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L-Kynureninium sulfate dihydrate

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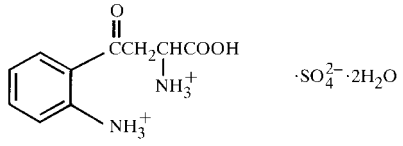
In the structure of the title compound, 2-(3-ammonio-3-carboxypropanoyl)-1-anilinium sulfate dihydrate, $C_{10}H_{14}N_2O_3^{2+}\cdot SO_4^{2-}\cdot 2H_2O$, the two amino groups are protonated. The molecule has a *trans* planar zigzag carbon-skeletal conformation elongated nearly in the benzene ring plane. The two amino and the carboxyl groups are located on the same side of the molecule. The crystal structure is stabilized by intermolecular hydrogen bonds involving the water molecules and the sulfate ion.

Comment

L-Kynurenine is a central metabolite of L-tryptophan derived from *N*-formylkynurenine catalyzed by kynurenine formylase. It is further metabolized through various metabolic pathways, such as 3-hydroxykynurenine anthranilic acid, anthranilic acid and kynurenic acid catalyzed by kynurenidase, kynurenine-3-hydroxylase and kynurenine transaminase, respectively (Mahler & Cordes, 1971). Metabolism of kynurenine in the rat brain (Guidetti *et al.*, 1995) or metabolism of L-tryptophan to kynurate and quinolinate in the central system (Naritsin *et al.*, 1995) have been reported. For the structural elucidation of the kynurenine metabolic pathway, accurate structural and conformational information for kynurenine is needed in order



(I)

to consider recognition of kynurenine by different enzymes. Accordingly, as the fundamental study, we have undertaken the structural analysis of the title compound, (I).

The molecule has a *trans* planar zigzag carbon-skeletal conformation, as indicated by the torsion angles [C1—C7—C8—C9 179.6 (5)° and C7—C8—C9—C10 168.4 (5)°]. The benzene ring and the zigzag carbon chain are nearly coplanar [C6—C1—C7—C8 20.8 (9)°]. The amino N1 and N2 and the carbonyl O1 atom are located on the same side of the molecule and weak intramolecular electrostatic interactions are formed between the two protonated amino groups and the electrically negative carbonyl O atom, as reflected by the intramolecular hydrogen bonds [N1···O1 2.684 (6), N2···O1 2.989 (6) and N2···O3 2.657 (6) Å]. This characteristic conformation of kynurenine may be predominant as the substrate for the related enzymes of kynurenine metabolism. In the crystal structure, the molecules are held together by intermolecular hydrogen bonds through sulfate ions and water molecules, but no significant ring stacking is observed.

Experimental

A very thin light-yellow needle crystal of the title compound was obtained by slow evaporation from a 50:50 ethanol–water solution of L-kynurenine in the presence of an equivalent molar amount of H_2SO_4 at room temperature.

Crystal data

$C_{10}H_{14}N_2O_3^{2+}\cdot SO_4^{2-}\cdot 2H_2O$
M_r = 342.32
Orthorhombic, $P2_12_12_1$
a = 11.353 (5) Å
b = 22.134 (5) Å
c = 5.897 (7) Å
V = 1481 (1) Å^3
Z = 4
D_x = 1.534 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10.3–14.5°
μ = 0.267 mm^{-1}
T = 296 K
Needle, light yellow
0.50 × 0.10 × 0.05 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.972, T_{max} = 0.998
4128 measured reflections
2072 independent reflections
1088 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.058
θ_{max} = 27.5°
$h = -14 \rightarrow 14$
$k = -28 \rightarrow 28$
$l = -7 \rightarrow 7$
3 standard reflections
every 150 reflections
intensity decay: 0.12%

Refinement

Refinement on F^2
$R(F)$ = 0.0565
$wR(F^2)$ = 0.1629
S = 1.153
2071 reflections
200 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + \{0.06100[\mathrm{Max}(F_o^2,0) + 2F_c^2]/3\}^2]$
$(\Delta/\sigma)_{max}$ = 0.0044
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S—O4	1.461 (4)	C1—C6	1.397 (9)
S—O5	1.455 (5)	C1—C7	1.500 (9)
S—O6	1.486 (5)	C2—C3	1.357 (9)
S—O7	1.447 (5)	C3—C4	1.405 (10)
O1—C7	1.212 (8)	C4—C5	1.36 (1)
O2—C10	1.309 (8)	C5—C6	1.374 (9)
O3—C10	1.189 (8)	C7—C8	1.503 (9)
N1—C2	1.480 (8)	C8—C9	1.527 (9)
N2—C9	1.458 (8)	C9—C10	1.537 (9)
C1—C2	1.398 (8)		
O4—S—O5	109.8 (3)	C1—C2—C3	122.0 (6)
O4—S—O6	109.3 (3)	C2—C3—C4	119.5 (6)
O4—S—O7	113.1 (3)	C3—C4—C5	119.9 (6)
O5—S—O6	108.3 (4)	C4—C5—C6	120.1 (6)
O5—S—O7	109.4 (3)	C1—C6—C5	121.7 (7)
O6—S—O7	106.8 (3)	O1—C7—C1	121.3 (5)
C2—C1—C6	116.8 (6)	O1—C7—C8	121.1 (6)
C2—C1—C7	122.3 (6)	C1—C7—C8	117.6 (5)
C6—C1—C7	120.9 (6)	C7—C8—C9	113.1 (5)
N1—C2—C1	119.8 (6)	N2—C9—C8	112.6 (5)
N1—C2—C3	118.2 (5)	N2—C9—C10	108.8 (5)

C8—C9—C10	111.9 (5)	O2—C10—C9	112.2 (6)
O2—C10—O3	126.1 (6)	O3—C10—C9	121.7 (6)
O1—C7—C1—C2	21.2 (10)	C1—C6—C5—C4	2 (1)
O1—C7—C1—C6	−159.6 (7)	C1—C7—C8—C9	−179.6 (5)
O1—C7—C8—C9	0.7 (9)	C2—C1—C6—C5	−1 (1)
O2—C10—C9—N2	178.6 (5)	C2—C1—C7—C8	−158.4 (6)
O2—C10—C9—C8	−56.5 (7)	C2—C3—C4—C5	−0 (1)
O3—C10—C9—N2	0.4 (8)	C3—C2—C1—C6	−1.1 (9)
O3—C10—C9—C8	125.3 (7)	C3—C2—C1—C7	178.1 (6)
N1—C2—C1—C6	178.6 (5)	C3—C4—C5—C6	−1 (1)
N1—C2—C1—C7	−2.2 (9)	C5—C6—C1—C7	179.7 (6)
N1—C2—C3—C4	−177.9 (6)	C6—C1—C7—C8	20.8 (9)
N2—C9—C8—C7	−68.7 (7)	C7—C8—C9—C10	168.4 (5)
C1—C2—C3—C4	1 (1)		

All H atoms were located from difference Fourier maps and were then fixed at the generated ideal positions, except for the water H atoms. The absolute structure was not determined [Flack parameter 0.9 (3)] by our X-ray analysis, but can be inferred from the known absolute configuration of L-kynurenine sulfate used in the synthesis which was confirmed by measuring the optical rotation.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999*a*); cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999*a*); data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1999*b*); program(s) used to solve structure: *SIR*88 (Burla *et al.*, 1989) and *DIRDIF* (Beurskens *et al.*, 1994); program(s) used to refine structure: *TEXSAN LS*; software used to prepare material for publication: *TEXSAN*.

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