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Structure of 1,1-Dichloro-2,2-bis(4-dimethylaminophenyl)ethylene

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Abstract. $C_{18}H_{20}Cl_2N_2$, $M_r = 335{\cdot}27$, monoclinic, $P2_1/c$, $a = 8{\cdot}091\,(4)$, $b = 6{\cdot}145\,(2)$, $c = 34{\cdot}384\,(10)$ Å, $\beta = 91{\cdot}50\,(3)°$, $V = 1709\,(1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}310$, $D_x = 1{\cdot}303$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}38$ mm^{-1}, $F(000) = 704$, $T = 297$ K, $R = 0{\cdot}046$ for 1053 observed reflections. The C—C ethylene bond distance is 1·328 (11) Å. The twist about the ethylenic bond is 7·1 (5)°. The C—Cl distances are 1·731 (9) and 1·743 (8) Å. One phenyl ring is planar and the other has an approximate boat conformation with a pseudo mirror plane.

Introduction. DDE [1,1-dichloro-2,2-bis(*p*-chlorophenyl)ethylene] is a stable primary degradation product of the important wide-spectrum insecticide DDT [1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane].

The X-ray structual study of the present compound, a structural analogue of DDE, was undertaken to provide additional stereochemical information and to study the effects of substitution on the ethylenic bond and the twist about this bond.

Experimental. Transparent, slightly yellowish needle-shaped crystals from alcohol, density by flotation (benzene–bromoform), space group $P2_1/c$, crystal $0{\cdot}14 \times 0{\cdot}24 \times 0{\cdot}55$ mm; cell parameters determined by least squares from setting angles of 15 reflections, $10 \leq 2\theta \leq 15°$, intensity data measured on a Syntex $P2_1$ diffractometer using graphite-monochromatized Mo $K\alpha$ radiation, 3550 unique reflections collected (*h*

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Table 1. *Fractional atomic coordinates with their e.s.d.'s in parentheses and equivalent isotropic temperature factors* (Å^2) *for the non*-H *atoms*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_i.$$

	x	y	z	B_{eq}
Cl(1)	0·5923 (3)	0·1442 (4)	0·3808 (1)	6·56
Cl(2)	0·4241 (3)	−0·0167 (4)	0·4467 (1)	6·25
N(1)	0·2191 (9)	−0·0001 (14)	0·2086 (2)	6·60
N(1′)	−0·1609 (8)	−0·5951 (12)	0·4553 (2)	5·61
C(1)	0·2404 (10)	−0·0182 (16)	0·2484 (2)	5·40
C(2)	0·1864 (11)	−0·1995 (16)	0·2688 (3)	5·45
C(3)	0·2103 (11)	−0·2169 (15)	0·3087 (3)	5·61
C(4)	0·2853 (9)	−0·0533 (13)	0·3312 (2)	4·60
C(5)	0·3350 (11)	0·1288 (16)	0·3111 (3)	5·79
C(6)	0·3155 (11)	0·1444 (18)	0·2711 (3)	6·20
C(7)	0·3022 (10)	−0·0846 (13)	0·3737 (2)	4·71
C(8)	0·4213 (10)	0·0016 (14)	0·3961 (2)	5·25
C(9)	0·2927 (17)	0·1787 (24)	0·1886 (4)	8·45
C(10)	0·1264 (16)	−0·1630 (21)	0·1874 (3)	7·00
C(1′)	−0·0528 (10)	−0·4826 (14)	0·4333 (2)	4·42
C(2′)	0·1085 (12)	−0·5515 (16)	0·4276 (2)	5·25
C(3′)	0·2204 (20)	−0·4305 (26)	0·4079 (4)	5·97
C(4′)	0·1773 (10)	−0·2235 (14)	0·3925 (2)	4·49
C(5′)	0·0156 (10)	−0·1613 (15)	0·3959 (2)	4·66
C(6′)	−0·0972 (11)	−0·2824 (15)	0·4158 (2)	4·56
C(9′)	−0·3130 (13)	−0·5009 (23)	0·4673 (4)	7·14
C(10′)	−0·1109 (15)	−0·7836 (19)	0·4779 (3)	6·63

Table 2. *Bond distances* (Å) *and angles* (°) *with their e.s.d.'s in parentheses*

Cl(1)—C(8)	1·731 (9)	C(2′)—C(3′)	1.365 (18)
Cl(2)—C(8)	1·743 (8)	C(3′)—C(4′)	1·418 (18)
C(7)—C(8)	1·328 (11)	C(4′)—C(5′)	1·371 (12)
C(7)—C(4)	1·477 (10)	C(5′)—C(6′)	1·374 (12)
C(7)—C(4′)	1·484 (11)	C(6′)—C(1′)	1·412 (12)
C(1)—C(2)	1·393 (13)	N(1)—(1)	1·379 (10)
C(2)—C(3)	1·385 (14)	N(1)—C(9)	1·434 (16)
C(3)—C(4)	1·397 (12)	N(1)—C(10)	1·438 (14)
C(4)—C(5)	1·381 (12)	N(1′)—C(1′)	1·360 (10)
C(5)—C(6)	1·384 (14)	N(1′)—C(9′)	1·430 (13)
C(6)—C(1)	1·397 (14)	N(1′)—C(10′)	1·447 (13)
C(1′)—C(2′)	1·391 (13)		
Cl(1)—C(8)—Cl(2)	110·3 (5)	C(6′)—C(1′)—N(1′)	121·3 (8)
Cl(1)—C(8)—C(7)	126·8 (6)	N(1′)—C(1′)—C(2′)	122·9 (8)
Cl(2)—C(8)—C(7)	122·9 (6)	C(2′)—C(1′)—C(6′)	115·7 (8)
C(4)—C(7)—C(8)	124·7 (7)	C(1′)—C(2′)—C(3′)	122·8 (9)
C(8)—C(7)—C(4′)	118·0 (6)	C(2′)—C(3′)—C(4′)	120·9 (12)
C(4)—C(7)—C(4′)	117·4 (6)	C(3′)—C(4′)—C(7′)	121·0 (9)
C(1)—C(6)—C(5)	122·9 (9)	C(5′)—C(4′)—C(7)	122·6 (7)
C(4)—C(5)—C(6)	121·8 (9)	C(3′)—C(4′)—C(5′)	116·4 (9)
C(3)—C(4)—C(5)	115·7 (8)	C(4′)—C(5′)—C(6′)	122·6 (8)
C(5)—C(4)—C(7)	125·5 (7)	C(1′)—C(6′)—C(5′)	121·3 (8)
C(3)—C(4)—C(7)	118·8 (7)	C(9)—N(1)—C(1)	119·7 (9)
C(2)—C(3)—C(4)	122·7 (8)	C(9)—N(1)—C(10)	120·5 (9)
C(1)—C(2)—C(3)	121·6 (8)	C(10)—N(1)—C(1)	119·8 (8)
C(2)—C(1)—C(6)	115·4 (8)	C(9′)—N(1′)—C(1′)	121·7 (8)
N(1)—C(1)—C(2)	122·1 (8)	C(9′)—N(1′)—C(10′)	113·6 (8)
C(6)—C(1)—N(1)	122·5 (9)	C(10′)—N(1′)—C(1′)	122·1 (8)

= −11 to 11, $k = 0$ to 8, $l = 0$ to 44; $3 \leq 2\theta \leq 53°$), $\bar{1}\bar{1}8$ used as intensity standard, 1053 observed reflections with $I \geq 3\sigma(I)$. No absorption corrections were applied. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Anisotropic full-matrix least-squares refinement with *SHELX*76 (Shledrick, 1976) based on F with H atoms (from ΔF synthesis) isotropic gave final $R = 0{\cdot}046$, $wR = 0{\cdot}046$ with $w = 1/\sigma^2(F_o)$. In the final cycle, maximum shift/σ is 0·1, peak heights in final ΔF map from −0·28 to 0·32 e Å^{-3}, $S = 1{\cdot}843$. Scattering factors for the non-H atoms from Cromer & Waber (1965) and for H atoms from Stewart, Davidson & Simpson (1965). All the molecular geometry, intra- and intermolecular calculations were performed with the program *PARST* (Nardelli, 1983).

Discussion. The final atomic parameters are listed in Table 1.[*] Fig. 1 shows a perspective view of the molecule with atomic numbering scheme. The bond lengths and angles are listed in Table 2.

The C—C ethylene bond distance [1·328 (11) Å] is slightly longer than the reported value of 1·314 (6) Å in unsubstituted ethylene (van Nes & Vos, 1977). An appreciable lengthening of this bond has been observed in the case of polarized ethylenes (Adhikesavalu & Venkatesan, 1981, 1982, 1983; Hazell & Mukhopadhyay, 1980). The twist about the ethylenic bond has been measured by the angle between the planes through Cl(1), C(8), Cl(2) and C(4), C(7), C(4′). In the present structure the twist angle is 7·1 (5)° while in DDE it is 6·3 (4)° as computed from the published data of Shields, Kennard & Robinson (1977). However, in the case of polarized ethylenes, the twist angles (Adhikesavalu & Venkatesan, 1981, 1982, 1983) are comparatively high.

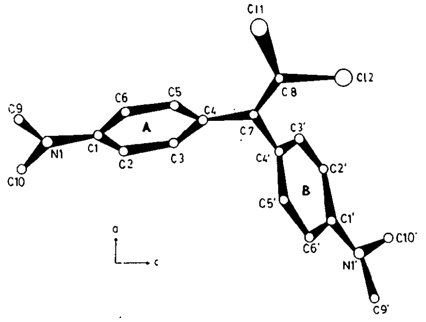

Fig. 1. Perspective view of the molecule down the *b* axis with atomic numbering scheme.

The phenyl ring *A* and the ethylenic group (=CCl$_2$) are planar but the phenyl ring *B* deviates significantly from planarity, having an approximate boat conformation with a pseudo mirror plane along C(1′)···C(4′).

The narrowing of the endocyclic angles at C(1) and C(1′) [115·4 (8) and 115·7 (8)° respectively] shows the extensive conjugation between the substituents and the ring in the case of *para*-disubstituted

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[*] Lists of H-atom coordinates, structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52079 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

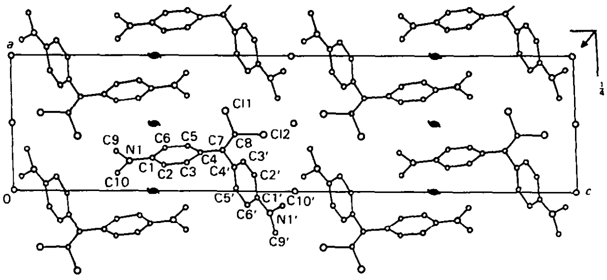

Fig. 2. The packing of the molecules as seen in projection down the *b* axis.

benzene derivatives. A similar effect has also been observed in *syn-p*-dimethylaminobenzaldehyde oxime (Bachechi & Zambonelli, 1972) and 2-(*p*-dimethylanilino)-4-phenyl-6a-thia-1,6-thiophthene (Hordvik & Saethre, 1972) which exemplified the effect of both π-donor and π-acceptor functional groups on the ring geometry (Domenicano, Vaciago & Coulson, 1975).

The molecular packing of the crystal viewed down the *b* axis is shown in Fig. 2. The structure is stabilized by van der Waals forces with no significant short intermolecular contacts.

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Structure of Benzyloxycarbonyl-L-alanyl-α-aminoisobutyric Acid, Z-L-Ala-Aib-OH

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Abstract. $C_{15}H_{20}N_2O_5$, $M_r = 308{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}545\,(1)$, $b = 10{\cdot}014\,(1)$, $c = 16{\cdot}692\,(1)$ Å, $V = 1595{\cdot}5\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}283$, $D_m = 1{\cdot}278$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}77$ mm^{-1}, $F(000) = 656$, $T = 295$ K, $R = 0{\cdot}047$ for 1508 reflections. The molecule has an extended form and no intramolecular hydrogen bonds. The α-aminoisobutyrate residue adopts a conformation close to that of an ideal right-handed 3_{10}-helix. The crystal structure is stabilized by three kinds of intermolecular hydrogen bonds.

Introduction. A number of short Aib-containing peptides studied so far adopt a β-turn conformation which characterizes a 3_{10}-helix (Prasad & Balaram, 1984). The conformation of the Aib residue is remarkably restricted owing to the bulky geminate methyl groups at the C^α atom. The torsion angles of the backbone at the Aib residue are close to the $\varphi = \pm 60$ and $\psi = \pm 30°$ angles of an ideal 3_{10}-helix. On the other hand, Ala residues are generally more flexible than the Aib residues, and the homo-oligomers have an extended conformation (Fawcett, Camerman & Camerman, 1975). However, the Ala residues in Aib-containing peptides tend to take torsion angles close to those of the 3_{10}-helix. As such, in an extreme example, Boc-L-Ala-Aib-OH (Bosh, Voges, Jung & Winter, 1983), the Ala residue has a conformation close to the 3_{10}-helix, although the Aib conformation is different. Thus, the structure of the

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