Synthesis and crystal structure of di-tert-butyl 1″-acetyl-2,2″,9′-trioxo-4α,9α′-dihydro-1′H,3′H,9′H-dispiro[indoline-3,2′-xanthene-4′,3″-indoline]-1,3′-dicarboxylate, C₃₉H₃₈N₂O₉

Abstract

C₃₉H₃₈N₂O₉, monoclinic, P2₁/c (no. 14), a = 9.9846(6) Å, b = 31.4308(13) Å, c = 11.5739(6) Å, β = 110.712(7)°, V = 3397.4(3) Å³, Z = 4, ρcalc = 1.172 g cm⁻³, ρmax = 1.245 g cm⁻³, ρmin = 0.884 g cm⁻³, T = 293 K.

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The molecular structure is shown in the figure (hydrogen atoms were omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The mixture of tert-butyl 2-oxo-3-(4-oxo-4H-chromen-3-yl) methylindoline-1-carboxylate (0.2 mmol), tert-butyl (E)-2-(1-acetyl-2-oxindolin-3-ylidene)acetate (0.3 mmol), 5 Å molecular sieves 125 mg, catalyst (3,5-bis(trifluoromethyl) phenyl)-3-((S)-(6-methoxyquinolin-4-yl)((1S,2S,4S,5R)-5-vinylquinuclidin-2-yl)methyl)thiourea (10 mol %) and 6.0 mL of freshly distilled Et₂O was maintained at room temperature for 84 h. Then concentration by evaporation under reduced pressure gave a crude product, which was purified by column chromatography on silica gel column using hexane/EtOAc (8/1, v/v) to give the corresponding pure products [3].

Experimental details

All hydrogen atoms were placed in geometrically idealized positions. The Uiso values of the hydrogen atoms of methyl
of 1,5 $U_{eq}(C)$ and the $U_{iso}$ values of all other hydrogen atoms were set to 1.2$U_{eq}(C)$.

### Comment

The spiroindolone system is the core structure of some natural alkaloids. Moreover, they have momentous medicinal properties including anticancer [4], antioxidant [5], antimicrobial [6], antifungal [7], anti HIV [8] and antitubercular activities [9]. Due to the aforesaid properties a variety of methods using diverse types of catalysts have been reported in the literature for the procurement of these types of compounds [10–13]. On the other hand, 4H-chromen-4-ones, a well-known class of oxygenated heterocyclic compounds, play an important role in nature due to their recognized biological, pharmacological and biocidal activities [14–16]. Due to the significance of hybrid systems in
drug discovery [17], there is an urgent need to assemble multiple pharmacophores into a single molecule. According to physiological activity structure combination strategy, spirooxindole skeleton and 4H-chromen-4-ones ring were joined together and the title compound was synthesized.

X-ray crystal structural analysis indicates that the molecular structure of the title structure consists of a 1,2,3,4,4a,9a-hexahydro-9H-xanthen-9-one ring, a 1-acetylindoline-2-one ring, a tert-butyl 2-oxoindoline-1-carboxylate ring and a tert-butylxoycarbonyl moiety (cf. the figure). The indoline-2-one rings are essentially planar, with a mean deviation from plane of 0.0168(3) Å for 1-acetylindoline-2-one ring and 0.0120(2) Å for tert-butyl 2-oxoindoline-1-carboxylate ring. Xanthen and indoline-2-one rings form spiro structural feature through atom C1 and C19. Because C1 and C19 are sp³ carbon atoms, the indoline-2-one rings are non-coplanar with the xanthen ring. Bond lengths and angles in the title molecule are all in the expected ranges [18, 19].

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**References**