Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,5-Bis(benzoylsulfanyl)-1,3-dithiol-2-one

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.065
Data-to-parameter ratio = 16.8

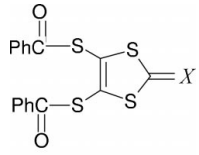
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 November 2004
Accepted 4 November 2004
Online 13 November 2004

The title compound [systematic name: *S*,*S*′-2-oxo-1,3-dithiol-4,5-diyl bis(thiobenzoate)], $C_{17}H_{10}O_3S_4$, obtained from 4,5-bis(benzoylsulfanyl)-1,3-dithiole-2-thione and mercury(II) acetate in acetic acid/chloroform, exists as isolated molecules with no significant intermolecular S···S, S···O or O···O interactions.

Comment

The title compound, 4,5-bis(benzoylsulfanyl)-1,3-dithiol-2-one, (I), and the zincate salts $[Q]_2[Zn(dmio)_2]$ (Q is the onium cation and dmio is 2-oxo-1,3-dithiole-4,5-dithiolate) are very useful stable sources of the dmio dianion and have found extended use as precursors of both organic dmio compounds and metal–dmio complexes. Additionally, dmio compounds, such as the title compound, are good sources of tetrathiafulvalenes on reaction with phosphites (Svenstrup & Becher, 1995).



(I) *X* = O
(II) *X* = S

While the crystal structure of a $Zn(dmio)_2$ salt has been reported (Candiota *et al.*, 2003), no previous study of the structure of (I) has been reported.

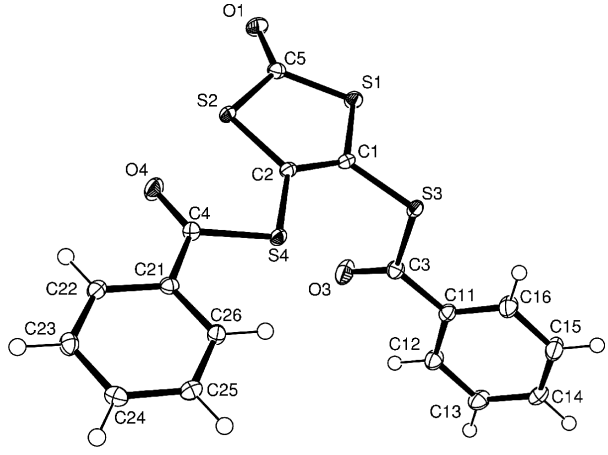


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as circles of arbitrary radii.

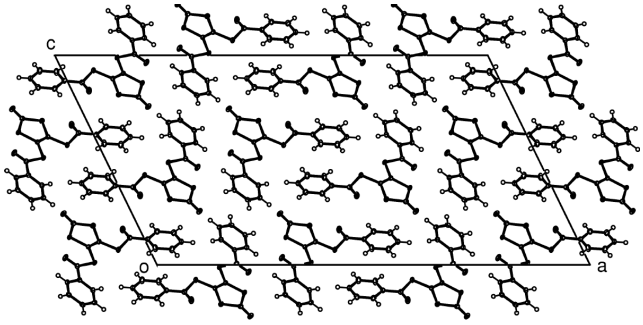


Figure 2
The unit cell contents of (I), projected on to the (101) plane.

Bond lengths and angles within the five-membered dmio ring in (I) are within the ranges found for other dmio compounds, such as [Q][Sn(dmio)$_3$] and [Q][Zn(dmio)$_2$] (Candiota *et al.*, 2003; Chohan *et al.*, 2003; Aupers *et al.*, 2002; de Assis *et al.*, 1999).

The dmio ring, together with the attached carbonyl O atom, is essentially planar, with S1 showing the largest deviation [0.0158 (6) Å] from the mean plane. The two phenyl rings are inclined at angles of 78.60 (4) (C11–C16) and 6.94 (8)° (C21–C26) to the dmio ring. Molecules of (I) show no strong association with each other, the closest intermolecular S···S, S···O and O···O separations being 3.6561 (5), 3.4524 (12) and 3.1479 (17) Å, respectively, all just outside the van der Waals radii sum for the appropriate atoms; van der Waals radii for S and O are taken as 1.80 and 1.52 Å, respectively (*PLATON*; Spek, 2004).

The structure of the analogous 4,5-bis(benzoylsulfanyl)-1,3-dithiole-2-thione compound, (II), has been reported at both 120 (Cox & Doidge-Harrison, 1996) and 288 K (Solans *et al.*, 1987). There are weak C—H···O and S···S intermolecular interactions in (II).

Experimental

The title compound was prepared using a modification of a published method (Hartke *et al.*, 1980). A solution of mercury(II) acetate (4.78 g, 15.0 mmol) in glacial acetic acid (120 ml) was added with vigorous agitation to a solution of 4,5-bis(benzoylsulfanyl)-1,3-dithiole-2-thione [(II); 6.09 g, 15.0 mmol] (Steimecke, 1979) in chloroform (120 ml). After refluxing for 5 h, the reaction mixture was filtered, and the filtrate was successively washed with water, saturated aqueous sodium bicarbonate solution and more water, dried over $MgSO_4$ and evaporated to leave a yellow solid, which was recrystallized from chloroform/methanol (yield 54%, m.p. 388–389 K). IR (CsI, cm^{-1}): 3083 (ν C—H), 1701, 1697, 1668, 1621 (ν C═O), 1467 (ν C═C), 896 (ν C—S).

Crystal data

$C_{17}H_{10}O_3S_4$
M_r = 390.49
Monoclinic, $C2/c$
a = 35.6460 (6) Å
b = 5.20360 (10) Å
c = 19.1402 (3) Å
β = 116.0945 (8)°
V = 3188.39 (10) Å^3
Z = 8
D_x = 1.627 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4041 reflections
θ = 2.7–27.5°
μ = 0.61 mm^{-1}
T = 120 (2) K
Block, colourless
0.60 × 0.30 × 0.20 mm

Data collection

Bruker–Nonius KappaCCD diffractometer with rotating-anode source
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.787, T_{max} = 0.883
25 707 measured reflections
3656 independent reflections
3191 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.5°
$h = -46 \rightarrow 46$
$k = -6 \rightarrow 6$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.06
3656 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 3.4602P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16···S3	0.95	2.59	3.0392 (15)	109
C26—H26···S4	0.95	2.58	3.0146 (14)	108

All H atoms were first identified in a difference map and then placed in geometrical positions and refined using a riding model with C–H distances of 0.95 Å. Analysis of molecular interactions was carried out using *PLATON* (Spek, 2004).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

We thank the EPSRC's X-ray Crystallographic Service, University of Southampton, for the collection of the data. The Brazil-based authors thank CNPq, CAPES and FUJB for support.

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