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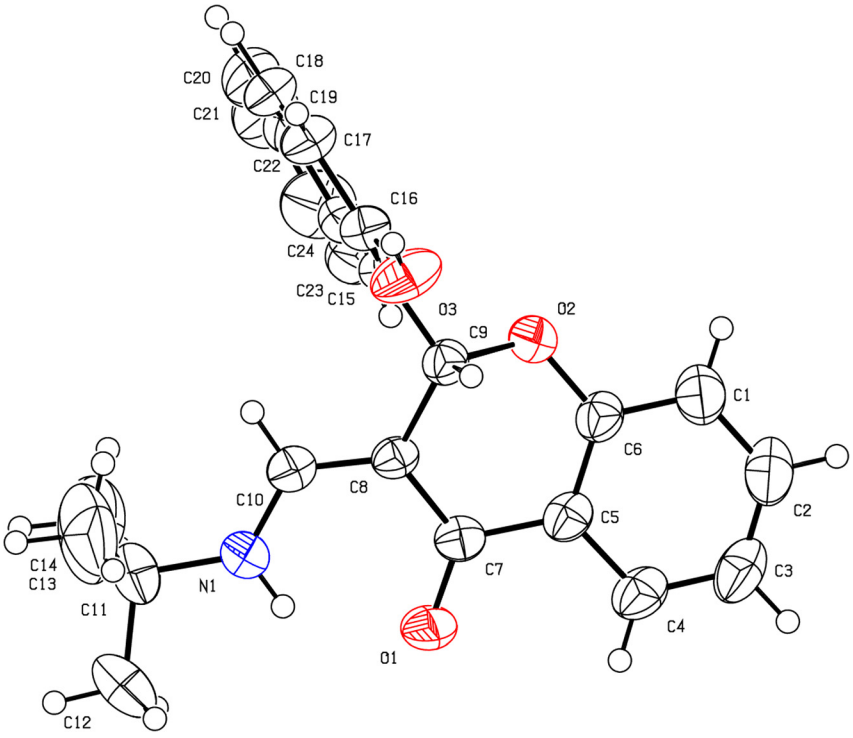
Crystal structure of (*Z*)-3-((*tert*-butylamino) methylene)-2-(2-hydroxynaphthalen-1-yl) chroman-4-one, $C_{24}H_{23}NO_3$



https://doi.org/10.1515/ncrs-2022-0097
Received February 28, 2022; accepted May 5, 2022;
published online May 31, 2022

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, β = 99.465(2)°, Z = 4, V = 2096.4(5) Å^3, $R_{gt}(F)$ = 0.0545, $wR_{ref}(F^2)$ = 0.1688, T = 273 K.

CCDC no.: 2075774

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.26 × 0.25 × 0.24 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10244, 3674, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2278
$N(param)_{refined}$:	257
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3, 4]

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

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.27097 (17)	0.08430 (9)	0.71931 (9)	0.0521 (4)
O2	0.55040 (19)	0.19361 (9)	0.56362 (9)	0.0526 (4)
O3	0.95646 (18)	0.13025 (11)	0.68919 (12)	0.0668 (5)
H3	1.0559	0.1222	0.7002	0.100*
N1	0.4423 (2)	0.17041 (12)	0.84362 (10)	0.0492 (5)
H1	0.3619	0.1368	0.8269	0.059*
C7	0.3742 (2)	0.11045 (13)	0.67470 (12)	0.0393 (5)
C8	0.5090 (2)	0.16202 (12)	0.70592 (12)	0.0366 (5)
C15	0.7641 (3)	0.24002 (14)	0.67316 (12)	0.0422 (5)
C5	0.3515 (2)	0.08888 (13)	0.58554 (13)	0.0421 (5)
C9	0.6341 (2)	0.17452 (13)	0.64831 (12)	0.0401 (5)
H9	0.6908	0.1206	0.6455	0.048*
C10	0.5349 (3)	0.19061 (13)	0.78780 (12)	0.0432 (5)
H10	0.6234	0.2264	0.8044	0.052*
C6	0.4370 (3)	0.13416 (14)	0.53279 (13)	0.0455 (6)
C24	0.7278 (3)	0.32772 (14)	0.67392 (12)	0.0470 (6)
C16	0.9254 (3)	0.21398 (15)	0.69197 (13)	0.0474 (6)
C4	0.2371 (3)	0.02859 (15)	0.55055 (15)	0.0537 (6)
H4	0.1792	−0.0021	0.5849	0.064*
C19	0.8603 (3)	0.38655 (16)	0.69339 (13)	0.0556 (7)
C3	0.2094 (3)	0.01421 (17)	0.46573 (17)	0.0646 (7)
H3A	0.1349	−0.0268	0.4429	0.078*
C18	1.0217 (3)	0.35558 (18)	0.71417 (15)	0.0644 (7)
H18	1.1079	0.3933	0.7287	0.077*
C1	0.4057 (3)	0.12052 (17)	0.44747 (14)	0.0603 (7)
H1A	0.4610	0.1517	0.4122	0.072*
C17	1.0542 (3)	0.27234 (17)	0.71351 (15)	0.0583 (7)
H17	1.1619	0.2534	0.7273	0.070*
C2	0.2929 (3)	0.06094 (18)	0.41502 (16)	0.0667 (7)
H2	0.2724	0.0520	0.3576	0.080*
C23	0.5672 (3)	0.35999 (17)	0.65437 (15)	0.0646 (7)
H23	0.4789	0.3232	0.6420	0.078*
C11	0.4595 (4)	0.19826 (18)	0.93121 (16)	0.0731 (8)
C20	0.8251 (5)	0.47277 (19)	0.69006 (17)	0.0792 (9)
H20	0.9112	0.5111	0.7014	0.095*
C21	0.6707 (5)	0.50176 (19)	0.6710 (2)	0.0913 (10)
H21	0.6508	0.5594	0.6694	0.110*
C22	0.5396 (4)	0.4449 (2)	0.6534 (2)	0.0861 (9)
H22	0.4326	0.4652	0.6408	0.103*
C13	0.6337 (5)	0.1809 (3)	0.9753 (2)	0.1375 (16)
H13A	0.6536	0.1213	0.9766	0.206*
H13B	0.6470	0.2021	1.0315	0.206*
H13C	0.7106	0.2083	0.9457	0.206*
C14	0.4241 (6)	0.2925 (2)	0.9330 (3)	0.1288 (15)
H14A	0.4958	0.3221	0.9020	0.193*
H14B	0.4422	0.3119	0.9898	0.193*
H14C	0.3119	0.3028	0.9083	0.193*
C12	0.3336 (6)	0.1495 (3)	0.9687 (2)	0.158 (2)
H12A	0.2259	0.1617	0.9387	0.237*
H12B	0.3392	0.1651	1.0262	0.237*
H12C	0.3551	0.0903	0.9651	0.237*

vial was cooled to room temperature, the solvent was removed and the residue was purified by silica gel column chromatography using a gradient of ethyl acetate/hexane (0–100%) to afford the targeted product (*Z*)-3-((*tert*-butylamino)methylene)-2-(2-hydroxynaphthalen-1-yl)chroman-4-one. The product was dissolved in methanol and crystals of the title compound were obtained by slow evaporation within seven days (Tables 1 and 2).

Experimental details

Using Olx2 [2], the structure was solved with the ShelXS 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

Chromene derivatives, as an important class of heterocycles, are used for various applications [4–7]. The chromene derivatives show various pharmacological properties [8–11]. Recently, for their pharmacological and biological applications, the synthesis of chromene 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].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was financially supported by the National Natural Science Foundation of China (Grant No. 51973042 and 51863002), the Major Project of Natural Science Foundation of Guizhou Province (Grant No. QKHJC–ZK[2021]ZD048). We would also like to thank Chongqing University of Arts and Sciences for the procedure of compound synthesis.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. Smart apex–ii ccd; Bruker AXS Inc.: Madison, WI, USA, 2006.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
5. Pratap R., Ram V. J. Natural and synthetic chromenes, fused chromenes, and versatility of dihydrobenzo[*h*]chromenes in organic synthesis. *Chem. Rev.* 2014, *114*, 10476–10526.
6. Costa M., Dias T. A., Brito A., Proença F. Biological importance of structurally diversified chromenes. *Eur. J. Med. Chem.* 2016, *123*, 487–507.
7. Movassagh B., Rooh H., Bijanzadeh H. R. A mild and highly efficient one-pot synthesis of 1,3,5-triaryl-2-pyrazolines. *Chem. Heterocycl. Compd.* 2013, *48*, 1719–1721.
8. Oliveira-Pinto S., Pontes O., Baltazar F., Costa M. *In vivo* efficacy studies of chromene-based compounds in triple-negative breast cancer – a systematic review. *Eur. J. Pharmacol.* 2020, *887*, 173452.
9. Das S. G., Srinivasan B., Hermanson D. L., Bleeker N. P., Doshi J. M., Tang R., Beck W. T., Xing C. Structure-activity relationship and molecular mechanisms of ethyl 2-amino-6-(3,5-dimethoxyphenyl)-4-(2-ethoxy-2-oxoethyl)-4*H*-chromene-3-carboxylate (CXL017) and its analogues. *J. Med. Chem.* 2011, *54*, 5937–5948.
10. Conti C., Proietti Monaco L., Desideri N. Synthesis and anti-rhinovirus activity of novel 3-[2-(pyridinyl)vinyl]substituted -2*H*-chromenes and -4*H*-chromen-4-ones. *Bioorg. Med. Chem.* 2014, *22*, 1201–1207.
11. Kashman Y., Gustafson K. R., Fuller R. W., Cardellina J. H., McMahon J. B., Currens M. J., Buckheit R. W., Hughes S. H., Cragg G. M., Boyd M. R. The calanolides, a novel HIV-inhibitory class of coumarin derivatives from the tropical rainforest tree, Calophyllum lanigerum. *J. Med. Chem.* 1992, *35*, 2735–2743.
12. Rouh H., Tang Y., Zhang S., Ali A. I. M., Surowiec K., Unruh D., Li G. Asymmetric [4 + 2] cycloaddition synthesis of 4*H*-chromene derivatives facilitated by group-assisted-purification (GAP) chemistry. *RSC Adv.* 2021, *11*, 39790–39796.
13. Xu Y.-Z., Tian J.-W., Sha F., Li Q., Wu X. Y. Concise synthesis of chromene/chromane-type Aryne precursors and their applications. *J. Org. Chem.* 2021, *86*, 6765–6779.
14. Zhong N.-J., Liu L., Wang D., Chen Y.-J. Enantioselective tandem reaction of chromone-derived Morita–Baylis–Hillman carbonates with benzylamines catalyzed by a trifunctional organocatalyst: the synthesis of chiral 3-aminomethylene-flavanones. *Chem. Commun.* 2013, *49*, 3697–3699.
15. Wu C., Liu Y., Zeng H., Liu L., Wang D., Chen Y. In(III)–catalyzed tandem reaction of chromone-derived Morita–Baylis–Hillman alcohols with amines. *Org. Biomol. Chem.* 2011, *9*, 253–256.