Crystal structure of 2*a*,4-diphenyl-5-benzoyl- 2,2-dichloro-2*a*,3,4,5-tetrahydroazeto[1,2-*a*][1,5]benzodiazepin-1(2*H*)-one, $C_{30}H_{22}Cl_2N_2O_2$

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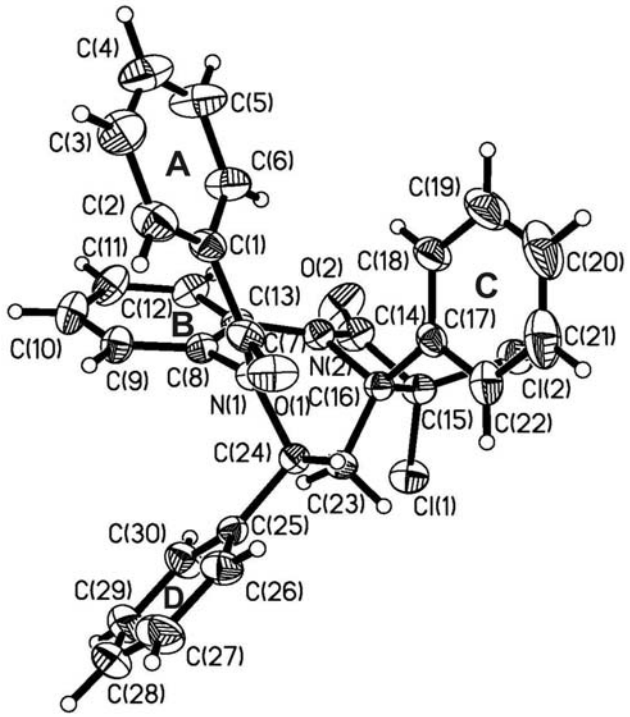

Abstract

$C_{30}H_{22}Cl_2N_2O_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.332(2) Å, b = 10.506(2) Å, c = 12.784(3) Å, α = 84.589(7)°, β = 86.443(7)°, γ = 87.747(7)°, V = 1244.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.047, $wR_{ref}(F^2)$ = 0.143, T = 296 K.

Source of material

The title compound was synthesized according to a published procedure [1]. 2,4-Diphenyl-1-benzoyl-1,5-benzodiazepine (1 mmol) and dichloroacetyl chloride (2 mmol) were stirred in anhydrous toluene (20 ml). Dried triethylamine (0.202 g, 2 mmol) in anhydrous toluene (10 ml) was added dropwise into the first solution over a period of 20 min, and the mixture was allowed to react at room temperature. After the addition was completed, the mixture was stirred at room temperature for further 5 h and cooled. The crystalline triethylamine hydrochloride formed was removed by filtration. The toluene solution was washed with water, saturated aqueous $NaHCO_3$, and then brine. The separated organic layer was dried over Na_2SO_4. The toluene was removed on the rotary evaporator at reduced pressure, the precipitate was recrystallized from methanol, and colorless single crystals suitable for X-ray diffraction were obtained.

Discussion

In the title crystal structure, the bond lengths of C7—O1 and C14—O2 are 1.216(2) Å and 1.191(3) Å, respectively, which is consistent with the values of the C=O double bond in carbonyl compounds [2]. Four benzene rings **A** (C1/C2/C3/C4/ C5/C6), **B** (C8/C9/C10/C11/C12/C13), **C** (C17/C18/C19/C20/C21/C22) and **D** (C25/C26/C27/C28/C29/C30) are not coplanar, with the dihedral angles of 60.2°, 106.5°, 109.3°, 79.2°, 50.7° and 51.1° for **AB**, **AC**, **AD**, **BC**, **BD** and **CD**, respectively. The conformation of seven-membered ring (C8/C13/N2/C16/C23/C24/ N1) is a twist chair, while the conformation of the four-membered ring (N2/C14/C15/C16) is a distorted square. There are three types of intramolecular hydrogen bonds: C12–H12···O2 (d(C12–H12···O2) = 3.0644(3) Å, ∠C12–H12···O2 = 123°), C18–H18···N2 (d(C18–H18···N2) = 2.8266(3) Å, ∠C18–H18···N2 = 103°), C24–H24···O1 (d(C24–H24···O1) = 2.6851(3) Å, ∠C24–H24···O1 = 102°). The weak intermolecular hydrogen bond C5–H5···Cl2 (d(C5–H5···Cl2) = 3.6013(3) Å, ∠C5– H5···Cl2 = 142°) assembles the molecules into a 3D network.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.23 × 0.24 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.92 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	55.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11208, 5772
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3999
$N(param)_{refined}$:	325
Programs:	SHELXS-97, SHELXL-97, SHELXTL [3]

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

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	0.0706	0.5368	0.1358	0.081
H(3)	2*i*	0.1728	0.4393	–0.0076	0.104
H(4)	2*i*	0.3657	0.5259	–0.1028	0.112
H(5)	2*i*	0.4659	0.7033	–0.0528	0.110
H(6)	2*i*	0.3732	0.7975	0.0932	0.079
H(9)	2*i*	0.3282	0.5662	0.3376	0.061
H(10)	2*i*	0.5652	0.4953	0.3424	0.076
H(11)	2*i*	0.7455	0.6376	0.3033	0.078
H(12)	2*i*	0.6923	0.8530	0.2742	0.064
H(18)	2*i*	0.3764	1.0353	0.0965	0.079

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(19)	2*i*	0.2456	1.0959	–0.0485	0.116
H(20)	2*i*	0.0134	1.1704	–0.0277	0.128
H(21)	2*i*	–0.0913	1.1865	0.1376	0.113
H(22)	2*i*	0.0366	1.1273	0.2838	0.079
H(23A)	2*i*	0.1584	1.0390	0.4270	0.051
H(23B)	2*i*	0.2925	0.9482	0.4490	0.051

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(24)	2*i*	0.0512	0.8973	0.3417	0.046
H(26)	2*i*	–0.0992	0.7456	0.4220	0.067
H(27)	2*i*	–0.1684	0.6016	0.5602	0.095
H(28)	2*i*	–0.0126	0.5405	0.6895	0.098
H(29)	2*i*	0.2056	0.6326	0.6854	0.090
H(30)	2*i*	0.2787	0.7725	0.5446	0.072

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)	2*i*	0.2133(2)	0.6759(2)	0.1309(2)	0.053(1)	0.053(1)	0.037(1)	–0.0063(9)	–0.0081(8)	–0.0050(9)
C(2)	2*i*	0.1520(3)	0.5700(3)	0.0992(2)	0.082(2)	0.071(2)	0.052(1)	–0.025(1)	–0.010(1)	–0.008(1)
C(3)	2*i*	0.2120(4)	0.5128(3)	0.0122(2)	0.125(3)	0.072(2)	0.069(2)	–0.015(2)	–0.020(2)	–0.029(2)
C(4)	2*i*	0.3273(4)	0.5634(4)	–0.0438(2)	0.105(2)	0.110(3)	0.067(2)	0.005(2)	0.006(2)	–0.041(2)
C(5)	2*i*	0.3867(4)	0.6688(4)	–0.0140(2)	0.088(2)	0.123(3)	0.066(2)	–0.020(2)	0.023(2)	–0.038(2)
C(6)	2*i*	0.3311(3)	0.7255(3)	0.0732(2)	0.071(2)	0.076(2)	0.053(1)	–0.021(1)	0.009(1)	–0.018(1)
C(7)	2*i*	0.1392(2)	0.7396(2)	0.2197(2)	0.044(1)	0.056(1)	0.037(1)	–0.0109(9)	–0.0057(8)	–0.0026(9)
C(8)	2*i*	0.3662(2)	0.7524(2)	0.3021(2)	0.039(1)	0.045(1)	0.038(1)	–0.0032(8)	–0.0046(7)	–0.0084(8)
C(9)	2*i*	0.4010(2)	0.6245(2)	0.3241(2)	0.052(1)	0.045(1)	0.058(1)	–0.0032(9)	–0.010(1)	–0.0097(9)
C(10)	2*i*	0.5428(3)	0.5817(2)	0.3265(2)	0.059(1)	0.050(1)	0.085(2)	0.009(1)	–0.022(1)	–0.017(1)
C(11)	2*i*	0.6502(2)	0.6670(2)	0.3053(2)	0.045(1)	0.069(2)	0.087(2)	0.010(1)	–0.017(1)	–0.029(1)
C(12)	2*i*	0.6185(2)	0.7956(2)	0.2871(2)	0.038(1)	0.063(1)	0.062(1)	–0.006(1)	–0.0046(9)	–0.020(1)
C(13)	2*i*	0.4762(2)	0.8402(2)	0.2877(2)	0.040(1)	0.049(1)	0.039(1)	–0.0044(8)	–0.0041(8)	–0.0096(8)
C(14)	2*i*	0.5303(3)	1.0716(2)	0.2897(2)	0.051(1)	0.059(1)	0.069(2)	–0.019(1)	0.009(1)	–0.016(1)
C(15)	2*i*	0.4021(2)	1.1561(2)	0.3225(2)	0.054(1)	0.046(1)	0.051(1)	–0.0169(9)	0.0043(9)	–0.0106(9)
C(16)	2*i*	0.3053(2)	1.0421(2)	0.3010(2)	0.043(1)	0.038(1)	0.040(1)	–0.0065(8)	0.0001(8)	–0.0044(8)
C(17)	2*i*	0.2189(2)	1.0736(2)	0.2053(2)	0.062(1)	0.036(1)	0.043(1)	–0.0074(9)	–0.0087(9)	–0.0014(8)
C(18)	2*i*	0.2818(3)	1.0654(2)	0.1054(2)	0.097(2)	0.058(1)	0.043(1)	–0.005(1)	–0.006(1)	–0.002(1)
C(19)	2*i*	0.2035(5)	1.1019(3)	0.0188(2)	0.162(4)	0.084(2)	0.046(2)	–0.006(2)	–0.027(2)	–0.001(1)
C(20)	2*i*	0.0655(5)	1.1463(3)	0.0311(3)	0.159(4)	0.076(2)	0.090(3)	0.003(2)	–0.071(3)	0.006(2)
C(21)	2*i*	0.0032(4)	1.1557(3)	0.1295(3)	0.095(2)	0.073(2)	0.116(3)	0.012(2)	–0.052(2)	0.004(2)
C(22)	2*i*	0.0793(3)	1.1201(2)	0.2169(2)	0.068(2)	0.054(1)	0.074(2)	0.005(1)	–0.018(1)	0.001(1)
C(23)	2*i*	0.2245(2)	0.9771(2)	0.3967(2)	0.045(1)	0.045(1)	0.038(1)	–0.0085(8)	0.0014(8)	–0.0052(8)
C(24)	2*i*	0.1404(2)	0.8628(2)	0.3717(1)	0.0330(9)	0.041(1)	0.040(1)	–0.0028(8)	–0.0020(7)	–0.0028(8)
C(25)	2*i*	0.0979(2)	0.7743(2)	0.4678(2)	0.039(1)	0.040(1)	0.044(1)	–0.0012(8)	0.0042(8)	–0.0050(8)
C(26)	2*i*	–0.0360(2)	0.7220(2)	0.4745(2)	0.050(1)	0.065(1)	0.053(1)	–0.016(1)	0.007(1)	–0.005(1)
C(27)	2*i*	–0.0777(3)	0.6359(3)	0.5570(2)	0.080(2)	0.080(2)	0.075(2)	–0.032(2)	0.021(2)	–0.002(2)
C(28)	2*i*	0.0143(4)	0.6007(3)	0.6345(2)	0.101(2)	0.062(2)	0.073(2)	–0.008(2)	0.032(2)	0.013(1)
C(29)	2*i*	0.1448(3)	0.6537(3)	0.6310(2)	0.083(2)	0.077(2)	0.060(2)	0.016(2)	0.002(1)	0.017(1)
C(30)	2*i*	0.1878(3)	0.7388(2)	0.5473(2)	0.048(1)	0.071(2)	0.058(1)	–0.002(1)	–0.002(1)	0.012(1)
Cl(1)	2*i*	0.41130(7)	1.18357(6)	0.45603(5)	0.0656(4)	0.0702(4)	0.0571(3)	–0.0170(3)	–0.0080(3)	–0.0188(3)
Cl(2)	2*i*	0.37667(8)	1.30330(5)	0.24788(5)	0.0988(5)	0.0438(3)	0.0621(4)	–0.0207(3)	0.0131(3)	–0.0048(2)
N(1)	2*i*	0.2200(2)	0.7924(2)	0.2897(1)	0.0353(8)	0.0425(8)	0.0388(8)	–0.0049(6)	–0.0035(6)	–0.0064(7)
N(2)	2*i*	0.4442(2)	0.9726(2)	0.2777(1)	0.0398(9)	0.0439(9)	0.0489(9)	–0.0091(7)	0.0030(7)	–0.0059(7)
O(1)	2*i*	0.0087(2)	0.7470(2)	0.2275(1)	0.0411(9)	0.098(1)	0.0520(9)	–0.0132(8)	–0.0061(6)	–0.0181(8)
O(2)	2*i*	0.6569(2)	1.0846(2)	0.2815(2)	0.049(1)	0.084(1)	0.152(2)	–0.0267(9)	0.021(1)	–0.043(1)

References

1. Xu, J. X.; Guo, Z.; Wing, L. C.: Reactions of 2,3-dihydro-1*H*-1,5-benzodiazepines and chloroacetyl chlorides: Synthesis of 2*a*,3,4,5-tetrahydroazeto[1,2-*a*][1,5]benzodiazepin-1(2*H*)-ones. Heteroatom. Chem. **7** (2001) 636-640.
2. Borowiak, B. T.; Wolska, I.: Structure of 6,6-diethylperhydro-1,4-diazepine. Acta Crystallogr. **C45** (1989) 936-938.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.