Crystal structure of 2-benzoyl-3-(3,4-dichlorophenyl)cyclopropane-1,1-dicarbonitrile, $C_{18}H_{10}Cl_2N_2O$

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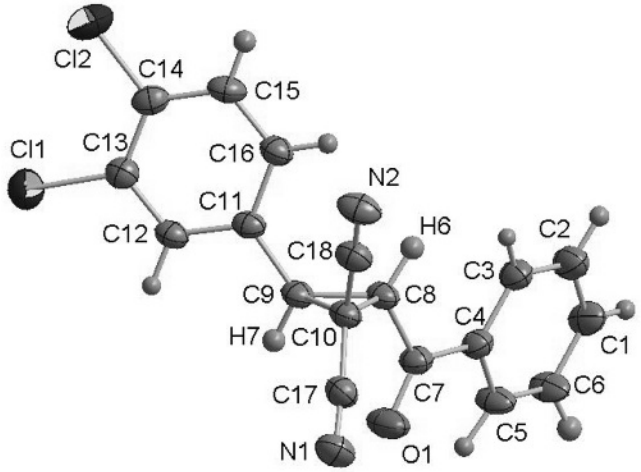

Abstract

$C_{18}H_{10}Cl_2N_2O$, triclinic, $P\bar{1}$ (no. 2), a = 6.8195(5) Å, b = 9.6727(8) Å, c = 13.268(1) Å, α = 98.491(2)°, β = 100.571(2)°, γ = 107.434(2)°, V = 801.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.0436, $wR_{ref}(F^2)$ = 0.1224, 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:	4.10 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	56.86°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6825, 4013
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2680
$N(param)_{refined}$:	248
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-(3,4-dichlorobenzylidene)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, 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 individual displacement parameters were refined.

Discussion

The synthesis and application of multisubstituted cyclopropanes have been subjects of great interest because of the roles of cyclopropane units 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–C10, and C9–C10 bond lengths are 1.532(2), 1.540(3) Å, respectively. The C8–C9 bond length is with 1.493(3) 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 61.17(12)°, 60.67(13)° and 58.16(12)°, only slightly deviating from the ideal 60°. With respect to the plane of the three-membered ring, 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(6)	2*i*	0.188(3)	0.609(2)	0.235(2)	0.044(5)
H(8)	2*i*	0.111(3)	0.924(2)	0.511(2)	0.050(5)
H(7)	2*i*	–0.025(3)	0.790(2)	0.336(1)	0.042(5)
H(9)	2*i*	0.653(3)	0.728(2)	0.565(2)	0.068(6)
H(3)	2*i*	0.304(3)	0.610(2)	0.105(2)	0.073(7)
H(10)	2*i*	0.403(3)	0.645(2)	0.407(2)	0.054(6)
H(4)	2*i*	–0.089(4)	0.824(3)	–0.004(2)	0.087(8)
H(2)	2*i*	0.486(4)	0.662(3)	–0.026(2)	0.092(8)
H(1)	2*i*	0.380(4)	0.790(3)	–0.147(2)	0.090(8)
H(5)	2*i*	0.078(5)	0.851(4)	–0.150(3)	0.14(1)

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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(2)	2*i*	0.3717(4)	0.6991(3)	–0.0212(2)	0.060(1)	0.112(2)	0.054(1)	0.040(1)	0.017(1)	0.020(1)
Cl(1)	2*i*	0.35358(9)	1.06310(7)	0.71230(5)	0.0780(4)	0.0709(4)	0.0586(3)	0.0319(3)	0.0159(3)	0.0062(3)
Cl(2)	2*i*	0.72946(9)	0.93401(7)	0.74742(5)	0.0635(3)	0.0852(4)	0.0677(4)	0.0224(3)	–0.0088(3)	0.0173(3)
C(12)	2*i*	0.2247(3)	0.8823(2)	0.5211(2)	0.0444(9)	0.050(1)	0.053(1)	0.0248(8)	0.0152(8)	0.0202(9)
C(13)	2*i*	0.3744(3)	0.9313(2)	0.6155(2)	0.050(1)	0.047(1)	0.050(1)	0.0177(8)	0.0149(9)	0.0178(9)
O(1)	2*i*	–0.1875(2)	0.7192(2)	0.1419(1)	0.0601(8)	0.106(1)	0.066(1)	0.0527(9)	0.0207(7)	0.0380(9)
C(9)	2*i*	0.0611(3)	0.7263(2)	0.3436(2)	0.0466(9)	0.052(1)	0.050(1)	0.0276(8)	0.0125(8)	0.0188(9)
C(8)	2*i*	0.0694(3)	0.6449(2)	0.2405(2)	0.0450(9)	0.059(1)	0.048(1)	0.0290(9)	0.0144(8)	0.0218(9)
C(7)	2*i*	–0.0239(3)	0.6918(2)	0.1447(2)	0.049(1)	0.060(1)	0.047(1)	0.0266(9)	0.0085(8)	0.0181(9)
C(16)	2*i*	0.3973(3)	0.7176(2)	0.4582(2)	0.050(1)	0.055(1)	0.056(1)	0.0287(9)	0.0145(9)	0.017(1)
C(11)	2*i*	0.2324(3)	0.7755(2)	0.4417(2)	0.0426(9)	0.050(1)	0.047(1)	0.0219(8)	0.0130(8)	0.0216(9)
C(14)	2*i*	0.5364(3)	0.8726(2)	0.6307(2)	0.046(1)	0.053(1)	0.053(1)	0.0149(8)	0.0062(9)	0.021(1)
C(4)	2*i*	0.0921(3)	0.7137(2)	0.0616(1)	0.047(1)	0.057(1)	0.041(1)	0.0199(8)	0.0061(8)	0.0136(9)
C(15)	2*i*	0.5470(3)	0.7666(2)	0.5529(2)	0.046(1)	0.061(1)	0.066(1)	0.0285(9)	0.010(1)	0.024(1)
C(3)	2*i*	0.2665(3)	0.6699(3)	0.0567(2)	0.056(1)	0.078(2)	0.044(1)	0.033(1)	0.0097(9)	0.017(1)
C(1)	2*i*	0.3072(4)	0.7725(4)	–0.0936(2)	0.075(2)	0.131(3)	0.062(2)	0.039(2)	0.028(1)	0.045(2)
C(5)	2*i*	0.0271(4)	0.7865(3)	–0.0138(2)	0.078(2)	0.129(2)	0.074(2)	0.063(2)	0.029(1)	0.059(2)
C(6)	2*i*	0.1349(5)	0.8163(4)	–0.0899(2)	0.109(2)	0.171(3)	0.085(2)	0.082(2)	0.045(2)	0.084(2)
C(10)	2*i*	–0.0804(3)	0.5628(2)	0.3019(2)	0.0439(9)	0.056(1)	0.049(1)	0.0264(8)	0.0150(8)	0.0189(9)
N(1)	2*i*	–0.4876(3)	0.4999(2)	0.2530(2)	0.050(1)	0.083(1)	0.080(1)	0.0283(9)	0.018(1)	0.017(1)
C(17)	2*i*	–0.3087(3)	0.5292(2)	0.2709(2)	0.053(1)	0.060(1)	0.055(1)	0.0283(9)	0.0196(9)	0.016(1)
N(2)	2*i*	0.0399(3)	0.3704(2)	0.3884(2)	0.065(1)	0.073(1)	0.099(2)	0.035(1)	0.031(1)	0.048(1)
C(18)	2*i*	–0.0181(3)	0.4523(2)	0.3495(2)	0.048(1)	0.057(1)	0.062(1)	0.0237(9)	0.0212(9)	0.023(1)

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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–H 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.