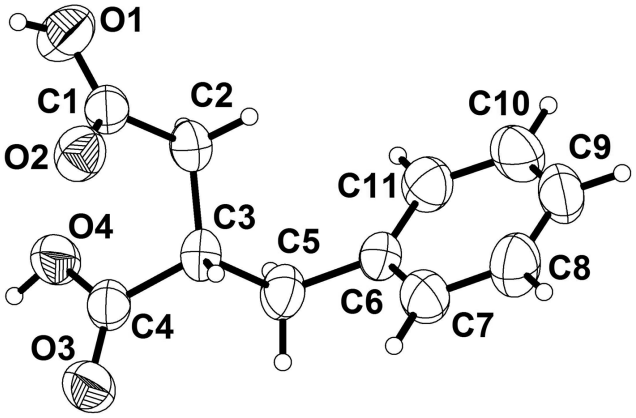
Yun-Peng Zhang and Bing Li*

The crystal structure of (*S*)-2-benzylsuccinic acid, $C_{11}H_{12}O_4$



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Abstract

$C_{11}H_{12}O_4$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 9.960(2)$ Å, $b = 9.960(2)$ Å, $c = 10.967(2)$ Å, $V = 1083.9(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0505$, $wR_{ref}(F^2) = 0.1861$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title compound was prepared from L-phenylalanine by diazotization, esterification, condensation, hydrolyzation and decarboxylation. It was recrystallized from water to give colorless crystals for single-crystal X-ray diffraction.

Experimental details

All H atoms were positioned geometrically and treated as riding, with C—H bond lengths constrained to 0.93 Å for phenyl hydrogen atoms, 0.97 Å for methylene H atoms and 0.97 Å for methyne H atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. The O—H bond length was constrained to 0.82 Å and the $U_{iso}(H)$ was set to $1.5U_{eq}(O)$. The presence of the *S* enantiomer was concluded from the synthesis.

Table 1: Data collection and handling.

Crystal:	Colourless platelet
Size:	0.40 × 0.40 × 0.19 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω scans
$2\theta_{max}$, completeness:	54.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10615, 2468, 0.040
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1466
$N(param)_{refined}$:	137
Programs:	Rigaku programs [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.4610(3)	0.3699(3)	0.3337(2)	0.0789(9)
H1A	0.4154	0.3020	0.3425	0.118*
O2	0.4629(3)	0.3607(2)	0.5359(2)	0.0642(7)
O3	0.8093(3)	0.3531(3)	0.6540(2)	0.0717(8)
O4	0.7878(2)	0.3354(2)	0.4531(2)	0.0644(7)
H4A	0.8434	0.2755	0.4631	0.097*
C1	0.4999(3)	0.4121(3)	0.4377(3)	0.0514(7)
C2	0.5946(3)	0.5294(3)	0.4387(4)	0.0568(8)
H2A	0.5426	0.6118	0.4409	0.068*
H2B	0.6467	0.5292	0.3638	0.068*
C3	0.6909(3)	0.5270(3)	0.5471(3)	0.0511(7)
H3A	0.6381	0.5377	0.6220	0.061*
C4	0.7670(3)	0.3959(3)	0.5545(3)	0.0532(7)
C5	0.7958(3)	0.6421(3)	0.5406(4)	0.0609(9)
H5A	0.8599	0.6320	0.6068	0.073*
H5B	0.8451	0.6355	0.4644	0.073*
C6	0.7307(3)	0.7790(3)	0.5490(3)	0.0523(8)
C7	0.6860(4)	0.8261(4)	0.6591(3)	0.0697(11)
H7A	0.6956	0.7731	0.7283	0.084*
C8	0.6263(5)	0.9523(4)	0.6689(4)	0.0788(12)
H8A	0.5948	0.9826	0.7438	0.095*
C9	0.6144(4)	1.0317(3)	0.5671(5)	0.0744(11)
H9A	0.5762	1.1167	0.5731	0.089*
C10	0.6587(4)	0.9857(4)	0.4576(5)	0.0763(11)
H10A	0.6504	1.0393	0.3886	0.092*
C11	0.7159(4)	0.8596(3)	0.4482(4)	0.0648(9)
H11A	0.7449	0.8289	0.3725	0.078*

***Corresponding author: Bing Li,** Department of Chemistry, Zhejiang A & F University, Lin'an, Zhejiang 311300, People's Republic of China, e-mail: libingzjfc@163.com
Yun-Peng Zhang: Department of Chemistry, Zhejiang A & F University, Lin'an, Zhejiang 311300, People's Republic of China

Comment

(*S*)-2-benzylsuccinic acid is a valuable compound, which was employed as a key intermediate for hypoglycemic KAD-1229 by means of asymmetric alkylation of N-acylsultam [3, 4]. (*S*)-2-benzylsuccinic acid can be synthesized by various procedures. The preparation of (*S*)-2-benzylsuccinic acid from *L*-phenylalanine is considered to be one of the most clean, efficient and convenient processes [5, 6].

The asymmetric unit of the title structure contains one independent molecule. All bond lengths and angles of this molecule are in the expected ranges. The molecules are packed in the crystal structure *via* two intermolecular strong classical hydrogen bonds, O4–H4A···O2^{i} and O1–H1A···O2^{ii}. The H···A distances are 1.80 and 1.87 Å, respectively and the angles are 172 and 174°, respectively. Symmetry codes: (i) $x+1/2, -y+1/2, -z+1$; (ii) $x-1/2, -y+1/2, -z+1$.

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