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## Structure Reports

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## 2-Methyl-2-(4-nitrophenylsulfanyl)-propanoic acid

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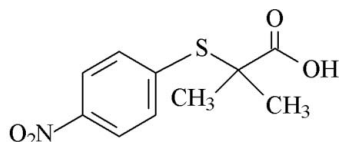
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Key indicators: single-crystal X-ray study;  $T = 273$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.048;  $wR$  factor = 0.123; data-to-parameter ratio = 13.8.

The title compound,  $\text{C}_{10}\text{H}_{11}\text{NO}_4\text{S}$ , is of interest with respect to its biological activity. The molecules are linked into centrosymmetric dimers by intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds and the dimers are further connected into chains by weak  $\text{C}-\text{H}\cdots\text{O}$  interactions.

### Related literature

For related literature on fibrate structures and biological activity, see: Henry *et al.* (2003); Rath *et al.* (2005); Djinović *et al.* (1989); Thorp (1962); Thorp & Waring (1962); Miller & Spence (1998); Forcheron *et al.* (2002). For related literature, see: Bernstein *et al.* (1995); Desiraju (2002).



### Experimental

#### Crystal data

$\text{C}_{10}\text{H}_{11}\text{NO}_4\text{S}$	$\gamma = 88.298$ (2)°
$M_r = 241.26$	$V = 578.60$ (12) Å <sup>3</sup>
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.9382$ (8) Å	Mo $K\alpha$ radiation
$b = 9.4500$ (11) Å	$\mu = 0.28$ mm <sup>-1</sup>
$c = 9.6395$ (11) Å	$T = 273$ (2) K
$\alpha = 66.371$ (2)°	$0.32 \times 0.23 \times 0.18$ mm
$\beta = 87.995$ (2)°	

#### Data collection

Bruker SMART CCD area detector diffractometer	5660 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	2036 independent reflections
$T_{\min} = 0.92$ , $T_{\max} = 0.95$	1718 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.020$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	148 parameters
$wR(F^2) = 0.123$	H-atom parameters constrained
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.28$ e Å <sup>-3</sup>
2036 reflections	$\Delta\rho_{\text{min}} = -0.17$ e Å <sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O4}-\text{H4}\cdots\text{O3}^{\text{i}}$	0.82	1.84	2.656 (3)	175
$\text{C2}-\text{H2}\cdots\text{O2}^{\text{ii}}$	0.93	2.46	3.211 (3)	138

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT-Plus NT (Bruker, 2000); data reduction: SAINT-Plus NT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL-NT (Bruker, 2000); software used to prepare material for publication: PLATON (Spek, 2003) and publCIF (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LN2010).

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## supporting information

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## 2-Methyl-2-(4-nitrophenylsulfanyl)propanoic acid

**Gabriel Navarrete-Vázquez, Rafael Villalobos-Molina, Samuel Estrada-Soto, Rolffy Ortiz-Andrade and Hugo Tlahuext**

### S1. Comment

Fibrates, such as bezafibrate, clofibric acid and fenofibrate (Henry *et al.*, 2003; Rath *et al.*, 2005; Djinović *et al.*, 1989), which are ligands for the nuclear receptor PPAR (peroxisome proliferator-activated receptor), are used as therapeutic agents in the treatment of hyperlipidemia, heart disease and diabetic complications in humans. The fibrates are a widely used class of lipid-modifying agents that decrease plasma triglycerides (Thorp, 1962; Miller & Spence, 1998; Forcheron *et al.*, 2002). The fibric acid pharmacophore has been of interest to medicinal chemists ever since the initial discovery that ethyl chlorophenoxyisobutyrate possessed hypolipidemic properties (Thorp & Waring, 1962).

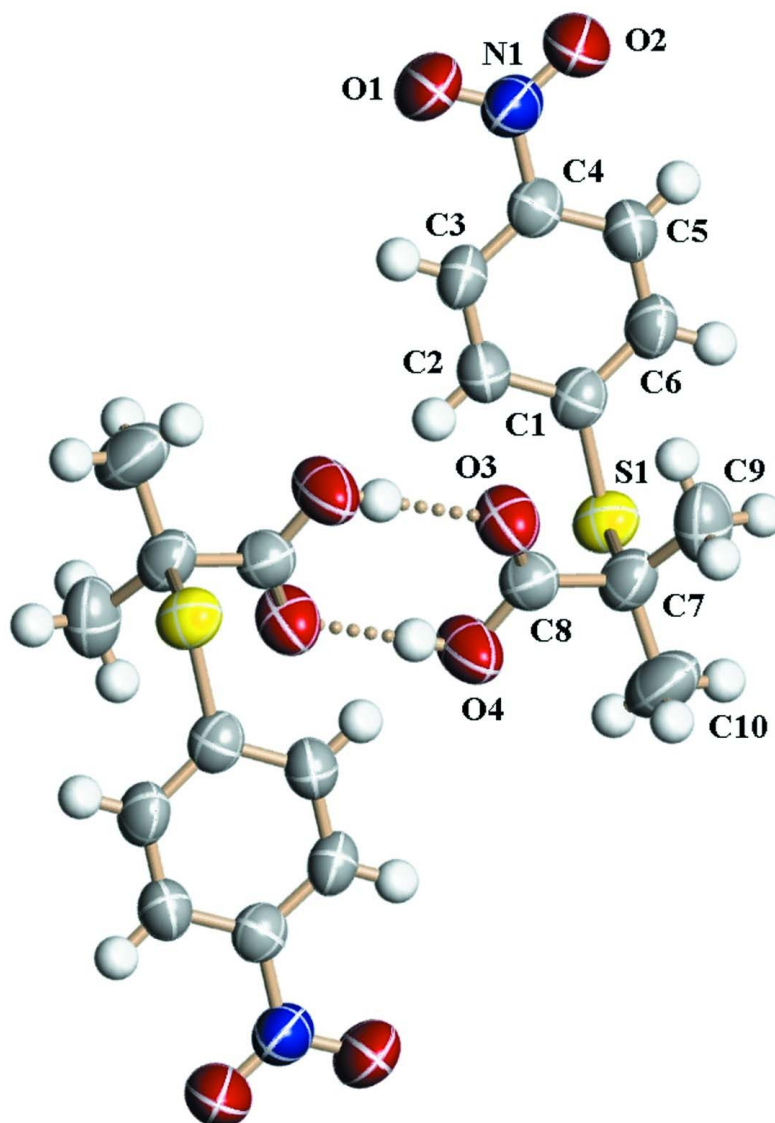
In order to assist our knowledge about the electronic and steric requirements for such compounds to show antihyperlipidemic activity, we have determined the crystal structure of the title compound, (I), which is an analogue of clofibric acid with a thioisobutirate side chain. A view of the molecular structure of (I) and hydrogen bonded dimers is given in Fig. 1. The crystal structure is permeated by strong O—H $\cdots$ O hydrogen-bonding interactions, as well as weak C—H $\cdots$ O interactions (Table 1) (Desiraju, 2002). The O—H $\cdots$ O hydrogen bonding interactions form centrosymmetric dimers and generate rings that can be described as having a graph set motif of  $R_2^2(8)$  (Bernstein *et al.*, 1995).

### S2. Experimental

A mixture of 4-nitrothiophenol (1.0 g, 6.40 mmol), potassium carbonate (1.94 g, 14.1 mmol) in acetonitrile, was added dropwise to 1.04 ml of ethyl 2-bromo-2-methylpropionate (1.37 g, 7.04 mmol). The mixture was stirred and heated under reflux for 6 h. After that, the mixture was poured onto cold water. The resulting oil was treated with a mixture of tetrahydrofuran/methanol/H<sub>2</sub>O (3:2:1, v/v/v, 6 ml/mmol), and LiOH was added (5 equiv). The mixture was stirred at room temperature for 3 h. Then, HCl solution (10% v/v) was added, and most of the organic solvents removed *in vacuo*. The partly solid residue was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 10 ml), dried with Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated *in vacuo* to give a yellow solid (m.p. 394.9 K). Single crystals of (I) were obtained from acetonitrile.

### S3. Refinement

All the H atoms were constrained using the riding-model approximation [ $C-H_{\text{aryl}} = 0.95 \text{ \AA}$ ,  $U_{\text{iso}}(H_{\text{aryl}}) = 1.2 U_{\text{eq}}(C_{\text{aryl}})$ ;  $C-H_{\text{methyl}} = 0.98 \text{ \AA}$ ,  $U_{\text{iso}}(H_{\text{methyl}}) = 1.5 U_{\text{eq}}(C_{\text{methyl}})$ ;  $O-H = 0.82 \text{ \AA}$ ,  $U_{\text{iso}}(H_{\text{hydroxyl}}) = 1.5 U_{\text{eq}}(O)$ ].



**Figure 1**

The molecular structure of (I) showing 50% probability displacement ellipsoids and the atomic numbering. H atoms are shown as small spheres of arbitrary radius. The intermolecular hydrogen bonds O4—H4···O3 forming the  $R_2^2(8)$  motif are shown as dotted lines.

### 2-Methyl-2-(4-nitrophenylsulfanyl)propanoic acid

#### Crystal data

$C_{10}H_{11}NO_4S$

$M_r = 241.26$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 6.9382$  (8) Å

$b = 9.4500$  (11) Å

$c = 9.6395$  (11) Å

$\alpha = 66.371$  (2)°

$\beta = 87.995$  (2)°

$\gamma = 88.298$  (2)°

$V = 578.60$  (12) Å<sup>3</sup>

$Z = 2$

$F(000) = 252$

$D_x = 1.385$  Mg m<sup>-3</sup>

Melting point: 394.9 K

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

Cell parameters from 2036 reflections

$\theta = 2.3$ – $25^\circ$

$\mu = 0.28 \text{ mm}^{-1}$   
 $T = 273 \text{ K}$

Plate, colourless  
 $0.32 \times 0.23 \times 0.18 \text{ mm}$

*Data collection*

Bruker CCD area detector  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.92, T_{\max} = 0.95$

5660 measured reflections  
 2036 independent reflections  
 1718 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$   
 $\theta_{\max} = 25.0^\circ, \theta_{\min} = 2.3^\circ$   
 $h = -8 \rightarrow 8$   
 $k = -11 \rightarrow 11$   
 $l = -11 \rightarrow 11$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.123$   
 $S = 1.08$   
 2036 reflections  
 148 parameters  
 0 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  
 $w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.156P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.17 \text{ e } \text{Å}^{-3}$

*Special details*

**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 ( $\text{Å}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1054 (3)	0.1894 (3)	0.6403 (2)	0.0531 (5)
C2	0.1738 (3)	0.0380 (3)	0.6935 (3)	0.0580 (6)
H2	0.3037	0.0168	0.7153	0.070*
C3	0.0525 (3)	-0.0797 (3)	0.7141 (3)	0.0576 (6)
H3	0.0982	-0.1813	0.7509	0.069*
C4	-0.1375 (3)	-0.0462 (3)	0.6797 (2)	0.0524 (5)
C5	-0.2096 (4)	0.1026 (3)	0.6234 (3)	0.0608 (6)
H5	-0.3388	0.1231	0.5988	0.073*
C6	-0.0871 (3)	0.2196 (3)	0.6045 (3)	0.0599 (6)
H6	-0.1336	0.3210	0.5669	0.072*
C7	0.2702 (4)	0.3441 (3)	0.8037 (3)	0.0624 (6)
C8	0.3733 (3)	0.1963 (3)	0.9005 (3)	0.0568 (6)
C9	0.0695 (4)	0.3533 (4)	0.8653 (3)	0.0837 (9)
H9A	0.0023	0.2602	0.8821	0.125*

H9B	0.0011	0.4402	0.7936	0.125*
H9C	0.0773	0.3653	0.9592	0.125*
C10	0.3903 (5)	0.4859 (3)	0.7791 (4)	0.0940 (10)
H10A	0.4076	0.4928	0.8745	0.141*
H10B	0.3247	0.5773	0.7119	0.141*
H10C	0.5140	0.4764	0.7354	0.141*
N1	-0.2678 (3)	-0.1730 (3)	0.7030 (2)	0.0674 (6)
O1	-0.2015 (3)	-0.3028 (2)	0.7498 (3)	0.0997 (7)
O2	-0.4350 (3)	-0.1430 (3)	0.6715 (3)	0.1118 (9)
O3	0.2868 (2)	0.0840 (2)	0.98479 (19)	0.0724 (5)
O4	0.5595 (3)	0.1977 (2)	0.8833 (2)	0.0839 (6)
H4	0.6034	0.1101	0.9283	0.126*
S1	0.26504 (9)	0.34147 (7)	0.61287 (7)	0.0636 (2)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0654 (14)	0.0463 (12)	0.0439 (11)	0.0029 (10)	-0.0021 (10)	-0.0142 (9)
C2	0.0524 (13)	0.0544 (14)	0.0660 (14)	0.0081 (11)	-0.0043 (11)	-0.0233 (11)
C3	0.0646 (15)	0.0425 (12)	0.0637 (14)	0.0088 (10)	0.0004 (11)	-0.0201 (10)
C4	0.0580 (13)	0.0509 (12)	0.0505 (12)	0.0010 (10)	0.0029 (10)	-0.0230 (10)
C5	0.0566 (13)	0.0572 (14)	0.0687 (15)	0.0103 (11)	-0.0110 (11)	-0.0252 (12)
C6	0.0675 (15)	0.0447 (12)	0.0632 (14)	0.0124 (11)	-0.0135 (11)	-0.0170 (11)
C7	0.0765 (16)	0.0474 (13)	0.0621 (14)	0.0032 (11)	-0.0115 (12)	-0.0203 (11)
C8	0.0599 (15)	0.0550 (14)	0.0535 (13)	-0.0011 (11)	-0.0059 (11)	-0.0192 (11)
C9	0.090 (2)	0.084 (2)	0.0815 (19)	0.0282 (16)	-0.0080 (15)	-0.0396 (16)
C10	0.126 (3)	0.0560 (16)	0.102 (2)	-0.0089 (16)	-0.0276 (19)	-0.0311 (16)
N1	0.0681 (14)	0.0593 (13)	0.0789 (14)	-0.0041 (11)	0.0051 (11)	-0.0323 (11)
O1	0.0975 (15)	0.0514 (12)	0.144 (2)	-0.0033 (10)	-0.0158 (13)	-0.0309 (12)
O2	0.0606 (13)	0.0851 (15)	0.197 (3)	-0.0033 (11)	-0.0088 (14)	-0.0639 (16)
O3	0.0667 (11)	0.0628 (11)	0.0653 (11)	-0.0002 (9)	0.0036 (8)	-0.0026 (9)
O4	0.0596 (11)	0.0673 (12)	0.0976 (15)	-0.0026 (9)	-0.0058 (9)	-0.0041 (10)
S1	0.0762 (5)	0.0503 (4)	0.0543 (4)	-0.0081 (3)	-0.0034 (3)	-0.0100 (3)

*Geometric parameters (Å, °)*

C1—C6	1.384 (3)	C7—C10	1.531 (4)
C1—C2	1.387 (3)	C7—S1	1.851 (2)
C1—S1	1.769 (2)	C8—O3	1.208 (3)
C2—C3	1.361 (3)	C8—O4	1.296 (3)
C2—H2	0.9300	C9—H9A	0.9600
C3—C4	1.368 (3)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C4—C5	1.374 (3)	C10—H10A	0.9600
C4—N1	1.462 (3)	C10—H10B	0.9600
C5—C6	1.364 (3)	C10—H10C	0.9600
C5—H5	0.9300	N1—O2	1.206 (3)
C6—H6	0.9300	N1—O1	1.207 (3)

C7—C8	1.508 (3)	O4—H4	0.8200
C7—C9	1.508 (4)		
C6—C1—C2	119.0 (2)	C9—C7—S1	111.51 (18)
C6—C1—S1	120.86 (17)	C10—C7—S1	103.62 (19)
C2—C1—S1	120.08 (18)	O3—C8—O4	122.9 (2)
C3—C2—C1	120.5 (2)	O3—C8—C7	121.8 (2)
C3—C2—H2	119.7	O4—C8—C7	115.3 (2)
C1—C2—H2	119.7	C7—C9—H9A	109.5
C2—C3—C4	119.0 (2)	C7—C9—H9B	109.5
C2—C3—H3	120.5	H9A—C9—H9B	109.5
C4—C3—H3	120.5	C7—C9—H9C	109.5
C3—C4—C5	122.1 (2)	H9A—C9—H9C	109.5
C3—C4—N1	118.9 (2)	H9B—C9—H9C	109.5
C5—C4—N1	119.0 (2)	C7—C10—H10A	109.5
C6—C5—C4	118.4 (2)	C7—C10—H10B	109.5
C6—C5—H5	120.8	H10A—C10—H10B	109.5
C4—C5—H5	120.8	C7—C10—H10C	109.5
C5—C6—C1	120.9 (2)	H10A—C10—H10C	109.5
C5—C6—H6	119.6	H10B—C10—H10C	109.5
C1—C6—H6	119.6	O2—N1—O1	123.1 (2)
C8—C7—C9	111.5 (2)	O2—N1—C4	118.7 (2)
C8—C7—C10	111.5 (2)	O1—N1—C4	118.2 (2)
C9—C7—C10	112.9 (2)	C8—O4—H4	109.5
C8—C7—S1	105.22 (16)	C1—S1—C7	102.66 (11)
C6—C1—C2—C3	-1.8 (3)	C9—C7—C8—O4	-160.9 (2)
S1—C1—C2—C3	-179.55 (18)	C10—C7—C8—O4	-33.6 (3)
C1—C2—C3—C4	0.8 (4)	S1—C7—C8—O4	78.0 (2)
C2—C3—C4—C5	0.8 (4)	C3—C4—N1—O2	-179.4 (2)
C2—C3—C4—N1	-179.5 (2)	C5—C4—N1—O2	0.3 (4)
C3—C4—C5—C6	-1.3 (4)	C3—C4—N1—O1	-1.1 (3)
N1—C4—C5—C6	179.0 (2)	C5—C4—N1—O1	178.6 (2)
C4—C5—C6—C1	0.3 (4)	C6—C1—S1—C7	97.2 (2)
C2—C1—C6—C5	1.2 (4)	C2—C1—S1—C7	-85.1 (2)
S1—C1—C6—C5	178.97 (19)	C8—C7—S1—C1	68.65 (18)
C9—C7—C8—O3	20.9 (3)	C9—C7—S1—C1	-52.4 (2)
C10—C7—C8—O3	148.1 (3)	C10—C7—S1—C1	-174.16 (18)
S1—C7—C8—O3	-100.2 (2)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4 $\cdots$ O3 <sup>i</sup>	0.82	1.84	2.656 (3)	175
C2—H2 $\cdots$ O2 <sup>ii</sup>	0.93	2.46	3.211 (3)	138

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