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Crystal structure of 3,10-bis(4-chlorophenyl)-6,12-dibenzyl-2,9-acetyl-6,12-diazapentacyclo[6.3.1.0^{2,7}.0^{4,11}.0^{5,9}]-dodecane, C₄₀H₃₆Cl₂N₂O₂

Table 1: Data collection and handling.

Crystal:	Colorless orthorhombic
Size:	0.26 × 0.22 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.231 cm ⁻¹
Diffractometer, scan mode:	Rigaku XtaLAB P200, ϕ and ω
2 θ max, completeness:	55.8°, 99.91%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	28826, 8023, 0.0531
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6441
$N(\text{param})_{\text{refined}}$:	418
Programs:	Rigaku [1], CrysAlis ^{PRO} [2], Olex2 [3], SHELX [4, 5]

<https://doi.org/10.1515/ncrs-2019-0834>

Received November 13, 2019; accepted December 12, 2019; available online January 18, 2020

Abstract

C₄₀H₃₆Cl₂N₂O₂, orthorhombic, *Pbca* (no. 61), $a = 10.6137(2)$ Å, $b = 20.1559(4)$ Å, $c = 31.4991(5)$ Å, $V = 6738.6(2)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0471$, $wR_{\text{ref}}(F^2) = 0.1242$, $T = 128$ K.

CCDC no.: 1936098

The structure of the title crystal is shown in the figure. The measurement methods and detail parameters are in Table 1 and Table 2.

Source of materials

The crystal of the title compound was synthesized by a [2+2] photocycloaddition reaction. 3-Acetyl-*N*-benzyl-4-chlorophenyl-1,4-dihydropyridin was irradiated under 365 nm UV-light in methanol/THF solvent for 21 days. The mixture thus obtained was then purified by Sephadex LH-20

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	3160.1(6)	4910.2(3)	6045.5(2)	53.03(16)
Cl2	5163.8(7)	1050.7(3)	1992.9(2)	69.1(2)
O1	976.9(12)	4393.1(7)	3876.9(4)	43.6(3)
O2	2278.9(12)	1272.1(6)	4159.7(4)	37.9(3)
N1	2711.2(12)	2889.8(6)	3412.5(4)	24.2(3)
N2	2660.1(12)	2973.6(6)	4543.5(4)	22.9(3)
C1	3552.2(14)	3421.4(7)	3554.1(4)	23.5(3)
C2	2887.9(14)	3761.5(7)	3948.9(4)	23.2(3)
C3	4010.4(14)	3922.0(7)	4247.4(5)	24.2(3)
C4	4704.2(14)	3249.9(7)	4220.3(5)	23.8(3)
C5	4779.2(14)	3102.1(7)	3742.1(5)	23.3(3)
C6	2366.5(14)	2583.5(7)	3817.5(4)	23.3(3)
C7	2108.2(14)	3171.1(7)	4137.7(4)	23.4(3)
C8	3849.0(14)	2682.7(7)	4407.8(4)	22.8(3)
C9	3549.2(14)	2227.0(7)	4008.7(5)	23.6(3)
C10	4715.1(14)	2337.8(7)	3725.9(5)	24.9(3)
C11	2100.0(16)	4358.8(8)	3814.4(5)	29.8(3)
C12	2782(2)	4920.0(8)	3595.2(6)	41.4(4)
C13	1615.0(15)	3121.3(8)	3170.7(5)	28.1(3)
C14	1990.6(16)	3438.5(8)	2754.2(5)	29.5(3)
C15	2969.6(17)	3175.4(9)	2513.8(5)	34.8(4)
C16	3278.6(19)	3448.4(11)	2122.5(6)	45.9(5)
C17	2622(2)	3984.3(12)	1966.7(6)	56.8(6)
C18	1657(2)	4257.1(12)	2203.4(7)	57.1(6)
C19	1344.5(19)	3988.4(10)	2598.8(6)	42.8(4)
C20	3753.6(15)	4179.6(7)	4694.4(5)	26.7(3)
C21	4694.2(16)	4117.7(8)	5002.6(5)	30.4(3)
C22	4525.1(17)	4343.8(8)	5416.2(5)	33.9(4)

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C23	3404.6(18)	4646.5(8)	5522.0(5)	34.4(4)
C24	2462.6(18)	4734.4(9)	5223.2(5)	36.9(4)
C25	2642.4(17)	4499.9(8)	4811.5(5)	33.7(4)
C26	3329.4(16)	1510.3(8)	4142.4(5)	28.7(3)
C27	4458.5(18)	1099.9(8)	4264.0(6)	40.5(4)
C28	1835.8(15)	2552.8(8)	4804.4(5)	27.5(3)
C29	2482.8(15)	2379.7(8)	5217.1(5)	29.2(3)
C30	2884.9(16)	1735.7(9)	5301.7(6)	37.0(4)
C31	3481.7(18)	1588.2(12)	5683.0(7)	49.4(5)
C32	3681(2)	2079.3(14)	5977.3(6)	56.6(6)
C33	3297(2)	2727.6(13)	5896.9(6)	51.5(5)
C34	2697.0(17)	2877.1(10)	5514.8(5)	38.0(4)
C35	4775.0(15)	2005.5(8)	3292.9(5)	27.2(3)
C36	3877.6(17)	1555.0(8)	3146.3(5)	33.1(4)
C37	3997.1(19)	1249.1(9)	2750.2(6)	38.8(4)
C38	5027(2)	1403.2(9)	2498.3(6)	41.2(4)
C39	5936.8(19)	1840.2(9)	2637.1(6)	41.6(4)
C40	5812.2(17)	2134.0(9)	3033.9(6)	35.7(4)

(C:M = 10:1, V/V) chromatography and the title compound was obtained. The title compound was recrystallized in acetone by slow evaporation after three days yielding colorless crystals.

Experimental details

A suitable crystal was selected and on a Rigaku Saturn 70 diffractometer. The crystal was kept at 128 K during data collection. Using Olex2 [3], the structure was solved with the ShelXT [4] structure solution program using intrinsic phasing and refined with the ShelXL [5] refinement package. All hydrogens were placed in idealized positions and added using the riding models implemented in the SHELX system.

Discussion

The title compound is a derivative of 6,12-diazatetrakisbomocubanes which have been reported to have interesting pharmacological effects: these include the carcinogenic effect of phenyl-substituted cubanes and substituted bishomocubanes and the recently observed anti-HIV effect of carboxylated cubanes [6–8]. Only a small number of structures of 6,12-diazatetrakisbomocubanes have been reported so far [6–9].

The X-ray diffraction analysis revealed that the compound crystallises in the orthorhombic crystal system. The two piperidine rings adopt a boat conformation, the apexes

(prows) being the N1, C7 and N2, C10 atoms, and the five-membered rings adopt an envelope conformation in the compound. Selected torsion angles include C1–N1–C13–C14: 64.85°, C6–N1–C13–C14: 179.78°, C7–N2–C28–C29: 62.45°, C8–N2–C28–C29: –179.57°. The bond lengths and bond angles are in the normal range and the overall molecular geometry is very similar to that of the unchlorinated analogue, which was crystallised as an acetone solvate [9, 10]. The structure is stabilised by several C–H···O hydrogen bonds.

Acknowledgements: We gratefully acknowledge support by the Natural Science Foundation of Hebei Province (H2019209496, H2019209571, H2015209094).

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