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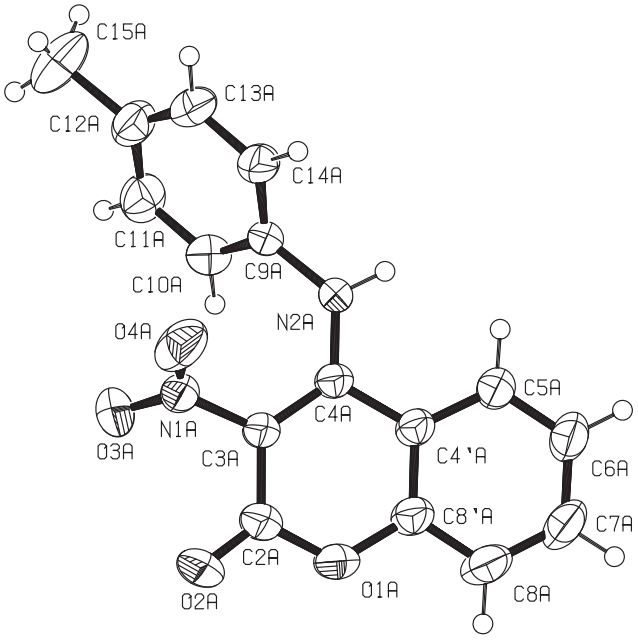
The crystal structure of 3-nitro-4-(*p*-tolylamino)-2*H*-chromen-2-one, $C_{16}H_{12}N_2O_4$



https://doi.org/10.1515/ncrs-2019-0592
Received August 15, 2019; accepted October 2, 2019; available online October 14, 2019

Abstract

$C_{16}H_{12}N_2O_4$, triclinic, $P\bar{1}$ (no. 2), $a = 10.7105(5)$ Å, $b = 11.2685(5)$ Å, $c = 13.0145(6)$ Å, $\alpha = 99.292(4)°$, $\beta = 109.607(4)°$, $\gamma = 96.771(4)°$, $V = 1435.01(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0427$, $wR_{ref}(F^2) = 0.1124$, $T = 295(2)$ K.

CCDC no.: 1957160

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

Table 1: Data collection and handling.

Crystal:	Trapezoid, yellow
Size:	0.60 × 0.37 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.1 mm^{-1}
Diffractometer, scan mode:	Gemini S, ω-scans
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14917, 5870, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4398
$N(param)_{refined}$:	399
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], PLATON [4]

Source of material

The synthesis and characterization of the title compound was previously reported [5]. The product was obtained in the reaction of 4-chloro-3-nitrocoumarin and *p*-toluidine in ethyl acetate, in the presence of triethylamine. The obtained solid was dissolved in chloroform and allowed to slowly evaporate to give yellow crystals of the title compound.

Experimental details

Hydrogen atoms were introduced in idealized positions and refined using riding model. Their U_{iso} values are aproximated as $U_{iso} = kU_{eq}$ of the parent atom ($k = 1.2$ for sp^2 and 1.5 for sp^3 atoms).

Comment

Coumarins are a large group of organic compounds of natural or synthetic origin. They are known to possess promising pharmacological activities, such as antimicrobial [6], antioxidant [7], anticancer [8], anti-inflammatory [9]. The coumarin framework is a promising scaffold for the synthesis of analogs, with an aim to study and improve their biological properties. Varying the types of substitution and substituent identity led to different conformations of the molecules that

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1B	1.16119(11)	0.91869(10)	0.56562(9)	0.0507(3)
O2B	1.15133(12)	1.03279(10)	0.44271(10)	0.0519(3)
O4B	0.81452(11)	0.84478(10)	0.23184(9)	0.0479(3)
O3B	1.00662(12)	0.91945(11)	0.22776(10)	0.0558(3)
N1B	0.93786(13)	0.87025(11)	0.27307(10)	0.0362(3)
N2B	0.87600(12)	0.63433(11)	0.33237(10)	0.0387(3)
H2B	0.836251	0.583155	0.359005	0.046*
C2B	1.10733(15)	0.93653(14)	0.45939(13)	0.0388(3)
C3B	1.00335(14)	0.84102(13)	0.38018(12)	0.0340(3)
C4B	0.95786(14)	0.73335(13)	0.40659(12)	0.0332(3)
C4′B	1.00456(15)	0.72883(14)	0.52459(12)	0.0375(3)
C5B	0.95315(18)	0.63703(15)	0.56831(14)	0.0475(4)
H5B	0.885596	0.572694	0.520475	0.057*
C6B	1.0014(2)	0.64103(18)	0.68098(15)	0.0593(5)
H6B	0.965341	0.580279	0.709059	0.071*
C7B	1.1031(2)	0.73457(19)	0.75265(15)	0.0673(6)
H7B	1.136165	0.735796	0.828719	0.081*
C8B	1.1560(2)	0.82598(18)	0.71280(15)	0.0615(5)
H8B	1.224721	0.889028	0.761301	0.074*
C8′B	1.10573(17)	0.82301(14)	0.59954(13)	0.0439(4)
C9B	0.84650(15)	0.60311(13)	0.21529(12)	0.0370(3)
C14B	0.71886(17)	0.54160(15)	0.14572(13)	0.0470(4)
H14B	0.652399	0.524001	0.175034	0.056*
C13B	0.69084(18)	0.50656(16)	0.03269(14)	0.0534(4)
H13B	0.605387	0.463541	−0.013288	0.064*
C12B	0.78597(18)	0.53343(15)	−0.01451(13)	0.0491(4)
C11B	0.91319(18)	0.59404(15)	0.05704(14)	0.0478(4)
H11B	0.979087	0.613224	0.027542	0.057*
C10B	0.94511(16)	0.62686(14)	0.17099(13)	0.0415(4)
H10B	1.032336	0.664676	0.217631	0.050*
C15B	0.7517(2)	0.4984(2)	−0.13898(15)	0.0696(6)
H15D	0.656614	0.492578	−0.176750	0.104*
H15E	0.776372	0.420753	−0.157462	0.104*
H15F	0.800415	0.559467	−0.161945	0.104*
O1A	0.53546(13)	0.31509(12)	0.23852(10)	0.0589(3)
O2A	0.71073(13)	0.43775(13)	0.36920(11)	0.0705(4)
O3A	0.76230(14)	0.37834(16)	0.57852(12)	0.0860(5)
O4A	0.58391(16)	0.43433(13)	0.59828(11)	0.0721(4)
N1A	0.64180(16)	0.38220(14)	0.54349(12)	0.0536(4)
N2A	0.39533(13)	0.19486(12)	0.47211(10)	0.0420(3)
H2A	0.310529	0.164988	0.448302	0.050*
C2A	0.60992(17)	0.36344(16)	0.34898(14)	0.0495(4)
C3A	0.56108(16)	0.32163(14)	0.42846(13)	0.0417(4)
C4A	0.43935(15)	0.24380(13)	0.40064(12)	0.0374(3)
C4′A	0.35433(16)	0.21171(13)	0.28208(12)	0.0405(4)
C5A	0.22108(17)	0.14846(15)	0.24021(14)	0.0512(4)
H5A	0.181416	0.124955	0.289111	0.061*
C6A	0.1483(2)	0.12077(18)	0.12750(15)	0.0641(5)
H6A	0.059217	0.080087	0.100911	0.077*
C7A	0.2058(2)	0.15255(18)	0.05325(15)	0.0678(6)
H7A	0.156086	0.131784	−0.023043	0.081*
C8A	0.3359(2)	0.21456(17)	0.09166(15)	0.0621(5)
H8A	0.375516	0.235287	0.041860	0.074*
C8′A	0.40807(18)	0.24621(14)	0.20584(13)	0.0472(4)
C9A	0.47220(15)	0.18618(14)	0.58334(12)	0.0388(3)
C10A	0.59289(17)	0.14569(15)	0.60714(14)	0.0471(4)
H10A	0.626842	0.124809	0.550974	0.056*
C11A	0.66338(19)	0.13623(17)	0.71500(16)	0.0562(5)
H11A	0.746203	0.111065	0.731199	0.067*
C12A	0.6135(2)	0.16335(17)	0.79925(15)	0.0571(5)
C13A	0.4899(2)	0.19918(19)	0.77235(15)	0.0613(5)
H13A	0.453202	0.215518	0.827419	0.074*
C14A	0.41943(17)	0.21134(17)	0.66578(14)	0.0523(4)
H14A	0.336571	0.236442	0.649507	0.063*
C15A	0.6927(3)	0.1566(3)	0.91842(18)	0.0957(8)
H15A	0.684746	0.224738	0.969055	0.144*
H15B	0.785947	0.159033	0.928055	0.144*
H15C	0.657656	0.081621	0.933552	0.144*

had a profound impact on their biological activity [10]. We have previously synthesized a number of coumarin derivatives with significant antimicrobial and/or antioxidant activities [5, 11–13].

The asymmetric part of the unit cell contains two molecules (A) and (B) of the 3-nitro-4-(*p*-tolylamino)-2*H*-chromen-2-one. Bond lengths within the coumarin core of these molecules are in good agreement with those found in other 3-nitro-4-(arylamino) coumarins [10]. The two molecules have very similar conformations, as RMSD of their fit is 0.2417 Å. For both molecules, the coumarin core of the molecule is approximately planar, while both substituent groups at C3 and C4 are turned out from the aforementioned planes. This is visualized through torsion angle C4—C3—N1—O3 (131.55(17)° for molecule A and −141.34(15)° for molecule B) for nitro group twisting, as well as torsion angle C4—N2—C9—C10 (−46.8(2)° for molecule A and 37.7(2)° for molecule B) for the *p*-tolylamino group twisting.

Hydrogen bonds N2B—H2B···O2A and N2A—H2A···O2B^i [symmetry code (i) = $x-1$, $y-1$, z] connect molecules in ···A···B···A···B··· chains.

Acknowledgements: This work was supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia (Grants Nos. 172061, 172014 and 45022).

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