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Diethyl 2,2′-(trisulfane-1,3-diyl)dibenzoate

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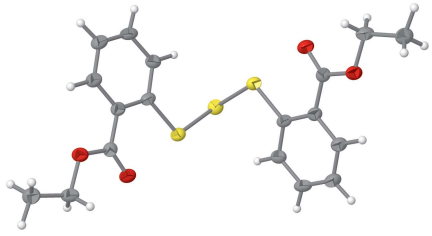
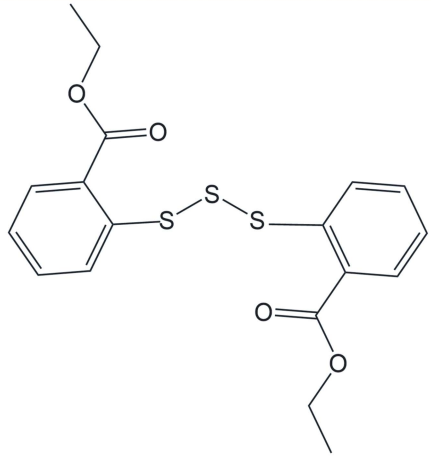
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{18}H_{18}O_4S_3$, was synthesized in the presence of chloridoauric acid from 3*H*-1,2-benzodithiole-3-thione as starting material. The asymmetric unit comprises one half of the molecule, the complete molecule being generated by the application of twofold rotation symmetry. The two benzene rings are inclined by 81.0 (2)°. In the crystal, slipped π–π stacking interactions are observed between the benzene rings of neighbouring molecules.

3D view



Chemical scheme



Structure description

3*H*-1,2-Benzodithiole-3-thione is a representative of 1,2-dithiole-3-thiones that define a bioactive family of compounds (Li *et al.*, 2016; Russell *et al.*, 2015). A bimolecular condensation reaction of 3*H*-1,2-benzodithiole-3-thione produced the title compound. The half-molecule present in the asymmetric unit is almost planar, with the maximum deviation being 0.114 (4) Å involving the carbonyl O atom of the ester group. The two equivalent S—S bond lengths of 2.0434 (17) Å and the S—S—S angle of 106.91 (11)° are typical for trisulfanyl groups (Fig. 1). The bent molecules are stacked along the *b* axis and interact through slightly displaced π–π stacking interactions [plane-to-plane separation between parallel benzene rings = 3.371 (7) Å] (Fig. 2).

Synthesis and crystallization

The title compound was prepared by a condensation reaction of two 3*H*-1,2-benzodithiole-3-thione molecules in the presence of $HAuCl_4{\cdot}3H_2O$ in ethanol. 3*H*-1,2-Benzodithiole-3-thione (90 mg) dissolved in 10 ml of absolute ethanol was added to 200 mg of $HAuCl_4{\cdot}3H_2O$ dissolved in 10 ml of THF. The mixture was stirred for 1 h under reflux, cooled to room temperature and left undisturbed for several hours. After filtra-

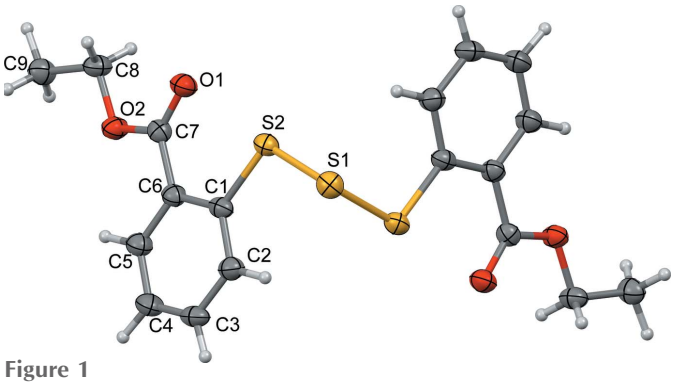


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius. Unlabelled atoms are related to the labelled atoms by symmetry code $(-x+1, y, -z+\frac{3}{2})$.

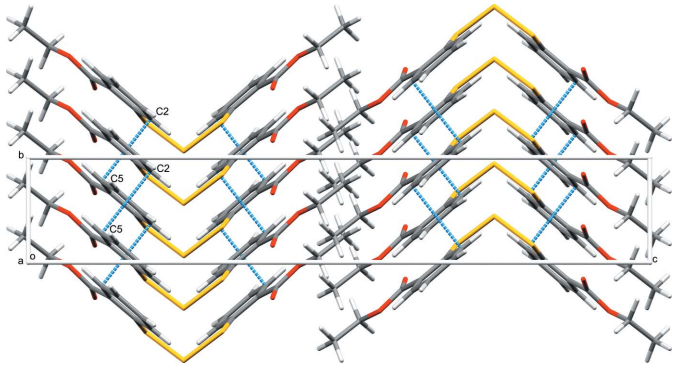


Figure 2
The packing of the molecules in the title structure in a view along the *a* axis, showing π–π stacking interactions as dashed blue lines.

tion, the remaining solution was evaporated and the product dissolved in diethyl ether from which crystals of the title compound were harvested after 5 d.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{18}O_4S_3$
M_r	394.54
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	150
a, *b*, *c* (Å)	15.071 (3), 4.4304 (11), 27.422 (9)
β (°)	106.59 (3)
V (Å^3)	1754.8 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.44
Crystal size (mm)	0.44 × 0.19 × 0.10
Data collection	
Diffractometer	Rigaku OF Xcalibur Atlas Gemini ultra
Absorption correction	Analytical (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.872, 0.963
No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections	6690, 2177, 1653
R_{int}	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.703
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.079, 0.166, 1.00
No. of reflections	2175
No. of parameters	142
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.89, −1.00

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SUPERFLIP* (Palatinus & Chapuis, 2007), *CRYSTALS* (Betteridge *et al.*, 2003) and *Mercury* (Macrae *et al.*, 2006).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161708 [https://doi.org/10.1107/S2414314616017089]

Diethyl 2,2′-(trisulfane-1,3-diyl)dibenzoate

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(I)

Crystal data

$C_{18}H_{18}O_4S_3$
M_r = 394.54
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 15.071 (3) Å
b = 4.4304 (11) Å
c = 27.422 (9) Å
β = 106.59 (3)°
V = 1754.8 (8) Å^3
Z = 4
F(000) = 824
D_x = 1.493 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1198 reflections
θ = 4.9–27.5°
μ = 0.44 mm^{-1}
T = 150 K
Lath, light yellow
0.44 × 0.19 × 0.10 mm

Data collection

Rigaku OF Xcalibur Atlas Gemini ultra diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.4685 pixels mm^{-1}
ω scans
Absorption correction: analytical (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min} = 0.872, T_{max} = 0.963
6690 measured reflections
2177 independent reflections
1653 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.058
θ_{max} = 30.0°, θ_{min} = 3.6°
h = −19→20
k = −6→6
l = −37→37

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.166
S = 1.00
2175 reflections
142 parameters
0 restraints
Primary atom site location: other
Hydrogen site location: difference Fourier map
All H-atom parameters refined
Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed belo*w* and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 0.372E + 04 0.520E + 04 0.373E + 04 0.166E + 04 752.
$(\Delta/\sigma)_{max}$ = 0.0002161
$\Delta\rho_{max}$ = 0.89 e Å^{-3}
$\Delta\rho_{min}$ = −1.00 e Å^{-3}
Extinction correction: Larson (1970), Equation 22
Extinction coefficient: 29 (8)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems open-flow nitrogen cryostat (Cosier & Glazer, 1986) with a nominal stability of 0.1K.
Cosier, J. & Glazer, A.M., 1986. J. Appl. Cryst. 105-107.

Refinement. Crystal data, data collection and structure refinement details are summarized in Table 1. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98 and N—H in the range 0.86–0.89 Å) and U_{iso}(H) (in the range 1.2-1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints (Cooper *et al.*, 2010).
Cooper, R. I., Thompson, A. L. & Watkin, D. J. (2010). *J. Appl. Cryst.* **43**, 1100–1107.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3715 (3)	1.0334 (11)	0.67128 (17)	0.0268
C2	0.3092 (3)	1.1298 (12)	0.69705 (18)	0.0321
C3	0.2187 (3)	1.0225 (12)	0.6825 (2)	0.0339
C4	0.1900 (3)	0.8208 (14)	0.6436 (2)	0.0386
C5	0.2512 (3)	0.7263 (12)	0.61804 (19)	0.0336
C6	0.3420 (3)	0.8314 (11)	0.63087 (16)	0.0279
C7	0.4044 (3)	0.7398 (10)	0.60108 (17)	0.0263
C8	0.4233 (3)	0.4435 (13)	0.5337 (2)	0.0342
C9	0.3649 (4)	0.2518 (13)	0.4921 (2)	0.0364
S1	0.5000	1.4389 (4)	0.7500	0.0320
S2	0.48814 (8)	1.1643 (3)	0.68860 (4)	0.0297
O2	0.3662 (2)	0.5346 (8)	0.56525 (12)	0.0319
O1	0.4814 (2)	0.8383 (9)	0.60692 (12)	0.0349
H21	0.330 (4)	1.268 (13)	0.726 (2)	0.0389*
H31	0.181 (4)	1.099 (13)	0.702 (2)	0.0410*
H41	0.127 (4)	0.736 (14)	0.632 (2)	0.0459*
H51	0.229 (4)	0.568 (13)	0.589 (2)	0.0403*
H81	0.473 (4)	0.340 (13)	0.554 (2)	0.0408*
H82	0.444 (4)	0.637 (14)	0.520 (2)	0.0408*
H91	0.402 (4)	0.177 (15)	0.468 (2)	0.0548*
H92	0.341 (4)	0.065 (16)	0.504 (2)	0.0551*
H93	0.314 (4)	0.370 (15)	0.471 (2)	0.0548*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0220 (19)	0.027 (2)	0.033 (2)	0.0025 (18)	0.0097 (17)	0.0072 (19)
C2	0.030 (2)	0.035 (3)	0.033 (2)	0.001 (2)	0.0129 (19)	0.002 (2)
C3	0.028 (2)	0.036 (3)	0.043 (3)	0.003 (2)	0.019 (2)	0.006 (2)
C4	0.025 (2)	0.051 (3)	0.041 (3)	−0.005 (2)	0.0115 (19)	0.002 (3)
C5	0.027 (2)	0.038 (3)	0.036 (2)	−0.003 (2)	0.0108 (19)	0.002 (2)
C6	0.025 (2)	0.029 (2)	0.030 (2)	−0.003 (2)	0.0088 (17)	0.002 (2)
C7	0.0258 (19)	0.023 (2)	0.029 (2)	−0.0018 (17)	0.0071 (17)	0.0015 (17)
C8	0.029 (2)	0.037 (3)	0.041 (3)	−0.004 (2)	0.017 (2)	−0.003 (2)
C9	0.033 (2)	0.035 (3)	0.041 (3)	−0.005 (2)	0.012 (2)	−0.009 (2)
S1	0.0334 (8)	0.0258 (9)	0.0365 (9)	0.0000	0.0094 (7)	0.0000

S2	0.0251 (5)	0.0321 (7)	0.0327 (6)	−0.0028 (5)	0.0098 (4)	0.0001 (5)
O2	0.0285 (15)	0.0348 (19)	0.0346 (17)	−0.0071 (15)	0.0127 (13)	−0.0075 (15)
O1	0.0261 (15)	0.042 (2)	0.0384 (17)	−0.0040 (15)	0.0120 (13)	−0.0035 (17)

Geometric parameters (Å, º)

C1—C2	1.394 (6)	C7—O2	1.342 (5)
C1—C6	1.395 (6)	C7—O1	1.208 (5)
C1—S2	1.782 (4)	C8—C9	1.490 (7)
C2—C3	1.390 (6)	C8—O2	1.442 (5)
C2—H21	0.98 (5)	C8—H81	0.92 (6)
C3—C4	1.364 (8)	C8—H82	1.02 (6)
C3—H31	0.95 (5)	C9—H91	1.02 (6)
C4—C5	1.373 (7)	C9—H92	0.99 (7)
C4—H41	0.99 (6)	C9—H93	0.97 (6)
C5—C6	1.393 (6)	S1—S2^{i}	2.0434 (17)
C5—H51	1.05 (6)	S1—S2	2.0434 (17)
C6—C7	1.467 (6)		
C2—C1—C6	119.4 (4)	C6—C7—O2	112.7 (4)
C2—C1—S2	121.4 (4)	C6—C7—O1	124.8 (4)
C6—C1—S2	119.1 (3)	O2—C7—O1	122.5 (4)
C1—C2—C3	119.8 (5)	C9—C8—O2	107.2 (4)
C1—C2—H21	120 (3)	C9—C8—H81	112 (4)
C3—C2—H21	120 (3)	O2—C8—H81	108 (3)
C2—C3—C4	121.0 (4)	C9—C8—H82	112 (3)
C2—C3—H31	115 (3)	O2—C8—H82	107 (3)
C4—C3—H31	124 (3)	H81—C8—H82	111 (4)
C3—C4—C5	119.3 (5)	C8—C9—H91	111 (3)
C3—C4—H41	124 (3)	C8—C9—H92	115 (4)
C5—C4—H41	117 (3)	H91—C9—H92	104 (5)
C4—C5—C6	121.6 (5)	C8—C9—H93	110 (4)
C4—C5—H51	119 (3)	H91—C9—H93	106 (5)
C6—C5—H51	120 (3)	H92—C9—H93	110 (5)
C1—C6—C5	118.8 (4)	S2^{i}—S1—S2	106.91 (11)
C1—C6—C7	120.7 (4)	C1—S2—S1	105.12 (16)
C5—C6—C7	120.4 (4)	C8—O2—C7	115.1 (3)

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.