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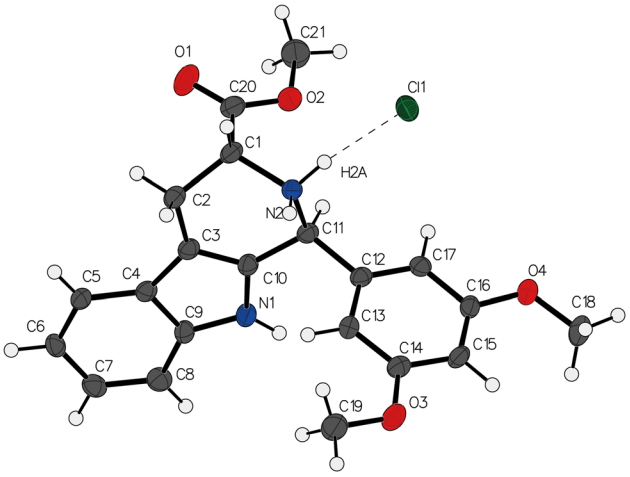
Crystal structure of (3*R*)-1-(3,5-dimethoxyphenyl)-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indole-3-carboxylate chloride, $C_{21}H_{23}ClN_2O_4$



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Abstract

$C_{21}H_{23}ClN_2O_4$, monoclinic, $P2_1$ (no. 4), a = 10.9270(4) Å, b = 8.0274(3) Å, c = 12.9691(6) Å, β = 114.914(1)°, V = 1031.73(7) Å^3, Z = 2, $R_{gt}(F)$ = 0.0362 $wR_{ref}(F^2)$ = 0.0898, T = 150 K.

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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:	0.16 × 0.08 × 0.05 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.21 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, 97 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6958, 3362, 0.048
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3137
$N(param)_{refined}$:	256
Programs:	Bruker,[1] SHELX,[2,3] Olex2[4]

1 Source of materials

To a solution of D-tryptophan methyl ester hydrochloride (2.55 g, 10 mmol) in methanol (30 mL) was added 3,5-dimethoxybenzaldehyde (1.99, 12 mmol). The mixture was refluxed for 3 h, until the TLC indicated the reaction was completed. The solvent was evaporated to dryness, yielding the crude product that was then purified. For crystal growth, the crude product was dissolved in a minimal amount of hot ethanol and slowly cooled to room temperature.

2 Experimental details

The crystal structure was determined using the Shelxt program[2] and subsequently underwent refinement via the Shelxl tools[3] available in the Olex2 suite.[4] Hydrogen atoms were theoretically positioned and subjected to refinement under the riding model, assigning U_{iso}(H) values at 1.5 times U_{eq}(C).

3 Comment

Tryptophan is an indispensable amino acid that is necessary for protein biosynthesis and myriad essential metabolic pathways across all forms of life. However, animals are deficient in the enzymatic apparatus required to synthesize tryptophan *de novo* from simpler precursors. Derivatives of tryptophan boast significant added value and find widespread utilization in the chemical, food, polymer, and

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2789(3)	0.5827(4)	0.5535(2)	0.0241(7)
H1A	0.248658	0.657525	0.485624	0.029*
C2	0.2593(3)	0.6790(4)	0.6474(2)	0.0236(6)
H2C	0.289682	0.795659	0.649459	0.028*
H2D	0.162581	0.680136	0.632172	0.028*
C3	0.3403(3)	0.5956(4)	0.7594(2)	0.0222(6)
C4	0.3277(3)	0.6080(4)	0.8653(2)	0.0213(6)
C5	0.2408(3)	0.6907(4)	0.9016(2)	0.0234(6)
H5	0.170640	0.759438	0.850614	0.028*
C6	0.2578(3)	0.6713(4)	1.0131(2)	0.0257(7)
H6	0.197775	0.725707	1.037896	0.031*
C7	0.3629(3)	0.5722(4)	1.0901(3)	0.0274(7)
H7	0.374068	0.563356	1.166573	0.033*
C8	0.4501(3)	0.4873(4)	1.0563(2)	0.0277(7)
H8	0.520183	0.418967	1.107822	0.033*
C9	0.4311(3)	0.5061(4)	0.9436(2)	0.0231(6)
C10	0.4453(3)	0.4922(4)	0.7768(2)	0.0217(6)
C11	0.4944(3)	0.4365(4)	0.6911(2)	0.0211(6)
H11	0.463215	0.319433	0.668732	0.025*
C12	0.6467(3)	0.4413(4)	0.7306(2)	0.0210(6)
C13	0.7189(3)	0.5811(4)	0.7880(2)	0.0252(7)
H13	0.674390	0.673344	0.802915	0.030*
C14	0.8583(3)	0.5819(4)	0.8230(3)	0.0278(7)
C15	0.9240(3)	0.4458(4)	0.8029(2)	0.0263(7)
H15	1.019339	0.446806	0.828887	0.032*
C16	0.8506(3)	0.3099(4)	0.7455(2)	0.0230(6)
C17	0.7102(3)	0.3068(4)	0.7090(2)	0.0219(6)
H17	0.659399	0.212466	0.669707	0.026*
C18	1.0470(3)	0.1717(6)	0.7508(3)	0.0487(10)
H18A	1.070735	0.265287	0.714214	0.073*
H18B	1.072945	0.066855	0.726760	0.073*
H18C	1.094749	0.182693	0.833535	0.073*
C19	0.8783(4)	0.8579(5)	0.8947(3)	0.0424(10)
H19A	0.815093	0.901477	0.820832	0.064*
H19B	0.947421	0.941951	0.933937	0.064*
H19C	0.829247	0.831380	0.940865	0.064*
C20	0.1914(3)	0.4273(5)	0.5176(2)	0.0289(7)
C21	0.1773(4)	0.1457(5)	0.4620(3)	0.0448(10)
H21A	0.101021	0.166962	0.388633	0.067*
H21B	0.143385	0.109778	0.517361	0.067*
H21C	0.234517	0.058185	0.452972	0.067*
Cl1	0.49020(7)	0.42042(10)	0.39131(6)	0.0294(2)
N1	0.5015(2)	0.4372(4)	0.88807(18)	0.0259(6)
H1	0.570750	0.369193	0.918262	0.031*
N2	0.4268(2)	0.5466(3)	0.58797(19)	0.0215(5)
H2A	0.435643	0.497670	0.528164	0.026*
H2B	0.471913	0.645314	0.601646	0.026*
O1	0.0754(2)	0.4280(4)	0.5026(2)	0.0466(6)
O2	0.2555(2)	0.2969(3)	0.50185(19)	0.0327(6)
O3	0.9408(2)	0.7115(3)	0.8783(2)	0.0410(7)
O4	0.9050(2)	0.1721(3)	0.71845(18)	0.0320(5)

pharmaceutical sectors, where they play a critical role in the management of diseases and the enhancement of quality of life.[5,6] Owing to their pronounced bioactivity, the crystallographic elucidation of these compounds has been extensively pursued.[7–9]

As depicted in the graphical representation, the indole moiety's carbon and nitrogen atoms verge on coplanarity, whereas the carbon and nitrogen atoms of the piperidine moiety manifest a dihedral angle of 42.2(4)°, delineated by the atoms C10–C11–N2–C1. This specific angle is complemented by the spatial arrangement of the indole ring with respect to the benzene ring, which is positioned at an angle of 76°, consistent with structures documented in previous literature.[10–15] The chloride ion interacts with the hydrogen ion and the nitrogen atom situated on the piperidine ring, wherein the bond angle between N2, H2A, and Cl1 is reported as 167.75(15) degrees. Additionally, the bond distance for H2A–Cl1 is measured at 2.1867(9) Å.

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