

Ethyl 2-cyano-2-(1,3-dithiepan-2-ylidene)acetate

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Received 27 November 2025

Accepted 18 December 2025

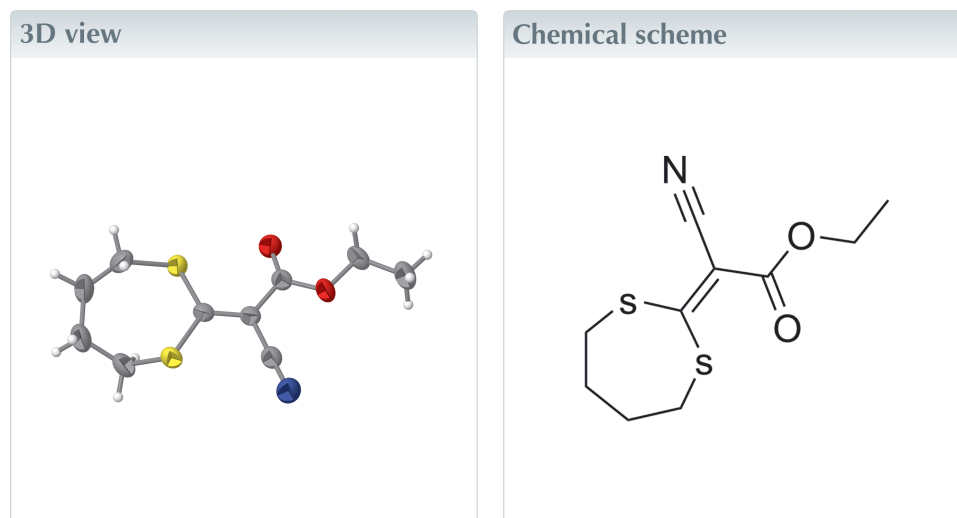
Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

Keywords: crystal structure; Hirshfeld surface analysis.

CCDC reference: 2517231

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

In the title compound, C₁₀H₁₃NO₂S₂, the seven-membered 1,3-dithiepane ring adopts a distorted chair conformation. In the extended structure, inversion dimers linked by pairwise C–H···O hydrogen bonds generate R₂²(14) loops.



Structure description

The title compound, C₁₀H₁₃NO₂S₂, is commercially available. It crystallizes in the triclinic space group $P\bar{1}$ with one molecule in the asymmetric unit (Fig. 1). The seven-membered heterocyclic ring adopts a distorted chair conformation with C4/C6/C7/S1/S2 roughly coplanar (r.m.s. deviation = 0.207 Å) and C5 and C8 deviating from their best plane by –0.849 (3) and 0.869 (3) Å, respectively. The C4–S1 [1.746 (2) Å] and C4–S3 [1.733 (2) Å] bond lengths show slight asymmetry. The cyanide group and O1 have an *anti* orientation [O1–C1–C2–C3 = 174.1 (2)°] and the ester chain is extended [C1–O2–C9–C10 = –179.4 (2)°]. In the extended structure, inversion dimers linked by pairwise C8–H8B···O1 hydrogen bonds (Table 1) generate R₂²(14) loops (Fig. 2). Otherwise, there are no notable short contacts.

The program *CrystalExplorer* (Turner *et al.*, 2017) was used to generate the Hirshfeld surface (supplementary Fig. 1) and two-dimensional fingerprint plots for the title compound. The full two-dimensional fingerprint plot for the title compound is given in Fig. 3(a) and the different contact types are shown in Fig. 3(b)–(f). The greatest contribution to the overall Hirshfeld surface is due to H···H contacts, which contribute 40.4%, followed by H···O/O···H (15.9%), N···H/H···N (15.8%), H···S/S···H (12.1%) while S···S gives a small contribution of 1.3%.

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C8-H8B\cdots O1^i$	0.95 (3)	2.58 (3)	3.484 (3)	160 (2)

Symmetry code: (i) $-x + 1, -y + 2, -z$.

Synthesis and crystallization

The title compound, obtained commercially, was recrystallized from ethanol solution, yielding colorless prismatic crystals suitable for single-crystal X-ray diffraction analysis.

Refinement

Crystallographic data and refinement parameters for the title compound are given in Table 2.

Acknowledgements

This work was supported by the Laboratoire de Cristallographie, Departement de Physique, Universite Constantine 1, Algeria. We thank Mr F. Saidi, Engineer at the Laboratory of Crystallography, University Constantine 1, for assistance in collecting data on the Xcalibur X-ray diffractometer.

References

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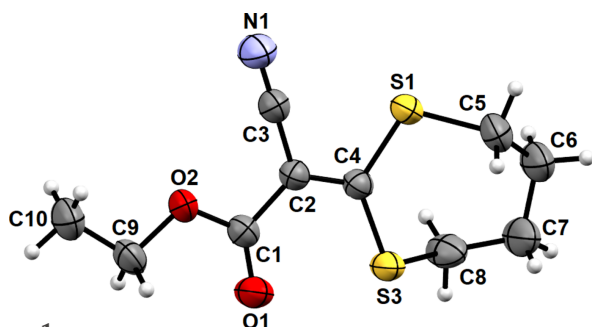


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

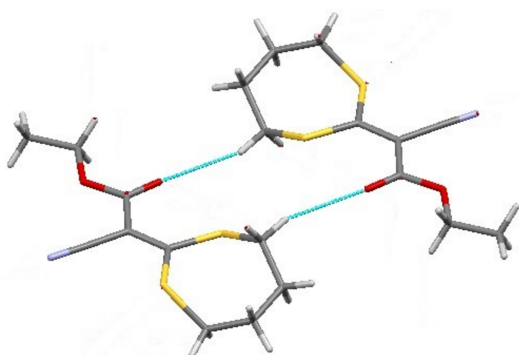


Figure 2

An inversion dimer in the extended structure of the title compound linked by pairwise $C-H\cdots O$ hydrogen bonds.

Table 2

Experimental details.

Crystal data	
Chemical formula	$C_{10}H_{13}NO_2S_2$
M_r	243.33
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
a, b, c (Å)	6.4761 (8), 9.5708 (11), 10.6436 (12)
α, β, γ (°)	71.347 (10), 78.799 (10), 71.986 (11)
V (Å ³)	591.08 (13)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.43
Crystal size (mm)	$0.5 \times 0.29 \times 0.15$
Data collection	
Diffractometer	Xcalibur, Eos
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2013)
T_{min}, T_{max}	0.986, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3567, 2274, 1684
R_{int}	0.029
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.042, 0.098, 0.99
No. of reflections	2274
No. of parameters	152
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	0.21, -0.20

Computer programs: *CrysAlis PRO* (Agilent, 2013), *SHELXT* (Sheldrick, 2015a), *SHELXL2014/7* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

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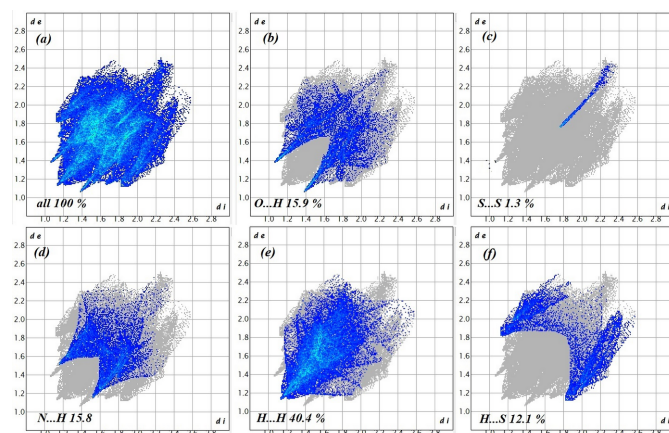


Figure 3

(a) The full two-dimensional Hirshfeld fingerprint plot for the title compound and those delineated into (b) $O\cdots H/H\cdots O$, (c) $S\cdots S$, (d) $N\cdots H/H\cdots N$, (e) $H\cdots H$ and (f) $H\cdots S/S\cdots H$ contacts.

full crystallographic data

IUCrData (2025). **10**, x251142 [<https://doi.org/10.1107/S2414314625011423>]

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Crystal data

$C_{10}H_{13}NO_2S_2$

$M_r = 243.33$

Triclinic, $P\bar{1}$

$a = 6.4761$ (8) Å

$b = 9.5708$ (11) Å

$c = 10.6436$ (12) Å

$\alpha = 71.347$ (10)°

$\beta = 78.799$ (10)°

$\gamma = 71.986$ (11)°

$V = 591.08$ (13) Å³

$Z = 2$

$F(000) = 256$

$D_x = 1.367$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å

Cell parameters from 1290 reflections

$\theta = 3.7$ – 26.3 °

$\mu = 0.43$ mm⁻¹

$T = 293$ K

Needle, yellow

$0.5 \times 0.29 \times 0.15$ mm

Data collection

Xcalibur, Eos

diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 8.0226 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(CrysAlisPro; Agilent, 2013)

$T_{\min} = 0.986$, $T_{\max} = 1.000$

3567 measured reflections

2274 independent reflections

1684 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.5$ °

$h = -7 \rightarrow 8$

$k = -11 \rightarrow 12$

$l = -12 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.098$

$S = 0.99$

2274 reflections

152 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent

and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.21$ e Å⁻³

$\Delta\rho_{\min} = -0.20$ e Å⁻³

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. The H atoms attached to C5 and C8 were freely refined. The other b H atoms were included in calculated positions and treated as riding (C—H = 0.95–0.98 Å) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.84626 (9)	0.63684 (6)	0.37317 (6)	0.0493 (2)
S3	0.54520 (10)	0.86994 (6)	0.17303 (6)	0.0526 (2)
O1	0.3040 (3)	0.70355 (19)	0.11035 (17)	0.0592 (6)
O2	0.3664 (2)	0.44937 (17)	0.19391 (15)	0.0495 (5)
N1	0.6992 (4)	0.2995 (2)	0.4281 (2)	0.0666 (8)
C1	0.3971 (3)	0.5847 (3)	0.1832 (2)	0.0412 (7)
C2	0.5574 (3)	0.5687 (2)	0.27179 (19)	0.0379 (7)
C3	0.6340 (3)	0.4190 (3)	0.3604 (2)	0.0442 (8)
C4	0.6379 (3)	0.6850 (2)	0.27224 (19)	0.0381 (7)
C5	0.7925 (5)	0.7955 (3)	0.4434 (3)	0.0614 (10)
C6	0.8948 (5)	0.9230 (3)	0.3654 (3)	0.0768 (11)
C7	0.7828 (5)	1.0306 (3)	0.2451 (3)	0.0806 (11)
C8	0.7696 (5)	0.9547 (3)	0.1443 (3)	0.0602 (10)
C9	0.2088 (4)	0.4526 (3)	0.1127 (2)	0.0534 (8)
C10	0.1962 (5)	0.2933 (3)	0.1393 (3)	0.0743 (11)
H5A	0.851 (4)	0.744 (3)	0.527 (2)	0.0631*
H5B	0.634 (4)	0.837 (3)	0.457 (2)	0.057 (7)*
H6A	0.89577	0.98185	0.42431	0.0922*
H6B	1.04552	0.87868	0.33590	0.0922*
H7A	0.86041	1.10835	0.20172	0.0968*
H7B	0.63584	1.08175	0.27513	0.0968*
H8A	0.903 (5)	0.880 (3)	0.126 (3)	0.086 (9)*
H8B	0.744 (4)	1.032 (3)	0.063 (3)	0.076 (8)*
H9A	0.06691	0.51712	0.13520	0.0640*
H9B	0.25414	0.49338	0.01901	0.0640*
H10A	0.15400	0.25341	0.23249	0.1116*
H10B	0.08997	0.29244	0.08811	0.1116*
H10C	0.33632	0.23112	0.11444	0.1116*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0545 (4)	0.0433 (3)	0.0528 (4)	−0.0111 (3)	−0.0257 (3)	−0.0071 (3)
S3	0.0595 (4)	0.0374 (3)	0.0624 (4)	−0.0137 (3)	−0.0307 (3)	−0.0011 (3)
O1	0.0602 (10)	0.0468 (10)	0.0693 (11)	−0.0136 (8)	−0.0301 (8)	−0.0016 (8)
O2	0.0539 (9)	0.0415 (9)	0.0615 (10)	−0.0106 (7)	−0.0250 (7)	−0.0166 (8)
N1	0.0785 (15)	0.0467 (13)	0.0755 (15)	−0.0181 (11)	−0.0331 (12)	−0.0024 (12)
C1	0.0392 (12)	0.0443 (13)	0.0411 (12)	−0.0115 (10)	−0.0061 (9)	−0.0119 (10)
C2	0.0398 (12)	0.0373 (12)	0.0360 (11)	−0.0083 (9)	−0.0066 (9)	−0.0096 (9)
C3	0.0448 (13)	0.0435 (14)	0.0475 (13)	−0.0124 (10)	−0.0104 (10)	−0.0130 (11)
C4	0.0402 (12)	0.0395 (12)	0.0339 (11)	−0.0078 (9)	−0.0075 (9)	−0.0099 (9)
C5	0.076 (2)	0.0617 (17)	0.0597 (17)	−0.0202 (15)	−0.0262 (14)	−0.0215 (14)
C6	0.094 (2)	0.0581 (17)	0.096 (2)	−0.0284 (15)	−0.0491 (17)	−0.0145 (16)
C7	0.096 (2)	0.0506 (16)	0.110 (2)	−0.0331 (15)	−0.0535 (18)	−0.0041 (16)
C8	0.0656 (18)	0.0481 (15)	0.0631 (18)	−0.0256 (14)	−0.0199 (14)	0.0075 (13)

C9	0.0493 (13)	0.0630 (16)	0.0568 (15)	-0.0130 (12)	-0.0194 (11)	-0.0225 (13)
C10	0.089 (2)	0.0657 (18)	0.087 (2)	-0.0281 (15)	-0.0379 (16)	-0.0226 (16)

Geometric parameters (Å, °)

S1—C4	1.746 (2)	C9—C10	1.484 (4)
S1—C5	1.812 (3)	C5—H5A	0.96 (2)
S3—C4	1.733 (2)	C5—H5B	0.98 (3)
S3—C8	1.806 (3)	C6—H6A	0.9700
O1—C1	1.205 (3)	C6—H6B	0.9700
O2—C1	1.337 (3)	C7—H7A	0.9700
O2—C9	1.448 (3)	C7—H7B	0.9700
N1—C3	1.139 (3)	C8—H8A	0.97 (3)
C1—C2	1.476 (3)	C8—H8B	0.95 (3)
C2—C3	1.437 (3)	C9—H9A	0.9700
C2—C4	1.369 (3)	C9—H9B	0.9700
C5—C6	1.502 (4)	C10—H10A	0.9600
C6—C7	1.510 (4)	C10—H10B	0.9600
C7—C8	1.503 (4)	C10—H10C	0.9600
C4—S1—C5	105.61 (12)	C7—C6—H6A	109.00
C4—S3—C8	105.23 (12)	C7—C6—H6B	109.00
C1—O2—C9	115.77 (18)	H6A—C6—H6B	108.00
O1—C1—O2	123.6 (2)	C6—C7—H7A	109.00
O1—C1—C2	125.1 (2)	C6—C7—H7B	109.00
O2—C1—C2	111.30 (19)	C8—C7—H7A	109.00
C1—C2—C3	116.38 (19)	C8—C7—H7B	109.00
C1—C2—C4	124.33 (19)	H7A—C7—H7B	108.00
C3—C2—C4	119.28 (19)	S3—C8—H8A	108.1 (19)
N1—C3—C2	178.2 (2)	S3—C8—H8B	103.0 (17)
S1—C4—S3	121.36 (12)	C7—C8—H8A	114.1 (19)
S1—C4—C2	116.79 (15)	C7—C8—H8B	107.2 (18)
S3—C4—C2	121.81 (16)	H8A—C8—H8B	106 (3)
S1—C5—C6	116.7 (2)	O2—C9—H9A	110.00
C5—C6—C7	114.9 (3)	O2—C9—H9B	110.00
C6—C7—C8	114.5 (2)	C10—C9—H9A	110.00
S3—C8—C7	117.2 (2)	C10—C9—H9B	110.00
O2—C9—C10	107.7 (2)	H9A—C9—H9B	108.00
S1—C5—H5A	100.8 (17)	C9—C10—H10A	109.00
S1—C5—H5B	108.1 (16)	C9—C10—H10B	109.00
C6—C5—H5A	111.1 (17)	C9—C10—H10C	109.00
C6—C5—H5B	109.3 (15)	H10A—C10—H10B	109.00
H5A—C5—H5B	111 (2)	H10A—C10—H10C	110.00
C5—C6—H6A	109.00	H10B—C10—H10C	109.00
C5—C6—H6B	109.00		
C4—S1—C5—C6	-90.0 (3)	O2—C1—C2—C4	173.05 (19)
C5—S1—C4—C2	-141.98 (18)	O1—C1—C2—C4	-7.2 (4)

C5—S1—C4—S3	40.31 (17)	O2—C1—C2—C3	-5.7 (3)
C8—S3—C4—S1	24.32 (17)	C3—C2—C4—S1	4.9 (3)
C4—S3—C8—C7	-88.0 (2)	C1—C2—C4—S3	3.9 (3)
C8—S3—C4—C2	-153.28 (18)	C3—C2—C4—S3	-177.39 (16)
C1—O2—C9—C10	-179.4 (2)	C1—C2—C4—S1	-173.78 (16)
C9—O2—C1—C2	178.94 (17)	S1—C5—C6—C7	76.9 (3)
C9—O2—C1—O1	-0.9 (3)	C5—C6—C7—C8	-58.4 (4)
O1—C1—C2—C3	174.1 (2)	C6—C7—C8—S3	84.3 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C8—H8B \cdots O1 ⁱ	0.95 (3)	2.58 (3)	3.484 (3)	160 (2)

Symmetry code: (i) $-x+1, -y+2, -z$.