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Crystal structure of (S)-pipecolic acid (2R,3R)-tartrate, $C_{10}H_{17}NO_8$

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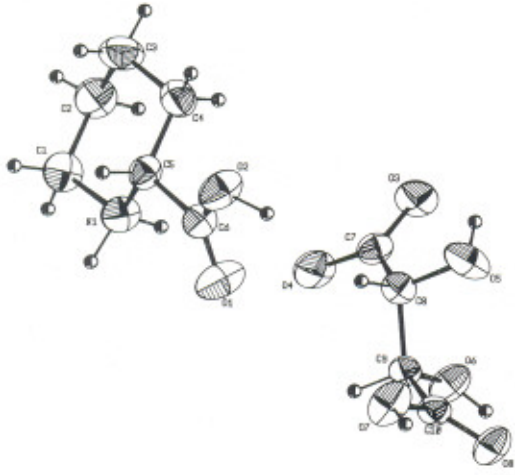

Source of material: Title compound was prepared by resolution of (R,S)-pipecolic acid with (2R,3R)-tartaric acid and recrystallized from ethanol. M.p. 182°.
The piperidine ring has a chair conformation with the carboxyl group in the equatorial position, as observed in (R,S)-pipecolic acid tetrahydrate (see ref. 1). The amino group of pipecolic acid and one carboxyl group of tartaric acid are charged.

Orthorhombic, $P2_12_12_1$ (no 19), $a = 18.627(3)$, $b = 9.064(3)$, $c = 7.600(3)$ Å, $V = 1283.1$ Å^3, $Z = 4$, $\varrho_m = 1.400$ g · cm^{-3}, $R = 0.045$.

Table 1. Parameters used for the X-ray data collection

Diffractometer type:	Philips PW1100	Number of unique reflections:	1325
Wave length:	Mo K_α radiation (0.7107 Å)	Criterion for unobserved reflections:	$F_o < 5\sigma(F_o)$
Crystal characteristics:	size $0.25 \times 0.15 \times 0.25$ mm	Number of refined parameters:	237
Temperature of measurement:	293 K	Scan mode:	2Θ
$2\theta_{max}$:	50°	μ:	0.83 cm^{-1}
		Structure solution program used:	SHELX

Table 2. Final atomic coordinates and displacement parameters (in $Å^2$)

Atom	x	y	z	U_{iso}/U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	0.3640(2)	0.5342(4)	1.0884(4)	0.054(2)	0.042(2)	0.043(2)	0.002(2)	−0.008(2)	0.002(2)
C(1)	0.4179(2)	0.5349(5)	1.2370(5)	0.055(2)	0.047(2)	0.041(2)	0.009(2)	−0.009(2)	0.008(2)
C(2)	0.3873(2)	0.6179(6)	13936(6)	0.061(3)	0.067(3)	0.038(2)	0.005(2)	−0.008(2)	0.000(2)
C(3)	0.3675(2)	0.7750(6)	1.3381(6)	0.071(3)	0.063(3)	0.044(2)	0.009(2)	−0.005(2)	−0.017(3)
C(4)	0.3144(2)	0.7713(5)	1.1838(6)	0.045(2)	0.052(2)	0.050(3)	0.009(2)	−0.001(2)	−0.004(2)
C(5)	0.3451(2)	0.6862(4)	1.0295(5)	0.037(2)	0.036(2)	0.038(2)	−0.000(2)	−0.006(2)	0.003(2)
C(6)	0.2938(2)	0.6735(4)	0.8739(5)	0.041(2)	0.040(2)	0.041(2)	−0.001(2)	−0.002(2)	0.000(2)
O(1)	0.2716(2)	0.5576(3)	0.8220(4)	0.083(2)	0.043(2)	0.060(2)	−0.009(2)	−0.028(2)	0.002(2)
O(2)	0.2780(2)	0.8017(3)	0.8084(4)	0.066(2)	0.042(2)	0.057(2)	−0.001(1)	−0.030(2)	0.008(2)
C(7)	0.1331(2)	0.8524(4)	0.5366(5)	0.044(2)	0.035(2)	0.032(2)	−0.001(2)	−0.008(2)	0.000(2)
O(3)	0.0934(1)	0.9033(4)	0.6547(3)	0.064(2)	0.066(2)	0.019(1)	0.007(1)	−0.001(1)	−0.002(1)
O(4)	0.1890(1)	0.7776(3)	0.5598(4)	0.062(2)	0.064(2)	0.041(2)	0.012(2)	−0.023(1)	−0.006(2)
C(8)	0.1126(2)	0.8838(4)	0.3491(4)	0.041(2)	0.039(2)	0.024(2)	0.003(2)	0.001(2)	0.004(2)
O(5)	0.0495(2)	0.9680(3)	0.3377(3)	0.076(2)	0.061(2)	0.032(1)	0.035(2)	−0.006(1)	−0.004(2)
C(9)	0.1036(2)	0.7390(4)	0.2477(4)	0.035(2)	0.040(2)	0.020(2)	0.003(2)	−0.001(2)	−0.002(2)
O(6)	0.0569(1)	0.6436(3)	0.3420(4)	0.057(2)	0.053(2)	0.034(1)	−0.013(1)	−0.013(1)	0.007(1)
C(10)	0.0776(2)	0.7692(4)	0.0630(4)	0.040(2)	0.041(2)	0.026(2)	0.006(2)	−0.005(2)	−0.006(2)
O(7)	0.1236(2)	0.8460(4)	−0.0253(3)	0.060(2)	0.087(2)	0.019(1)	−0.022(2)	−0.002(1)	0.010(2)
O(8)	0.0212(1)	0.7223(3)	0.0068(4)	0.048(1)	0.062(2)	0.031(1)	−0.009(1)	−0.013(1)	0.002(2)
H(11)	0.466(2)	0.578(4)	1.192(4)	0.032(9)					
H(12)	0.429(2)	0.434(4)	1.242(6)	0.06(1)					
H(21)	0.424(2)	0.624(4)	1.458(5)	0.04(1)					
H(22)	0.345(3)	0.567(6)	1.431(7)	0.09(2)					
H(31)	0.408(2)	0.813(6)	1.289(7)	0.08(1)					
H(32)	0.345(2)	0.817(5)	1.438(5)	0.05(1)					
H(41)	0.274(3)	0.725(6)	1.249(8)	0.07(2)					
H(42)	0.306(2)	0.877(5)	1.127(6)	0.05(1)					
H(50)	0.388(2)	0.720(5)	0.976(6)	0.05(1)					
H(1)	0.324(2)	0.488(5)	1.112(6)	0.06(1)					
H(1')	0.392(2)	0.487(6)	0.978(6)	0.10(1)					
H(2)	0.234(3)	0.794(6)	0.738(8)	0.11(2)					
H(5)	0.042(2)	1.002(6)	0.435(6)	0.06(1)					
H(6)	0.022(2)	0.614(6)	0.288(6)	0.07(1)					
H(8)	0.151(2)	0.941(4)	0.289(5)	0.033(9)					
H(9)	0.151(2)	0.696(4)	0.229(5)	0.013(8)					
H(10)	0.1095	0.8605	0.8798	0.08(1)					

Further details of the structure determination (e.g. structure factors) have been deposited within the relevant database and can be accessed as Collection No. 320355 or ordered from the Fachinformationszentrum Karlsruhe, D-7514 Eggenstein-Leopoldshafen.

References:

1. Bhattacharjee, S.K., Chacko, K.K.: DL-Homoproline Tetrahydrate. Acta Crystallogr. **B35** (1979) 396–398.