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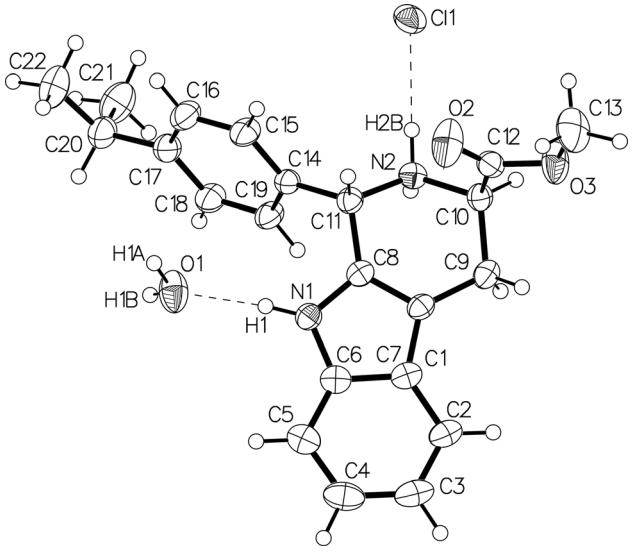
The crystal structure of (1S,3*R*)-1-(4-isopropylphenyl)-3-(methoxycarbonyl)-2,3,4,9-tetrahydro-1H-pyrido[3,4-b]indol-2-iumchloride monohydrate, $C_{22}H_{27}ClN_2O_3$



https://doi.org/10.1515/ncrs-2022-0069
Received February 11, 2022; accepted April 25, 2022; published online May 5, 2022

Abstract

$C_{22}H_{27}ClN_2O_3$, orthorhombic, $P2_12_12_1$ (no. 19), a = 8.6797(3) Å, b = 9.8809(3) Å, c = 24.7824(8) Å, V = 2125.42(12) Å^3, Z = 4, $R_{gt}(F)$ = 0.0478, $wR_{ref}(F^2)$ = 0.1082, T = 170 K.

CCDC no.: 2168514

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.19 × 0.12 × 0.08 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.20 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	24639, 4345, 0.074
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3314
$N(param)_{refined}$:	259
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

The reaction substrate D-Tryptophan methyl ester hydrochloride (10 mmol) was dissolved evenly in methanol (40 mL) with stirring, then 4-isopropylbenzaldehyde (20 mmol) was added and refluxed for 12 h. After the reaction was completed (monitored by TLC), the mixture was concentrated under vacuum and then purified by silica gel column chromatography with dichloromethane and ethyl acetate (v/v = 3/1) as the eluent. The product was light-yellow solid with a yield of 52%. The single crystal configuration of the title compound was obtained by recrystallization using the solvent volatilization method in four days.

Experimental details

All hydrogen atoms were added in their geometrically idealized positions and refined by the riding models on their parent atoms with U_{iso} = 1.2 U_{eq}. The anomalous scattering determined the absolute configuration of the title compound (Flack parameter 0.05(5)).

Comment

Tryptophan, also known as β-Indolyl alanine, whose molecular configuration is similar to 3-Indoleacetic acid,

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5727 (4)	0.4381 (4)	0.40848 (14)	0.0297 (8)
C2	0.5681 (5)	0.3964 (4)	0.46242 (15)	0.0372 (10)
H2	0.657926	0.400264	0.484247	0.045*
C3	0.4308 (6)	0.3496 (4)	0.48334 (17)	0.0438 (11)
H3	0.426803	0.320925	0.519909	0.053*
C4	0.2971 (5)	0.3434 (4)	0.45167 (18)	0.0451 (11)
H4	0.204387	0.310461	0.467203	0.054*
C5	0.2974 (5)	0.3844 (4)	0.39828 (16)	0.0371 (10)
H5	0.206826	0.380409	0.376834	0.045*
C6	0.4360 (4)	0.4315 (4)	0.37734 (15)	0.0302 (9)
C7	0.6904 (4)	0.4907 (4)	0.37379 (14)	0.0282 (8)
C8	0.6232 (4)	0.5120 (3)	0.32466 (14)	0.0264 (8)
C9	0.8587 (4)	0.5133 (4)	0.38437 (14)	0.0324 (9)
H9A	0.911517	0.424797	0.387332	0.039*
H9B	0.871459	0.561587	0.419079	0.039*
C10	0.9328 (4)	0.5963 (4)	0.33900 (14)	0.0284 (8)
H10	1.045464	0.575213	0.339044	0.034*
C11	0.6978 (4)	0.5716 (4)	0.27634 (14)	0.0280 (8)
H11	0.673360	0.670473	0.275650	0.034*
C12	0.9162 (4)	0.7469 (4)	0.34691 (14)	0.0311 (8)
C13	1.0128 (6)	0.9353 (4)	0.3941 (2)	0.0607 (14)
H13A	1.063084	0.982298	0.364050	0.091*
H13B	1.068773	0.954057	0.427605	0.091*
H13C	0.906330	0.967336	0.397521	0.091*
C14	0.6512 (4)	0.5119 (4)	0.22257 (14)	0.0273 (8)
C15	0.6069 (5)	0.5975 (4)	0.18144 (15)	0.0364 (10)
H15	0.609918	0.692631	0.186896	0.044*
C16	0.5576 (5)	0.5461 (4)	0.13199 (15)	0.0418 (11)
H16	0.527547	0.606749	0.104170	0.050*
C17	0.5520 (5)	0.4078 (4)	0.12279 (15)	0.0338 (9)
C18	0.5978 (5)	0.3229 (4)	0.16453 (16)	0.0365 (9)
H18	0.596482	0.227767	0.159037	0.044*
C19	0.6454 (4)	0.3734 (4)	0.21387 (15)	0.0336 (9)
H19	0.674287	0.312882	0.241891	0.040*
C20	0.4974 (5)	0.3467 (4)	0.06993 (15)	0.0428 (10)
H20	0.416691	0.278213	0.079102	0.051*
C21	0.6281 (6)	0.2705 (5)	0.04117 (17)	0.0572 (13)
H21A	0.706663	0.335258	0.029290	0.086*
H21B	0.586573	0.222530	0.009774	0.086*
H21C	0.674537	0.205224	0.066105	0.086*
C22	0.4245 (6)	0.4466 (5)	0.03189 (17)	0.0596 (14)
H22A	0.337711	0.491159	0.049881	0.089*
H22B	0.387448	0.399018	−0.000288	0.089*
H22C	0.500894	0.514624	0.021315	0.089*
N1	0.4704 (3)	0.4756 (3)	0.32606 (11)	0.0283 (7)
H1	0.405566	0.479786	0.298803	0.034*
N2	0.8704 (3)	0.5563 (3)	0.28468 (11)	0.0286 (7)
H2A	0.895701	0.468152	0.278688	0.034*
H2B	0.919335	0.606632	0.259151	0.034*
O2	0.8275 (4)	0.8186 (3)	0.32400 (13)	0.0615 (10)
O3	1.0129 (4)	0.7903 (3)	0.38385 (12)	0.0521 (8)
Cl1	1.02330 (11)	0.75272 (11)	0.20249 (4)	0.0437 (3)
O1	0.2293 (4)	0.5255 (3)	0.25344 (12)	0.0512 (8)
H1A	0.198073	0.597013	0.236153	0.077*
H1B	0.223748	0.455323	0.232290	0.077*

is a significant neurotransmitter in the human body as the precursor of serotonin [5]. It is also an essential precursor of auxin biosynthesis in plants [6]. It can be used as a nutritional supplement for pregnant women and young children. As a tranquilizer, it can adjust the mental rhythm and promote sleep. In addition, tryptophan scaffolds are widely found in bioactive natural products, including halogenated and non-halogenated indoles. Modification of these indoles may lead to novel pharmaceutically active compounds [7]. Some derivatives are commonly used to design antitumor and anti-inflammatory compounds [8, 9]. This paper reports a novel crystal structure of a tryptophan derivative, which formed a 2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indole skeleton through the ring-closing reaction. It has significant potential application for related drugs and drug intermediates, and similar structures have been reported abundantly [10–19].

The molecule structure of the title compound is shown in the figure consisting of the protonated target molecule, a couter chlorid anion and a water molecule. The amino group on indole participates in the closed-loop to form a hexatomic ring, similar to the reported systems [10–12], whose one hydrogen atom is replaced by a cumene group, and the length of the C11–C14 bond is 1.512(6) Å. The isopropyl-phenyl ring plane on the cumene group has obvious torsion with the indole plane, and the dihedral angle is 73.17°. There are two hydrogen bonds formed between a bonded water molecule and the organic molecules in the title crystal. The hydrogen bond N1–H1···O1 is between the amino group on indole and the water molecule with bond length 1.952(4) Å and bond angle 162.6(3)°. The other hydrogen bond O1–H1B···O2 is between the water molecule and the ester group with bond length 1.992(4) Å and bond angle 166.9(3)°. In addition, the chloride anion Cl1 stabilizes the structure with two hydrogen bonds O1–H1A···Cl1 with bond length 2.3161(11) Å and bond angle 157.1(3)° and N2–H2B···Cl1 with bond length 2.2067(11) Å and bond angle 172.22(19)°. The results illustrate that the forces of these hydrogen bonds are the main factor in stabilizing the crystal structure.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was financially supported by the Key Scientific Research Projects of Colleges and Universities in Henan Province (22A430032).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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