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Instrumental Achievements

Crystal Structure of 3,3-Dichloro-*N*-*p*-methoxyphenyl-4-(2-phenylstryl)-2-azetidinone

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The β-lactam ring (2-azetidinone) has a key role in the most widely employed class of antimicrobial agents. The activity and the selectivity of the β-lactam ring can be decisively influenced by the attached subtituents to the β-lactam ring[1] and depend on some quantitave geometrical parameters of β-lactam structures (such as the deviation of the N1 atom from the surrounding C atoms and the sum of the bond angles at the N1 atom).[2] Recently we reported some structural investigations which were made by changing the substituents around the β-lactam ring to determine whether the substituents change the activity and selectivity of the monocyclic β-lactams.[3] Here, we wish to report a new crystal structure of 3,3-dichloro-*N*-*p*-methoxyphenyl-4-(2-phenylstryl)-2-azetidinone ($C_{24}H_{19}Cl_2NO_2$) (Fig. 1).

The compound was prepared as follows. A solution of dichloroacetyl chloride (0.002 mol, 1.78 ml) in dry benzene (20 ml) was added dropwise over 1 h at room temperature to a mixture of β-phenylcinnamaldehyde *N*-*p*-methoxyphenylimine (0.001 mol, 0.313 g) and triethylamine (0.002 mol, 2.78 ml) in dry benzene. The mixture was stirred for 2 h at room temperature and amine salt was removed by filtration. The filtrate was washed with 5% HCl and water and dried over sodium sulfate. The title compound was crystallized from ethanol. Spectroscopic data for the title compound are as follows: ^{1}H-NMR, 3.783 (s, 3H); 4.917 (d, 1H); 6.066 (d, 1H); 7.427 (m, 14H) and IR, 1790 cm^{-1} (C=O); m.p. (C) = 135˚C.

The X-ray data were collected by a graphite-monochromated Mo K_α radiation (λ = 0.71069 Å). The crystal structure was solved by direct methods.[4] All the non-hydrogen atoms were refined anisotropically (hydrogen atoms were included but not refined). All hydrogen atoms were placed geometrically at the corresponding C atoms (except for the H which is located from difference Fourier map near the C9 atom). The crystal and experimental data are listed in Table 1. The final fractional

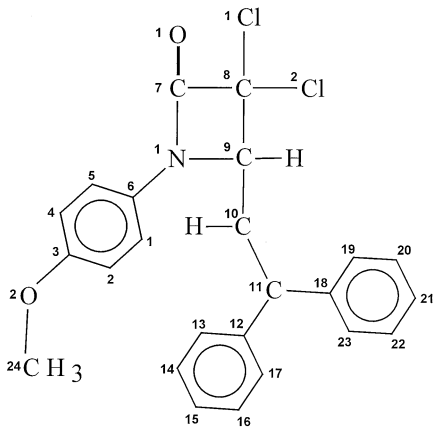

Fig. 1 Chemical structure of the title molecule.

Table 1 Crystal data and structure refinement for the title compound

Formula: $C_{24}H_{19}NO_2Cl_2$	
Formula weight: 424.3	
Crystal system: triclinic	
Space group: *P*-1	Z = 2
a = 11.513(2)Å	α = 92.712(9)˚
b = 11.724(2)Å	β = 101.298(9)˚
c = 8.6930(7)Å	γ = 68.431(8)˚
V = 1069.7(2)Å^3	
D_x = 1.317 g/cm^3	
F(0 0 0) = 440.0	
μ(Mo K_α) = 3.23 cm^{-1}	
R = 0.043	
wR^2 = 0.053	
$2\theta_{max}$ = 60˚	
$(\Delta/\sigma)_{max}$ = 0.00	
$(\Delta\rho)_{max}$ = 0.31 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.21 eÅ^{-3}	
No. of reflections used: 3609 ($I > 2.0\ \sigma(I)$)	
No. variables: 263	
Measurement: Rigaku AFC7S	
Program system: TEXSAN	
Structure determination: direct methods (SIR92)	
Refinement: full-matrix least-squares	

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Table 2 Final atomic fractional coordinates and equivalent isotropic displacement parameters for the title compound

Atom	*x*	*y*	*z*	B_{eq}
Cl1	0.80873(3)	0.40252(3)	0.96768(4)	6.053(9)
Cl2	0.70715(4)	0.39519(3)	0.63669(4)	6.62(1)
O1	1.00933(8)	0.19322(8)	0.7506(1)	6.59(3)
O2	0.98132(8)	-0.37339(8)	0.6611(1)	6.52(3)
N1	0.85785(8)	0.11766(8)	0.8071(1)	4.42(2)
C1	0.8293(1)	-0.0747(1)	0.8303(1)	5.48(3)
C2	0.8566(1)	-0.1968(1)	0.7943(1)	5.57(4)
C3	0.9473(1)	-0.2536(1)	0.7054(1)	4.87(3)
C4	1.0113(1)	-0.1880(1)	0.6522(1)	5.08(3)
C5	0.9836(1)	-0.0664(1)	0.6881(1)	4.57(3)
C6	0.8923(1)	-0.0091(1)	0.7767(1)	4.10(3)
C7	0.9103(1)	0.2015(1)	0.7836(1)	4.82(3)
C8	0.7898(1)	0.3070(1)	0.8109(1)	4.58(3)
C9	0.7362(1)	0.2066(1)	0.8415(1)	4.09(3)
C10	0.7134(1)	0.1916(1)	1.0006(1)	4.09(3)
C11	0.5996(1)	0.21574(9)	1.0356(1)	3.89(3)
C12	0.4799(1)	0.2762(1)	0.9211(1)	4.34(3)
C13	0.4579(1)	0.3871(1)	0.8470(1)	5.40(3)
C14	0.3471(2)	0.4431(1)	0.7407(2)	7.53(4)
C15	0.2574(2)	0.3916(2)	0.7058(2)	8.77(5)
C16	0.2764(1)	0.2825(2)	0.7781(2)	8.10(5)
C17	0.3871(1)	0.2256(1)	0.8861(1)	6.08(4)
C18	0.5873(1)	0.1823(1)	1.1932(1)	4.10(3)
C19	0.4895(1)	0.2573(1)	1.2643(1)	5.52(3)
C20	0.4772(1)	0.2244(1)	1.4100(2)	6.68(4)
C21	0.5602(2)	0.1170(2)	1.4836(1)	6.56(4)
C22	0.6579(1)	0.0412(1)	1.4150(1)	6.16(4)
C23	0.6715(1)	0.0737(1)	1.2704(1)	5.02(3)
C24	0.9260(1)	-0.4462(1)	0.7222(2)	7.60(5)

$B_{eq} = (8/3)\pi^2\Sigma_i\Sigma_j U_{ij} a_i a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j^*)$.

Table 3 Selected bond lengths (Å) and angles (˚) for the title molecule

Cl1-C8	1.753(2)
Cl2-C8	1.770(2)
O1-C7	1.199(2)
N1-C7	1.365(2)
N1-C6	1.415(2)
N1-C9	1.483(2)
C7-C8	1.531(3)
C8-C9	1.569(3)
C9-C10	1.490(2)
C6-N1-C7	132.6(2)
C6-N1-C9	129.1(2)
C7-N1-C9	97.0(2)
O1-C7-N1	133.7(2)
N1-C7-C8	90.8(2)
O1-C7-C8	135.4(2)
Cl1-C8-C7	116.7(2)
Cl2-C8-C7	111.3(1)
C7-C8-C9	87.0(2)
Cl1-C8-Cl2	110.6(1)
Cl1-C8-C9	116.6(1)
Cl2-C8-C9	112.8(1)
N1-C9-C10	114.4(2)
N1-C9-C8	85.2(1)
C8-C9-C10	119.3(2)

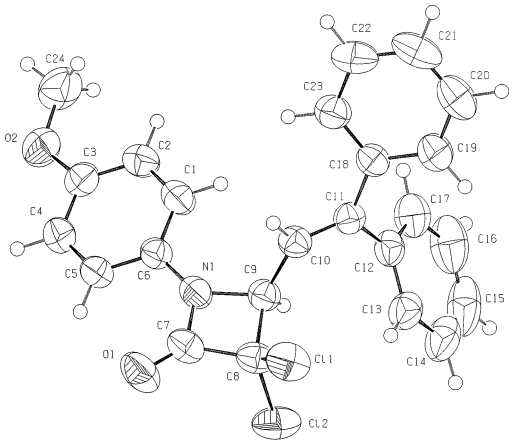

Fig. 2 Molecular structure of the title compound with the atom labeling. Thermal ellipsoids are drawn at the 50% probability level.

atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms are given in Table 2 and selected bond distances and bond angles are listed in Table 3. The molecular structure of the title molecule is shown in Fig. 2 with the atom-labeling schemes.

Brufani and Cella[2] concluded that, when the N1 atom is deviated 0.4 – 0.5 Å from the plane, surrounding C atoms at the β-lactam molecules could be biologically active. Here, the sum of the bond angles at the N1 atom (358.7), deviation of the N1 atom is 0.088 Å below the C6, C7, and C9 plane. The torsion angles of C6–N1–C9–C8 [167.0(2)˚] and C6–N1–C9–C8 [166.4(2)˚] support that there is no significant deviation of the N1 atom from the surrounding C atoms (0.088 Å). All these results indicate that our molecule is inactive. However, in the solid state, introduction of these substituents does not change the activity property of the β-lactam ring. There is neither intermolecular nor intramolecular proximity between molecules and atoms.

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