Crystal structure of (*S*)-2-amino-3-(1*H*-indol-3-yl)propanoic acidate — acetic acid — water (1:1:1), $C_{11}H_{12}N_2O_2 \cdot CH_3COOH \cdot H_2O$

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Abstract

$C_{13}H_{18}N_2O_5$, monoclinic, *C*121 (no. 5), $a = 11.277(1)$ Å, $b = 6.8760(8)$ Å, $c = 18.527(2)$ Å, $\beta = 102.680(2)^\circ$, $V = 1401.6$ Å³, $Z = 4$, $R_g(F) = 0.053$, $wR_{ref}(F^2) = 0.130$, $T = 298$ K.

Source of material

The solution of (*S*)-2-amino-3-(1*H*-indol-3-yl)propanoic acid (0.1 mol) in acetic acid (95 %, 30 ml) was stirred under reflux for 10 min. After cooling and filtration, filtrate was kept at room temperature for 10 days. Natural evaporation gave colourless single crystals of the title compound, suitable for X-ray analysis.

Discussion

Amino acids play an important role in methylation, detoxication and antioxidation. They are very useful in the pharmaceutical and food industries [1]. Tryptophan is the only one complicated amino acid with general-purpose physiological and biochemical functions [2].

The asymmetric unit of the title crystal structure contains one (*S*)-2-amino-3-(1*H*-indol-3-yl)propanoic acidate, one acetic acid and one water molecule. The geometry of molecule is close to the related compounds [Fe(L-trp)₂(HL-trp)₂]_n [2] and (*S*)-2-[(*S*)-2-acetamido-3-phenylpropanamido]-3-phenylpropanoic acid [3].

The indole ring is essentially plane. The crystal structure is stabilized by hydrogen bonds. The protonated N atoms interact with the (*S*)-2-amino-3-(1*H*-indol-3-yl)propanoic acidate anions, acetic acid and water through N—H...O hydrogen bonds. The water interacts with the (*S*)-2-amino-3-(1*H*-indol-3-yl)propanoic acidate, acetic acid and another water through C—H...O, O—H...O and N—H...O hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.05 × 0.08 × 0.35 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.03 cm ^{−1}
Diffraction, scan mode:	Bruker SMART CCD, ϕ/ω
$2\theta_{max}$:	50.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3543, 2357
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1505
$N(param)_{refined}$:	183
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1A)	4c	0.0839	0.3793	0.0625	0.054
H(1B)	4c	0.0038	0.2291	0.0798	0.054
H(1C)	4c	0.1175	0.2721	0.1316	0.054
H(2)	4c	0.4637	0.5666	0.2652	0.061
H(4A)	4c	0.3129	0.1065	0.2095	0.094
H(5A)	4c	0.8247	0.0048	0.0127	0.074
H(5B)	4c	0.9455	0.0099	0.0085	0.074
H(2A)	4c	−0.0252	0.4127	0.1805	0.040
H(3A)	4c	0.0920	0.7013	0.1142	0.048
H(3B)	4c	0.0266	0.7422	0.1789	0.048
H(4)	4c	0.3158	0.6027	0.1510	0.058
H(8)	4c	0.4608	0.5503	0.4144	0.078
H(9)	4c	0.3296	0.5502	0.4927	0.091
H(10)	4c	0.1237	0.5769	0.4473	0.086
H(11)	4c	0.0435	0.6054	0.3228	0.063
H(13A)	4c	0.1766	0.1331	0.3742	0.099
H(13B)	4c	0.3178	0.1424	0.3823	0.099
H(13C)	4c	0.2509	−0.0589	0.3726	0.099

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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
N(1)	4c	0.0558(3)	0.3229(4)	0.0987(2)	0.026(2)	0.042(2)	0.038(2)	−0.001(2)	0.004(1)	0.002(2)
N(2)	4c	0.3867(3)	0.5763(6)	0.2621(2)	0.030(2)	0.070(3)	0.055(2)	−0.010(2)	0.017(2)	−0.008(2)
O(1)	4c	−0.1431(3)	0.4935(5)	0.0185(1)	0.050(2)	0.092(2)	0.036(2)	0.025(2)	0.001(1)	−0.011(2)
O(2)	4c	−0.2010(3)	0.6159(6)	0.1138(2)	0.056(2)	0.115(3)	0.052(2)	0.052(2)	0.014(2)	0.003(2)
O(3)	4c	0.1274(2)	0.1035(4)	0.2353(1)	0.035(2)	0.062(2)	0.056(2)	0.005(2)	0.011(1)	0.003(2)
O(4)	4c	0.3282(2)	0.0954(5)	0.2547(1)	0.035(2)	0.102(2)	0.050(2)	0.016(2)	0.012(1)	−0.003(2)
O(5)	4c	0.8972(3)	0.0114(4)	0.0379(2)	0.047(2)	0.074(2)	0.061(2)	−0.008(2)	0.002(1)	−0.007(2)
C(1)	4c	−0.1274(3)	0.5272(5)	0.0849(2)	0.038(2)	0.037(3)	0.035(2)	0.002(2)	0.007(2)	−0.002(2)
C(2)	4c	−0.0077(3)	0.4714(5)	0.1358(2)	0.033(2)	0.037(2)	0.034(2)	−0.002(2)	0.016(2)	−0.002(2)
C(3)	4c	0.0726(4)	0.6456(5)	0.1584(2)	0.045(3)	0.037(3)	0.039(2)	−0.005(2)	0.010(2)	−0.002(2)
C(4)	4c	0.3001(4)	0.5973(7)	0.1982(2)	0.045(2)	0.061(3)	0.041(2)	−0.016(3)	0.015(2)	−0.008(2)
C(5)	4c	0.1887(3)	0.6091(6)	0.2131(2)	0.038(2)	0.029(2)	0.037(2)	−0.007(2)	0.006(2)	−0.002(2)
C(6)	4c	0.2049(3)	0.5912(6)	0.2914(2)	0.033(2)	0.044(2)	0.045(2)	−0.002(2)	0.008(2)	−0.007(2)
C(7)	4c	0.3306(3)	0.5731(6)	0.3203(2)	0.034(2)	0.057(3)	0.046(2)	−0.005(2)	0.006(2)	−0.004(2)
C(8)	4c	0.3775(4)	0.5597(8)	0.3958(2)	0.042(3)	0.095(4)	0.051(3)	0.004(3)	−0.004(2)	−0.006(3)
C(9)	4c	0.2994(5)	0.5607(9)	0.4419(2)	0.067(3)	0.122(5)	0.036(2)	0.000(4)	0.007(2)	−0.004(3)
C(10)	4c	0.1754(5)	0.5770(9)	0.4145(2)	0.069(3)	0.105(4)	0.049(3)	0.002(3)	0.028(2)	−0.007(3)
C(11)	4c	0.1270(4)	0.5932(7)	0.3403(2)	0.045(2)	0.067(3)	0.048(2)	−0.001(3)	0.017(2)	−0.005(3)
C(12)	4c	0.2273(4)	0.0929(7)	0.2777(2)	0.052(3)	0.041(2)	0.049(2)	0.008(3)	0.016(2)	−0.001(2)
C(13)	4c	0.2447(4)	0.0759(8)	0.3588(2)	0.076(3)	0.075(3)	0.045(2)	0.002(3)	0.009(2)	0.006(3)

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