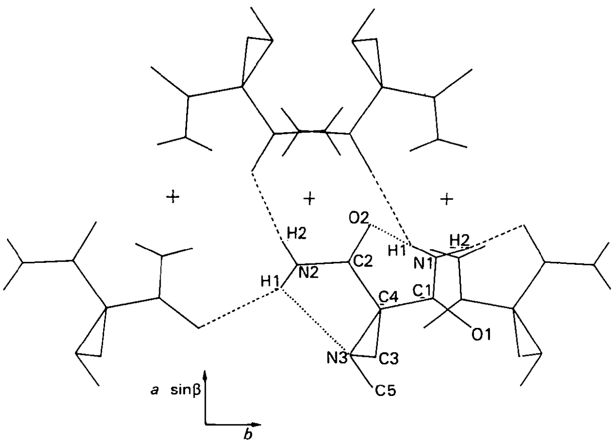

Fig. 3. Schematic representation of the molecular network formed by intra- (dotted line) and intermolecular (dashed line) hydrogen bonds.

Two intermolecular contacts involve H(2)—N, acting as a single hydrogen donor, and O(2) and their geometry implies two weak hydrogen bonds. There are also some short distances of the type C—H···O, all involving the O(1) atom; the relative parameters are reported in Table 3. The packing arrangement in the crystal is shown in Fig. 3, together with the hydrogen-bonding scheme.

References

Allen, F. H. (1980). *Acta Cryst.* B**36**, 81–96.
Allen, F. H. (1982). *Tetrahedron*, **38**, 2843–2853.
Bak, B. & Skaarup, S. (1971). *J. Mol. Struct.* **10**, 383–391.
Brückner, S. (1982). *Acta Cryst.* B**38**, 2405–2408.
Brückner, S., Malpezzi, L., Prosyanik, A. V. & Bondarenko, S. V. (1985). *Acta Cryst.* C**41**, 215–219, 219–222.
Deakyne, C. A., Allen, L. C. & Laurie, V. W. (1977). *J. Am. Chem. Soc.* **99**, 1343–1349.
Delker, G. L., Wang, Y., Stucky, G. D., Lambert, R. L., Haas, C. K. & Seyferth, D. (1976). *J. Am. Chem. Soc.* **98**, 1779–1784.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
Ito, T. & Sakurai, T. (1973). *Acta Cryst.* B**29**, 1594–1603.
Lauher, J. W. & Ibers, J. A. (1975). *J. Am. Chem. Soc.* **97**, 561–567.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Stalick, J. K. & Ibers, J. A. (1970). *J. Am. Chem. Soc.* **92**, 5333–5338.

Acta Cryst. (1985). C**41**, 215–219

The Structures of Two Crystalline Forms of 1-Phenyl-2,2-aziridinedicarboxamide, $C_{10}H_{11}N_3O_2$

By S. Brückner and L. Malpezzi*

Dipartimento di Chimica, Politecnico di Milano, Piazza Leonardo da Vinci 32, 20133 *Milano, Italy*

and A. V. Prosyanik and S. V. Bondarenko

Institute of Chemical Technology, Dniepropetrovsk, USSR

(*Received* 13 *April* 1984; *accepted* 25 *September* 1984)

Abstract. $M_r = 205{\cdot}2$. (*A*): monoclinic, $P2_1/n$, $a = 6{\cdot}180\,(5)$, $b = 28{\cdot}147\,(12)$, $c = 5{\cdot}919\,(2)$ Å, $\beta = 105{\cdot}90\,(4)°$, $V = 990\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}376$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}928$ cm^{-1}, $F(000) = 432$, room temperature, final $R = 0{\cdot}054$ for 813 observed reflections. (*B*): orthorhombic, $P2_12_12_1$, $a = 7{\cdot}236\,(4)$, $b = 8{\cdot}761\,(1)$, $c = 15{\cdot}700\,(4)$ Å, $V = 995{\cdot}3\,(6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}370$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}923$ cm^{-1}, room temperature, final $R = 0{\cdot}050$ for 1576 observed reflections. Two crystalline forms of the title compound have been obtained from ethanol in dilute solution by fast evaporation (form *A*) and in concentrated solution by slow evaporation (form *B*). The pyramidal geometry of the N ring atom is discussed and compared with previous data. The molecular dimensions of the two structures are similar, the orientation of the phenyl plane with respect to the aziridine ring is perpendicular in *A* and quasi perpendicular in *B*.

Introduction. The crystal structures of the title compound were determined as part of a continuing study into aziridine systems in order to investigate the pyramidal nature of the ring nitrogen atom and the

---

* To whom correspondence should be addressed.

geometry of the substituted three-membered heterocyclic ring. The structures of 2,2-aziridinedicarboxamide (Brückner, 1982) and of 1-methyl-2,2-aziridinedicarboxamide (Brückner, Malpezzi, Prosyanik & Bondarenko, 1985) have already been determined.

Experimental. Crystals 0·5 × 0·2 × 0·6 mm (*A*), 0·2 × 0·3 × 0·5 mm (*B*); Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo *K*α, least-squares fit of 17 (*A*) and 18 (*B*) reflections with $8 \leq \theta \leq 20°$ to obtain cell parameters; 1942 (*A*) and 2146 (*B*) independent reflections with $\theta \leq 26°$ (*A*) $-7 \leq h \leq 7$, $0 \leq k \leq 34$, $0 \leq l \leq 7$ and $\theta \leq 30°$ (*B*) $0 \leq h \leq 11$, $0 \leq k \leq 13$ and $0 \leq l \leq 24$, 813 (*A*) and 1576 (*B*) with $I > 2{\cdot}5\sigma(I)$; $\theta/2\theta$ scan technique, three periodically monitored reflections with no significant intensity variation, Lp correction, absorption ignored; direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Delcercq & Woolfson, 1980), 260 (*A*) and 202 (*B*) normalized structure factors with $E > 1{\cdot}7$; anisotropic blocked full-matrix refinement, isotropic for H (located from difference Fourier map); function minimized $\sum w(|F_o| - |F_c|)^2$, calculations performed with *SHELX*76 (Sheldrick, 1976). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Structure *A*: last cycle with 178 refined parameters converged at $R = 0{\cdot}054$ and $R_w = 0{\cdot}047$ (omitting unobserved) with $w = 2{\cdot}3562[\sigma^2(F_o) + 0{\cdot}00012 \times (F_o)^2]^{-1}$; final difference synthesis: $(\Delta/\sigma)_{\rm mean} = 0{\cdot}06$, $\Delta\rho_{\rm max} = 0{\cdot}25$ e Å^{-3} at 1·28 Å from N(1), $\Delta\rho_{\rm min} = -0{\cdot}22$ 3 Å^{-3}.

Structure *B*: last cycle with 176 refined parameters converged at $R = 0{\cdot}050$ and $R_w = 0{\cdot}054$ (omitting unobserved), with $w = 2{\cdot}5884[\sigma^2(F_o) + 0{\cdot}00073 \times (F_o)^2]^{-1}$; $\Delta\rho_{\rm min} = -0{\cdot}22$ e Å^{-3}, $\Delta\rho_{\rm max} = 0{\cdot}21$ e Å^{-3} at 1·28 Å from N(1), $(\Delta/\sigma)_{\rm mean} = 0{\cdot}06$.[*]

Discussion. The atom numbering scheme used for both structures is shown in Fig. 1. Atomic coordinates with isotropic thermal parameters are given in Table 1 and bond lengths, angles and some torsion angles are reported in Table 2. The molecular dimensions of the two structures agree with one another within the limit of experimental error, the main difference, from the point of view of intramolecular geometry, being the orientation of the phenyl group with respect to the aziridine ring; bond lengths and angles will consequently be discussed in terms of numerical values averaged over the two independent observations. The orientation of the phenyl ring has been examined in phenyl-substituted cyclopropane compounds (Lauher & Ibers, 1975; Jason & Ibers, 1977; Allen, 1980). These papers indicate that the perpendicular or distorted perpendicular conformation dominates, presumably because steric interactions prohibit the angle-bisector conformer and also because the possible interactions in the perpendicular conformation might stabilize the geometry of the strained three-membered ring system. The conformation of structures *A* and *B* suggests a similar

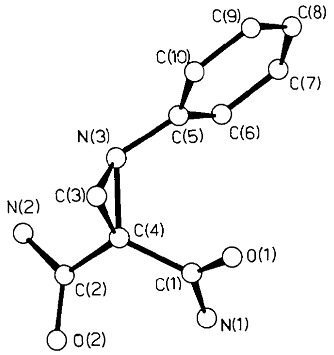

Fig. 1. Perspective view of a molecule of structure *A* showing atomic labelling.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2)

	x	*y*	*z*	$B_{\rm eq}$†
Structure *A*				
O(1)	4233 (6)	631 (1)	1439 (7)	3·8 (3)
O(2)	−1585 (6)	229 (1)	−3532 (7)	5·0 (4)
N(1)	3153 (7)	326 (2)	−2206 (8)	3·9 (4)
N(2)	−2692 (7)	990 (2)	−4136 (8)	3·8 (4)
N(3)	358 (6)	1290 (1)	−127 (7)	2·8 (3)
C(1)	2793 (9)	563 (2)	−420 (10)	2·8 (4)
C(2)	−1358 (8)	645 (2)	−2999 (9)	3·1 (5)
C(3)	−202 (9)	918 (2)	1289 (10)	3·8 (5)
C(4)	484 (8)	792 (2)	−840 (9)	2·6 (4)
C(5)	2330 (7)	1572 (2)	829 (8)	2·6 (4)
C(6)	3914 (9)	1636 (2)	−413 (9)	3·2 (4)
C(7)	5740 (10)	1931 (2)	476 (11)	4·3 (6)
C(8)	5970 (10)	2173 (2)	2585 (12)	4·7 (6)
C(9)	4359 (10)	2114 (2)	3762 (10)	4·4 (5)
C(10)	2566 (9)	1814 (2)	2910 (9)	3·7 (5)
Structure *B*				
O(1)	−2869 (2)	−289 (3)	671 (2)	4·4 (2)
O(2)	2479 (2)	−384 (2)	−39 (1)	3·7 (1)
N(1)	−584 (3)	−2005 (2)	746 (2)	4·0 (2)
N(2)	3454 (3)	861 (3)	1130 (2)	3·2 (1)
N(3)	−72 (2)	1745 (2)	1559 (1)	2·5 (1)
C(1)	−1226 (3)	−599 (3)	735 (2)	2·7 (1)
C(2)	2164 (3)	316 (2)	615 (1)	2·3 (1)
C(3)	−476 (3)	2252 (3)	701 (2)	3·0 (2)
C(4)	184 (3)	663 (2)	852 (1)	2·3 (1)
C(5)	−1534 (3)	1569 (3)	2153 (2)	2·6 (1)
C(6)	−1490 (4)	366 (3)	2724 (2)	3·4 (2)
C(7)	−2846 (5)	255 (4)	3350 (2)	4·2 (2)
C(8)	−4202 (4)	1348 (4)	3413 (2)	4·4 (2)
C(9)	−4238 (4)	2545 (4)	2850 (2)	3·8 (2)
C(10)	−2911 (3)	2668 (3)	2219 (2)	3·2 (2)

† $B_{\rm eq} = \frac{1}{3}\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

---

[*] Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39771 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

	Structure *A*	Structure *B*
C(1)–C(1)	1·226 (6)	1·224 (3)
O(2)–C(2)	1·211 (7)	1·218 (3)
N(1)–C(1)	1·319 (8)	1·316 (3)
N(2)–C(2)	1·332 (7)	1·324 (3)
N(3)–C(3)	1·440 (8)	1·449 (3)
N(3)–C(4)	1·471 (6)	1·471 (3)
N(3)–C(5)	1·436 (6)	1·419 (3)
C(1)–C(4)	1·523 (7)	1·516 (3)
C(2)–C(4)	1·517 (7)	1·511 (3)
C(3)–C(4)	1·479 (8)	1·491 (3)
C(5)–C(6)	1·387 (8)	1·384 (4)
C(5)–C(10)	1·380 (7)	1·389 (3)
C(6)–C(7)	1·383 (7)	1·392 (4)
C(7)–C(8)	1·395 (10)	1·375 (5)
C(8)–C(9)	1·373 (10)	1·372 (5)
C(9)–C(10)	1·375 (8)	1·384 (4)
C(4)–N(3)–C(5)	122·4 (4)	121·3 (2)
C(3)–N(3)–C(5)	119·1 (4)	119·6 (2)
C(3)–N(3)–C(4)	61·0 (3)	61·4 (2)
O(1)–C(1)–N(1)	123·9 (5)	123·5 (2)
N(1)–C(1)–C(4)	116·1 (5)	116·3 (2)
O(1)–C(1)–C(4)	119·8 (5)	120·1 (2)
O(2)–C(2)–N(2)	124·2 (5)	124·4 (2)
N(2)–C(2)–C(4)	116·4 (5)	116·4 (2)
O(2)–C(2)–C(4)	119·3 (5)	119·1 (2)
N(3)–C(3)–C(4)	60·5 (4)	60·0 (2)
C(2)–C(4)–C(3)	117·3 (5)	116·9 (2)
C(1)–C(4)–C(3)	115·9 (5)	116·5 (2)
C(1)–C(4)–C(2)	118·4 (4)	117·5 (2)
N(3)–C(4)–C(3)	58·4 (3)	58·6 (1)
N(3)–C(4)–C(2)	114·5 (4)	115·8 (2)
N(3)–C(4)–C(1)	118·5 (4)	118·5 (2)
N(3)–C(5)–C(10)	119·7 (5)	120·6 (2)
N(3)–C(5)–C(6)	120·6 (4)	119·4 (2)
C(6)–C(5)–C(10)	119·5 (5)	119·8 (2)
C(5)–C(6)–C(7)	119·8 (5)	119·6 (3)
C(6)–C(7)–C(8)	120·4 (6)	120·3 (3)
C(7)–C(8)–C(9)	119·1 (6)	120·0 (3)
C(8)–C(9)–C(10)	120·7 (6)	120·5 (3)
C(5)–C(10)–C(9)	120·5 (5)	119·8 (3)
C(4)–N(3)–C(5)–C(6)	56·5 (7)	69·6 (3)
C(3)–N(3)–C(5)–C(6)	128·8 (5)	142·2 (2)
C(4)–N(3)–C(5)–C(10)	−128·7 (5)	−116·5 (3)
C(3)–N(3)–C(5)–C(10)	−56·4 (6)	−43·9 (3)
C(5)–N(3)–C(4)–C(1)	3·4 (7)	3·8 (3)
C(3)–N(3)–C(4)–C(1)	−104·5 (5)	−105·3 (2)
C(5)–N(3)–C(4)–C(2)	−143·9 (5)	−143·9 (2)
C(3)–N(3)–C(4)–C(2)	108·2 (5)	107·1 (2)
O(1)–C(1)–C(4)–C(2)	−171·4 (5)	−158·5 (2)
N(1)–C(1)–C(4)–C(3)	160·8 (5)	170·3 (2)
O(2)–C(2)–C(4)–N(3)	−169·9 (5)	−169·2 (2)
N(2)–C(2)–C(4)–C(3)	73·1 (7)	74·4 (3)
N(3)–C(5)–C(10)–C(9)	−175·1 (5)	−174·5 (2)
N(3)–C(5)–C(6)–C(7)	176·4 (5)	175·1 (3)

behaviour for 1-phenyl-substituted aziridine systems. In *A* the phenyl ring assumes the perpendicular conformation and in *B* the distorted one, revealed from the acute vector–plane angle θ between the normal to the phenyl ring and the distal C(3)—C(4) vector (Lauher & Ibers, 1975; Jason & Ibers, 1977); these angles being 89·0 (2) and 77·5 (1)° for *A* and *B*, respectively. The phenyl ring is planar in both structures, with no atom deviating more than 0·01 Å from the best least-squares plane and with bond lengths and angles at standard values.

The three-membered ring shows the characteristic behaviour of analogous substituted aziridines where the actual geometry is the result of a sum of several effects due to each substituent (Brückner *et al.*, 1985, and references therein). The N(3)—C(3) bond length in both structures is significantly shorter than N(3)—C(4) as found in 1-methyl-2,2-aziridinedicarboxamide. The C(3)—C(4) bond has the usual short C—C bond in aziridine compounds, while the difference between structures *A* and *B* is within experimental error.

The pyramidal nature of the N ring atom is identical in *A* and *B*, the N(3) atom lying 0·603 (3) Å out of the plane defined by its three substituents. The N(3)—C(5) bond makes an angle of 126·3 (2)° with the aziridine ring and the mean valence ring angle at N(3) is 120·6 (3)°; in the 1-methyl compound the corresponding values were respectively 122·3 (3) and 117·4 (3)°, while in the 1-unsubstituted compound they were respectively 107·5 (5) and 105·1 (5)°, showing a steady increase as H, CH_3 and C_6H_5 are bonded to N. The plane defined by C(1)—C(4)—C(2) is almost perpendicular to the aziridine plane, the twist of 81·7 (3)° for *A* and 88·4 (2)° for *B* being nearly equal to that found in the 1-methyl compound. These planes through C(1)—C(4)—C(2) and through the aziridine ring nearly bisect the angles C(3)—C(4)—N(3) and C(1)—C(4)—C(2), respectively.

The bent-back angle between the normal to the C(1)—C(4)—C(2) plane and the vector C(3)—C(4) (Stalick & Ibers, 1970; Allen, 1982) is 61·1 (4)° for *A* and 60·0 (2)° for *B* [1-methyl compound 60·6 (4)°].

The carboxamide groups are nearly planar, the largest deviation from the best plane being 0·027 (5) Å for C(1) in *A* and the angles between these two planes are 48·7 (2) in *A* and 56·1 (1)° in *B*. Unlike the orientation in the 1-methyl compound, these groups are not symmetrical with respect to the aziridine plane as can be seen from the Newman projection of Fig. 2.

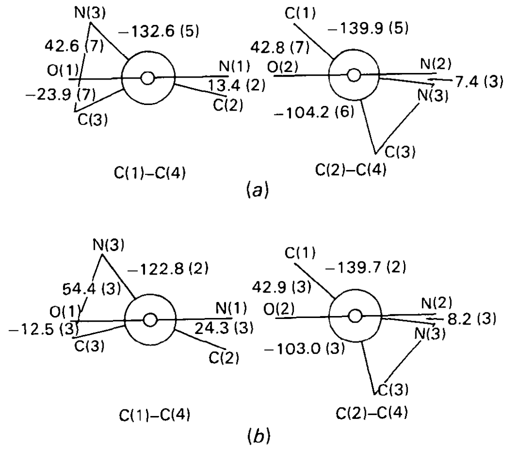

Fig. 2. Newman projection along the bonds C(1)—C(4) and C(2)—C(4) of (*a*) structure *A*, (*b*) structure *B*. (Angles in deg.)

The angles between the aziridine ring and the two carboxamide planes are 82·5 (3) and 69·1 (3)° for *A* and 69·6 (1) and 77·1 (1)° for *B*. The dimensions of the two carboxamide groups are equal in *A* and *B*: the C=O bond length is typical of that found in many amides (Long, Maddox & Trueblood, 1969), while the C—N distances are shorter than the mean value of 1·333 Å. The asymmetry in the angles centred on C(1) and C(2) is the same as that found in the 1-methyl compound.

The crystal packing of *A* and *B* is illustrated in Figs. 3 and 4, respectively. The different packing is the consequence of the presence in *A* of molecules of opposite optical activity in a racemic mixture, while in *B* only one of the two enantiomers is present. The two similar densities indicate an equivalent space-filling ability for the two structures. An attempt was made to measure the optical activity in solution of the enantiomer present in *B*, but it failed, thus preventing any further investigation of the absolute configuration.

Table 3. *Some non-bonded parameters*

A—H···*B*	*A*···*B* (Å)	H···*B* (Å)	∠*A*—H···*B*(°)	Symmetry operation for *B*
Hydrogen bonding				
Structure *A*				
N(1)—H(2)···O(2)	2·830 (6)	2·10 (5)	132 (2)	x, y, z
N(2)—H(1)···N(3)	2·728 (6)	2·34 (4)	109 (2)	x, y, z
N(1)—H(1)···O(1)	3·109 (6)	2·36 (5)	136 (4)	$1 - x, -y, -z$
N(1)—H(2)···O(2)	2·898 (6)	2·18 (5)	130 (4)	$-x, -y, -1 - z$
N(2)—H(2)···O(1)	2·961 (5)	1·98 (4)	176 (4)	$x - 1, y, z - 1$
Structure *B*				
N(1)—H(2)···O(2)	2·906 (3)	2·31 (3)	120 (3)	x, y, z
N(2)—H(1)···N(3)	2·750 (3)	2·28 (4)	110 (2)	x, y, z
N(2)—H(2)···O(1)	2·935 (3)	1·94 (4)	173 (3)	$1 + x, y, z$
N(1)—H(1)···O(2)	2·903 (3)	2·10 (3)	167 (3)	$x - \frac{1}{2}, -y - \frac{1}{2}, -z$
Some intra- and intermolecular contacts				
Structure *A*				
C(3)—H(2)···O(1)	2·836 (7)	2·41 (5)		x, y, z
C(3)—H(1)···O(1)	3·558 (5)	2·68 (5)		$x - 1, y, z$
Structure *B*				
C(3)—H(2)···O(1)	2·821 (3)	2·42 (3)		x, y, z
C(3)—H(2)···O(2)	3·287 (3)	2·81 (3)		$x - \frac{1}{2}, \frac{1}{2} - y, -z$
C(6)—H···N(3)	3·551 (3)	2·82 (3)		$-x, -\frac{1}{2} + y, \frac{1}{2} - z$
C(8)—H···O(3)	3·498 (3)	2·59 (4)		$-\frac{1}{2} - x, -y, z + \frac{1}{2}$

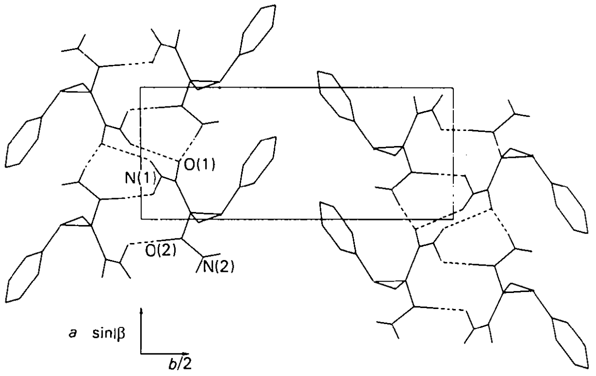

Fig. 3. Crystal packing of *A* projected along the *c* axis, showing intra- (dotted line) and intermolecular (dashed line) hydrogen bonds.

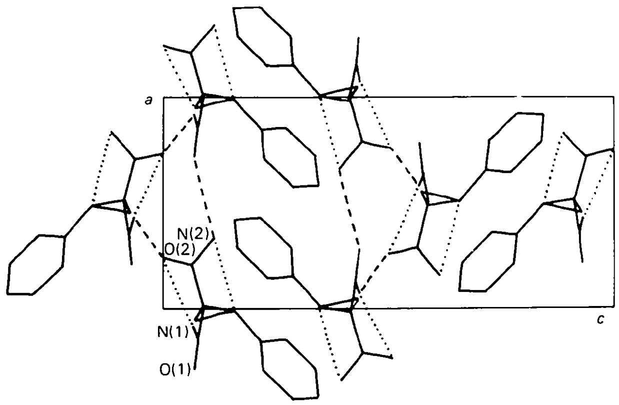

Fig. 4. Crystal packing of *B* projected along the *b* axis, with hydrogen bonds as in Fig. 3. For clarity, hydrogen atoms have been omitted.

The crystal packing in both structures is governed by some intra- and intermolecular hydrogen bonds of the type N—H···O and N—H···N as reported in Table 3. All H atoms linked to N(1) and N(2) in *A* and in *B* are involved in hydrogen bonding and in *A* N(1)—H acts as a bifurcated donor to one intra- and one intermolecular oxygen atom. The shortest non-bonded distance, in *A* and in *B*, is the intramolecular contact N(3)···N(2), which is a common feature in aziridine carboxamide compounds. As in the 1-methyl compound, this interaction, however, appears to be rather weak because of the unfavourable N(2)—H···N(3) angle. The other intramolecular contact, N(1)···O(2), shows an analogous small value for the N(1)—H···O(2) angle, but the residual electron density in the final difference map in the vicinity of N(1) suggests a disordered hydrogen atom, which implies little reliability in its final position. The other hydrogen bonds show a better geometry. In addition, in both structures there are some intra- and intermolecular non-bonded interactions characterized by the values reported in Table 3. The short C—H···O and C—H···N contacts have been discussed statistically by Taylor & Kennard (1982) in a survey on 113 organic crystal structures and in a quantitative approach by Berkovitch-Yellin & Leiserowitz (1984) using atom–atom potential-energy calculations. The authors concluded that the C—H···O and the C—H···N interactions are more attractive than repulsive and can reasonably be described as hydrogen bonds. Taylor & Kennard also pointed out that C—H groups adjacent to a nitrogen atom are especially likely to form C—H···*X* hydrogen bonds (*X* = O,N) while Berkovitch-Yellin & Leiserowitz remarked that the amide O atom is a far stronger proton acceptor than the corresponding O atom in a carbonyl group. Our studies on aziridinedicarboxamide systems support these conclusions, giving evidence of the significant role of C—H···*X* contacts in determining the packing arrangement in similar compounds.

References

Allen, F. H. (1980). *Acta Cryst.* B**36**, 81–96.

Allen, F. H. (1982). *Tetrahedron*, **38**, 2843–2853.

Berkovitch-Yellin, Z. & Leiserowitz, L. (1984). *Acta Cryst.* B**40**, 159–165.

Brückner, S. (1982). *Acta Cryst.* B**38**, 2405–2408.

Brückner, S., Malpezzi, L., Prosyanik, A. V. & Bondarenko, S. V. (1985). *Acta Cryst.* C**41**, 213–215.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Jason, M. E. & Ibers, J. A. (1977). *J. Am. Chem. Soc.* **98**, 6012–6018.

Lauher, J. W. & Ibers, J. A. (1975). *J. Am. Chem. Soc.* **97**, 561–567.

Long, R. E., Maddox, H. & Trueblood, K. N. (1969). *Acta Cryst.* B**25**, 2083–2094.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Stalick, J. K. & Ibers, J. A. (1970). *J. Am. Chem. Soc.* **92**, 5333–5338.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Acta Cryst. (1985). C**41**, 219–222

The Structure of Diethyl 1-(*p*-Methoxyphenylsulphonyl)-2,2-aziridinedicarboxylate, $C_{15}H_{19}NO_7S$

By S. Brückner and L. Malpezzi*

Dipartimento di Chimica, Politecnico di Milano, Piazza Leonardo da Vinci 32, 20133 *Milano, Italy*

and A. V. Prosyanik and S. V. Bondarenko

Institute of Chemical Technology, Dniepropetrovsk, USSR

(*Received* 18 *May* 1984; *accepted* 25 *September* 1984)

Abstract. $M_r = 357{\cdot}4$, monoclinic, $P2_1/c$, $a = 17{\cdot}124\,(2)$, $b = 10{\cdot}364\,(1)$, $c = 10{\cdot}144\,(1)$ Å, $\beta = 106{\cdot}41\,(3)°$, $V = 1727{\cdot}0\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}374$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}122$ cm^{-1}, $F(000) = 752$, room temperature, final $R = 0{\cdot}041$ for 1347 observed reflections. The N ring atom lies 0·700 (3) Å out of the plane of its three substituents and the N—S bond makes a mean valence angle of 117·6 (3)° with the two N—C bonds of the three-membered ring. The lone pair of this N atom is *trans* to O(5) across the S—N bond.

Introduction. The structure of the title compound was determined as part of a continuing study on aziridine systems in order to investigate the pyramidal structure of the ring N atom and the change of geometry in several different *N*-substituted compounds. The structures of non-substituted 2,2-aziridinedicarboxamide (Brückner, 1982), 1-methyl- and 1-phenyl-2,2-aziridinedicarboxamide (Brückner, Malpezzi, Prosyanik & Bondarenko, 1985) have been previously determined.

Experimental. Colourless crystal, 0·4 × 0·4 × 0·05 mm, Philips PW1100 diffractometer, graphite-monochromated Mo $K\alpha$, cell constants by least-squares analysis of θ angles of 21 strong reflections with $7 \leq \theta \leq 16°$; 3033 independent reflections $-17 \leq h \leq 17$, $0 \leq k \leq 10$, $0 \leq l \leq 12$ with $\theta \leq 25°$, 1347 with $I \geq 2{\cdot}5\sigma(I)$; $\theta/2\theta$ scan technique, three periodically monitored reflections with no intensity variation; Lp correction, absorption ignored; direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), 296 normalized structure factors with $|E| \geq 1{\cdot}69$; anisotropic blocked full-matrix refinement, isotropic for H [some located from difference electron density map and those of methyl groups involving C(6), C(8) and C(15) calculated], phenyl group treated as rigid body, 271 parameters refined; function minimized $\sum w(|F_o|^2 - |F_c|^2)$ with $w = 1{\cdot}5739\,[\sigma^2(F_o) + 0{\cdot}00007 \times (F_o)^2]^{-1}$; final $R = 0{\cdot}041$ and $R_w = 0{\cdot}033$; final $\Delta\rho$ excursion $-0{\cdot}22$–$0{\cdot}19$ e Å^{-3}, $(\Delta/\sigma)_{\text{mean}} = 0{\cdot}119$, $S = 1{\cdot}31$; refinement calculations with *SHELX*76 (Sheldrick, 1976); atomic scattering factors as supplied by the program.†

---

* To whom correspondence should be addressed.

† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39772 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/85/020219-04$01.50