2-(1-Mesitylthioethyl)-1-cyclopentanoic Acid

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Abstract. $C_{17}H_{24}O_2S$, $M_r = 292.44$, triclinic, $P\bar{1}$, $a = 8.504$ (1), $b = 12.236$ (2), $c = 7.964$ (1) Å, $\alpha = 84.65$ (1), $\beta = 84.39$ (1), $\gamma = 84.02$ (1)°, $V = 817.3$ (2) Å^3, $Z = 2$, $D_x = 1.19$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 17.0$ cm^{-1}, $F(000) = 316$, $T = 291$ K, $R = 0.072$ for 1982 observed reflections. The configuration of the substituents is *cis*. Cyclopentane adopts an envelope conformation with C(1) at the flap and the carboxylic group in a pseudoaxial position. There is dimer formation *via* hydrogen bonds between two carboxylic groups [O⋯O = 2.678 (3) Å].

Experimental. Crystals obtained by evaporation from isooctane. D_m not measured. Plate crystal with dimensions $0.6 \times 0.4 \times 0.1$ mm. Lattice parameters refined using 15 reflections in the range $8° \le 2\theta \le 30°$. Syntex $P2_1$, Ni-filtered Cu $K\alpha$ radiation. 2061 $h \pm k \pm l$ independent reflections with $\sin\theta/\lambda \le 0.53$ Å^{-1}, 1982 with $I \ge 2.5\sigma(I)$. Standard reflection $2\bar{2}1$ checked every 50 reflections: no significant deviation. Structure solved by Patterson map and *DIRDIF*81 (Beurskens *et al.*, 1981). 20 H atoms from difference Fourier synthesis; 4 H atoms calculated. Anisotropic least-squares refinement (*SHELX*76, Sheldrick, 1976) using F; H isotropic

with common refined temperature factor, $U = 0.078$ Å^2. $w = 1/(\sigma^2 + 0.03382F^2)$, $R = 0.072$, $wR = 0.089$ for 1982 observed reflections. Final $(\Delta/\sigma)_{max} = 1.57$. Max. and min. heights in final difference Fourier synthesis 0.37 and -0.49 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The high Δ/σ and R values are a result of the poor quality of the crystal. The atomic parameters are given in Table 1.* Fig. 1 shows the molecule and numbering scheme; Fig. 2 shows the packing in the unit cell (*PLUTO*, Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42745 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
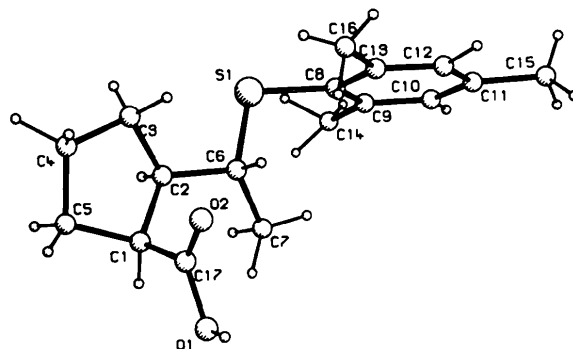
Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = (8/3)\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq}
C(1)	1643 (3)	5186 (2)	2849 (4)	3.66 (5)
C(2)	1590 (3)	4265 (2)	1655 (3)	3.13 (4)
C(3)	2585 (3)	4633 (2)	43 (4)	3.84 (5)
C(4)	2573 (3)	5880 (2)	−20 (4)	4.69 (6)
C(5)	1629 (4)	6232 (2)	1598 (4)	4.72 (6)
C(6)	2029 (3)	3103 (2)	2421 (3)	3.43 (5)
C(7)	1009 (4)	2805 (3)	4058 (4)	4.96 (6)
S(1)	1881 (1)	2146 (1)	821 (1)	4.38 (2)
C(8)	2720 (3)	886 (2)	1806 (3)	3.57 (5)
C(9)	1730 (3)	73 (2)	2496 (3)	4.27 (5)
C(10)	2449 (4)	−900 (2)	3263 (4)	4.97 (6)
C(11)	4061 (4)	−1094 (2)	3382 (4)	4.99 (6)
C(12)	4988 (4)	−268 (2)	2671 (5)	4.98 (6)
C(13)	4373 (3)	696 (2)	1904 (3)	3.85 (5)
C(14)	−26 (4)	198 (4)	2442 (5)	6.23 (7)
C(15)	4816 (7)	−2163 (3)	4159 (7)	8.16 (10)
C(16)	5496 (4)	1519 (3)	1131 (5)	5.01 (6)
C(17)	3115 (3)	5062 (2)	3780 (3)	3.46 (4)
O(1)	2849 (3)	5163 (2)	5412 (3)	5.05 (4)
O(2)	4454 (2)	4912 (2)	3086 (2)	4.96 (4)

Fig. 1. View of the molecule with the atom numbering.

Fig. 2. Stereoview of the crystal packing in the unit cell.

Table 2. *Bond distances (Å) and angles (°)*

C(2)—C(1)	1·548 (3)	C(13)—C(8)	1·409 (4)
C(5)—C(1)	1·547 (4)	C(10)—C(9)	1·397 (4)
C(17)—C(1)	1·504 (4)	C(14)—C(9)	1·490 (5)
C(3)—C(2)	1·525 (3)	C(11)—C(10)	1·378 (5)
C(6)—C(2)	1·518 (4)	C(12)—C(11)	1·391 (5)
C(4)—C(3)	1·522 (4)	C(15)—C(11)	1·502 (5)
C(5)—C(4)	1·523 (5)	C(13)—C(12)	1·355 (4)
C(7)—C(6)	1·530 (3)	C(16)—C(13)	1·509 (4)
S(1)—C(6)	1·831 (2)	O(1)—C(17)	1·312 (3)
C(8)—S(1)	1·777 (3)	O(2)—C(17)	1·219 (3)
C(9)—C(8)	1·408 (4)		
C(5)—C(1)—C(2)	102·1 (2)	C(13)—C(8)—C(9)	119·8 (2)
C(17)—C(1)—C(2)	113·3 (2)	C(10)—C(9)—C(8)	117·6 (3)
C(17)—C(1)—C(5)	110·2 (2)	C(14)—C(9)—C(8)	123·5 (3)
C(3)—C(2)—C(1)	104·6 (2)	C(14)—C(9)—C(10)	118·9 (3)
C(6)—C(2)—C(1)	115·0 (2)	C(11)—C(10)—C(9)	123·3 (3)
C(6)—C(2)—C(3)	116·0 (2)	C(12)—C(11)—C(10)	116·8 (3)
C(4)—C(3)—C(2)	107·3 (2)	C(15)—C(11)—C(10)	122·4 (3)
C(5)—C(4)—C(3)	106·5 (2)	C(12)—C(11)—C(15)	120·7 (3)
C(4)—C(5)—C(1)	105·8 (2)	C(11)—C(12)—C(13)	123·1 (3)
C(7)—C(6)—C(2)	113·0 (2)	C(8)—C(13)—C(12)	119·4 (3)
S(1)—C(6)—C(2)	108·1 (2)	C(8)—C(13)—C(16)	122·1 (3)
S(1)—C(6)—C(7)	111·4 (2)	C(12)—C(13)—C(16)	118·5 (3)
C(8)—S(1)—C(6)	101·5 (1)	C(1)—C(17)—O(1)	114·4 (2)
C(9)—C(8)—S(1)	119·9 (2)	C(1)—C(17)—O(2)	123·5 (2)
C(13)—C(8)—S(1)	120·4 (2)	O(1)—C(17)—O(2)	122·1 (2)

Related literature. The synthesis and crystal structure of the related 2-(1-phenylthioethyl)pent-3-ene-1-carboxylic acid has been described by Michel *et al.* (1980).

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Structure of 2-(2-Chloro-3-pyridylthio)-3,5,6-trimethylphenol at 163 K

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Abstract. $C_{14}H_{14}ClNOS$, $M_r = 279·78$, monoclinic, $P2_1/c$, $a = 7·5049$ (10), $b = 20·487$ (3), $c = 8·6131$ (10) Å, $\beta = 90·527$ (8)°, $V = 1324·2$ (3) Å^3, $Z = 4, D_x(163$ K$) = 1·403, D_m(295$ K$) = 1·343$ g cm^{-3}, λ(Mo Kα) = 0·71069 Å, $\mu = 4·242$ cm^{-1}, $F(000) = 584$, $R = 0·0407$ for 2930 observed reflections. The molecule is a precursor to 6,7,9-trimethyl-4-azaphenoxathiin through displacement of the chloro substituent through release of HCl. The angle between the aromatic rings is 87·52 (6)°; the C—S—C plane is nearly parallel to the pyridine plane [dihedral angle: 4·33 (9)°]. The O atom is 5·579 (2) Å from the Cl atom. Bond lengths are normal: C—S, 1·766, 1·774 Å; C—Cl, 1·744 Å; aromatic C—C, 1·370–1·407 Å; other C—C, 1·506–1·511 Å; C—N, 1·319, 1·342 Å. The C—S—C angle is 102·98 (8)°.

Experimental. A summary of data collection and structural refinement is given in Table 1. Density measured by flotation. S and Cl positions obtained from *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Electron density map afforded remaining non-H positions. Electron density difference map at $R = 0·061$ provided H positions as peaks of 0·57–0·84 e Å^{-3}. All positions and thermal parameters (isotropic H atoms) refined by full-matrix