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Crystal structure of 5-nitro-2-(pyrrolidin-1-yl)benzaldehyde, C_{11}H_{12}N_{2}O_{3}

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Abstract
C_{11}H_{12}N_{2}O_{3}, monoclinic, P2_1/n (no. 14), a = 8.1726(2) Å, b = 7.1419(2) Å, c = 18.4875(6) Å, β = 100.376(1), V = 1061.43(4) Å³, Z = 4, R_{gt}(F) = 0.0533, wR_{ref}(F^2) = 0.1212, T = 298 K.

CCDC no.: 1536055

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
Pyrrolidine (5.46 g, 66 mmol) and sodium hydrogen carbonate (5.54 g, 66 mmol) were added to 2-chloro-5-nitrobenzaldehyde (8 g, 42 mmol) dissolved in ethanol. The mixture was heated to reflux during 24 h under inert conditions. After cooling to ambient temperature, the mixture was poured into 150 ml of dichloromethane, then washed twice with 50 ml of water. The organic layer was dried on magnesium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel using DCM/hexane (80/2). Yellow crystals (7 g) of the title compound were obtained with 72.31% yield.

Table 1: Data collection and handling.

<table>
<thead>
<tr>
<th>Crystal: Yellow prism</th>
<th>Size: 0.25 × 0.20 × 0.10 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength: Mo Kα radiation (0.71073 Å)</td>
<td>µ: 1.0 cm⁻¹</td>
</tr>
<tr>
<td>Diffractometer, scan mode: Nonius KappaCCD, ω/2θ-scans</td>
<td>2θ_{max}, completeness: 50°, &gt;92%</td>
</tr>
<tr>
<td>N(hkl) measured, N(hkl) unique, R_{int}:</td>
<td>1729, 1729, 0.045</td>
</tr>
<tr>
<td>Criterion for I_{obs}, N(hkl)_{gt}:</td>
<td>I_{obs} &gt; 2 σ(I_{obs}), 1475</td>
</tr>
<tr>
<td>N(param) refined:</td>
<td>149</td>
</tr>
<tr>
<td>Programs:</td>
<td>SIR2004 [1], PLATON [2], SHELX [3]</td>
</tr>
</tbody>
</table>

Experimental details
H atoms were placed at chemically acceptable positions using instructions AFIX 43 for the aromatic group and AFIX 23 for methylene groups. The hydrogen atom attached to C7 was located from difference fourier map and refined isotropically. All C atoms involved in the pyrrolidine ring are disordered over two positions with different occupancy factors (0.58 and 0.42), their temperature factors were restrained using the DELU instruction of SHELX-2014/7 [3].

Discussion
A variety of tricyclic indolinic derivatives have been discovered and approved to be useful in the treatment of many diseases [4–6]. But the biological activity shown by a set of analogue compounds depends on position and nature of substituents on a common scaffold [7]. In intending to synthesize new tricyclic indolinic derivatives, especially those with a nitro-aryl function, the title compound has been synthesized.
carbon atoms C91 and C92 being out of the plane formed by the four remaining atoms. The puckering parameters are: q2 = 0.424(14)° and φ = 254.1(19)° for N2/C81–C111 ring, and q2 = 0.407(16)° and φ = 71(2)° for N2/C82–C112. The crystal packing is supported by weak C··· H... O hydrogen bonds and π–π interactions.

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References

10. Abdulrahman, I. A.; Raju, S. K.; Natarajan, A.; Ramachandran, A. N.; Janakiraman, S.: Crystal structure of 8-[(2-methylphenyl)-11-[(E)-(2-methylphenyl)-methylene]-14-hydroxy-3,13-diazaheteptacyclo-[13.7.1.9,3.0.3.0.5.8.0.24.6.3.0.16.8.3.0.23.5.7.3.4.9.12]-tetracosa-1(22),15,17,19(23),20-pentaeno-10- one methanol monosolvate, C19H14N2O2·CH3OH, C19H13N2O2·Z. Kristallogr. – NCS 229 (2014) 175–177.