Crystal structure of *erythro*-2-hydroxy-3-phenylbutyric acid, $C_6H_5CH(CH_3)CH(OH)COOH$

R. Ludwig* and D. Lentz*

Freie Universität Berlin, Institut für Chemie / Analytische und Anorganische Chemie, Fabockstr. 34-36, D-14195 Berlin, Germany

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applied for analytical purposes. We were able to purify one of the diastereomers of 2-hydroxy-3-phenylbutyric acid [3] by fractionated crystallization. The *erythro*-isomer is less soluble in toluene compared with the *threo* form.

Table 1. Data collection and handling.

Crystal:	colourless needle, size 0.2 × 0.3 × 0.8 mm
Wavelength:	Mo K_{α} radiation (0.71069 Å)
μ :	0.97 cm ⁻¹
Diffractometer, scan mode:	Stoe, ω
$2\theta_{\max}$:	52°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	1916, 1790
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1172
$N(\text{param})_{\text{refined}}$:	167
Programs:	SHELXS-86 [4], SHELXL-93 [5]

Abstract

$C_{10}H_{12}O_3$, monoclinic, $P12_1/c1$ (No. 14), $a = 8.427(2)$ Å, $b = 5.967(2)$ Å, $c = 18.008(2)$ Å, $\beta = 90.43(2)^\circ$, $V = 905.5$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.033$, $wR_{\text{all}}(F^2) = 0.096$, $T = 293$ K.

Source of material

The title compound was synthesized from the hydrogensulfite adduct of 2-phenylpropionaldehyde (Merck) via the corresponding nitrile [1]. It was crystallized 8 times from toluene to separate it from the more soluble *threo*-form, and twice from water (mp 395 K, Lit. 395 K for the racemate). NMR spectroscopic data is included in the deposited CIF-file.

Discussion

α -Hydroxycarboxylic acids are of interest as complexing ligands, e.g. for lanthanoids [2]. In these studies, it turned out that the stability of adducts between a lanthanoid ion, a β -diketone and an α -hydroxycarboxylic acid is influenced by the structure of the latter. The adduct formation can improve the mutual separation and

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1A)	4e	0.654(3)	0.066(4)	0.472(1)	0.058(6)
H(1B)	4e	0.745(2)	−0.079(4)	0.532(1)	0.048(5)
H(1C)	4e	0.837(2)	0.063(3)	0.469(1)	0.049(5)
H(2)	4e	0.756(2)	0.399(3)	0.5249(9)	0.031(4)
H(3)	4e	0.995(2)	0.269(3)	0.5641(9)	0.028(4)
H(12)	4e	0.544(2)	0.591(4)	0.570(1)	0.046(5)
H(13)	4e	0.315(3)	0.622(4)	0.640(1)	0.060(6)
H(14)	4e	0.232(2)	0.325(3)	0.715(1)	0.046(5)
H(15)	4e	0.391(2)	−0.007(4)	0.720(1)	0.053(6)
H(16)	4e	0.618(2)	−0.026(3)	0.650(1)	0.044(5)
H(1O)	4e	0.947(3)	0.123(5)	0.691(2)	0.089(9)
H(3O)	4e	0.936(3)	0.789(5)	0.628(1)	0.073(7)

* (e-mails: rludwig@mail.chemie.fu-berlin.de,
lentz@chemie.fu-berlin.de)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	4e	0.9226(2)	0.0811(2)	0.64847(7)	0.0504(8)	0.0176(6)	0.0420(8)	0.0027(5)	-0.0149(6)	-0.0001(5)
O(2)	4e	0.9354(2)	0.4880(2)	0.71146(7)	0.0568(8)	0.0297(7)	0.0411(8)	0.0005(6)	-0.0086(6)	-0.0075(5)
O(3)	4e	0.9320(2)	0.6593(2)	0.60114(7)	0.078(1)	0.0179(6)	0.0493(8)	-0.0024(6)	-0.0055(7)	0.0014(6)
C(1)	4e	0.7483(2)	0.0607(3)	0.5037(1)	0.047(1)	0.034(1)	0.035(1)	0.0017(8)	-0.0058(9)	-0.0081(8)
C(2)	4e	0.7530(2)	0.2648(3)	0.55542(9)	0.0363(9)	0.0227(8)	0.0278(8)	0.0016(7)	-0.0026(7)	0.0019(7)
C(3)	4e	0.9097(2)	0.2687(3)	0.60068(9)	0.0337(9)	0.0191(7)	0.0332(9)	0.0028(7)	-0.0005(7)	0.0001(7)
C(4)	4e	0.9262(2)	0.4832(3)	0.64496(9)	0.0269(8)	0.0219(8)	0.040(1)	0.0027(7)	-0.0031(7)	-0.0030(7)
C(11)	4e	0.6059(2)	0.2812(3)	0.60293(8)	0.0325(8)	0.0275(8)	0.0285(8)	0.0001(7)	-0.0075(6)	-0.0026(7)
C(12)	4e	0.5120(2)	0.4721(3)	0.6002(1)	0.041(1)	0.0312(9)	0.042(1)	0.0035(8)	-0.0046(8)	0.0025(8)
C(13)	4e	0.3740(2)	0.4881(4)	0.6407(1)	0.039(1)	0.047(1)	0.056(1)	0.0138(9)	-0.0035(9)	-0.005(1)
C(14)	4e	0.3276(2)	0.3126(4)	0.6856(1)	0.0319(9)	0.062(1)	0.044(1)	-0.002(1)	0.0019(8)	-0.006(1)
C(15)	4e	0.4193(2)	0.1230(4)	0.6895(1)	0.041(1)	0.048(1)	0.041(1)	-0.0081(9)	-0.0012(8)	0.0050(9)
C(16)	4e	0.5566(2)	0.1064(3)	0.6486(1)	0.039(1)	0.0303(9)	0.042(1)	0.0008(8)	-0.0034(8)	0.0035(8)

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References

1. Corson, B. B.; Dodge, R. A.; Harris, S. A.; Yeaw, J. S.: Mandelic acid. *Org. Synth. Coll. I* (1956) 336-340.
2. Hasegawa, Y.; Harada, Y.; Ludwig, R.: Application of α -hydroxy-carboxylic acids as masking and synergistic agents in mutual separation of lanthanoids(III). *Int. Symp. Trace Analysis*; Tokyo Univ., 31.7.-1.8. Japan 1998.
3. Beilstein Handbook of organic chemistry. Springer Verlag, Berlin 1971, 4. Ed. Vol. 10, Suppl. E III, 595.
4. Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct methods for Larger Structure. *Acta Crystallogr. A* **46** (1990) 467-473.
5. Sheldrick, G. M.: SHELXL-93, a program for refining crystal structures. University of Göttingen, pre-release version, Germany 1992.