

trans*-(±)-2-Carboxymethyl-3-phenylcyclopropane-1-carboxylic acid*Thomas D. Avery, Dennis K. Taylor and Edward R. T. Tiekink***

Department of Chemistry, The University of Adelaide, Australia 5005

Correspondence e-mail:
edward.tiekink@adelaide.edu.au

The carboxyl and carboxymethyl substituents in the title compound, C₁₂H₁₂O₄, lie on opposite sides of the cyclopropyl ring and each carboxyl group participates in a centrosymmetric hydrogen-bonding scheme, typical for carboxylic acids, leading to the formation of zigzag chains in the crystal.

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Key indicators

Single-crystal X-ray study

T = 173 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.037

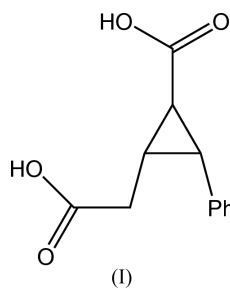
wR factor = 0.114

Data-to-parameter ratio = 17.2

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

Comment

The title compound, (I), was prepared by the controlled hydrolysis of the parent methyl diester (Avery *et al.*, 2001). The structure determination confirms the overall stereochemistry, notably the positioning of the substituents around the cyclopropyl ring as shown in Fig. 1. The plane of the C11/O11/O11A carboxylic acid group is almost orthogonal to the cyclopropane plane, as seen in the value of the $X-\text{C}1-\text{C}11-\text{O}11$ torsion angle of $4.1(2)^\circ$, where X is the midpoint of the C2—C3 bond. By contrast, the dihedral angle formed by the planes of the phenyl and cyclopropyl rings is $68.70(11)^\circ$. The conformation is thus described as *cis*-bisected in accord with Allen (1980). The distal C2—C3 bond distance of 1.5027 (19) Å is shorter, as expected (Allen, 1980), than the respective vicinal C1—C2 and C1—C3 bond distances of 1.5196 (19) and 1.5195 (17) Å, reflecting the influence of the π -acceptor carboxylic acid group upon the C—C bonds within the cyclopropyl ring.



Molecules associate in the lattice *via* hydrogen-bonding interactions involving both carboxylic acid residues so that each carboxylic acid group associates with its centrosymmetrically related mate. Thus, $\text{O}-\text{H}11\text{A} = 0.92 \text{ \AA}$, $\text{H}11\text{A} \cdots \text{O}11^{\text{i}} = 1.74 \text{ \AA}$ and $\text{O}11\text{A} \cdots \text{O}11^{\text{i}} = 2.653(2) \text{ \AA}$, and the angle at H11A = 174° [symmetry code: (i) $1 - x, -1 - y, -z$]. The comparable parameters for H22A and O22ⁱⁱ are 0.87, 1.78 and 2.646(2) Å, and 175° [symmetry code: (ii) $2 - x, -y, -z$]. Such association leads to the formation of zigzag chains extending in the *ab* plane.

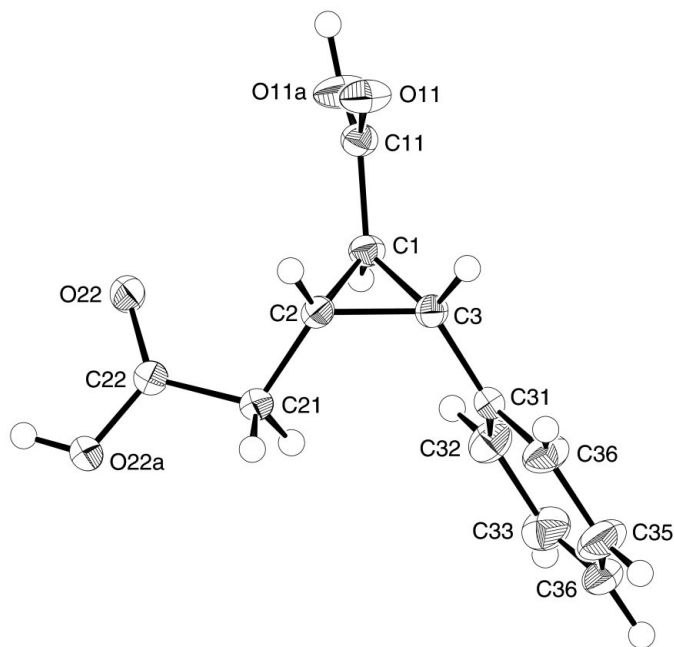


Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

Experimental

The title compound was prepared according to the literature procedure of Avery *et al.* (2001). Colourless crystals were obtained by slow evaporation of a dichloromethane/heptane solution of the compound (m.p. 458–460 K).

Crystal data

$C_{12}H_{12}O_4$
 $M_r = 220.22$
Monoclinic, $P2_1/n$
 $a = 8.555$ (4) Å
 $b = 8.814$ (4) Å
 $c = 14.849$ (2) Å
 $\beta = 101.70$ (2)°
 $V = 1096.5$ (6) Å³
 $Z = 4$
 $D_x = 1.334$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 24 reflections
 $\theta = 7.3$ – 10.7 °
 $\mu = 0.10$ mm⁻¹
 $T = 173$ K
Block, colourless
 $0.45 \times 0.29 \times 0.16$ mm

Data collection

Rigaku AFC-7R diffractometer
 ω - 2θ scans
4704 measured reflections
2510 independent reflections
1829 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.020$
 $\theta_{max} = 27.5$ °

$h = -11 \rightarrow 6$
 $k = 0 \rightarrow 11$
 $l = -19 \rightarrow 19$
3 standard reflections
every 400 reflections
intensity decay: 0.3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.114$
 $S = 0.90$
2510 reflections
146 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2 + 0.3498P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.21$ e Å⁻³
 $\Delta\rho_{min} = -0.23$ e Å⁻³

The C-bound H atoms were placed in geometrically calculated positions and included in the final refinement in the riding-model approximation with an overall isotropic displacement parameter. The H atoms on O atoms were located from a difference map but were not refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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