Lian-Man He and Chuan-Yu Gao*

Crystal structure of 2-amino-4-(2, 4-dichlorophenyl)-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile, C₁₆H₁₂Cl₂N₂O₂

Table 1: Data collection and handling.

Crystal:	Colourless blocks
Wavelength:	Size 0.20 × 0.19 × 0.17 mm
μ :	Mo K_{α} radiation (0.71073 Å)
Diffractometer, scan mode:	4.2 cm ⁻¹
2 $\theta_{\max}$, completeness:	Bruker FRAMBO, φ and ω
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	53.2°, >99%
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	5570, 2793, 0.037
$N(\text{param})_{\text{refined}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1882
Programs:	200
	SHELX [7], Bruker programs [8]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.06792(13)	0.8370(3)	0.06568(19)	0.0425(7)
C2	0.05615(12)	0.7717(3)	0.12960(18)	0.0363(6)
C3	0.07528(12)	0.8405(3)	0.22419(18)	0.0378(6)
H3A	0.0387	0.8385	0.2317	0.045*
C4	0.09035(12)	1.0038(3)	0.22175(19)	0.0373(6)
C5	0.09920(12)	1.0609(3)	0.15442(19)	0.0403(6)
C6	0.11578(14)	1.2191(3)	0.1484(2)	0.0502(8)
H6A	0.0796	1.2723	0.1015	0.060*
H6B	0.1468	1.2214	0.1292	0.060*
C7	0.14054(16)	1.2965(3)	0.2430(2)	0.0608(9)
H7A	0.1817	1.2596	0.2857	0.073*
H7B	0.1433	1.4037	0.2352	0.073*
C8	0.09920(17)	1.2678(3)	0.2848(2)	0.0601(9)
H8A	0.1183	1.3100	0.3477	0.072*
H8B	0.0601	1.3187	0.2471	0.072*
C9	0.08742(12)	1.1042(3)	0.2897(2)	0.0438(7)
C10	0.12472(13)	0.7512(3)	0.30576(19)	0.0439(7)
C11	0.11146(17)	0.6937(3)	0.3725(2)	0.0590(9)
H11A	0.0737	0.7181	0.3675	0.071*
C12	0.1507(2)	0.6034(4)	0.4452(2)	0.0763(11)
H12A	0.1397	0.5666	0.4879	0.092*
C13	0.2065(2)	0.5686(4)	0.4537(3)	0.0802(12)
C14	0.22379(16)	0.6238(4)	0.3916(3)	0.0795(12)
H14A	0.2622	0.6004	0.3985	0.095*
C15	0.18242(15)	0.7158(3)	0.3180(2)	0.0575(8)
C16	0.02733(13)	0.6299(3)	0.11006(18)	0.0409(7)
O1	0.09360(9)	0.97797(18)	0.08005(13)	0.0491(6)

DOI 10.1515/ncrs-2016-0065

Received March 2, 2016; accepted June 9, 2016; available online June 22, 2016

Abstract

C₁₆H₁₂Cl₂N₂O₂, monoclinic, *C*2/*c* (no. 15), *a* = 24.944(3) Å, *b* = 8.9007(8) Å, *c* = 16.3770(18) Å, β = 119.710(10)°, *V* = 3158.0(6) Å³, *Z* = 8, $R_{\text{gt}}(F)$ = 0.0527, $wR_{\text{ref}}(F^2)$ = 0.1602, *T* = 293(2) K.

CCDC no.: 1484303

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized according to a reported procedure [6]. A mixture of 3,5-cyclohexanedione (10 mmol),

*Corresponding author: Chuan-Yu Gao, Department of Hypertension, Henan Province People's Hospital, Zhengzhou 450003, China, e-mail: chuanyu_gao66@yeah.net; and Department of Cardiology, Henan Province People's Hospital, Zhengzhou 450003, China

Lian-Man He: Department of Hypertension, Henan Province People's Hospital, Zhengzhou 450003, China

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O2	0.07229(10)	1.0553(2)	0.34518(15)	0.0604(6)
N1	0.05784(13)	0.7818(3)	−0.01600(16)	0.0595(8)
H1A	0.0419	0.6939	−0.0336	0.071*
H1B	0.0673	0.8339	−0.0513	0.071*
N2	0.00267(13)	0.5162(3)	0.09732(18)	0.0607(8)
Cl1	0.20610(4)	0.78282(13)	0.24155(9)	0.0944(4)
Cl2	0.25718(7)	0.45027(13)	0.54251(9)	0.1448(7)

2,4-dichlorobenzaldehyde (10 mmol), malononitrile (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

Carbon-bound and Nitrogen-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with *U*_{iso}(H) set to 1.2 *U*_{eq}(C).

Discussion

The progress achieved in the synthesis of heterocyclic compounds with biological potential is due to improvement of the methodological study of tested substances too [1, 2]. It is known that many pyran derivatives have biological activity, with their antibacterial, antimycobacterial, antimycotic, antifungal, antidepressive and cardiotonic action being notable [3–5]. Recent research has also established an analgesic and anti-inflammatory activity for such compounds [5].

In the crystal structure of the title compound, the 4*H*-pyran ring is nearly planar. The cyclohex-2-en-1-on-2,3-diyl moiety is folded. The plane of the 4*H*-pyran ring is almost perpendicular to the plane of the dichlorophenyl moiety. All bond lengths and angles are in the expected ranges.

References

- Li, J.; Xue, X. Y.; Li, X.; Hou, Z.; Yang, X. H.; Qu, D.; Zhou, Y.; Zhang, Z. D.; Luo, X. X.; Li, J. T.; Li, M. K.: Synthesis of biscoumarin and dihydropyran derivatives as two novel classes of potential anti-bacterial derivatives. *Arch. Pharm. Res.* (2015) DOI 10.1007/s12272-015-0614-7.
- Li, J.; Meng, J.; Qu, D.; Zhang, Z.-D.; Li, F.; Yang, X.-H.; Li, J.-T.; Li, M.-K.: New biscoumarin and dihydropyran derivatives as antimicrobials. *Res. Chem. Intermed.* **41** (2015) 8257–8267.
- Li, J.; Lv, C. W.; Li X. J.; Qu, D.; Hou, Z.; Jia, M.; Luo, X. X.; Li, X.; Li, M. K.: Synthesis of biscoumarin and dihydropyran derivatives and evaluation of their antibacterial activity. *Molecules* **20** (2015) 17469–17482.
- Khan, A. T.; Lal, M.; Ali, S.; Khan, Md. M.: One-pot three-component reaction for the synthesis of pyran annulated heterocyclic compounds using DMAP as a catalyst. *Tetrahedron Lett.* **52** (2011) 5327–5332.
- Matsuno, T.; Saito, M.; Matsumoto, Y.; Morikawa, J.: A new benzo-γ-pyran derivative isolated from propolis. *Z. Naturforsch.* **B53** (1998) 1037–1039.
- Li, J.; Sui, Y. P.; Xin, J. J.; Du, X. L.; Li, J. T.; Huo, H. R.; Ma, H.; Wang, W. H.; Zhou, H. Y.; Zhan, H. D.; Wang, Z. J.; Li, C.; Sui, F.; Li, X.: Synthesis of biscoumarin and dihydropyran derivatives with promising antitumor and antibacterial activities. *Bioorg. Med. Chem. Lett.* **25** (2015) 5520–5523.
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.
- Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA, 2009.