



Crystal structures of 2-(benzenecarbothioxy)-ethyl benzenecarbothioate and 2-(benzenecarbothioxy)ethyl benzoate

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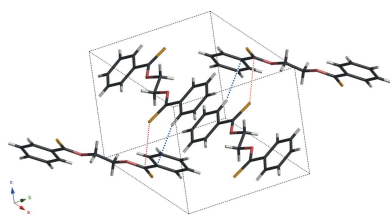
Keywords: crystal structure; thionoester; all-*trans* structure; C···S close contact; C—H··· π interaction.**CCDC references:** 1572676; 1572675**Supporting information:** this article has supporting information at journals.iucr.org/e

The title compounds, C₁₆H₁₄O₂S₂ and C₁₆H₁₄O₃S, which are monomeric models (models **D** and **E**) for a polythionoester and a poly(ester-*co*-thionoester), respectively, crystallize in the space group *P*2₁/*c* and are isostructural with each other. The molecule in each crystal is located on an inversion centre and has an all-*trans* structure. The asymmetric unit comprises one half-molecule. In the crystal, there are intermolecular C···S contacts [3.391 (3) and 3.308 (3) Å for models **D** and **E**, respectively] and C—H··· π interactions, which form a layer structure parallel to the *bc* plane. The carbonyl and thiocarbonyl groups of the model **E** compound are each disordered over two equivalent sites about the inversion centre with equal occupancies.

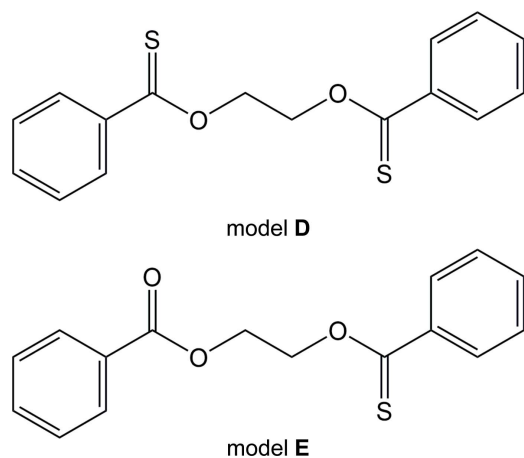
1. Chemical context

Compounds expressed as C₆H₅—C(=X)—Y—CH₂—CH₂—Y—C(=X)—C₆H₅ (*X*, *Y* = O or S) can be considered to be monomeric models for polymers, [—C(=X)—C₆H₄—C(=X)—Y—CH₂—CH₂—Y—]_{*n*}, namely, *X* = *Y* = O, poly(ethylene terephthalate) (designated herein as polymer **A**); *X* = O and *Y* = S, poly(ethylene dithioterephthalate) (polymer **B**); *X* = *Y* = S, poly(ethylene tetrathioterephthalate) (polymer **C**); *X* = S and *Y* = O, poly(ethylene dithionoterephthalate) (polymer **D**). It is well established that the solution, mechanical and thermal properties of such aromatic polymers are essentially determined by the conformational characteristics of the Y—CH₂—CH₂—Y unit (referred hereafter to as the spacer) and intermolecular interactions between the benzene rings (Sasanuma, 2009; Sasanuma *et al.*, 2013). In expectation that replacement of oxygen by sulfur at the *X* or *Y* site would affect the spacer conformation, and π – π and C—H··· π interactions of the benzene rings, and thus lead to variations in the physical properties, we synthesized polymers **B** and **C**, and characterized them by X-ray diffraction, NMR spectroscopy, thermal analyses, molecular orbital calculations and statistical mechanics of the chain molecules (Abe & Sasanuma, 2012). Herein, the monomeric models for polymers **A**–**D** are termed models **A**–**D**, respectively.

By molecular orbital calculations at the second-order Møller–Plesset perturbation (MP2) level with moderate-size basis sets, we have determined the most stable conformations of the Y—CH₂—CH₂—Y parts of the models and evaluated their free energies relative to that of the all-*trans* form as follows: model **A**, *tgt* and –1.1 kcal mol^{–1} (Sasanuma, 2009); model **B**, *g*[±]*tg*[±] and –3.1 kcal mol^{–1} (Abe & Sasanuma, 2012);



model **C**, $g^{\pm}tg^{\mp}$ and $-2.1 \text{ kcal mol}^{-1}$ (Abe & Sasanuma, 2012); model **D**, tgt and $-1.7 \text{ kcal mol}^{-1}$ (this study). We have also predicted that an asymmetric model compound, $C_6H_5-C(=O)-O-CH_2-CH_2-O-C(=S)-C_6H_5$ (model **E**), would be most stabilized in the $tg^{\pm}g^{\mp}$ conformation with a free energy of $-1.8 \text{ kcal mol}^{-1}$ (this study). However, not all the models and polymers crystallize in the lowest-energy conformations: model (polymer) **A**, ttt (ttt) (Pérez & Brisse, 1976; Daubeny *et al.*, 1954); model (polymer) **B**, $g^{\pm}tg^{\mp}$ ($g^{\pm}tg^{\mp}$) (Deguire & Brisse, 1988; Abe & Sasanuma, 2012); model (polymer) **C**, $g^{\pm}tg^{\mp}$ (amorphous) (Abe *et al.*, 2011; Abe & Sasanuma, 2012). In the crystals, the molecules adopt conformations so as to form intermolecular interactions effectively and minimize the total of intramolecular and intermolecular interaction energies. Interestingly, however, models **A–C** crystallize in the same spacer conformation as those of the corresponding polymers; therefore, the crystal structure of the model suggests the polymer conformation. This study has aimed to determine crystal structures of the title compounds (models **D** and **E**) to predict the crystal conformations of polymers **D** and **E** on the above hypothesis.



2. Structural commentary

The molecule of model **D** lies on an inversion centre and the asymmetric unit contains one half-molecule. The central $O-CH_2-CH_2-O$ unit adopts an all-*trans* conformation (Fig. 1). The molecule of model **E** is also located on an inversion centre and the $O-CH_2-CH_2-O$ bond sequence is in an all-*trans* conformation. Since the molecule has carbonyl and thiocarbonyl groups, the atoms S and O (S1 and O2) are each assumed to be disordered over two equivalent sites about the inversion centre with equal occupancies (Fig. 2). Consequently, it was proved that all the models (**A**, **D** and **E**) with the $O-CH_2-CH_2-O$ spacer crystallize with all-*trans* structures, although models **A**, **D** and **E** in the free state are most stabilized in tgt , tgt , and $tg^{\pm}g^{\mp}$ conformations, respectively.

3. Supramolecular features

The compounds of models **D** and **E** are isotypic and crystallize in the space group $P2_1/c$. There are no classical hydrogen

Table 1

C–H... π interaction geometry (\AA , $^\circ$) for model **D**.

Cg1 is the centroid of the C2–C7 ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C3-H3 \cdots Cg1^i$	0.95	2.92	3.721 (3)	143

Symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$

Table 2

C–H... π interaction geometry (\AA , $^\circ$) for model **E**.

Cg1 is the centroid of the C2–C7 ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C3-H3 \cdots Cg1^i$	0.95	2.89	3.641 (3)	137

Symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$

bonds but intermolecular close contacts between atoms C and S [$C1-S1^i = 3.391$ (3) and 3.308 (3) \AA for models **D** and **E**, respectively; symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$]. Both compounds also have C–H... π interactions (Tables 1 and 2) and form layer structures parallel to the bc plane *via* these intermolecular interactions (Figs. 3 and 4).

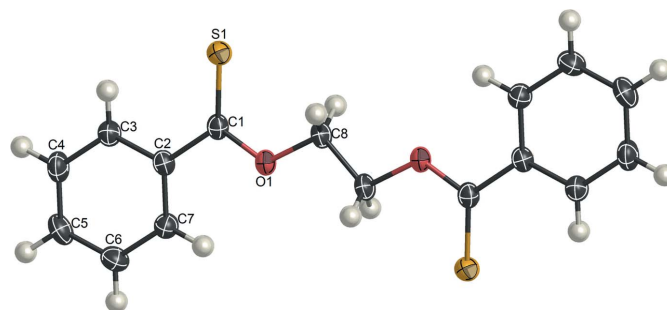


Figure 1

The molecular structure of model **D**, showing atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The unlabelled atoms are related to the labelled atoms by inversion symmetry (symmetry code: $2 - x, 2 - y, 2 - z$). H atoms are represented by spheres of arbitrary size.

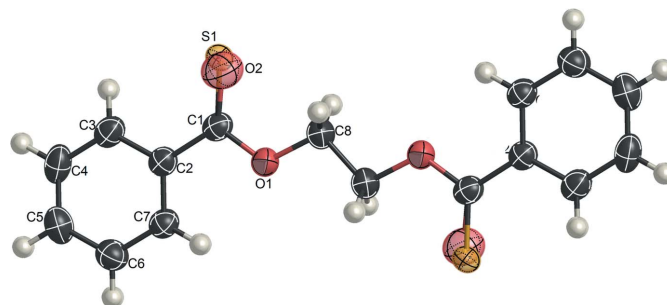


Figure 2

The molecular structure of model **E**, showing atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The unlabelled atoms are related to the labelled atoms by inversion symmetry (symmetry code: $2 - x, 2 - y, 2 - z$). H atoms are represented by spheres of arbitrary size.

Table 3
Experimental details.

	model D	model E
Crystal data		
Chemical formula	C ₁₆ H ₁₄ O ₂ S ₂	C ₁₆ H ₁₄ O ₃ S
<i>M_r</i>	302.39	286.33
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Monoclinic, <i>P</i> 2 ₁ / <i>c</i>
Temperature (K)	173	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.829 (5), 11.680 (7), 7.727 (5)	8.800 (5), 11.403 (6), 7.506 (4)
β (°)	113.475 (10)	113.831 (6)
<i>V</i> (Å ³)	730.9 (8)	689.0 (6)
<i>Z</i>	2	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.36	0.24
Crystal size (mm)	0.40 × 0.40 × 0.20	0.40 × 0.40 × 0.10
Data collection		
Diffractometer	Bruker APEXII CCD area detector	Bruker APEXII CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	Multi-scan (<i>SADABS</i> ; Sheldrick, 1996)
<i>T_{min}</i> , <i>T_{max}</i>	0.88, 0.93	0.84, 0.98
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	4017, 1617, 1487	3265, 1523, 1335
<i>R_{int}</i>	0.014	0.015
(sin θ/λ) _{max} (Å ⁻¹)	0.648	0.653
Refinement		
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.029, 0.080, 1.03	0.038, 0.090, 1.08
No. of reflections	1617	1523
No. of parameters	91	100
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.39, -0.20	0.19, -0.16

Computer programs: *APEX2*, *SAINT*, *XSHEL* and *XCIF* (Bruker, 2012) *SHELXS97* (Sheldrick, 2008) and *SHELXL2013* (Sheldrick, 2015).

3.1. Synthesis and crystallization

Benzoyl chloride (10.0 ml, 87 mmol) was added dropwise under a nitrogen atmosphere to ethylene glycol (2.4 ml, 43 mmol) and pyridine (7.0 ml, 87 mmol) placed in a four-necked flask connected to a drying tube filled with calcium chloride, and the mixture was stirred at room temperature overnight. Water was added to the reaction mixture to yield a precipitate, which was collected by filtration, dissolved in chloroform, washed thrice with 5% aqueous solution of sodium bicarbonate, and dried over anhydrous magnesium sulfate overnight. The liquid phase was separated by filtration and condensed on a rotary evaporator, and the residue was

recrystallized from ethanol (15 ml). The white crystallites thus obtained were dried under reduced pressure at room temperature overnight to yield ethane-1,2-diyl dibenzoate (8.3 g, 71%). The synthesized ethane-1,2-diyl dibenzoate (0.10 g, 0.37 mmol) was ground in a mortar and mixed thoroughly with Lawesson's reagent (0.24 g, 0.59 mmol), and the powder mixture was moved to a 15 ml vial container and placed in a Yuasa PRE-7017R microwave oven. The powder was heated under the following microwave irradiation at 500 W: on for 2.0 min – off for several seconds – on for 1.0 min. The above handling was repeated ten times to obtain the product sufficiently.

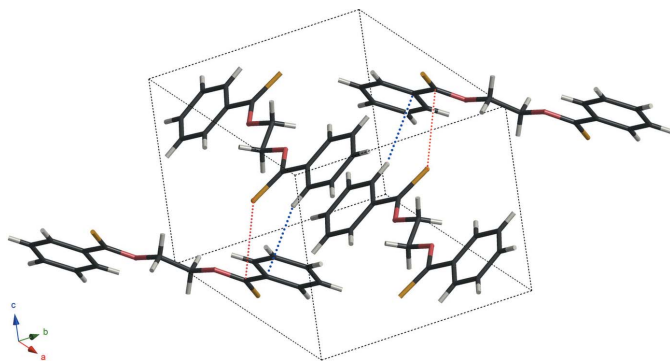


Figure 3
A packing diagram of model **D**, showing intermolecular C...S contacts (red dotted lines) and C–H...π interactions (blue dotted lines).

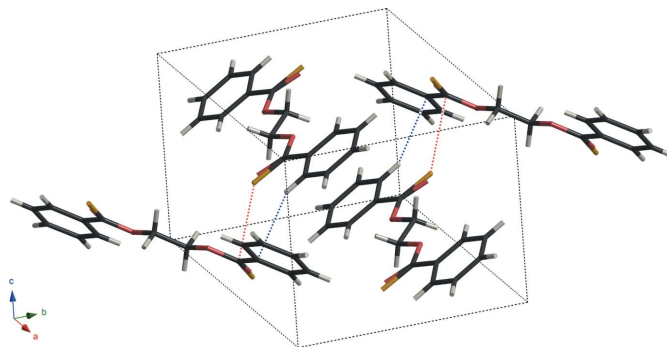


Figure 4
A packing diagram of model **E**, showing intermolecular C...S contacts (red dotted lines) and C–H...π interactions (blue dotted lines).

The crude product was extracted with chloroform and condensed under reduced pressure. The residue was dissolved in a mixed solvent of ethyl acetate and *n*-hexane (1:9 *v/v*) and subjected to column chromatography. The yellowish fraction ($R_f = 0.5$) was collected and condensed, and the residue underwent column chromatography again with a mixed solvent of toluene and *n*-hexane (1:5 *v/v*). Two yellow fractions [(1) $R_f = 0.1$ and (2) 0.3 – 0.5] were stratified and collected separately. The layer (1) was condensed and recrystallized from ethanol to yield a yellow solid, which was identified as 2-(benzenecarbothioxy)ethyl benzoate (model **E**, yield 23%) by ^1H and ^{13}C NMR, and the layer (2) was condensed and dried at room temperature overnight to yield a red solid, which was identified as 2-(benzenecarbothioxy)ethyl benzenecarbothioate (model **D**, yield 0.9%). A small quantity of model **D** was dissolved in chloroform in a thin vial container. The vessel was placed in a larger vial containing a small amount of *n*-hexane, and the outer container was capped. After a week, single crystals were found to be formed in the inner vessel. Single crystals of model **E** were prepared similarly.

4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were geometrically

positioned with $\text{C}-\text{H} = 0.95$ and 0.99 \AA for the aromatic and methylene groups, respectively, and were refined as riding with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

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supporting information

Acta Cryst. (2017). E73, 1430-1433 [https://doi.org/10.1107/S2056989017012701]

Crystal structures of 2-(benzenecarbothioyloxy)ethyl benzenecarbothioate and 2-(benzenecarbothioyloxy)ethyl benzoate

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINTE* (Bruker, 2012). Data reduction: *SAINTE* for model_D; *SAINTE* (Bruker, 2012) for model_E. For both structures, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *XSHEL* (Bruker, 2012); software used to prepare material for publication: *XCIF* (Bruker, 2012).

2-(Benzenecarbothioyloxy)ethyl benzenecarbothioate (model_D)

Crystal data

$C_{16}H_{14}O_2S_2$	$F(000) = 316$
$M_r = 302.39$	$D_x = 1.374 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.829 (5) \text{ \AA}$	Cell parameters from 2339 reflections
$b = 11.680 (7) \text{ \AA}$	$\theta = 2.5\text{--}27.6^\circ$
$c = 7.727 (5) \text{ \AA}$	$\mu = 0.36 \text{ mm}^{-1}$
$\beta = 113.475 (10)^\circ$	$T = 173 \text{ K}$
$V = 730.9 (8) \text{ \AA}^3$	Prismatic, yellow
$Z = 2$	$0.40 \times 0.40 \times 0.20 \text{ mm}$

Data collection

Bruker APEXII CCD area detector	4017 measured reflections
diffractometer	1617 independent reflections
Radiation source: fine-focus sealed tube	1487 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.014$
Detector resolution: $8.3333 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.4^\circ$, $\theta_{\text{min}} = 2.5^\circ$
φ and ω scans	$h = -10 \rightarrow 11$
Absorption correction: multi-scan	$k = -13 \rightarrow 15$
(SADABS; Sheldrick, 1996)	$l = -9 \rightarrow 5$
$T_{\text{min}} = 0.88$, $T_{\text{max}} = 0.93$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.2706P]$
$wR(F^2) = 0.080$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1617 reflections	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
91 parameters	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
0 restraints	

*Special details***Experimental.** SADABS (Sheldrick 1996)

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.70662 (15)	0.86862 (11)	0.98706 (17)	0.0209 (3)
C2	0.52641 (15)	0.87852 (10)	0.86705 (17)	0.0205 (3)
C3	0.41444 (16)	0.80285 (12)	0.89460 (19)	0.0271 (3)
H3	0.4532	0.7468	0.9916	0.033*
C4	0.24591 (17)	0.80973 (13)	0.7796 (2)	0.0312 (3)
H4	0.1705	0.7586	0.7991	0.037*
C5	0.18837 (16)	0.89124 (13)	0.6367 (2)	0.0309 (3)
H5	0.0741	0.8947	0.5572	0.037*
C6	0.29824 (17)	0.96779 (13)	0.6101 (2)	0.0316 (3)
H6	0.2585	1.024	0.5136	0.038*
C7	0.46690 (16)	0.96209 (11)	0.72503 (19)	0.0259 (3)
H7	0.5413	1.0148	0.707	0.031*
C8	0.97120 (15)	0.94978 (12)	1.04144 (19)	0.0267 (3)
H8A	1.0184	0.8771	1.0194	0.032*
H8B	1.0041	0.9599	1.1788	0.032*
O1	0.79324 (11)	0.95006 (8)	0.94319 (13)	0.0252 (2)
S1	0.79118 (4)	0.77062 (3)	1.15081 (5)	0.02945 (13)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0204 (6)	0.0227 (6)	0.0205 (6)	-0.0032 (5)	0.0091 (5)	-0.0036 (5)
C2	0.0189 (6)	0.0224 (6)	0.0205 (6)	-0.0013 (5)	0.0080 (5)	-0.0036 (5)
C3	0.0232 (6)	0.0300 (7)	0.0265 (7)	-0.0040 (5)	0.0081 (5)	0.0023 (5)
C4	0.0221 (6)	0.0373 (8)	0.0334 (7)	-0.0072 (6)	0.0103 (6)	-0.0024 (6)
C5	0.0191 (6)	0.0376 (8)	0.0318 (7)	0.0020 (5)	0.0057 (5)	-0.0050 (6)
C6	0.0274 (7)	0.0315 (7)	0.0314 (7)	0.0051 (6)	0.0068 (6)	0.0049 (6)
C7	0.0244 (7)	0.0244 (6)	0.0283 (7)	-0.0010 (5)	0.0099 (5)	0.0014 (5)
C8	0.0170 (6)	0.0314 (7)	0.0281 (6)	-0.0041 (5)	0.0051 (5)	0.0030 (5)
O1	0.0174 (4)	0.0269 (5)	0.0282 (5)	-0.0042 (3)	0.0057 (4)	0.0037 (4)
S1	0.02236 (19)	0.0332 (2)	0.0302 (2)	-0.00007 (13)	0.00763 (14)	0.00936 (13)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.3458 (16)	C5—C6	1.393 (2)
C1—C2	1.4931 (18)	C5—H5	0.95
C1—S1	1.6481 (14)	C6—C7	1.399 (2)
C2—C3	1.4042 (19)	C6—H6	0.95

C2—C7	1.4049 (19)	C7—H7	0.95
C3—C4	1.399 (2)	C8—O1	1.4474 (17)
C3—H3	0.95	C8—C8 ⁱ	1.518 (3)
C4—C5	1.392 (2)	C8—H8A	0.99
C4—H4	0.95	C8—H8B	0.99
O1—C1—C2	111.15 (11)	C6—C5—H5	120.0
O1—C1—S1	123.72 (10)	C5—C6—C7	120.22 (13)
C2—C1—S1	125.13 (9)	C5—C6—H6	119.9
C3—C2—C7	119.27 (12)	C7—C6—H6	119.9
C3—C2—C1	119.84 (12)	C6—C7—C2	120.09 (12)
C7—C2—C1	120.89 (11)	C6—C7—H7	120.0
C4—C3—C2	120.17 (13)	C2—C7—H7	120.0
C4—C3—H3	119.9	O1—C8—C8 ⁱ	104.66 (13)
C2—C3—H3	119.9	O1—C8—H8A	110.8
C5—C4—C3	120.21 (13)	C8 ⁱ —C8—H8A	110.8
C5—C4—H4	119.9	O1—C8—H8B	110.8
C3—C4—H4	119.9	C8 ⁱ —C8—H8B	110.8
C4—C5—C6	120.02 (13)	H8A—C8—H8B	108.9
C4—C5—H5	120.0	C1—O1—C8	118.26 (10)

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

2-(Benzenecarbothioxyloxy)ethyl benzoate (model_E)

Crystal data

$C_{16}H_{14}O_3S$

$M_r = 286.33$

Monoclinic, $P2_1/c$

$a = 8.800$ (5) Å

$b = 11.403$ (6) Å

$c = 7.506$ (4) Å

$\beta = 113.831$ (6)°

$V = 689.0$ (6) Å³

$Z = 2$

$F(000) = 300$

$D_x = 1.380$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1991 reflections

$\theta = 2.5$ – 27.6 °

$\mu = 0.24$ mm⁻¹

$T = 173$ K

Prismatic, yellow

$0.40 \times 0.40 \times 0.10$ mm

Data collection

Bruker APEXII CCD area detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.84$, $T_{\max} = 0.98$

3265 measured reflections

1523 independent reflections

1335 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 27.7$ °, $\theta_{\min} = 2.5$ °

$h = -11 \rightarrow 11$

$k = -6 \rightarrow 14$

$l = -8 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.090$

$S = 1.08$

1523 reflections

100 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0288P)^2 + 0.2342P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. SADABS (Sheldrick 1996)

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.78695 (19)	0.76827 (18)	1.1323 (3)	0.0410 (3)	0.5
O2	0.7837 (8)	0.7828 (6)	1.0959 (10)	0.087 (3)	0.5
O1	0.79258 (12)	0.94978 (9)	0.93503 (15)	0.0404 (3)	
C1	0.70747 (18)	0.86474 (13)	0.9769 (2)	0.0372 (3)	
C2	0.52662 (17)	0.87657 (12)	0.86227 (19)	0.0338 (3)	
C3	0.4186 (2)	0.79742 (14)	0.8908 (2)	0.0428 (4)	
H3	0.461	0.7372	0.9857	0.051*	
C4	0.2497 (2)	0.80604 (15)	0.7815 (2)	0.0486 (4)	
H4	0.1762	0.7516	0.8014	0.058*	
C5	0.1873 (2)	0.89288 (16)	0.6442 (3)	0.0492 (4)	
H5	0.0711	0.8978	0.5681	0.059*	
C6	0.2934 (2)	0.97304 (15)	0.6168 (2)	0.0470 (4)	
H6	0.2498	1.0336	0.5229	0.056*	
C7	0.46254 (18)	0.96570 (12)	0.7252 (2)	0.0381 (3)	
H7	0.5351	1.0213	0.7064	0.046*	
C8	0.97020 (18)	0.94635 (14)	1.0342 (2)	0.0440 (4)	
H8A	1.0153	0.8744	0.9999	0.053*	
H8B	1.0047	0.9485	1.1771	0.053*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0345 (5)	0.0398 (5)	0.0452 (6)	0.0040 (4)	0.0125 (4)	0.0086 (5)
O2	0.094 (4)	0.073 (4)	0.086 (5)	-0.007 (3)	0.028 (3)	0.007 (3)
O1	0.0368 (5)	0.0394 (6)	0.0392 (6)	-0.0059 (4)	0.0093 (4)	0.0043 (4)
C1	0.0457 (8)	0.0330 (7)	0.0324 (8)	-0.0042 (6)	0.0153 (6)	-0.0013 (6)
C2	0.0419 (8)	0.0332 (7)	0.0270 (7)	-0.0048 (6)	0.0144 (6)	-0.0058 (5)
C3	0.0536 (9)	0.0417 (8)	0.0333 (8)	-0.0113 (7)	0.0176 (7)	-0.0028 (6)
C4	0.0475 (9)	0.0563 (10)	0.0462 (10)	-0.0191 (8)	0.0233 (7)	-0.0124 (8)
C5	0.0380 (8)	0.0592 (10)	0.0489 (10)	-0.0037 (7)	0.0159 (7)	-0.0100 (8)
C6	0.0445 (8)	0.0470 (9)	0.0464 (10)	0.0036 (7)	0.0151 (7)	0.0021 (7)
C7	0.0427 (8)	0.0343 (7)	0.0380 (8)	-0.0027 (6)	0.0171 (6)	-0.0010 (6)
C8	0.0365 (8)	0.0462 (9)	0.0437 (9)	-0.0044 (7)	0.0103 (6)	0.0033 (7)

Geometric parameters (Å, °)

S1—C1	1.549 (3)	C4—H4	0.95
O2—C1	1.279 (6)	C5—C6	1.380 (2)
O1—C1	1.3381 (18)	C5—H5	0.95
O1—C8	1.4349 (19)	C6—C7	1.380 (2)
C1—C2	1.478 (2)	C6—H6	0.95
C2—C3	1.389 (2)	C7—H7	0.95
C2—C7	1.393 (2)	C8—C8 ⁱ	1.501 (3)
C3—C4	1.381 (2)	C8—H8A	0.99
C3—H3	0.95	C8—H8B	0.99
C4—C5	1.374 (3)		
C1—O1—C8	117.01 (12)	C4—C5—C6	119.98 (15)
O2—C1—O1	120.5 (3)	C4—C5—H5	120.0
O2—C1—C2	127.6 (3)	C6—C5—H5	120.0
O1—C1—C2	111.73 (13)	C5—C6—C7	120.36 (16)
O1—C1—S1	124.48 (13)	C5—C6—H6	119.8
C2—C1—S1	123.75 (13)	C7—C6—H6	119.8
C3—C2—C7	119.33 (14)	C6—C7—C2	119.85 (14)
C3—C2—C1	119.61 (14)	C6—C7—H7	120.1
C7—C2—C1	121.06 (13)	C2—C7—H7	120.1
C4—C3—C2	120.12 (15)	O1—C8—C8 ⁱ	104.96 (15)
C4—C3—H3	119.9	O1—C8—H8A	110.8
C2—C3—H3	119.9	C8 ⁱ —C8—H8A	110.8
C5—C4—C3	120.33 (15)	O1—C8—H8B	110.8
C5—C4—H4	119.8	C8 ⁱ —C8—H8B	110.8
C3—C4—H4	119.8	H8A—C8—H8B	108.8

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