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Crystal structure of (Z)-4-((E)-(4-chlorobenzylidene)hydrazono)-1-p-tolylpyrrolidine-3-carbonitrile, C_{19}H_{17}ClN_{4}

Abstract

C_{19}H_{17}ClN_{4}, triclinic, P\bar{1} (no. 2), a = 6.9042(5) Å, b = 7.1990(5) Å, c = 18.2633(13) Å, \(\alpha = 86.727(6)\) °, \(\beta = 79.214(6)\) °, \(\gamma = 69.876(7)\) °, V = 837.25(11) Å^3, Z = 2, \(R_{gt}(F) = 0.0553, wR_{ref}(F^2) = 0.1406, T = 296(2)\) K.

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

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Table 1: Data collection and handling.

| Crystal: | Colourless plate |
| Wavelength: | Mo K\alpha radiation (0.71073 Å) |
| \(\mu\): | 2.4 cm\(^{-1}\) |
| Diffractometer, scan mode: | SuperNova, \(\omega\)-scans |
| 2\(\theta_{\text{max}}\), completeness: | 59.6°, >83% |
| \(N(hkl)_{\text{measured}}, N(hkl)_{\text{unique,}}\ R_{\text{gt}}:\ | 8852, 3988, 0.028 |
| Criterion for \(I_{\text{obs}}\), \(I_{\text{obs}} > 2\sigma(I_{\text{obs}}), 2319\) |
| \(N(\text{param})_{\text{refined}}: | 218 |
| Programs: | CrysAlis \text{PRO} [12], SHELX [13], WinGX [14] |

Source of material

(Z)-4-((E)-(4-chlorobenzylidene)hydrazono)-1-p-tolylpyrrolidine-3-carbonitrile was synthesized from reaction of equimolar quantities of 4-hydrazono-1-p-tolylpyrrolidine-3-carbonitrile and 4-chlorobenzaldehyde in ethanol in the presence of few drops of glacial acetic acid under reflux for 1 h. The solid produced was filtered, dried and recrystallized from dimethylformamide to give colourless crystals of the title compound (Mp 210–211 °C) [1].

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. Methylene C—H bonds were fixed at 0.97 Å and methyl C—H bonds at 0.96 Å with 1.5 \(U_{eq}\) (C). Methyl groups were allowed to spin about the C—C bond. Aromatic C—H distances were set to 0.93 Å and N—H set to 0.86 Å with \(U_{iso}\) set to 1.2 \(U_{eq}\) (N/C).

Discussion

The most efficient syntheses of pyrrolidines involve reactions of primary amines with diols in the presence of a metal complex catalyst [2, 3], of primary amines with dihaloalkanes in the presence of potassium carbonate under microwave conditions [4], of cyclization of amino alcohols in the presence of thionyl chloride [5] and of N-tosylhydrazones with vinyl iodides in the presence of a Pd-catalyst [6]. They can be
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References


