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O-Ethyl *S*-(3-oxoindan-1-yl) dithiocarbonate

Jens Hartung,[a]* Kristina Daniel,[a] Ingrid Svoboda[b] and Hartmut Fuess[b]

[a]Fachbereich Chemie, Organische Chemie, Technische Universität Kaiserslautern, Erwin-Schrödinger-Strasse, D-67663 Kaiserslautern, Germany, and [b]Strukturforschung, FB11 Material- und Geowissenschaften, Technische Universität Darmstadt, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: hartung@chemie.uni-kl.de

Key indicators

Single-crystal X-ray study
T = 300 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.145
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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The title compound, $C_{12}H_{12}O_2S_2$, is a chiral *O*,*S*-dialkyl dithiocarbonate. The *O*-ethyl substituent and the dithiocarbonate functionality form a plane, which is twisted by 60.7 (2)° from the S-(3-oxoindan-1-yl) group.

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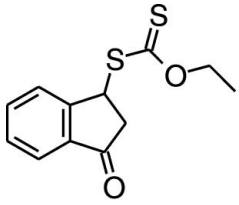
Comment

Atom C4 of the title compound is a stereogenic center. The racemate was prepared in the course of a study directed toward the development of novel thiazole-2(3*H*)-thione-derived alkoxyl radical precursors (Hartung, Daniel *et al.*, 2005). Its solid-state geometry was investigated by X-ray diffraction analysis.



(I)

The title compound, (I), crystallizes in the monoclinic space group $P2_1/c$ (Z = 4) as a 1:1 mixture of *S* and *R* enantiomers. The angle between the 3-oxoindan-1-yl plane [atom C4 deviates by 0.052 (4) Å and C5 by 0.066 (4) Å from the mean plane formed by atoms C6–C12] and the dithiocarbonate group is 62.1 (2)° [atom O1 deviates by 0.001 (6) Å from the plane formed by atoms C1/S1/S2] (Fig. 1). The C1═S1 distance [1.624 (3) Å] is slightly smaller than mean value of 1.67 (2) Å for the C═S double bond in other *O*,*S*-dialkyl dithiocarbonates (Abrahamsson & Innes, 1974; Allen *et al.*, 1987; Duarte *et al.*, 1989). The difference in the S2—C1 [1.716 (3) Å] and S2—C4 [1.808 (3) Å] bond lengths is explained by a change in hybridization from sp^2 at C1 to sp^3 at C4 (Zhang *et al.*, 2003; Hartung, Schmidt *et al.* 2005). The stereogenic bonds associated with the dithiocarbonate functionality exhibit an *E* configuration for C1—S2 and a *Z* geometry for C1—O2.

Experimental

A solution of 3-bromo-1-indanone (Minuti *et al.*, 2003) (0.30 g, 3.17 mmol) in acetone (10 ml) was added dropwise (10 min) to a suspension of potassium *O*-ethyl dithiocarbonate (6.0 g, 3.80 mmol) in acetone (20 ml). Stirring was continued for 1 h at 293 K. The solution was subsequently concentrated under reduced pressure to furnish a yellow residue, which was taken up in H_2O and Et_2O. The phases were separated. The aqueous phase was extracted with Et_2O. The combined organic phases were washed with H_2O and dried ($MgSO_4$). The solvent was removed *in vacuo* to furnish a crude

product, which was purified by chromatography [R_F = 0.80, SiO_2, petroleum ether/Et_2O (3:1, *v*/*v*)] to afford 0.63 g (79%) of (I) as a yellow solid. Yellow prism-shaped crystals suitable for X-ray diffraction were obtained by slowly concentrating a saturated solution of (I) in EtOH at 298 K. Analysis calculated for $C_{12}H_{12}O_2S_2$: C 57.11, H 8.79, S 25.41%; found: C 57.30, H 9.09, S 25.08%. MS (EI, 70 eV): *m*/*z* 252 (M^+, 11), 163 (8), 131 (100), 103 (27). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.42 (*t*, 3H, *J* = 7.2 Hz), 2.86 (*dd*, 1H, *J* = 19.6, 3.3 Hz), 3.44 (*dd*, 1H, *J* = 19.6 and 7.6 Hz), 4.68 (*dq*, 2H, *J* = 3.0 and 7.1 Hz), 5.36 (*dd*, 1H, *J* = 3.3, 7.7 Hz), 7.45–7.50 (*m*, 1H), 7.64–7.68 (*m*, 2H), 7.79 (m_c, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.1, 45.9, 46.4, 70.8, 124.2, 127.0, 129.5, 135.7, 137.7, 152.1, 203.3, 213.3.

Crystal data

$C_{12}H_{12}O_2S_2$	D_x = 1.403 Mg m^{-3}
M_r = 252.34	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 703 reflections
a = 11.287 (6) Å	θ = 2.9–19.1°
b = 11.606 (6) Å	μ = 0.43 mm^{-1}
c = 9.864 (6) Å	T = 300 (2) K
β = 112.39 (7)°	Prism, yellow
V = 1194.7 (11) Å^3	0.78 × 0.76 × 0.42 mm
Z = 4	

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector	6892 measured reflections
	2426 independent reflections
	1561 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.042
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2002)	θ_{max} = 26.4°
	$h = -9 \rightarrow 14$
	$k = -14 \rightarrow 14$
T_{min} = 0.718, T_{max} = 0.881	$l = -12 \rightarrow 11$

Refinement

Refinement on F^2	Only H-atom coordinates refined
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.0844P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.145	
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.010
2426 reflections	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
182 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.312 (3)	C2—O1	1.455 (3)
C1—S1	1.624 (3)	C4—S2	1.808 (3)
C1—S2	1.716 (3)	C6—O2	1.204 (3)
O1—C1—S1	127.2 (2)	O2—C6—C5	125.4 (2)
O1—C1—S2	114.66 (18)	C7—C6—C5	108.1 (2)
S1—C1—S2	118.13 (16)	C1—O1—C2	118.7 (2)
O2—C6—C7	126.4 (2)	C1—S2—C4	107.27 (12)
C8—C4—C5—C6	−2.2 (2)	O1—C1—S2—C4	2.2 (2)
C4—C5—C6—O2	−176.4 (2)	S1—C1—S2—C4	−177.74 (14)
S1—C1—O1—C2	1.4 (4)	C8—C4—S2—C1	60.7 (2)
S2—C1—O1—C2	−178.6 (2)	C5—C4—S2—C1	−62.1 (2)

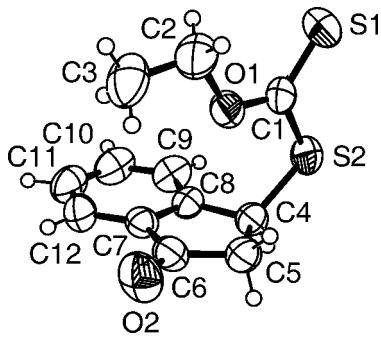


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

All H atoms were located in a difference Fourier map and refined. Their displacement parameters were constrained with U_{iso}(H) values set at 1.2U_{eq} of the parent atom.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON2002* (Spek, 2003) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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