

2-Oxo-2-(2-thienyl)acetic acid

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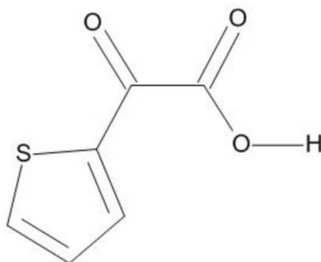
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; disorder in main residue; R factor = 0.037; wR factor = 0.106; data-to-parameter ratio = 18.5.

The structure of the title compound, $\text{C}_6\text{H}_4\text{O}_3\text{S}$, displays intermolecular hydrogen-bonding dimers. The structure exhibits a thienyl-ring flip disorder of the main molecule [occupancy ratio = 91.3 (2):8.7 (2)].

Related literature

For a discussion of ring-flip disorder in unsubstituted 2- and 3-thienyl rings, see: Crundwell *et al.* (2003). For information on simple $\text{O}-\text{H}\cdots\text{O}$ interactions, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_6\text{H}_4\text{O}_3\text{S}$	$V = 614.7$ (3) Å ³
$M_r = 156.15$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 3.7481$ (10) Å	$\mu = 0.46$ mm ⁻¹
$b = 15.314$ (3) Å	$T = 293$ K
$c = 10.727$ (3) Å	$0.34 \times 0.21 \times 0.11$ mm
$\beta = 93.30$ (2)°	

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer	6475 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2009)	1927 independent reflections
$T_{\min} = 0.944$, $T_{\max} = 1.000$	1512 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	12 restraints
$wR(F^2) = 0.106$	H-atom parameters constrained
$S = 1.09$	$\Delta\rho_{\text{max}} = 0.51$ e Å ⁻³
1927 reflections	$\Delta\rho_{\text{min}} = -0.29$ e Å ⁻³
104 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	1.82	2.637 (2)	176

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); 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) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

This work was funded by a CSU-AAUP Faculty Research Grant.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2317).

References

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supplementary materials

Acta Cryst. (2010). E66, o3056 [doi:10.1107/S160053681004403X]

2-Oxo-2-(2-thienyl)acetic acid

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Comment

The structure of 2-oxo-2-(2-thienyl)acetic acid, C₆H₄O₃S, has monoclinic (*P*2₁/*c*) symmetry. The structure displays intermolecular hydrogen bonding dimers. The structure exhibits a thienyl-ring flip disorder of the main molecule.

The structure of the title compound displays centrosymmetric *R*₂²(8) dimers by a simple O—H...O interactions (Bernstein *et al.*, 1995). The structure exhibits a thienyl-ring flip disorder of the main molecule with occupancy ratios of 91.3 (2)% to 8.7 (2)%.

Experimental

The title compound was purchased as 2-thiopheneglyoxylic acid from Aldrich (95% purity). Crystals for this *x*-ray diffraction study were harvested from methanol during routine recrystallization.

Refinement

During refinement, the thienyl ring showed evidence of ring-flip disorder which is common for unsubstituted 2- and 3-thienyl rings (Crundwell *et al.*, 2003). After finding three of the flipped disordered atoms in the difference map, the rest of the ring was generated and modeled. The final model suggested that the thienyl ring disorder was 8.7 (2)%.

Hydrogen atoms on carbons were included in calculated positions with a C—H distance of 0.93 Å and were included in the refinement in riding motion approximation with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of the carrier atom.

The hydroxyl hydrogen was included in a calculated position with a O—H distance of 0.82 Å and was included in the refinement in riding motion approximation with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of the carrier atom.

Figures

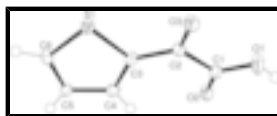


Fig. 1. A view of the title compound (Farrugia, 1997). Displacement ellipsoids are drawn at the 50% probability level.

2-Oxo-2-(2-thienyl)acetic acid

Crystal data

C₆H₄O₃S

F(000) = 320

M_r = 156.15

D_x = 1.687 Mg m⁻³

supplementary materials

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
 $a = 3.7481$ (10) Å
 $b = 15.314$ (3) Å
 $c = 10.727$ (3) Å
 $\beta = 93.30$ (2)°
 $V = 614.7$ (3) Å³
 $Z = 4$

Melting point: 361 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6632 reflections
 $\theta = 3.8$ – 32.0 °
 $\mu = 0.46$ mm⁻¹
 $T = 293$ K
Plate, yellow
 $0.34 \times 0.21 \times 0.11$ mm

Data collection

Oxford Diffraction Xcalibur Sapphire3
diffractometer
Radiation source: Enhance (Mo) X-ray Source
graphite
Detector resolution: 16.1790 pixels mm⁻¹
 ω scans
Absorption correction: multi-scan
(*CrysAlis PRO*; Oxford Diffraction, 2009)
 $T_{\min} = 0.944$, $T_{\max} = 1.000$
6475 measured reflections

1927 independent reflections
1512 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\max} = 32.0$ °, $\theta_{\min} = 3.8$ °
 $h = -5 \rightarrow 5$
 $k = -22 \rightarrow 16$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.106$
 $S = 1.09$
1927 reflections
104 parameters
12 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
 $w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.018P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.51$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

Special details

Experimental. Hydrogen atoms on carbons were included in calculated positions with a C—H distance of 0.93 Å and were included in the refinement in riding motion approximation with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of the carrier atom.

The hydroxyl hydrogen was included in a calculated position with a O—H distance of 0.82 Å and was included in the refinement in riding motion approximation with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of the carrier atom.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.1858 (3)	0.98317 (6)	0.36658 (10)	0.0261 (3)	
H1	0.2759	1.0208	0.4128	0.039*	
O2	0.5095 (3)	0.89316 (7)	0.49326 (9)	0.0207 (2)	
C1	0.2916 (4)	0.90590 (8)	0.40596 (12)	0.0167 (3)	
C2	0.1123 (4)	0.83170 (8)	0.32857 (12)	0.0156 (3)	
O3	-0.0839 (3)	0.85113 (7)	0.23733 (9)	0.0207 (2)	
C3	0.1813 (3)	0.74199 (8)	0.36784 (12)	0.0153 (3)	0.9131 (17)
C4	0.3362 (8)	0.70703 (19)	0.4767 (2)	0.0176 (4)	0.9131 (17)
H4	0.4368	0.7412	0.5413	0.021*	0.9131 (17)
C5	0.3282 (10)	0.61491 (14)	0.4812 (2)	0.0158 (3)	0.9131 (17)
H5	0.4176	0.5815	0.5484	0.019*	0.9131 (17)
C6	0.1687 (5)	0.58091 (10)	0.37166 (15)	0.0158 (3)	0.9131 (17)
H6	0.1417	0.5214	0.3564	0.019*	0.9131 (17)
S1	0.02766 (10)	0.65987 (2)	0.26826 (3)	0.01633 (14)	0.9131 (17)
C3B	0.1813 (3)	0.74199 (8)	0.36784 (12)	0.0153 (3)	0.0869 (17)
C4B	0.057 (4)	0.6842 (10)	0.2959 (14)	0.01633 (14)	0.0869 (17)
H4B	-0.0609	0.6947	0.2187	0.020*	0.0869 (17)
C5B	0.122 (6)	0.5982 (11)	0.350 (2)	0.0158 (3)	0.0869 (17)
H5B	0.0539	0.5454	0.3127	0.019*	0.0869 (17)
C6B	0.303 (13)	0.6081 (13)	0.464 (3)	0.0158 (3)	0.0869 (17)
H6B	0.3739	0.5612	0.5151	0.019*	0.0869 (17)
S1B	0.384 (3)	0.7158 (6)	0.5057 (7)	0.0176 (4)	0.0869 (17)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0349 (6)	0.0124 (4)	0.0291 (6)	0.0006 (4)	-0.0140 (4)	-0.0003 (4)
O2	0.0264 (5)	0.0153 (5)	0.0193 (5)	0.0004 (4)	-0.0080 (4)	-0.0003 (4)
C1	0.0180 (6)	0.0151 (6)	0.0170 (6)	-0.0003 (5)	0.0004 (5)	-0.0007 (5)
C2	0.0166 (6)	0.0150 (6)	0.0152 (6)	0.0009 (5)	-0.0005 (4)	-0.0003 (5)
O3	0.0245 (5)	0.0203 (5)	0.0166 (5)	0.0016 (4)	-0.0060 (4)	0.0009 (4)
C3	0.0159 (6)	0.0132 (6)	0.0165 (6)	0.0006 (5)	-0.0005 (5)	-0.0015 (5)
C4	0.0204 (11)	0.0172 (9)	0.0147 (13)	-0.0002 (7)	-0.0017 (9)	-0.0020 (9)
C5	0.0181 (9)	0.0144 (7)	0.0147 (10)	0.0001 (6)	-0.0009 (7)	-0.0009 (6)
C6	0.0160 (8)	0.0128 (7)	0.0184 (8)	0.0012 (6)	-0.0017 (6)	0.0023 (6)
S1	0.0178 (2)	0.0142 (2)	0.0167 (2)	-0.00080 (14)	-0.00148 (14)	-0.00162 (13)
C3B	0.0159 (6)	0.0132 (6)	0.0165 (6)	0.0006 (5)	-0.0005 (5)	-0.0015 (5)
C4B	0.0178 (2)	0.0142 (2)	0.0167 (2)	-0.00080 (14)	-0.00148 (14)	-0.00162 (13)
C5B	0.0160 (8)	0.0128 (7)	0.0184 (8)	0.0012 (6)	-0.0017 (6)	0.0023 (6)

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C6B	0.0181 (9)	0.0144 (7)	0.0147 (10)	0.0001 (6)	-0.0009 (7)	-0.0009 (6)
S1B	0.0204 (11)	0.0172 (9)	0.0147 (13)	-0.0002 (7)	-0.0017 (9)	-0.0020 (9)

Geometric parameters (Å, °)

O1—C1	1.3102 (16)	C5—C6	1.389 (2)
O1—H1	0.8200	C5—H5	0.9300
O2—C1	1.2223 (16)	C6—S1	1.7041 (15)
C1—C2	1.5387 (19)	C6—H6	0.9300
C2—O3	1.2265 (17)	C4B—C5B	1.452 (16)
C2—C3	1.4558 (18)	C4B—H4B	0.9300
C3—C4	1.382 (3)	C5B—C6B	1.380 (17)
C3—S1	1.7272 (13)	C5B—H5B	0.9300
C4—C5	1.412 (3)	C6B—S1B	1.730 (18)
C4—H4	0.9300	C6B—H6B	0.9300
C1—O1—H1	109.5	C6—C5—H5	124.6
O2—C1—O1	124.58 (12)	C4—C5—H5	124.6
O2—C1—C2	123.18 (12)	C5—C6—S1	112.77 (15)
O1—C1—C2	112.23 (11)	C5—C6—H6	123.6
O3—C2—C3	123.25 (12)	S1—C6—H6	123.6
O3—C2—C1	118.33 (12)	C6—S1—C3	91.96 (7)
C3—C2—C1	118.42 (11)	C5B—C4B—H4B	124.7
C4—C3—C2	132.00 (15)	C6B—C5B—C4B	108.5 (16)
C4—C3—S1	110.46 (14)	C6B—C5B—H5B	125.8
C2—C3—S1	117.44 (10)	C4B—C5B—H5B	125.8
C3—C4—C5	114.04 (18)	C5B—C6B—S1B	113.7 (16)
C3—C4—H4	123.0	C5B—C6B—H6B	123.1
C5—C4—H4	123.0	S1B—C6B—H6B	123.1
C6—C5—C4	110.76 (18)		

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>
O1—H1 \cdots O2 ⁱ	0.82	1.82	2.637 (2)	176

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

Fig. 1

