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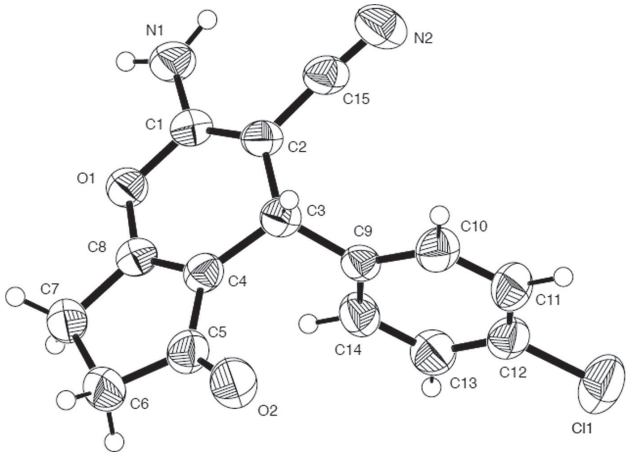
Crystal structure of 2-amino-5-oxo-4-(4-chlorophenyl)-4,5,6,7-tetrahydro-cyclopenta[*b*]pyran-3-carbonitrile, $C_{15}H_{11}ClN_2O_2$



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Abstract

$C_{15}H_{11}ClN_2O_2$, monoclinic, $C2/c$ (no. 15), $a = 12.955(7)$ Å, $b = 10.425(7)$ Å, $c = 20.149(13)$ Å, $\beta = 102.415(14)°$, $V = 2658(3)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0361$, $wR_{ref}(F^2) = 0.0917$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized according to a reported procedure [6]. A mixture of 1,3-cyclopentadione (10 mmol),

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.26 × 0.24 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.29 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6549, 2328, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1813
$N(param)_{refined}$:	182
Programs:	Olex2 [7], SHELX [8], Bruker [9]

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

Atom	x	y	z	U_{iso}*/U_{eq}
N1	0.40831(13)	−0.08472(17)	0.49442(10)	0.0652(5)
N2	0.38399(11)	0.25049(15)	0.43688(8)	0.0540(4)
H2A	0.438827	0.206820	0.455673	0.065*
H2B	0.390766	0.328919	0.425288	0.065*
C1	0.14267(14)	−0.29775(18)	0.28576(10)	0.0498(5)
C2	0.15125(15)	−0.32151(18)	0.35386(11)	0.0564(5)
H2	0.156579	−0.405151	0.370297	0.068*
C3	0.15183(15)	−0.21899(17)	0.39753(10)	0.0502(5)
H3	0.157048	−0.234528	0.443588	0.060*
C4	0.14482(12)	−0.09389(15)	0.37416(9)	0.0383(4)
C5	0.13709(14)	−0.07347(17)	0.30547(9)	0.0484(5)
H5	0.132841	0.010033	0.288852	0.058*
C6	0.13556(15)	−0.17488(19)	0.26097(10)	0.0537(5)
H6	0.129775	−0.159865	0.214818	0.064*
C7	0.15226(12)	0.01796(16)	0.42388(9)	0.0404(4)
H7	0.134466	−0.013128	0.465938	0.049*
C8	0.26338(13)	0.07436(16)	0.44074(8)	0.0405(4)
C9	0.34554(14)	−0.01045(18)	0.47073(9)	0.0470(5)
C10	0.28815(13)	0.19641(17)	0.42569(8)	0.0415(4)
C11	0.10983(13)	0.24216(16)	0.38367(9)	0.0414(4)
C12	0.07892(13)	0.12499(16)	0.39616(9)	0.0412(4)
C13	−0.03618(14)	0.12100(18)	0.37821(10)	0.0498(5)
C14	−0.07477(15)	0.24969(18)	0.34637(12)	0.0595(5)
H14A	−0.125975	0.287439	0.369336	0.071*
H14B	−0.107384	0.239285	0.298632	0.071*
C15	0.02440(14)	0.33452(18)	0.35508(10)	0.0520(5)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H15A	0.033161	0.368696	0.311853	0.062*
H15B	0.021876	0.404852	0.386213	0.062*
Cl1	0.14294(5)	−0.42584(5)	0.23024(3)	0.0780(2)
O1	−0.09200(10)	0.03086(13)	0.38695(9)	0.0701(5)
O2	0.21221(9)	0.28365(11)	0.39566(6)	0.0467(3)

4-chlorobenzaldehyde (10 mmol), malononitrile (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) in ethanol (100 mL) was refluxed for 2−3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2 times U_{eq}(C) and 1.2 times U_{eq}(N).

Comment

Natural products have impact on chemical biology and drug discovery, and the structural diversity of natural products has provided medicinal chemists an important source of inspiration in their search for molecules with pharmacological activity [1–3]. Pyranes cover a wide range of biological properties, including anti-oxidant, anti-inflammatory and anti-microbial as well as anticancer activities [4].

In the crystal structure of the title compound (Figure), the pyran ring and the adjacent cyclopentenone moiety both are basically planar, and roughly coplanar. The bond distances and the bond angles in the title compound are comparable with those in the reported compound $C_{15}H_{10}Cl_2N_2O_2$ [5]. The structural features of the title compound are very similar to those in $C_{15}H_{10}Cl_2N_2O_2$. In published compound, two Cl atoms at the phenyl moiety are present. While one Cl atom was found in the title compound.

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