Crystal structure of 2-(benzo[*d*][1,3]dioxol-5-yl)-3-benzoylcyclopropane-1,1-dicarbonitrile, $C_{19}H_{12}N_2O_3$

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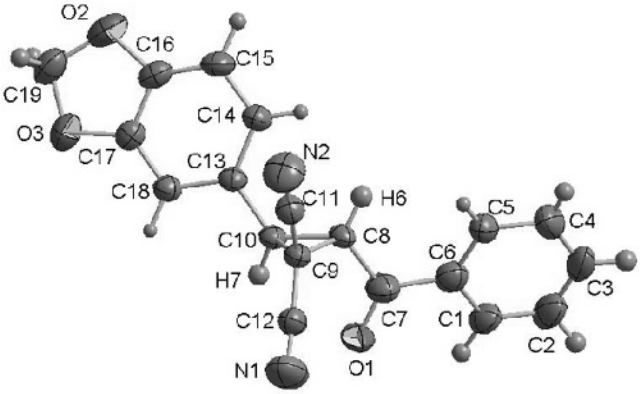

Abstract

$C_{19}H_{12}N_2O_3$, monoclinic, *C*2/*c* (no. 15), a = 19.7091(7) Å, b = 12.0956(7) Å, c = 15.9463(8) Å, β = 123.038(2)°, V = 3186.9 Å^3, Z = 8, $R_{gt}(F)$ = 0.0483, $wR_{ref}(F^2)$ = 0.1428, T = 296 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.23×0.29×0.42 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.91 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	57.38°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13392, 4113
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2729
$N(param)_{refined}$:	265
Programs:	SHELX [4], DIAMOND [5]

Source of material

A mixture of triphenylarsine (0.167 g, 0.15 mmol), phenacyl bromide (0.238 g, 1.2 mmol), 2-(benzo[*d*][1,3]dioxol-5-ylmethylene)malononitrile (1 mmol), and $NaHCO_3$ (0.240 g, 3 mmol) was stirred in CH_3CN at room temperature. The progress of reaction was monitored by thin-layer chromatography (*TLC*). After completion, water was added and the product was extracted with ethyl acetate. The organic layer was separated, dried over anhydrous sodium sulfate, and the solvent was evaporated under reduced pressure to afford the crude product. Colourless crystals of the title compound, which were suitable for X-ray analysis, were obtained by recrystallizing the deposit from a mixture of methylene chloride and ethyl acetate solution at room temperature.

Experimental details

The hydrogen atoms were located by geometrically calculations, and their positions and displacement parameters were refined during the structure refinement.

Discussion

The synthesis and application of multisubstituted cyclopropanes have been subjects of great interest because of their roles as basic structural elements in a wide range of biologically active compounds and important intermediates in organic synthesis [1–3]. As part of our work, we report the synthesis and crystal structure of the title compound. Within the crystal structure, the geometric parameters of cyclopropane are in the usual ranges. The C8–C9 and C9–C10 bond lengths are 1.543(2), 1.5519 (19) Å respectively. The C8–C10 bond length is 1.480(2) Å, slightly shorter than the other two C–C bonds. The bond angles of C9–C8–C10, C8–C9–C10 and C8–C10–C9 are 61.74(9)°, 57.13(9)° and 61.12(9)°, only slightly deviating from the ideal 60°. With respect to the plane of the three-membered ring, and the benzoyl and aryl groups are situated above and below the central cyclopropane ring.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*f*	0.177(2)	0.497(2)	0.124(2)	0.119(8)
H(2)	8*f*	0.109(2)	0.375(3)	0.174(2)	0.139(9)
H(3)	8*f*	0.002(2)	0.443(2)	0.176(2)	0.15(1)
H(4)	8*f*	–0.047(2)	0.631(2)	0.113(2)	0.116(9)
H(5)	8*f*	0.013(1)	0.735(2)	0.065(1)	0.073(6)
H(6)	8*f*	0.1008(9)	0.851(1)	0.072(1)	0.055(4)
H(7)	8*f*	0.2332(9)	0.852(1)	0.047(1)	0.051(4)
H(8)	8*f*	0.140(1)	1.016(2)	0.142(1)	0.073(5)
H(9)	8*f*	0.173(1)	1.193(2)	0.208(2)	0.094(6)
H(10)	8*f*	0.299(1)	1.028(1)	0.056(1)	0.063(5)
H(11)	8*f*	0.325(2)	1.397(2)	0.122(2)	0.120(8)
H(12)	8*f*	0.399(2)	1.346(2)	0.234(2)	0.119(8)

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.1291(1)	0.5229(2)	0.1229(1)	0.096(1)	0.053(1)	0.072(1)	0.0055(9)	0.049(1)	0.0072(8)
C(2)	8*f*	0.0919(2)	0.4553(2)	0.1548(2)	0.150(2)	0.062(1)	0.088(1)	–0.011(1)	0.075(2)	0.005(1)
C(3)	8*f*	0.0279(2)	0.4921(2)	0.1557(2)	0.148(2)	0.076(2)	0.097(2)	–0.045(2)	0.086(2)	–0.022(1)
C(4)	8*f*	0.0003(2)	0.5971(2)	0.1272(2)	0.099(2)	0.094(2)	0.124(2)	–0.023(1)	0.082(2)	–0.023(1)
C(5)	8*f*	0.0375(1)	0.6677(2)	0.0949(2)	0.069(1)	0.061(1)	0.088(1)	–0.0076(9)	0.051(1)	–0.010(1)
C(6)	8*f*	0.10185(9)	0.6305(1)	0.0919(1)	0.0595(8)	0.0472(8)	0.0480(7)	–0.0029(7)	0.0299(7)	–0.0056(6)
C(7)	8*f*	0.14369(9)	0.6981(1)	0.0569(1)	0.0596(8)	0.0456(8)	0.0629(9)	0.0090(7)	0.0391(7)	0.0019(7)
C(8)	8*f*	0.12955(9)	0.8213(1)	0.0460(1)	0.0571(8)	0.0446(8)	0.0598(8)	0.0093(6)	0.0408(7)	0.0037(6)
C(9)	8*f*	0.10828(8)	0.8739(1)	–0.0537(1)	0.0493(7)	0.0468(8)	0.0532(8)	0.0032(6)	0.0319(6)	–0.0005(6)
C(10)	8*f*	0.19068(8)	0.8927(1)	0.0463(1)	0.0455(7)	0.0464(8)	0.0538(8)	0.0076(6)	0.0303(6)	–0.0003(6)
C(11)	8*f*	0.04997(9)	0.9626(1)	–0.0910(1)	0.0525(8)	0.0567(9)	0.0583(9)	0.0049(7)	0.0296(7)	0.0067(7)
C(12)	8*f*	0.1064(1)	0.8079(1)	–0.1304(1)	0.0605(9)	0.062(1)	0.0616(9)	–0.0051(8)	0.0369(8)	–0.0082(8)
C(13)	8*f*	0.21308(8)	1.0053(1)	0.0906(1)	0.0458(7)	0.0481(8)	0.0464(7)	0.0082(6)	0.0240(6)	0.0008(6)
C(14)	8*f*	0.1778(1)	1.0547(2)	0.1358(1)	0.0654(9)	0.059(1)	0.072(1)	0.0008(8)	0.0463(9)	–0.0089(8)
C(15)	8*f*	0.1986(1)	1.1617(2)	0.1750(1)	0.084(1)	0.063(1)	0.077(1)	0.0055(9)	0.051(1)	–0.0156(9)
C(16)	8*f*	0.2553(1)	1.2147(1)	0.1669(1)	0.070(1)	0.0472(8)	0.0556(9)	0.0042(8)	0.0265(8)	–0.0057(7)
C(17)	8*f*	0.29094(9)	1.1666(1)	0.1223(1)	0.0548(8)	0.0539(9)	0.0528(8)	–0.0009(7)	0.0244(7)	0.0009(7)
C(18)	8*f*	0.27224(9)	1.0619(1)	0.0842(1)	0.0509(8)	0.0535(9)	0.0550(8)	0.0027(7)	0.0304(7)	–0.0033(7)
C(19)	8*f*	0.3439(2)	1.3350(2)	0.1725(2)	0.104(2)	0.064(1)	0.075(1)	–0.019(1)	0.039(1)	–0.010(1)
N(1)	8*f*	0.1042(1)	0.7601(2)	–0.1930(1)	0.107(1)	0.103(1)	0.086(1)	–0.018(1)	0.064(1)	–0.035(1)
N(2)	8*f*	0.00469(9)	1.0335(1)	–0.1185(1)	0.0700(9)	0.073(1)	0.090(1)	0.0222(8)	0.0350(8)	0.0180(9)
O(1)	8*f*	0.18950(8)	0.6579(1)	0.0368(1)	0.1004(9)	0.0535(7)	0.131(1)	0.0201(6)	0.0912(9)	0.0094(7)
O(2)	8*f*	0.2879(1)	1.3185(1)	0.2018(1)	0.105(1)	0.0541(7)	0.0927(9)	–0.0076(7)	0.0487(8)	–0.0201(7)
O(3)	8*f*	0.34625(8)	1.2378(1)	0.1247(1)	0.0889(9)	0.0647(8)	0.107(1)	–0.0234(7)	0.0578(8)	–0.0153(7)

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