89 (10)
N21A—C2A—C3A—C31A	−0.43 (19)	C31A—O32A—C33A—C34A	−176.70 (11)
S1A—C2A—C3A—C31A	179.11 (9)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21···O31	0.882 (19)	1.971 (18)	2.6872 (13)	137.3 (15)
N21A—H21A···O31A	0.91 (2)	1.960 (19)	2.6859 (14)	135.6 (16)

Fig. 1

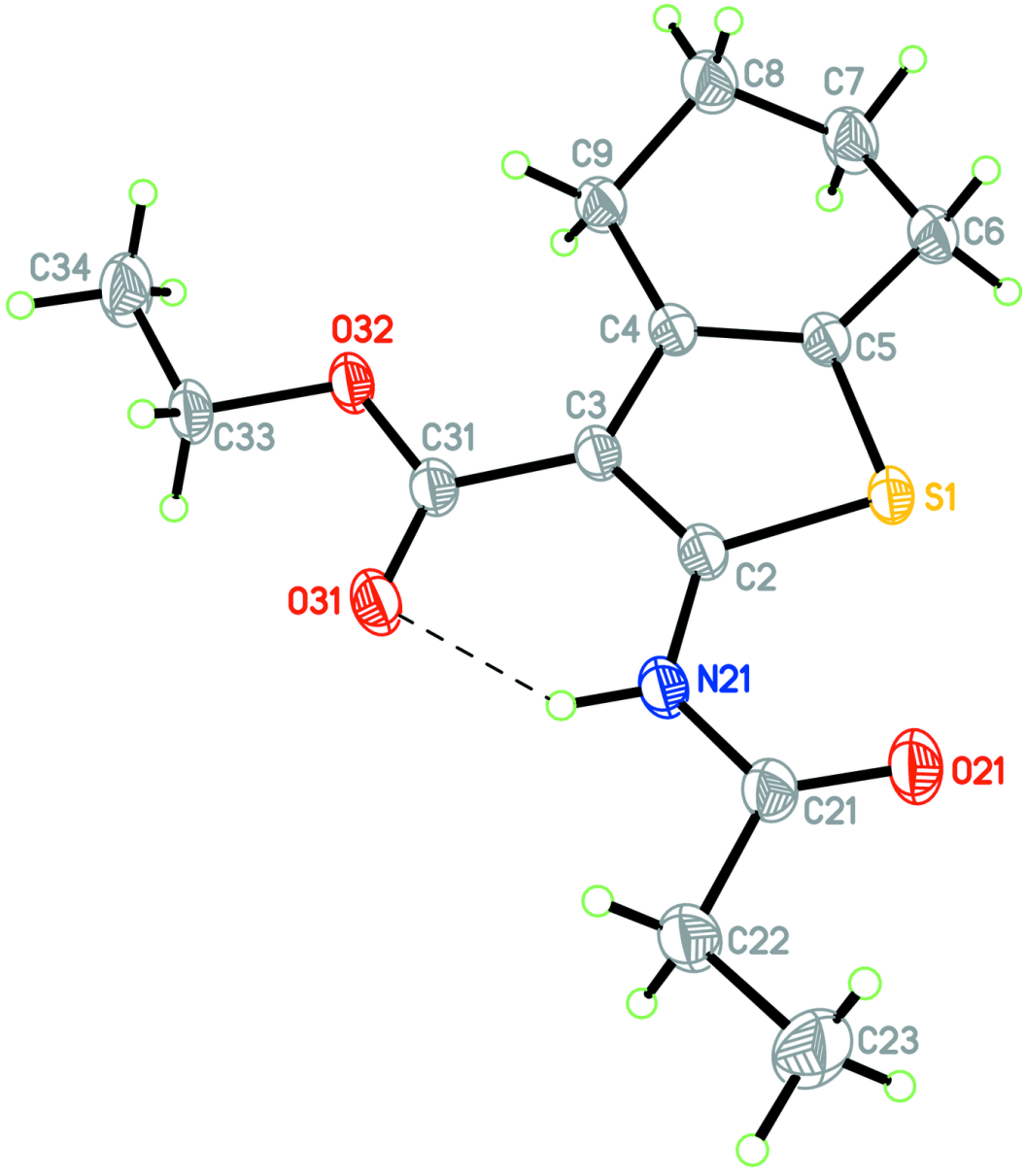


Fig. 2

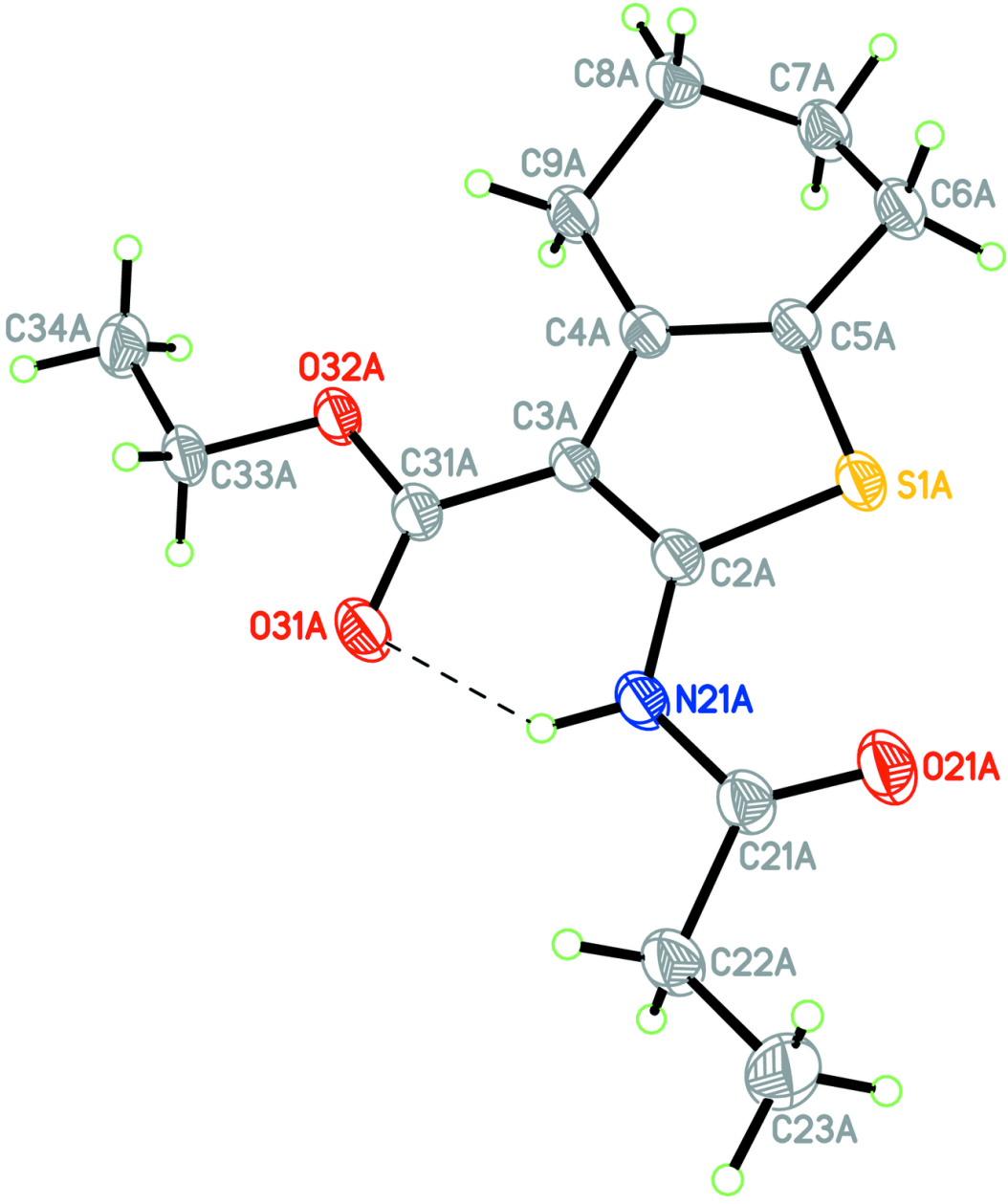

C8A
C7A
C9A
C6A
C34A
C4A
C5A
O32A
C3A
C31A
S1A
C33A
C2A
O31A
N21A
O21A
C21A
C22A
C23A

Fig. 3

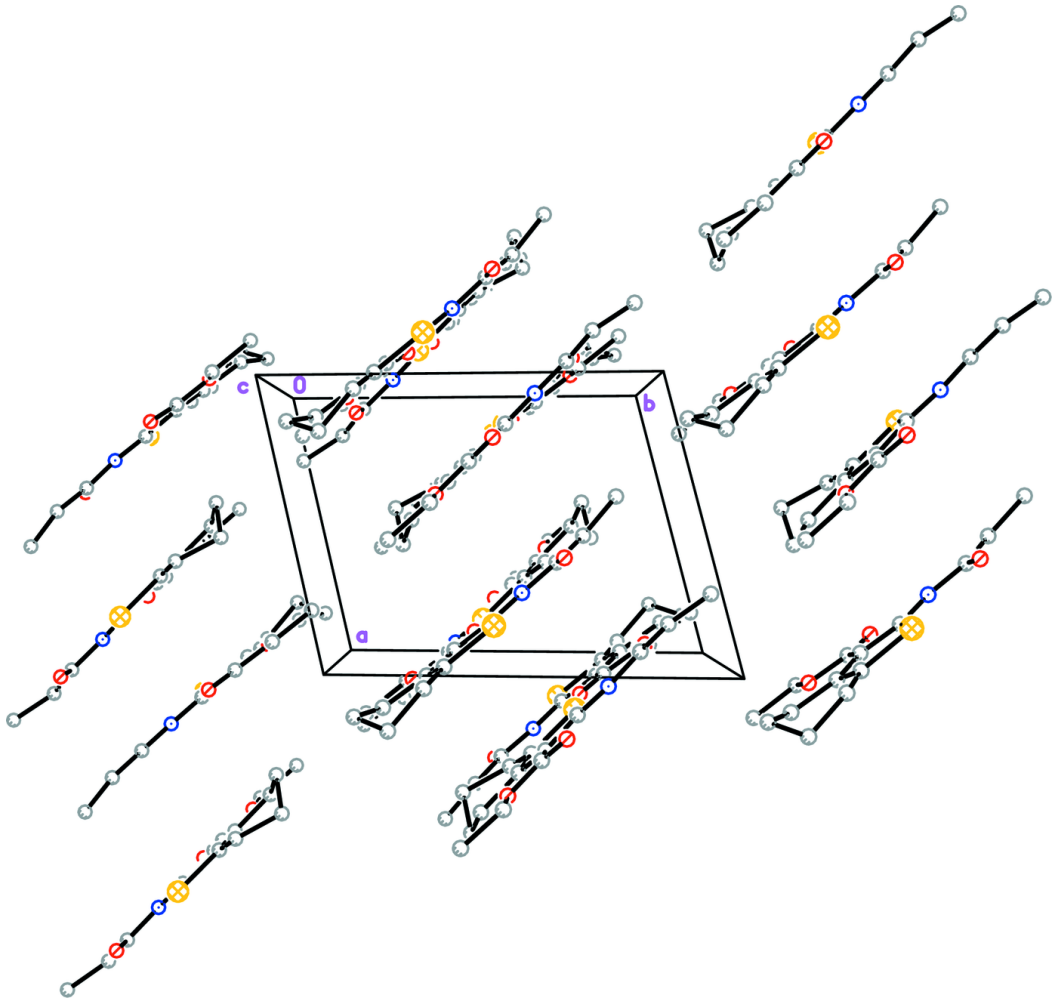
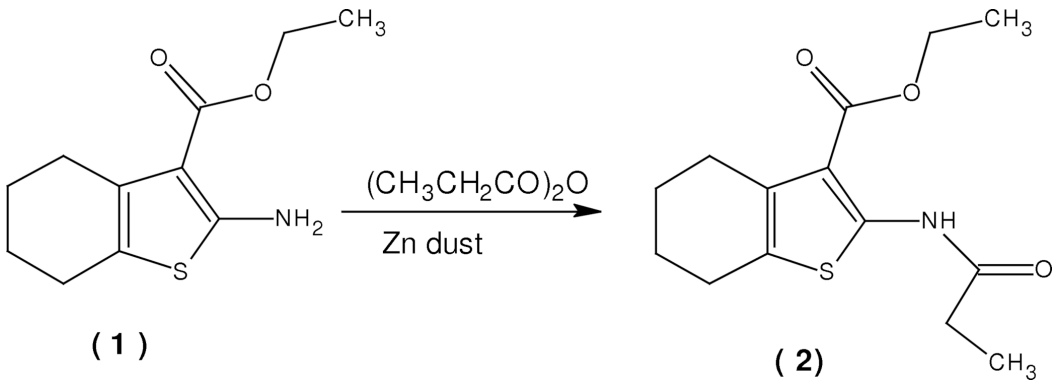


Fig. 4



(1) **(2)**