J. Cryst. Mol. Struct. (1977) 7, 95–106

Determination of the crystal and molecular structure of 1,4-bis(*p*-chlorobenzoyl)-5-methylene-7-methyl-2,3,4,5-tetrahydro-1*H*-1,4-diazepine

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(Received January 1, 1977)

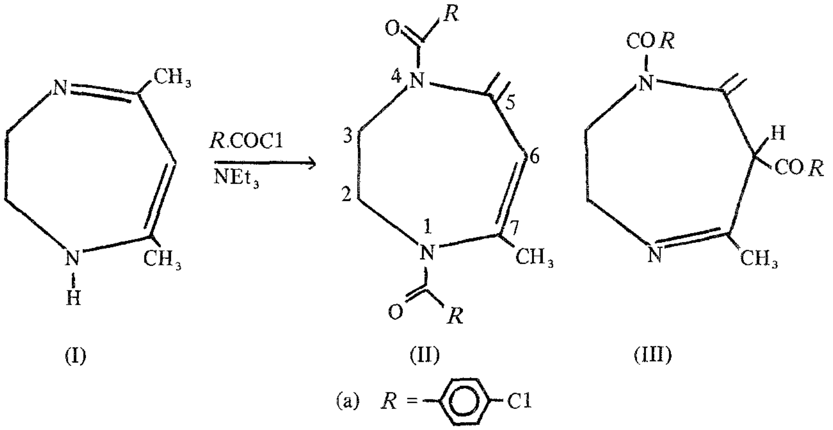
Abstract

The crystal and molecular structure of 1,4-bis(*p*-chlorobenzoyl)-5-methylene-7-methyl-2,3,4,5-tetrahydro-1*H*-1,4-diazepine has been determined by X-ray methods. The crystals are triclinic, $P\bar{1}$, $a = 10.605$, $b = 13.054$, $c = 7.351$ Å, $\alpha = 94.24$, $\beta = 101.53$, $\gamma = 101.55°$, $Z = 2$. The structure was solved by direct methods, and refined by full-matrix least squares to $R = 5.6\%$ on 3495 "observed" reflections. The structure is of the enamide form with diacylation on nitrogen. There are no abnormal bond lengths or intermolecular contacts.

Introduction

The acylation of 2,3-dihydro-5,7-dimethyl-1*H*-1,4-diazepine (I) was first studied by Lloyd and co-workers (1966). Using the Schotten–Baumann procedure, they attempted to benzoylate the perchloric acid salt of (I), but fragmentation of the ring occurred and only *sym*-dibenzoylethylenediamine was isolated. A similar ring opening occurs (Moore and Mitchell, 1967) with the corresponding 1*H*-1,5-benzodiazepine derivative under these conditions.

We have reinvestigated (Jarvis, 1970) the acylation of (I) in an effort to prepare 1-acyl derivatives which were required for photochemical studies. Under both hydrolytic and nonhydrolytic conditions it was found possible to retain the ring, but in all cases diacylation rather than monoacylation resulted independent of the acylating agent.



(I) (II) (III)

The diacylated products were tentatively assigned structure (II), there being ample precedent (Miescher et al., 1951; Breederveld, 1960; Anderson et al., 1974) for the formation of enamides from appropriately substituted imines. Although mechanistically reasonable, an alternative structure (III) involving substitution (Lloyd et al., 1974) at C(6) and on one of the nitrogens was considered less likely on the basis of the spectroscopic data. Diacylation on nitrogen has now been verified by X-ray crystallographic analysis of the bis-*p*-chlorobenzoyl derivative (IIa).

Experimental

The melting point was determined with a Yanagimoto micromelting point apparatus and the analysis was performed by the Australian Microanalytical Service, Melbourne.

The infrared spectrum was recorded with a Perkin-Elmer 577 grating spectrophotometer and the ultraviolet spectrum with a Cary 17 spectrophotometer. The pmr spectrum was run at 100 MHz with a Jeol JNM-4H-100 spectrometer using $CDCl_3$ as solvent and tetramethylsilane as internal standard. The mass spectrum was run at 70 eV on an A.E.I. MS903 mass spectrometer.

*1,4-Bis(*p*-chlorobenzoyl)-5-methylene-7-methyl-2,3,4,5-tetrahydro-1*H*-1,4 diazepine (IIa)*

To a stirred, ice-cooled solution of 2,3-dihydro-5,7-dimethyl-1*H*-1,4 diazepine (I) (10 g, 0.08 mol) and triethylamine (16.3 g, 0.16 mol) in the minimum quantity of ether, was added, in three portions over 1 hr, a solution of *p*-chlorobenzoyl chloride (28 g, 0.16 mol) in ether. The mixture was stirred for a further 12 hr at room temperature and then ice water was added. The precipitated product was filtered off, dried and crystallized from ethanol to afford the compound (IIa) (18.6 g, 58%), m.p. 152–153°C. Found: C, 62.8; H, 4.5; Cl, 17.2; N, 6.9; O, 8.3; M^+, 400.07269 (error, 4.6 ppm). $C_{21}H_{18}Cl_2N_2O_2$ requires C, 62.9; H, 4.5; Cl, 17.7; N, 7.0; O, 8.0%. Molecular weight (^{35}Cl) 400.0745. $\lambda_{\max}^{\mathrm{MeOH}}$ (ϵ) 225 (24,302), 255 nm (sh, 18,031). $\nu_{\max}^{\mathrm{nujol}}$ 1625 cm^{-1} ($c = 0$). Pmr (δ): 7.60–7.30 (m, 8H, aromatic protons), 5.78 (br.s, 1H, $=CH$), 5.00 and 4.64 (two br.s, 2H, $=CH_2$), 3.98 (m, 4H, CH_2–CH_2), and 1.83 (s, 3H, CH_3).

The crystals were triclinic and unit-cell parameters were obtained with a Philips PW1100 computer-controlled X-ray diffractometer. Crystal data are listed in Table 1; Cu *Kα* radiation ($\bar{\lambda} = 1.5418$ Å) was used throughout the experimental work.

Intensities were collected from a crystal having dimension 0.2, 0.4, 0.1 mm with a Philips PW1100 diffractometer and graphite monochromated Cu *Kα* radiation. The intensities of 3546 independent reflections were measured to $2\theta = 137°$. Of these, 3495 had $|F_o|^2 > 3\sigma(|F_o|^2)$; the remaining 51 were classified as unobserved and were omitted from the analysis. Three standard reflections, monitored every 2 h throughout data collection, showed no significant variation in intensity. Data were collected with a θ–2θ

Table 1. *Crystal data*

Molecular formula	$C_{21}H_{18}Cl_2N_2O_2$
FW	401.30
Crystal system	Triclinic
Space group	$P\bar{1}$
a	10.605(11) Å
b	13.054(14)
c	7.351(9)
α	94.24(10)°
β	101.53(10)
γ	101.55(10)
V_c	974.3 Å^3
Z	2
D_m	1.40 g cm^{-3}
D_x	1.37
F(000)	416
μ(Cu *Kα*)	14 cm^{-1}

Table 2. *Mean planes and deviations (Å)*[a]

		Δ		Δ
Plane 1	C(5)	0.061	C(8)	0.064
	C(6)	0.032	C(2)	−1.49
	C(7)	−0.066	C(3)	−0.876
	C(16)	0.019	N(1)	−0.441
	C(18)	−0.046	N(4)	0.229
			O(1)	−0.455
	$0.9651X + 0.2321Y + 0.1210Z = -4.0955$			
Plane 2	C(2)	−0.116	C(6)	−0.694
	C(7)	0.094	C(16)	1.130
	C(8)	0.019		
	C(11)	−0.113		
	N(1)	0.007		
	O(1)	0.108		
	$-0.8114X + 0.5835Y + 0.0354Z = 7.7253$			
Plane 3	C(3)	0.189	C(9)	−0.310
	C(5)	−0.222	C(6)	0.739
	C(13)	−0.034	C(2)	0.902
	C(20)	0.207		
	N(4)	0.090		
	O(2)	−0.229		
	$0.2869X + 0.8932Y - 0.3464Z = -1.4084$			
Plane 4	C(10)	0.003	C(8)	−0.094
	C(11)	0.009	N(1)	0.645
	C(12)	−0.008	O(1)	−0.863
	C(15)	0.007	Cl(1)	−0.002
	C(17)	0.011	H(101)	0.019
	C(23)	0.000	H(121)	−0.019
			H(231)	−0.071
			H(171)	0.005
	$-0.5427X + 0.3945Y + 0.7415Z = 7.4185$			
Plane 5	C(9)	0.002	C(13)	−0.248
	C(14)	−0.008	N(4)	0.126
	C(19)	0.002	O(2)	−0.868
	C(20)	−0.008	Cl(2)	−0.090
	C(21)	0.005	H(91)	0.004
	C(22)	0.005	H(191)	−0.025
			H(211)	0.039
			H(221)	−0.050
	$-0.3351X - 0.4304Y + 0.8381Z = 4.5671$			
Plane 6	C(5)	−0.008		
	C(18)	0.030		
	H(181)	−0.010		
	H(182)	−0.012		
	$0.9553X + 0.1869Y + 0.2289Z = -3.4353$			

Table 2–(*cont.*)

		Δ		Δ
Plane 7	C(5)	0.025	C(8)	0.317
	C(6)	0.025	C(13)	1.21
	C(7)	0.019	C(2)	−1.35
	C(16)	0.123	C(3)	−0.771
	C(18)	−0.177	O(1)	−0.138
	N(1)	−0.279	O(2)	1.09
	N(4)	0.263		

$$0.9614X + 0.2688Y + 0.0589Z = -4.3243$$

Angles between planes (deg)

1–2	50.0						
1–3	63.8	2–3	74.0				
1–4	70.0	2–4	45.8	3–4	86.6		
1–5	71.2	2–5	87.1	3–5	39.6	4–5	50.7
1–6	6.7	2–6	48.9	3–6	68.8	4–6	74.0
						5–6	77.9
1–7	4.1	2–7	51.6	3–7	60.3	4–7	68.2
				5–7	67.1	6–7	10.8

[a] The columns on the left-hand side refer to the atoms fitted, and the other columns refer to the deviations of other atoms from the planes. X is parallel to a, Y is perpendicular to the ac plane, and Z is perpendicular to a and lies in the ac plane.

scan and symmetric scan range of ±1.20° in 2θ from the calculated scattering angle at a scan rate of 0.0168° s^{-1}.

Intensity data were processed with a program (Hornstra and Stubbe) written specifically for the PW1100 diffractometer. The background-corrected intensities were assigned standard deviations according to the formula

$$\sigma(I) = [CT + (t_c/t_b)^2(B_1 + B_2) + (pI)^2]^{1/2}$$

where CT is the total integrated peak count obtained in scan time t_c, B_1 and B_2 are background counts each obtained in time t_b, and

$$I = CT - (t_c/t_b)(B_1 + B_2).$$

The value of p was selected as 0.04 to prevent unduly high weight being given to strong reflections. The values of I and $\sigma(I)$ were then corrected for Lorentz and polarization effects in the normal fashion. No correction for absorption was made.

Structure determination and refinement

The structure was solved by direct methods; $|E|$ values were calculated by the K-curve method. The statistics showed good agreement with those for the idealized centric distribution Thus, the space group $P\bar{1}$ was chosen and subsequently confirmed by analysis.

A Fourier map was computed with a set of 200 E values ($|E| \geqslant 1.5$). From this map, a total of 31 possible atom positions were identified. The two largest of these were assigned as chlorines and a further 26 initially treated as carbon atoms. After three cycles of least-squares refinement, four of the positions were assigned to N and O atoms from isotropic temperature factor and bond-length scans. The structure was then refined on $|F|$ by full-matrix anisotropic least squares. The hydrogen atoms were located from a difference synthesis. In particular, the methylene group on C(5) was identified unambiguously. The hydrogen atoms were subjected to isotropic refinement and corrections for anomalous dispersion were applied to the chlorine atoms. A satisfactory weighting scheme was obtained with the equation

$$W^{-1} = a + |F_o| + b|F_o{}^2| + c|F_o|^3$$

where $a = 6.005$, $b = -0.0020$ and $c = 1.4 \times 10^{-3}$. The least-squares refinement converged to an R of 5.6%. The scale factor for $|F_o|$ was 1.010. Dispersion corrections were taken from the *International Tables for X-ray Crystallography* (1974) and scattering factors from Cromer and Mann (1968) for C, N, O and Cl, and Stewart et al. (1965) for H.

Mean planes have been calculated through seven sets of atoms and are listed in Table 2. Computations were performed by local programs and the X RAY-72 system (1972). The atomic parameters are listed in Table 3, and bond lengths and angles in Table 4: Figure 1 illustrates the atomic numbering scheme. The reflections which were designated unobserved were calculated to be less than 3σ; they are included with the structure factor tables.

Discussion

Figure 2 is a stereographic illustration of the compound as seen along [001]. Clearly the structure is that of (IIa) with diacylation on nitrogen rather than the alternative enamide structure (IIIa).

The heterocyclic ring adopts a bent conformation with the nitrogen atoms and carbons 5, 6 and 7 being approximately coplanar. Both aryl rings are on the same side of this plane, with the amide carbonyl groups pointing away from the π-system in the ring. The bond lengths and bond angles are close to the standard values of these quantities, and there are no abnormal

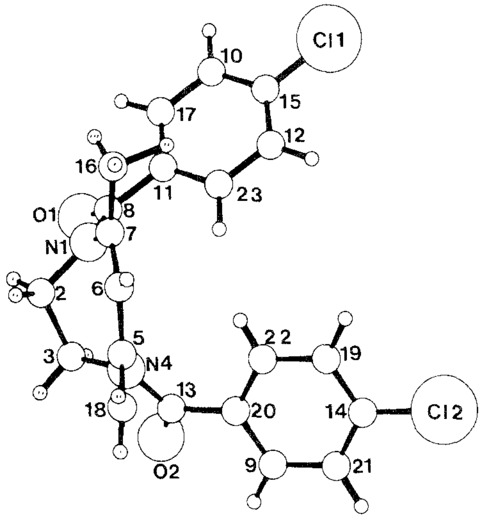


Fig. 1. Atom numbering scheme: carbon atoms have only numbers. The hydrogen atom numbering is described in footnote (b) to Table 3. Where there are two or more hydrogen atoms attached to one and the same carbon, the numbers are shown on the figure by the appropriate primes on the atom circle.

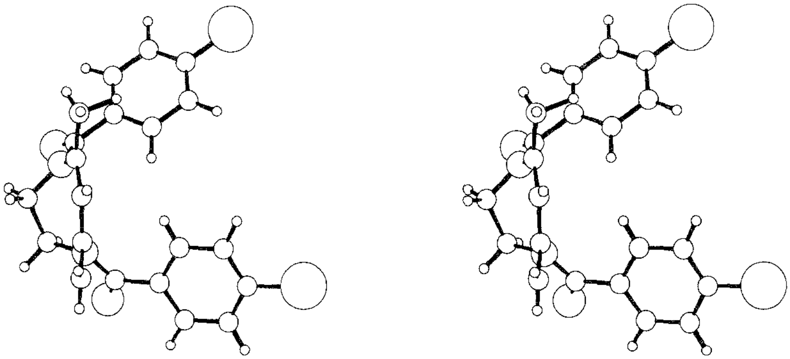


Fig. 2. Stereoview of the molecule, as seen along [001].

Table 3. *Final positional and thermal parameters,*[a] *with esd in parentheses*

Atom[b]	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	−0.5212(2)	0.2442(2)	0.5607(4)	0.032(1)	0.047(1)	0.050(1)	0.002(1)	0.004(1)	0.002(1)
C(3)	−0.4617(2)	0.1523(2)	0.5173(4)	0.039(1)	0.045(1)	0.048(1)	0.003(1)	−0.000(1)	−0.003(1)
C(5)	−0.3525(2)	0.1747(2)	0.8573(3)	0.032(1)	0.049(1)	0.041(1)	0.005(1)	0.019(1)	0.004(1)
C(6)	−0.3542(2)	0.2792(2)	0.9244(3)	0.044(1)	0.055(1)	0.037(1)	0.012(1)	0.014(1)	0.006(1)
C(7)	−0.3710(2)	0.3622(2)	0.8253(3)	0.038(1)	0.046(1)	0.038(1)	0.006(1)	0.013(1)	−0.000(1)
C(8)	−0.3768(2)	0.3955(2)	0.4947(3)	0.044(1)	0.045(1)	0.040(1)	0.009(1)	0.010(1)	0.004(1)
C(9)	−0.0434(3)	0.0455(2)	0.7170(4)	0.055(2)	0.054(2)	0.056(2)	0.018(1)	0.005(1)	−0.008(1)
C(10)	−0.1478(3)	0.6596(2)	0.5005(4)	0.064(2)	0.043(1)	0.057(2)	0.007(1)	0.015(1)	0.013(1)
C(11)	−0.2565(2)	0.4866(2)	0.5492(3)	0.043(1)	0.041(1)	0.038(1)	0.009(1)	0.011(1)	0.003(1)
C(12)	−0.0282(3)	0.5565(2)	0.7000(4)	0.042(1)	0.063(2)	0.051(1)	0.005(1)	0.011(1)	0.015(1)
C(13)	−0.2538(2)	0.1007(2)	0.6138(3)	0.042(1)	0.037(1)	0.046(1)	0.002(1)	0.014(1)	0.002(1)
C(14)	0.1438(3)	0.1578(2)	0.9182(4)	0.041(1)	0.070(2)	0.052(2)	0.018(1)	0.010(1)	0.006(1)
C(15)	−0.0341(3)	0.6470(2)	0.6188(4)	0.051(1)	0.052(1)	0.046(1)	−0.002(1)	0.019(1)	0.003(1)
C(16)	−0.3604(3)	0.4673(2)	0.9157(4)	0.066(2)	0.057(2)	0.048(2)	0.022(1)	0.019(1)	0.001(1)
C(17)	−0.2585(3)	0.5778(2)	0.4652(4)	0.051(1)	0.047(1)	0.052(1)	0.012(1)	0.008(1)	0.011(1)
C(18)	−0.3552(3)	0.0941(2)	0.9682(4)	0.057(2)	0.055(2)	0.056(2)	0.008(1)	0.018(1)	0.017(1)
C(19)	0.0708(3)	0.2314(2)	0.9462(4)	0.042(1)	0.054(1)	0.055(2)	0.007(1)	0.009(1)	−0.004(1)
C(20)	−0.1194(2)	0.1187(2)	0.7399(3)	0.038(1)	0.042(1)	0.046(1)	0.008(1)	0.013(1)	0.004(1)
C(21)	0.0883(3)	0.0645(3)	0.8063(5)	0.055(2)	0.068(2)	0.072(2)	0.031(1)	0.009(1)	−0.004(1)
C(22)	−0.0605(2)	0.2110(2)	0.8568(4)	0.040(1)	0.045(1)	0.056(1)	0.012(1)	0.013(1)	0.000(1)
C(23)	−0.1404(3)	0.4758(2)	0.6659(4)	0.046(1)	0.048(1)	0.050(1)	0.010(1)	0.013(1)	0.011(1)
O(1)	−0.4358(2)	0.3803(2)	0.3307(3)	0.067(1)	0.073(1)	0.042(1)	−0.005(1)	−0.002(1)	0.011(1)
O(2)	−0.2768(2)	0.0492(1)	0.4602(3)	0.047(1)	0.055(1)	0.052(1)	0.003(1)	0.008(1)	−0.015(1)
N(1)	−0.4178(2)	0.3408(2)	0.6274(3)	0.036(1)	0.040(1)	0.038(1)	0.004(1)	0.007(1)	0.000(1)
N(4)	−0.3458(2)	0.1507(2)	0.6659(3)	0.035(1)	0.042(1)	0.040(1)	0.007(1)	0.007(1)	−0.000(1)
Cl(1)	0.1048(1)	0.7486(1)	0.6597(1)	0.066(1)	0.072(1)	0.064(1)	−0.023(1)	0.011(1)	0.008(1)
Cl(2)	0.3115(1)	0.1882(1)	1.0243(1)	0.043(1)	0.108(1)	0.090(1)	0.026(1)	−0.004(1)	−0.002(1)

				U
H(101)	−0.1492(30)	0.7273(24)	0.4435(43)	0.063(9)
H(91)	−0.0843(34)	−0.0218(27)	0.6346(47)	0.074(10)
H(121)	0.0567(30)	0.5457(23)	0.7872(41)	0.059(8)
H(61)	−0.3374(30)	0.2944(24)	1.0527(43)	0.060(8)
H(21)	0.4202(28)	0.2575(22)	0.4550(40)	0.053(8)
H(22)	0.4281(29)	0.2335(23)	0.6616(41)	0.055(8)
H(191)	0.1102(32)	0.3009(25)	1.0259(46)	0.070(9)
H(211)	0.1397(37)	0.0079(29)	0.7886(52)	0.085(11)
H(221)	−0.1057(31)	0.2644(24)	0.8717(43)	0.065(9)
H(231)	−0.1423(34)	0.4035(27)	0.7151(48)	0.076(10)
H(171)	−0.3419(31)	0.5831(25)	0.3851(45)	0.067(9)
H(31)	−0.5303(34)	0.0834(27)	0.5006(47)	0.077(10)
H(32)	−0.4319(32)	0.1563(25)	0.4011(45)	0.067(9)
H(181)	−0.3616(33)	0.1112(26)	1.1051(47)	0.073(10)
H(182)	−0.3615(42)	0.0234(34)	0.9253(60)	0.109(14)
H(161)	−0.3553(31)	0.4693(24)	1.0388(43)	0.061(9)
H(162)	−0.2493(69)	0.5177(51)	0.9499(94)	0.187(25)
H(163)	−0.3980(45)	0.5091(35)	0.8433(63)	0.114(15)

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[a] The anisotropic temperature factor was of the form $\exp\{-2\pi^2 \lambda^{-2}(h^2 a^{*2} U_{11} + \ldots + 2hka^*b^* U_{12} + \ldots\}$; the isotropic temperature factor was of the form $8\pi^2 \lambda^{-2} U \sin^2 \theta$.

[b] In the hydrogen atom numbering scheme, the first digits (or the first two digits where there are three) indicate the numbers of the atoms to which the hydrogen atoms are bonded.

Table 4. *Bond lengths*[a] *(Å) and bond angles (deg), with esd in parentheses*

		C(15)–C(12)–H(121)	123(2)
		C(23)–C(12)–H(121)	118(2)
C(2)–C(3)	1.505(4)	C(13)–C(20)	1.500(3)
C(2)–N(1)	1.474(3)	C(13)–N(4)	1.376(4)
C(2)–H(21)	0.94(3)	C(13)–O(2)	1.226(3)
C(2)–H(22)	1.00(3)	C(20)–C(13)–N(4)	119.1(2)
C(3)–C(2)–N(1)	110.9(2)	C(20)–C(13)–O(2)	120(2)
C(3)–C(2)–H(21)	112(2)	N(4)–C(13)–O(2)	120.7(2)
C(3)–C(2)–H(22)	113(2)	C(14)–C(19)	1.378(5)
N(1)–C(2)–H(21)	108(2)	C(14)–C(21)	1.369(4)
N(1)–C(2)–H(22)	106(1)	C(14)–Cl(2)	1.746(3)
H(21)–C(2)–H(22)	107(3)	C(19)–C(14)–C(21)	121.5(3)
C(3)–N(4)	1.478(3)	C(19)–C(14)–Cl(2)	118.0(2)
C(3)–H(31)	1.02(3)	C(21)–C(14)–Cl(2)	120.5(3)
C(3)–H(32)	0.97(4)	C(15)–Cl(1)	1.732(3)
C(2)–C(3)–N(4)	110.7(2)	C(10)–C(15)–C(12)	122.0(2)
C(2)–C(3)–H(31)	110(2)	C(10)–C(15)–Cl(1)	118.3(2)
C(2)–C(3)–H(32)	111(2)	C(12)–C(15)–Cl(1)	119.7(2)
N(4)–C(3)–H(31)	111(2)	C(16)–H(161)	0.89(3)
N(4)–C(3)–H(32)	107(2)	C(16)–H(162)	1.20(7)
H(31)–C(3)–H(32)	106(3)	C(16)–H(163)	0.89(5)
C(5)–C(6)	1.421(4)	C(7)–C(16)–H(161)	113(2)
C(5)–C(18)	1.377(4)	C(7)–C(16)–H(162)	112(4)
C(5)–N(4)	1.437(3)	C(7)–C(16)–H(163)	115(3)
C(6)–C(5)–C(18)	123.0(2)	H(161)–C(16)–H(162)	88(4)
C(6)–C(5)–N(4)	119.4(2)	H(161)–C(16)–H(163)	125(4)
C(18)–C(5)–N(4)	117.6(2)	H(162)–C(16)–H(163)	99(4)
C(6)–C(7)	1.371(4)	C(17)–H(171)	0.98(3)
C(6)–H(61)	0.92(3)	C(10)–C(17)–C(11)	120.8(3)
C(5)–C(6)–C(7)	128.9(2)	C(10)–C(17)–H(171)	122(2)
C(5)–C(6)–H(61)	116(2)	C(11)–C(17)–H(171)	117.0(2)
C(7)–C(6)–H(61)	115(2)	C(18)–H(181)	1.03(4)
C(7)–C(16)	1.452(4)	C(18)–H(182)	0.94(4)
C(7)–N(1)	1.427(3)	C(5)–C(18)–H(181)	117(2)
C(6)–C(7)–C(16)	122.4(2)	C(5)–C(18)–H(182)	125(3)
C(6)–C(7)–N(1)	118.1(2)	H(181)–C(18)–H(182)	117(3)
C(16)–C(6)–N(1)	119.0(2)	C(19)–C(22)	1.379(4)
C(8)–C(11)	1.493(3)	C(19)–H(191)	1.01(3)
C(8)–N(1)	1.369(3)	C(14)–C(19)–C(22)	118.9(2)
C(8)–O(1)	1.224(3)	C(14)–C(19)–H(191)	123(2)
C(11)–C(8)–N(1)	119.7(2)	C(22)–C(19)–H(191)	118(2)
C(11)–C(8)–O(1)	119.3(2)	C(20)–C(22)	1.383(3)
N(1)–C(8)–O(1)	121.0(2)	C(9)–C(20)–C(13)	117.7(2)
C(9)–C(20)	1.389(4)	C(9)–C(20)–C(22)	118.3(2)
C(9)–C(21)	1.384(4)	C(13)–C(20)–C(22)	123.3(2)
C(9)–H(91)	0.99(3)	C(21)–H(211)	1.02(4)
C(20)–C(9)–C(21)	121.2(3)	C(9)–C(21)–C(14)	118.8(3)
C(20)–C(9)–H(91)	120.0(2)	C(9)–C(21)–H(211)	119(2)
C(21)–C(9)–H(91)	119(2)	C(14)–C(21)–H(211)	123(2)
C(10)–C(15)	1.385(4)	C(22)–H(221)	0.93(2)

Table 4.—*cont.*

C(10)–C(17)	1.386(4)	C(19)–C(22)–C(20)	121.3(3)
C(10)–H(101)	1.01(3)	C(19)–C(22)–H(221)	117.0(2)
C(15)–C(10)–C(17)	118.5(3)	C(20)–C(22)–H(221)	122.0(2)
C(15)–C(10)–H(101)	120(2)	C(23)–H(231)	1.03(4)
C(17)–C(10)–H(101)	121(2)	C(11)–C(23)–C(12)	120.4(3)
C(11)–C(23)	1.392(4)	C(11)–C(23)–H(231)	116(2)
C(11)–C(17)	1.384(4)	C(12)–C(23)–H(231)	124(2)
C(8)–C(11)–C(23)	122.8(3)	C(8)–N(1)–C(7)	127.2(2)
C(8)–C(11)–C(17)	117.6(2)	C(8)–N(1)–C(2)	117.2(2)
C(23)–C(11)–C(17)	119.3(2)	C(7)–N(1)–C(2)	115.6(2)
C(12)–C(15)	1.370(4)	C(5)–N(4)–C(13)	123.3(2)
C(12)–C(23)	1.388(4)	C(5)–N(4)–C(3)	118.5(2)
C(12)–H(121)	1.04(3)	C(13)–N(4)–C(3)	116.1(2)
C(15)–C(12)–C(23)	119.0(2)		

[a] See footnote *b* to Table 3.

intermolecular contact distances. The asymmetry of the double bonds causes the seven-membered ring to depart appreciably from *m* (c_s) symmetry; the amide groups, however, do maintain a planar conformation.

Acknowledgments

The authors wish to thank Dr. P. G. Leverett and Dr. B. M. Gatehouse of Monash University for the use of the crystallographic facilities, Dr. C. P. Whittle for assistance with the high resolution mass spectrum, and the Australian Research Grants Committee for a grant in partial support of this work. The award of a Commonwealth Scholarship (to W. R. C. Jarvis) is gratefully acknowledged.

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