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The crystal structure of \(\text{N-(7-(4-fluorobenzylidene)-3-(4-fluorophenyl)-3,3a,4,5,6,7-hexahydro-2H-indazole-2-carbonothioyl)benzamide, C}_{28}\text{H}_{23}\text{F}_{2}\text{N}_{3}\text{OS} \)

Abstract

\(\text{C}_{28}\text{H}_{23}\text{F}_{2}\text{N}_{3}\text{OS}, \) monoclinic, \(I2/a\) (no. 15), \(a = 20.3481(8)\ \text{Å}, \ b = 10.2647(4)\ \text{Å}, \ c = 23.6975(11)\ \text{Å}, \ \beta = 105.317(5)\)°, \(V = 4773.8(4)\ \text{Å}^3, \ Z = 8, \ R_{int}(F) = 0.0489, \ wR_{ref}(F^2) = 0.1543, \ T = 296(2) \text{ K}. \)

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The crystal 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.

Table 1: Data collection and handling.

| Crystal: Colourless block |
| Size: \(0.27 \times 0.26 \times 0.12\ \text{mm} \) |
| Wavelength: \(\text{Mo Kα radiation (0.71073 Å)} \) |
| \(\mu\): \(0.18\ \text{mm}^{-1}\) |
| Diffractometer, scan mode: \(\text{SuperNova, ω-scans} \) |
| \(\theta_{\text{max}}, \) completeness: \(29.7°, >93\% \) (up to 25.2, >99%) |
| \(N(hkl)_\text{measured}, N(hkl)_\text{unique}, R_{\text{int}}: \) 39559, 6270, 0.032 |
| Criterion for \(I_{\text{abs}}, N(hkl)_\text{gt}: \) \(I_{\text{obs}} > 2 \sigma(I_{\text{obs}}), 3722 \) |
| \(N(\text{param})_{\text{refined}}: \) 264 |
| Programs: \(\text{CrystAlis PRO [1], SHELX [2, 3], WinGX and ORTEP [4]} \) |

Source of materials

The title compound was synthesized from reaction of an equimolar mixture of \(7-(4\text{-fluorobenzylidene})-3-(4\text{-fluorophenyl})-3,3a,4,5,6,7\text{-hexahydro-2H-indazole} \) and benzoyl isothiocyanate in anhydrous ethanol under reflux for \(2\) h. The crude product was recrystallized from dimethylformamide to give colourless crystals in \(82\%\) yield (Mp. \(246–248\ °\text{C})\).

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. The N—H bond was fixed at 0.86 Å (AFIX 43 instruction in SHELXL [2, 3]), with displacement parameters set to 1.2 times \(U_{eq}(N)\). C—H distances for \(\text{sp}^2\) carbon atoms were set to 0.93 Å (AFIX 43) and \(U_{eq}(C)\) set to 1.2 times \(U_{eq}(C)\). The methylene C—H distances were set to 0.97 Å (AFIX 23) and \(U_{eq}(H)\) set to 1.2 times \(U_{eq}(C)\). The phenyl ring is disordered and was refined with restrained geometry to form regular hexagons and restrained displacement parameters.

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References