Open Access

Changming You and Changqing Wang*

Crystal structure of 2-benzoyl-3-(4-methoxyphenyl)cyclopropane-1,1-dicarbonitrile, $C_{19}H_{14}N_2O_2$

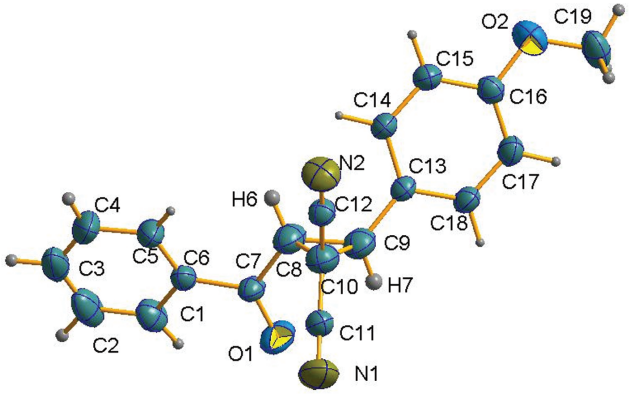

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEXII, ω
θ_{max}	28.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13416, 4055, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2291
$N(param)_{refined}$:	253
Programs:	SHELX [4], DIAMOND [5], BRUKER [6]

DOI 10.1515/ncrs-2017-0052
Received April 19, 2017; accepted August 12, 2017; available online September 16, 2017

Abstract

$C_{19}H_{14}N_2O_2$, monoclinic $C2/c$ (no. 15), $a = 19.8907(8)$ Å, $b = 12.0860(5)$ Å, $c = 15.5260(7)$ Å, $\beta = 120.588(1)°$, $V = 3213.06(20)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0504$, $wR_{ref}(F^2) = 0.1426$, $T = 296$ K.

CCDC no.: 1568605

The crystal structure 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.

Source of material

A mixture of phenacyl bromide (0.238 g, 1.2 mmol), triphenylarsine (0.167 g, 0.15 mmol), 2-(4-methoxybenzylidene) malononitrile (1 mmol), and $NaHCO_3$ (0.240 g, 3 mmol) was stirred in CH_3CN at room temperature. Completion of the reaction was monitored by TLC using hexane/ethyl acetate (6:1). The solution was filtered on a pad of Celite and washed with ethyl acetate (5 mL) followed by dichloromethane (5 mL). The filtrate was concentrated under reduced pressure to afford the crude product, which was purified by column chromatography using hexane/ethyl acetate and silica gel as the stationary phase. The pure product was a white solid (0.25 g, yield 86%). Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of the title compound in a mixture of dichloromethane with hexane at room temperature.

Experimental details

All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located by geometrical calculations, and their positions and thermal parameters were fixed during the structure refinement.

Discussion

The synthesis and application of multisubstituted cyclopropanes have been subjects of great interest because of the roles as basic structural elements in a wide range of biologically active compounds and important intermediates in organic synthesis [1–3]. We have been interested in this type of derivatives and reported several crystallographic analyses of the products. As a continuation of our work, the multisubstituted cyclopropane derivative 2-

***Corresponding author: Changqing Wang,** College of Chemistry and Environment Engineering, Jiujiang University, Jiujiang, Jiangxi 332005, P. R. China, e-mail: pharmawang@163.com
Changming You: College of Chemistry and Environment Engineering, Jiujiang University, Jiujiang, Jiangxi 332005, P. R. China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.12536(16)	0.46622(17)	0.13246(17)	0.0817(6)
C2	0.0798(2)	0.5313(2)	0.1555(2)	0.1070(9)
C3	0.0119(3)	0.4929(3)	0.1426(3)	0.1237(13)
C4	−0.0117(2)	0.3895(3)	0.1082(3)	0.1339(13)
C5	0.03325(14)	0.32125(19)	0.0857(2)	0.0872(7)
C6	0.10155(10)	0.35982(13)	0.09677(12)	0.0559(4)
C7	0.14958(10)	0.29359(13)	0.06900(14)	0.0603(4)
C8	0.13583(10)	0.17085(13)	0.05529(13)	0.0543(4)
C9	0.19223(9)	0.10020(13)	0.04533(12)	0.0516(4)
C10	0.11153(9)	0.12321(12)	−0.04863(13)	0.0524(4)
C11	0.10767(11)	0.19373(15)	−0.12574(15)	0.0672(5)
C12	0.05336(10)	0.03694(14)	−0.08464(13)	0.0592(4)
C13	0.21478(9)	−0.01325(13)	0.08595(12)	0.0503(4)
C14	0.17542(11)	−0.07426(14)	0.12244(14)	0.0625(5)
C15	0.19799(12)	−0.18060(16)	0.15694(16)	0.0700(5)
C16	0.26002(10)	−0.22877(14)	0.15505(14)	0.0628(4)
C17	0.30008(11)	−0.16970(16)	0.12021(15)	0.0685(5)
C18	0.27735(10)	−0.06263(16)	0.08619(14)	0.0623(5)
C19	0.33745(14)	−0.39023(19)	0.18261(18)	0.0970(7)
H19A	0.3252	−0.3885	0.1143	0.146*
H19B	0.3408	−0.4657	0.2038	0.146*
H19C	0.3866	−0.3540	0.2242	0.146*
H1	0.1737(16)	0.485(2)	0.140(2)	0.123(10)*
H2	0.0933(18)	0.610(3)	0.178(3)	0.168(12)*
H3	−0.0172(19)	0.540(2)	0.160(2)	0.154(11)*
H4	−0.061(2)	0.355(3)	0.088(3)	0.174(13)*
H5	0.0155(12)	0.2539(17)	0.0579(16)	0.081(6)*
H6	0.1090(9)	0.1384(12)	0.0842(11)	0.051(4)*
H7	0.2308(9)	0.1426(13)	0.0423(12)	0.057(4)*
H8	0.1320(11)	−0.0418(14)	0.1267(14)	0.071(5)*
H9	0.1698(12)	−0.2197(17)	0.1826(16)	0.090(6)*
H10	0.3045(11)	−0.0213(15)	0.0617(15)	0.077(6)*
H11	0.3446(13)	−0.2021(16)	0.1176(16)	0.094(7)*
N1	0.10417(13)	0.24665(18)	−0.18834(18)	0.1087(7)
N2	0.00791(10)	−0.03160(14)	−0.11037(14)	0.0850(5)
O1	0.19882(9)	0.33535(10)	0.05534(14)	0.0966(5)
O2	0.27790(9)	−0.33477(11)	0.19075(12)	0.0872(4)

benzoyl-3-(4-methoxyphenyl)cyclopropane-1,1-dicarbonitrile was synthesized from 2-(4-methoxybenzylidene)malononitrile and the corresponding phenacyl bromide.

There is one complete molecule in the asymmetric unit (*cf*. the figure). The C8—C10, and C9—C10 bond lengths are 1.541(2), 1.549(2) Å, respectively. The C8—C9 bond length is 1.479(2), slightly shorter than the other two C—C bonds in the cyclopropanyl moiety. The bond angles of C9—C8—C10, C8—C9—C10 and C8—C10—C9 are 61.66(11)°, 61.13(11)° and 57.2(1)°, respectively. Thus angles of ca. 60° are present. The benzoyl and aryl groups are situated in an anti configuration at the cyclopropane ring.

Acknowledgements: The authors thank the Natural Science Foundation of China (no. 21662019) for financial support.

References

1. Shi, M.; Lu, J. M.; Wei, Y.; Shao, L. X.: Rapid generation of molecular complexity in the Lewis or Brønsted acid-mediated reactions of methylenecyclopropanes. Acc. Chem. Res. **45** (2012) 641–652.
2. Parsons, A. T.; Smith, A. G.; Neel, A. N.; Johnson, J. S.: Dynamic kinetic asymmetric synthesis of substituted pyrrolidines from racemic cyclopropanes and aldimines: reaction development and mechanistic insights. J. Am. Chem. Soc. **132** (2010) 9688–9692.
3. Maghsoodlou, M. T.; Khorassani, S. M. H.; Heydari, R.; Charati, F. R.; Hazeri, N.; Lashkari, M.; Rostamizadeh, M.; Marandi, G.; Sobolev, A.; Makha, M.: Highly stereoselective construction of functionalized cyclopropanes from the reaction between acetylenic esters and C—CH acids in the presence of triphenylarsine. Tetrahedron Lett. **50** (2009) 4439–4442.
4. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
5. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2i. Crystal Impact, Bonn, Germany (2012).
6. Bruker. APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, Wisconsin, USA, 2009.