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Crystal structure of (Z)-3-((tert-butylamino)methylene)-2-(2-hydroxynaphthalen-1-yl)chroman-4-one, C$_{24}$H$_{23}$NO$_3$

Abstract

C$_{24}$H$_{23}$NO$_3$, orthorhombic, $Pna2_1$, $a = 8.2490(11)$ Å, $b = 15.886(2)$ Å, $c = 16.218(2)$ Å, $\beta = 99.465(2)^\circ$, $Z = 4$, $V = 2096.4(5)$ Å$^3$, $R_{gt}(F) = 0.0545$, $wR_{ref}(F^2) = 0.1688$, $T = 273$ K.

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Source of material

A solution of 1-iodonaphthalen-2-ol (1.0 mmol), 4-oxo-4$H$-chromene-3-carbaldehyde (1.0 mmol) and tert-butyl isocyanide (1.0 mmol) in DMF (2.0 mL) was heated under microwave irradiation at 150 °C for 20 min. After the microwave irradiation, the mixture was cooled to room temperature, and the solvent was removed by vacuum distillation. The resulting dark yellow solid was recrystallized from ethanol to give colorless needles.
 Experimental details

Using Olex2 [2], the structure was solved with the ShelX refinement package using Least-Squares minimization. All hydrogen atoms were added using a riding model using the default parameters of the SHELX program [3, 4].

 Comment

Chromone derivatives, as an important class of heterocycles, are used for various applications [4–7]. The chromone derivatives show various pharmacological properties [8–11]. Recently, for their pharmacological and biological applications, the synthesis of chromone derivatives has drawn more attention [12, 13]. In this work, we report the synthesis of the title compound using a three-component reaction with assistance of microwave irradiation in high yield.

The asymmetric unit consists of a chromone core and a naphthyl moiety. It is clear to find that the double bond of chromone is transferred from the inner to the outer, which leads the bond angles of C6–O2–C9 is 112.91(15)° and C8–C9–O2 is 109.99(15)°, respectively. And the double bond length of C8–C10 is 1.386(3) Å. The main chromene plane and the second naphthol plane are almost vertical to each other. In addition, the amino group does not rotate freely and is almost coplanar with chromone unit. The reason is that the amino group forms an intramolecular N1–H1···O1 hydrogen bond with the carbonyl group and N1–H1 and O1–H1 bond lengths are 0.860 and 1.968 Å, respectively. The N1···O1 separation is 2.645(3) Å. The bond angle of C8–C10–N1 is 124.3(2)°. For this crystal, all bond distances and angles are consistent with the expectation and in accord with the reported in the references [14, 15].

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References