V = 1375.4 (2) Å^3	Wedge
Z = 4	0.25 × 0.25 × 0.10 mm
D_x = 1.276 Mg m^{-3}	Colourless

Data collection

Enraf–Nonius CAD-4 MACHS diffractometer	R_{int} = 0.0177
$\omega/2\theta$ scans	θ_{max} = 74.89°
Absorption correction: none	$h = -1 \rightarrow 16$
3794 measured reflections	$k = -7 \rightarrow 1$
2822 independent reflections	$l = -21 \rightarrow 21$
2295 observed reflections [$I > 2\sigma(I)$]	3 standard reflections frequency: 150 min intensity decay: none

Refinement

Refinement on F^2	$\Delta\rho_{max}$ = 0.169 e Å^{-3}
$R(F)$ = 0.0359	$\Delta\rho_{min}$ = −0.119 e Å^{-3}
$wR(F^2)$ = 0.0958	Extinction correction: *SHELXL*93 (Sheldrick, 1993)
S = 1.024	Extinction coefficient: 0.0029 (3)
2822 reflections	Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
246 parameters	
All H-atom parameters refined	
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.2514P]$ where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{max}$ = 0.001	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O(1)	0.28961 (9)	−0.0157 (2)	0.64459 (7)	0.0645 (3)
O(2)	0.33628 (10)	0.3196 (2)	0.62674 (6)	0.0664 (3)
C(1)	0.05631 (11)	−0.0672 (3)	0.39314 (8)	0.0491 (3)
C(2)	−0.01914 (11)	0.0808 (3)	0.39970 (8)	0.0571 (4)
C(3)	0.01138 (12)	0.2848 (3)	0.42788 (9)	0.0576 (4)
C(4)	0.11781 (11)	0.3438 (2)	0.45098 (8)	0.0487 (3)
C(4a)	0.19316 (10)	0.1979 (2)	0.44376 (7)	0.0403 (3)
C(5)	0.41210 (11)	0.2981 (3)	0.36443 (8)	0.0493 (3)
C(6)	0.44212 (12)	0.2183 (3)	0.30073 (9)	0.0595 (4)
C(7)	0.41031 (12)	0.0168 (3)	0.27080 (9)	0.0601 (4)
C(8)	0.34835 (11)	−0.1110 (3)	0.30400 (8)	0.0514 (3)
C(8a)	0.31885 (9)	−0.0325 (2)	0.36797 (7)	0.0416 (3)
C(9)	0.25558 (10)	−0.1491 (2)	0.41385 (7)	0.0419 (3)
C(9a)	0.16273 (9)	−0.0088 (2)	0.41480 (7)	0.0408 (3)
C(10)	0.31233 (10)	0.2326 (2)	0.46794 (7)	0.0401 (3)
C(10a)	0.35064 (9)	0.1724 (2)	0.39810 (7)	0.0407 (3)
C(11)	0.36020 (10)	0.0645 (2)	0.53548 (7)	0.0404 (3)
C(12)	0.32958 (11)	−0.1631 (2)	0.50203 (8)	0.0451 (3)
C(13)	0.32422 (10)	0.1116 (2)	0.60785 (8)	0.0458 (3)
C(14)	0.2993 (3)	0.3896 (5)	0.69251 (14)	0.0895 (7)

Table 2. *Selected geometric parameters (Å, °)*

O(1)—C(13)	1.198 (2)	C(9)—C(12)	1.561 (2)
O(2)—C(13)	1.336 (2)	C(10)—C(10a)	1.509 (2)
O(2)—C(14)	1.451 (2)	C(10)—C(11)	1.564 (2)
C(4a)—C(10)	1.513 (2)	C(11)—C(13)	1.514 (2)
C(8a)—C(9)	1.511 (2)	C(11)—C(12)	1.542 (2)
C(9)—C(9a)	1.509 (2)		
C(4)—C(4a)—C(10)	126.49 (12)	C(10a)—C(10)—C(4a)	108.20 (10)
C(9a)—C(4a)—C(10)	113.08 (11)	C(10a)—C(10)—C(11)	106.99 (10)
C(8)—C(8a)—C(9)	126.81 (13)	C(5)—C(10a)—C(10)	126.25 (12)
C(10a)—C(8a)—C(9)	112.87 (11)	C(8a)—C(10a)—C(10)	113.52 (11)
C(9a)—C(9)—C(8a)	107.95 (10)	O(1)—C(13)—O(2)	123.09 (13)
C(9a)—C(9)—C(12)	106.60 (10)	O(1)—C(13)—C(11)	126.26 (13)
C(8a)—C(9)—C(12)	106.04 (10)	O(2)—C(13)—C(11)	110.64 (11)

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1205). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Methyl-2-thiophenecarboxylic Acid

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Abstract

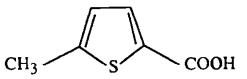
The present X-ray diffraction study establishes the molecular structure of 5-methyl-2-thiophenecarboxylic acid, $C_6H_6O_2S$. The crystal structure is stabilized by an O—H···O hydrogen bond and C—H···O and S···S non-bonded intermolecular interactions.

Comment

The crystal and molecular structure of 5-methyl-2-thiophenecarboxylic acid, (I), was first reported by Simonsen, Cordell & Boggs (1980) without atomic

† Contribution No. 1338 of the Instituto de Química, UNAM.

coordinates and packing information. The present work describes the structure determination and crystal packing in detail.



(I)

A view of the molecule with the atom-numbering scheme is shown in Fig. 1. The average C—S bond length is 1.712 (3) Å, which is similar to that found in a related compound (Danielsen, 1969). The C7—O9 bond length of 1.287 (3) Å is significantly longer than the C7—O8 length of 1.251 (3) Å, and the C2—C7—O9 angle of 115.8 (3)° is significantly smaller than the commonly observed mean value of 121.6° for ionized carboxy groups. These values indicate that the carboxy group is not ionized. The carboxylic acid group is planar, as is the five-membered ring (within 0.002 Å), and the interplanar angle between their planes is 4.9 (2)°.

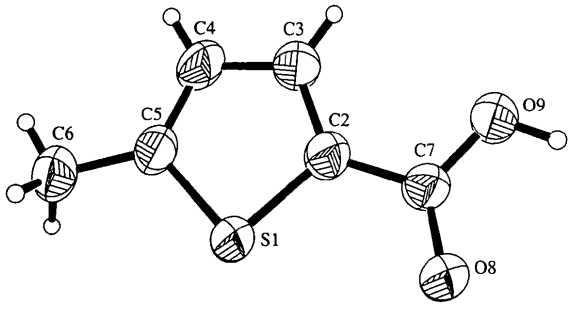


Fig. 1. The molecular structure of the title compound with the atom-labelling scheme, showing 50% probability displacement ellipsoids.

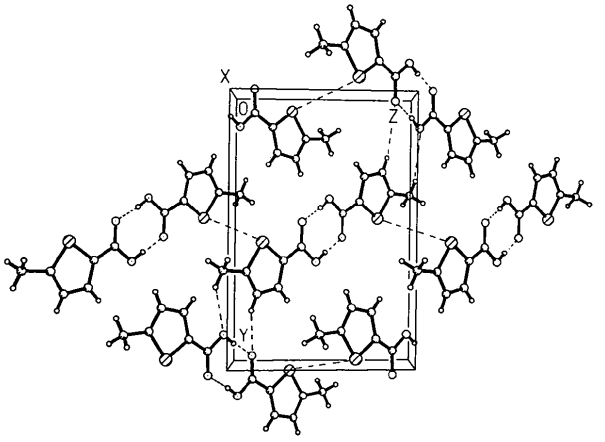


Fig. 2. A unit-cell drawing of the packing arrangement, with dashed lines indicating O—H···O, C—H···O and S···S intermolecular interactions.

The molecular packing viewed down the *a* axis is shown in Fig. 2. The crystal structure is stabilized by O—H···O dimer and C—H···O and S···S non-bonded interactions. The hydrogen-bonding details are given in Table 3. The hydroxy O9 atom is involved in an intermolecular hydrogen bond with atom O8 at a distance of 2.617 (3) Å (Allen, Kennard & Taylor, 1983). There is also an S···S($-x$, $1-y$, $-z$) non-bonded intermolecular interaction of 3.793 (2) Å. There are two C—H···O intermolecular interactions (Desiraju, 1991), with distances of 3.558 (4) and 3.559 (4) Å. One dimer is nearly perpendicular to another (Fig. 2) and they are arranged in a zigzag manner.

Experimental

The title compound was recrystallized from acetone at room temperature.

Crystal data

$C_6H_6O_2S$
M_r = 142.17
Monoclinic
$P2_1/c$
a = 5.079 (1) Å
b = 13.925 (3) Å
c = 9.450 (2) Å
β = 93.30 (1)°
V = 667.2 (2) Å^3
Z = 4
D_x = 1.415 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 10–30°
μ = 3.670 mm^{-1}
T = 293 (2) K
Needle
0.22 × 0.20 × 0.17 mm
Colourless

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction:
ψ scan
T_{min} = 0.064, T_{max} = 0.261
1129 measured reflections
1004 independent reflections
932 observed reflections
[$I > 2\sigma(I)$]

R_{int} = 0.024
θ_{max} = 62.97°
$h = -5 \rightarrow 5$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 10$
3 standard reflections
monitored every 100 reflections
intensity decay: 2.5%

Refinement

Refinement on F^2
$R(F)$ = 0.0388
$wR(F^2)$ = 0.1628
S = 1.242
981 reflections
106 parameters
All H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.4489P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.272 e Å^{-3}
$\Delta\rho_{min}$ = −0.399 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
S1	0.1099 (1)	0.5438 (1)	0.1841 (1)	0.0414 (4)
C2	−0.0817 (6)	0.6095 (2)	0.2912 (3)	0.0396 (7)
C3	−0.0583 (7)	0.7063 (2)	0.2655 (3)	0.0486 (8)
C4	0.1130 (6)	0.7254 (2)	0.1588 (4)	0.0491 (8)
C5	0.2200 (6)	0.6456 (2)	0.1042 (3)	0.0415 (7)
C6	0.4109 (8)	0.6393 (3)	−0.0101 (4)	0.0530 (9)
C7	−0.2523 (6)	0.5617 (2)	0.3866 (3)	0.0394 (7)
O8	−0.2671 (4)	0.47208 (13)	0.3891 (2)	0.0476 (6)
O9	−0.3845 (5)	0.61708 (15)	0.4653 (2)	0.0538 (7)

Table 2. *Selected geometric parameters (Å, °)*

S1—C2	1.710 (3)	C4—C5	1.353 (4)
S1—C5	1.715 (3)	C5—C6	1.494 (4)
C2—C3	1.376 (4)	C7—O8	1.251 (3)
C2—C7	1.448 (4)	C7—O9	1.287 (3)
C3—C4	1.394 (4)		
C2—S1—C5	91.7 (1)	C4—C5—C6	128.0 (3)
C3—C2—C7	128.4 (3)	C4—C5—S1	111.3 (2)
C3—C2—S1	111.2 (2)	C6—C5—S1	120.8 (2)
C7—C2—S1	120.3 (2)	O8—C7—O9	123.6 (3)
C2—C3—C4	112.3 (3)	O8—C7—C2	120.6 (3)
C5—C4—C3	113.6 (3)	O9—C7—C2	115.8 (3)

Table 3. *Hydrogen-bonding geometry (Å, °) and intermolecular interactions*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H9···O8^{i}	0.90 (5)	1.72 (5)	2.617 (3)	177 (5)
C4—H4···O8^{ii}	0.93 (4)	2.65 (4)	3.558 (4)	164 (3)
C6—H63···O9^{iii}	0.86 (5)	2.73 (6)	3.559 (4)	161 (5)

Symmetry codes: (i) $-1-x, 1-y, 1-z$; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $1+x, \frac{3}{2}-y, z-\frac{1}{2}$.

Cell refinement: *XSCANS* (Siemens, 1992). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A *de novo* Designed Possible 5-Lipoxygenase Inhibitor: α-Acetoxy-*N*-[1-(1-tricyclo[3.3.1.1^{3,7}]dec-1-yl)ethyl]benzeneacetamide

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Abstract

The structure of the title compound {alternative IUPAC name: α-[1-(tricyclo[3.3.1.1^{3,7}]dec-1-yl)ethylaminocarbonyl]benzyl acetate}, $C_{22}H_{29}NO_3$, a *de novo* designed non-acidic anti-inflammatory agent, has been determined. Data collection was performed utilizing methods common in macromolecular crystallography (rotation method and an imaging plate area detector) and provided data of good quality.

Comment

Leukotrienes are products of arachidonic acid metabolism and are frequently mediators of inflammation. They are derived from arachidonic acid through the action of the enzyme 5-lipoxygenase (5-LO). Pharmacological intervention in the conversion of arachidonic acid to leukotrienes by the development of 5-LO inhibitors seems to be a promising clinical approach (Gasland & Salmon, 1991; Musser & Kreff, 1992). In an attempt to produce possible 5-LO inhibitors, we are involved in a study of the design and synthesis of compounds of the general type (*A*). Linkage of the highly hydrophobic adamantane ring or the 1-adamantylethyl group to a phenyl ring through different bridges produces molecules with anti-inflammatory activity. The title compound, (*B*), a member of the series (*A*) compounds,

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