DRIEDONKS, R. A., KRIJGSMAN, P. C. J. & MELLEMA, J. E. (1976). *Philos. Trans. R. Soc. London Ser. B,* **276,** 131–141.

DRIEDONKS, R. A., KRIJGSMAN, P. C. J. & MELLEMA, J. E. (1977). *J. Mol. Biol.* **113,** 123–140.

DRIEDONKS, R. A., KRIJGSMAN, P. C. J. & MELLEMA, J. E. (1978). *Eur. J. Biochem.* **82,** 405–417.

FUKUYAMA, K., ABDEL-MEGUID, S. S. & ROSSMANN, M. G. (1981). *J. Mol. Biol.* **150,** 33–41.

GIBBS, A. J., NIXON, H. L. & WOODS, R. D. (1963). *Virology,* **19,** 441–449.

HARRISON, S. C. (1968). *J. Appl. Cryst.* **1,** 84–90.

HULL, R. (1969). *Adv. Virus Res.* **15,** 365–433.

HULL, R. (1970). *Virology,* **40,** 34–47.

HULL, R., HILLS, G. J. & MARKHAM, R. (1969). *Virology,* **37,** 416–428.

JASPARS, E. M. J. (1974). *Adv. Virus Res.* **19,** 37–149.

LEBEURIER, G., FRAENKEL-CONRAT, H., WURTZ, M. & HIRTH, L. (1971). *Virology,* **43,** 51–61.

LENTZ, P. J. JR & STRANDBERG, B. (1974). *Acta Cryst.* A**30,** 552–559.

MELLEMA, J. E. & VAN DEN BERG, H. J. N. (1974). *J. Supramol. Struct.* **2,** 17–31.

ROSSMANN, M. G. (1979). *J. Appl. Cryst.* **12,** 225–238.

ROSSMANN, M. G. & BLOW, D. M. (1962). *Acta Cryst.* **15,** 24–31.

ROSSMANN, M. G., LESLIE, A. G. W., ABDEL-MEGUID, S. S. & TSUKIHARA, T. (1979). *J. Appl. Cryst.* **12,** 570–581.

SCHUTT, C. & WINKLER, F. K. (1977). *The Rotation Method in Crystallography,* edited by U. W. ARNDT & A. J. WONACOTT, pp. 173–186. Amsterdam: North-Holland.

VAN BEYNUM, G. M. A., DE GRAAF, J. M., CASTEL, A., KRAAL, B. & BOSCH, L. (1977). *Eur. J. Biochem.* **72,** 63–78.

Structures of Three Schiff-Base Diazastilbenes:
(I) *trans-N*-(2-Pyridylmethylene)aniline, (II) *trans-N*-(4-Pyridylmethylene)aniline and (III) *trans-N*-Benzylidene-3-pyridinamine

BY MICHAEL WIEBCKE AND DIETRICH MOOTZ

Institut für Anorganische Chemie und Strukturchemie, Universität Düsseldorf, Universitätsstrasse 1, D-4000 Düsseldorf, Federal Republic of Germany

Abstract

The crystal structures of the three isomeric diazastilbenes ($C_{12}H_{10}N_2$) were determined from Mo $K\alpha$ diffractometer data. (I) is monoclinic, space group $P2_1/c$, with $a = 15{\cdot}391$ (4), $b = 5{\cdot}690$ (2), $c = 12{\cdot}288$ (3) Å, $\beta = 114{\cdot}17$ (2)° at 293 K, $Z = 4$. (II) is orthorhombic, space group $P2_12_12_1$, with $a = 5{\cdot}933$ (2), $b = 7{\cdot}682$ (3), $c = 21{\cdot}616$ (8) Å at 293 K, $Z = 4$. (III) is monoclinic, space group $P2_1/c$ with $a = 10{\cdot}413$ (4), $b = 8{\cdot}432$ (3), $c = 21{\cdot}842$ (12) Å, $\beta = 90{\cdot}85$ (4)° at 193 K, $Z = 8$. The unweighted R values obtained are 0·086 for (I), 0·073 for (II) and 0·071 for (III) with 1195, 656 and 2374 observed independent reflections, respectively. The non-planar molecular conformations are described in terms of the N torsion (8·9 to 51·3°) and C torsion (−17·9 to 13·4°) of the respective rings relative to the central part C–N=C–C of the molecules. The results are discussed in comparison with others obtained in solution and on related molecules.

Introduction

Of the diazastilbenes those of the Schiff-base type have non-planar molecular conformations as found by electron excitation (Pitea, Favini & Grasso, 1970) and ^{13}C NMR spectroscopy (Denecke, Müller & Bluhm, 1982). From the chemical shifts of the *para* C atoms the N torsion (the angle between the planes of the ring at the N atom in the bridge and of the central C–N=C–C part of the molecule) was found to be in the range 33 to 59°, depending on the position of the second N atom in the rings. A smaller value of about 10° was determined for the C torsion. Comparison of these findings with the molecular conformations in the solid state appeared of interest, and crystal structure determinations of three Schiff-base diazastilbenes are reported in this paper. The crystallographic numbering system is shown below for (I) (second N atom at position 10), (II) (second N at 12) and (III) (second N at 2).
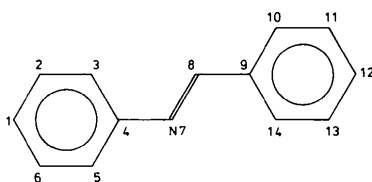
The conformation of *trans-N*-benzylideneaniline (BA), the parent monoaza compound, falls in the same range as given above and is approximately identical for the free molecule, the molecule in solution and in the crystalline state (Bürgi & Dunitz, 1970, 1971; Trætteberg, Hilmo, Abraham & Ljunggren, 1978; Bluhm, Knop & Behjati, 1979; Wolf, 1980). This, however, is not a general fact for molecules with torsional degrees of freedom, because intermolecular forces, specifically in the solid state, are capable of altering conformations significantly (Bernstein & Hagler, 1978, 1979).

Experimental

All compounds were prepared by known literature methods (Perkampus & Behjati, 1974). Single crystals of (I) and (II) suitable for X-ray studies were obtained by sublimation; crystals of (III) (liquid at room temperature) were grown on a Syntex $P2_1$ automatic diffractometer with a modified LT-1 low-temperature device. The same instrument (Mo $K\alpha$ radiation, graphite monochromator) was used for the X-ray measurements, which were carried out at room temperature for (I) and (II) and at 193 K for (III). The cell dimensions were calculated from the angular coordinates of 15 strong reflections in the range $20° < 2\theta < 30°$.

Table 1. *Additional crystal data and details of the structure refinements*

	(I)	(II)	(III)
V (Å^3)	981·8	985·2	1917·6
D_m (Mg m^{-3})	1·24	1·22	–
D_x (Mg m^{-3})	1·23	1·23	1·26
$F(000)$	384	384	768
μ(Mo $K\alpha$) (mm^{-1})	0·08	0·08	0·08
$2\theta_{max}$ (°) (Mo $K\alpha$)	55	50	50
Independent reflections measured	2259	1046	3376
Independent reflections considered observed $[I > 1·5\sigma(I)]$	1195	656	2374
Variables in the last run	159	157	333
Observed data:			
R	0·086	0·073	0·071
R_w	0·079	0·071	0·071
All data:			
R	0·160	0·119	0·109
R_w	0·088	0·078	0·076
Maximum residual electron density (e Å^{-3})	±0·26	±0·25	±0·30

Three-dimensional intensities of all crystals were measured by a variable ω-scan technique: scan range 0·7–0·8°, scan speed 0·49–29·3° min^{-1} selected by a rapid prescan. Background was counted on both sides of each reflection with one half of the time needed for the total scan. Alignment and crystal stability were checked by reference reflections. For (III) two sets of independent reflections were collected and averaged. The intensities were corrected for Lorentz and polarization factors, but not for absorption. Crystal data are summarized in the *Abstract* and in Table 1.

Structure determination and refinement

The structures were solved with the program *MULTAN* (Germain, Main & Woolfson, 1971) in an automatic phase-determination process. Refinement was carried out by the full-matrix least-squares method minimizing the function $\sum w(|F_o| - 1/k|F_c|)^2$ with the weights $w^{-1} = \sigma^2(F_o) + (0·02F_o)^2$ and $\sigma(F_o)$ derived from counting statistics. The scattering factors were those of Cromer & Waber (1974). The residuals (Table 1) are defined by $R = \sum ||F_o| - |F_c||/\sum |F_o|$ and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$. For all computations an Eclipse computer (Data General) with the program system *EXTL* (Syntex) was used. Final coordinates and equivalent isotropic thermal parameters of the non-H atoms are given in Table 2.*

Compound (I)

An E map yielded all non-H atoms of two independent molecules with their centres on crystallographic centres of symmetry. Because of the unequal bridging atoms and aromatic rings this implies orientational disorder of the whole molecules. Additional rotational disorder of the pyridine rings around the C(8)–C(9) bonds with two different positions for the ring N atoms could be ruled out by refinement of the respective occupancy factors. In the final cycles superimposed atoms were treated as C atoms with occupancy factors of 6·5/6. Ten H atoms were located in a difference electron density map; the two missing ones were placed in geometrically reasonable positions. The refinement was completed with anisotropic thermal parameters for all non-H atoms and isotropic ones for the H atoms, except for the half-occupied positions whose positional and thermal parameters were kept

Table 2. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\text{Å}^2 \times 10$), *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}(B_{11} a^2 a^{*2} + B_{12} aba^* b^* \cos \gamma + \ldots). \ X = \tfrac{1}{2}(C + N), \text{ treated as C with occupancy factor } 6\cdot5/6.$$

	x	y	z	B_{eq}		x	y	z	B_{eq}
(I), molecule A					(III), molecule A				
C(1)	1806 (3)	−2319 (9)	3826 (4)	46 (2)	C(1)	2552 (4)	3352 (5)	4761 (2)	35 (1)
C(2)	1304 (3)	−242 (9)	3511 (4)	44 (1)	N(2)	1470 (3)	2565 (5)	4620 (1)	38 (1)
C(3)	749 (3)	261 (8)	2331 (4)	37 (1)	C(3)	1231 (4)	2289 (5)	4032 (2)	34 (1)
C(4)	709 (3)	−1357 (7)	1466 (4)	34 (1)	C(4)	2051 (3)	2734 (4)	3566 (2)	26 (1)
X(5)	1201 (3)	−3436 (7)	1769 (3)	41 (1)	C(5)	3176 (4)	3509 (5)	3719 (2)	31 (1)
C(6)	1742 (3)	−3862 (9)	2943 (5)	50 (2)	C(6)	3430 (4)	3838 (5)	4330 (2)	33 (1)
X(7)	144 (3)	−978 (6)	207 (3)	36 (1)	N(7)	1804 (3)	2342 (4)	2937 (1)	30 (1)
					C(8)	676 (4)	2591 (5)	2726 (2)	28 (1)
					C(9)	308 (3)	2145 (4)	2097 (2)	26 (1)
(I), molecule B					C(10)	−852 (4)	2705 (5)	1847 (2)	34 (1)
C(1)	3158 (4)	653 (11)	2108 (5)	54 (2)	C(11)	−1223 (4)	2315 (5)	1257 (2)	37 (1)
C(2)	3753 (4)	−1206 (10)	2189 (5)	56 (2)	C(12)	−455 (4)	1365 (5)	911 (2)	33 (1)
C(3)	4262 (4)	−1228 (9)	1502 (5)	52 (2)	C(13)	689 (4)	789 (5)	1144 (2)	33 (1)
C(4)	4165 (3)	572 (8)	709 (4)	46 (1)	C(14)	1076 (4)	1169 (5)	1739 (2)	30 (1)
X(5)	3560 (3)	2411 (8)	611 (4)	53 (2)					
C(6)	3077 (4)	2421 (11)	1315 (5)	59 (2)					
X(7)	4677 (3)	683 (7)	−44 (4)	46 (1)					
					(III), molecule B				
					C(1)	4938 (4)	3251 (5)	793 (2)	31 (1)
(II)					N(2)	4572 (3)	1980 (4)	1110 (1)	34 (1)
C(1)	6002 (17)	3362 (12)	569 (4)	52 (3)	C(3)	4890 (4)	1979 (5)	1709 (2)	28 (1)
C(2)	4045 (16)	4265 (11)	686 (4)	47 (3)	C(4)	5563 (3)	3179 (4)	1994 (2)	24 (1)
C(3)	3151 (15)	4203 (10)	1279 (4)	43 (2)	C(5)	5880 (4)	4492 (5)	1664 (2)	27 (1)
C(4)	4135 (13)	3193 (10)	1728 (3)	37 (2)	C(6)	5576 (4)	4522 (5)	1038 (2)	29 (1)
C(5)	6077 (14)	2332 (11)	1594 (3)	40 (2)	N(7)	5934 (3)	3117 (3)	2623 (1)	26 (1)
C(6)	7018 (16)	2373 (12)	1006 (4)	48 (3)	C(8)	6526 (3)	1875 (5)	2804 (2)	26 (1)
N(7)	3373 (12)	3034 (9)	2352 (3)	41 (2)	C(9)	7000 (3)	1638 (4)	3435 (2)	24 (1)
C(8)	1357 (16)	3402 (10)	2513 (3)	38 (2)	C(10)	7665 (3)	260 (5)	3569 (2)	29 (1)
C(9)	554 (14)	3296 (10)	3145 (3)	35 (2)	C(11)	8123 (4)	−28 (5)	4154 (2)	33 (1)
C(10)	−1495 (15)	3953 (13)	3319 (4)	46 (3)	C(12)	7899 (4)	1048 (5)	4618 (2)	32 (1)
C(11)	−2120 (17)	3913 (11)	3930 (4)	44 (3)	C(13)	7231 (5)	2419 (5)	4492 (2)	34 (1)
N(12)	−845 (12)	3252 (9)	4378 (3)	43 (2)	C(14)	6782 (4)	2730 (5)	3905 (2)	28 (1)
C(13)	1149 (18)	2622 (11)	4211 (4)	46 (3)					
C(14)	1890 (14)	2594 (12)	3608 (4)	39 (3)					

fixed. An attempted refinement of an ordered structure in the lower-symmetry space group Pc was not convincing.

Compounds (II) and (III)

All non-H atoms of both structures appeared in E maps. C and N atoms were distinguished by their thermal parameters during refinement and by location of all H atoms from difference electron density maps. At a later stage of the refinement of (III) seven reflections with large differences of $|F_o|$ and $|F_c|$ were eliminated because of their poor intensity agreement in the two data sets collected. In the final cycles anisotropic thermal parameters for all non-H atoms and isotropic ones for the H atoms of (III) were used while the isotropic parameters of the H atoms of (II) were kept fixed at $B = 4\cdot0$ Å^2.

Results and discussion

Bond distances and bond angles are summarized in Fig. 1.* Least-squares planes through the non-H atoms of the rings and the four non-H atoms of the central parts of the molecules were calculated for all five molecules. The displacements of these atoms from the respective planes do not exceed 0·009 Å in (I), 0·015 Å in (II) and 0·021 Å in (III). Resulting torsion angles, defined as illustrated in Fig. 2, are given in Table 3.

Stereoscopic drawings of the crystal structures are shown in Fig. 3. (I) is isostructural with *trans*-azobenzene (TA) (Brown, 1966), *trans*-stilbene (TS) (Finder, Newton & Allinger, 1974; Bernstein, 1975; Hoekstra, Meertens & Vos, 1975) and tolane (TO) (Mavridis & Moustakali-Mavridis, 1977). The orienta-
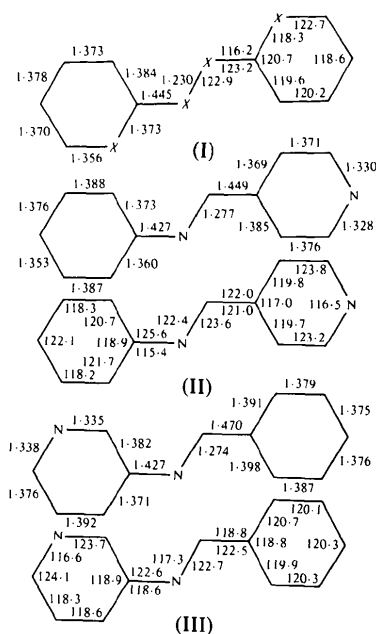
---

* See deposition footnote.

Fig. 1. Bond distances (Å) and bond angles (°). For (I) and (III) only averages are given, for the differences of structurally equivalent single values are within the range of 4·0 e.s.d.'s with the exception of the angle N(7)—C(8)—C(9) in (III) (7·3 e.s.d.'s). E.s.d.'s are 0·005–0·008 Å and 0·4–0·5° for (I) (single values), 0·009–0·012 Å and 0·7–0·9° for (II), 0·004–0·006 Å and 0·3–0·4° for (III) (single values). $X = \frac{1}{2}(C + N)$, owing to disorder.
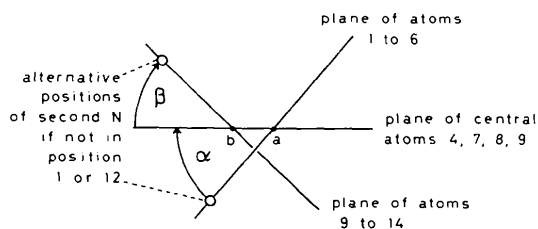

Fig. 2. Schematic representation of a molecule with hypothetical conformation. The points a and b represent the view down the C(4)—N(7) bond and C(8)—C(9) bond, respectively. The discussion is referred to the enantiomer with either $0° \le \alpha \le 90°$ or $-180° \le \alpha \le -90°$ as the angle for the N torsion. The angle β for the C torsion is not restricted ($-180° < \beta \le 180°$). Positive signs of α and β define clockwise rotation of the plane in front in order to eclipse the rear plane.

Fig. 3. ORTEP stereoscopic drawings (Johnson, 1976) of the three diazastilbene crystal structures [(I) without H atoms for clarity]. The probability level of the thermal ellipsoids of the non-H atoms is 30%.

tional disorder of the two independent molecules on crystallographic centres of symmetry is similar to that in the crystal structures of three substituted benzylideneanilines (Bernstein & Schmidt, 1972; Bernstein & Izak, 1975; Bernstein, Bar & Christensen, 1976). The observed absence of rotational disorder of the pyridine ring around the C(8)—C(9) bond in favour of a 'trans' position of the two N atoms in both molecules is probably due to electronic repulsion, because the 'cis' separation would be less than 3 Å.

This is also suggested from dipole-moment measurements (Pitea, Favini & Grasso, 1970).

A positional disorder with two or more random orientations of one of the two independent molecules was proposed for TA (Brown, 1966) to explain some unusual features found in the structure and was later established for TS from difference electron density calculations (Finder, Newton & Allinger, 1974), especially with low-temperature data (Hoekstra, Meertens & Vos, 1975). (I) shows remarkable parallels of thermal parameters and e.s.d.'s of positional parameters with those of TA and TS (room-temperature data). In all cases these values are larger for one

Table 3. *Torsion angles* α *and* β (°) *as defined in Fig. 2, with e.s.d.'s in parentheses*

For comparison, corresponding values in crystal structures of three related compounds are also listed.

		α	β
(I),	molecule A	17·9 (7)	−17·9 (7)
	molecule B	8·9 (8)	−8·9 (8)
(II)		19·2 (12)	−9·8 (12)
(III),	molecule A	46·2 (5)	13·4 (6)
	molecule B	51·3 (5)	2·5 (6)
BA (a)		55·2	10·3
TA (b),	molecule A	17·1	−17·1
	molecule B	6·0	−6·0
TS (c),	molecule A	5·0	−5·0
	molecule B	3·3	−3·3

(a) Bürgi & Dunitz (1970); (b) Brown (1966); (c) Finder, Newton & Allinger (1974).

molecule [molecule B in (I)]. However, a final difference electron density map for (I) gave no hint of additional disorder in this region and the central bond lengths of the two molecules are equal, in contrast to TA and TS.

A rotational disorder of the pyridine ring around the C(4)—N(7) bond resulting in two opposed positions of the ring N atom as was found in N-(5-methoxy-salicylidene)-3-pyridinamine (Moustakali-Mavridis, Hadjoudis & Mavridis, 1980) is not observed for either molecule of (III), probably because of specific dipole–dipole interactions (Fig. 3) in this crystal structure. In the free molecule for both ring orientations the intramolecular repulsion between an 'ortho' H atom [at C(3) or C(5)] and the bridge H atom at C(8) would apparently be the same, as supported by dipole-moment measurements (Pitea, Favini & Grasso, 1970).

No intermolecular distance in (I), (II) and (III) is shorter than the sum of the respective van der Waals radii (Zefirov, 1976). The molecular volumes are similar and comparable with those of BA, TA, TS and TO: 245 (I), 246 (II), 240 (III), 253 (BA), 246 (TA), 258 (TS, room temperature) and 262 Å^3 (TO).

The conformations of all molecules in the crystal structures are apparently more or less influenced by lattice forces. This follows from a comparison with the α values of the dissolved molecules as derived from ^{13}C NMR spectra: 34 (I), 33 (II) and 41° (III) (Denecke, Müller & Bluhm, 1982).* Furthermore, the differences of corresponding torsion angles of the two independent molecules in the quasi-heteromolecular compounds (Zorkii & Razumaeva, 1979) (I) and (III) are large and statistically significant. The angles of (I) are in good

---

* From the chemical shifts α cannot be distinguished from $\alpha - 180°$, which is relevant for (III), and β cannot be distinguished from $-\beta, \beta - 180°$ and $-\beta + 180°$, which is relevant for (I).

agreement with those of TA. The series of the isostructural compounds (I), TA, TS and TO suggests that their molecular packing is an effective arrangement for this kind of molecule and that crystal forces decisively determine the conformations. The angle β is small in all three diazastilbene crystal structures and is in accordance with the corresponding angle of approximately 10° for the dissolved molecules (Denecke, Müller & Bluhm, 1982).* Small values of β were also determined in the crystal structures of BA and most of the substituted benzylideneanilines (Bar & Bernstein, 1977; Nakai, Ezumi & Shiro, 1981).

The bonds in the central parts of the molecules of (II) and (III) show the same trend as was observed for substituted benzylideneanilines: the N(7)=C(8) bonds are lengthened while the C(4)—N(7) and C(8)—C(9) bonds are shortened in comparison with those of BA (Bar & Bernstein, 1977). The central bonds of both molecules in (I) are unexpectedly short but this may be caused by the disorder in the crystal structure. The geometries of the pyridine rings in (II) and (III) are compatible with those of pyridine (Mootz & Wussow, 1981) and some salicylideneaminopyridines (Moustakali-Mavridis, Hadjoudis & Mavridis, 1978, 1980).

---

* See previous footnote.

References

BAR, I. & BERNSTEIN, J. (1977). *Acta Cryst.* B33, 1738–1744.
BERNSTEIN, J. (1975). *Acta Cryst.* B31, 1268–1271.
BERNSTEIN, J., BAR, I. & CHRISTENSEN, A. (1976). *Acta Cryst.* B32, 1609–1611.
BERNSTEIN, J. & HAGLER, A. T. (1978). *J. Am. Chem. Soc.* 100, 673–681.
BERNSTEIN, J. & HAGLER, A. T. (1979). *Mol. Cryst. Liq. Cryst.* 50, 223–234.
BERNSTEIN, J. & IZAK, I. (1975). *J. Cryst. Mol. Struct.* 5, 257–266.
BERNSTEIN, J. & SCHMIDT, G. M. J. (1972). *J. Chem. Soc. Perkin Trans.* 2, pp. 951–955.
BLUHM, T., KNOP, J. V. & BEHJATI, B. (1979). *Croat. Chem. Acta*, 52, 29–34.
BROWN, C. J. (1966). *Acta Cryst.* 21, 146–152.
BÜRGI, H. B. & DUNITZ, J. D. (1970). *Helv. Chim. Acta*, 53, 1747–1764.
BÜRGI, H. B. & DUNITZ, J. D. (1971). *Helv. Chim. Acta*, 54, 1255–1260.
CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71–98. Birmingham: Kynoch Press.
DENECKE, E., MÜLLER, K. & BLUHM, T. (1982). *Org. Magn. Reson.* 18, 68–70.

FINDER, C. J., NEWTON, M. G. & ALLINGER, N. L. (1974). *Acta Cryst.* B30, 411–415.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368–376.

HOEKSTRA, A., MEERTENS, P. & VOS, A. (1975). *Acta Cryst.* B31, 2813–2817.

JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

MAVRIDIS, A. & MOUSTAKALI-MAVRIDIS, I. (1977). *Acta Cryst.* B33, 3612–3615.

MOOTZ, D. & WUSSOW, H.-G. (1981). *J. Chem. Phys.* 75, 1517–1522.

MOUSTAKALI-MAVRIDIS, I., HADJOUDIS, E. & MAVRIDIS, A. (1978). *Acta Cryst.* B34, 3709–3715.

MOUSTAKALI-MAVRIDIS, I., HADJOUDIS, E. & MAVRIDIS, A. (1980). *Acta Cryst.* B36, 1126–1130.

NAKAI, H., EZUMI, K. & SHIRO, M. (1981). *Acta Cryst.* B37, 193–197.

PERKAMPUS, H. H. & BEHJATI, B. (1974). *J. Heterocycl. Chem.* 11, 511–514.

PITEA, D., FAVINI, G. & GRASSO, D. (1970). *Gazz. Chim. Ital.* 100, 301–316.

TRÆTTEBERG, M., HILMO, I., ABRAHAM, R. J. & LJUNGGREN, S. (1978). *J. Mol. Struct.* 48, 395–405.

WOLF, A. (1980). *J. Mol. Struct.* 67, 89–99.

ZEFIROV, YU. V. (1976). *J. Gen. Chem. USSR*, 46, 2519–2523.

ZORKII, P. M. & RAZUMAEVA, A. E. (1979). *J. Struct. Chem.* 20, 390–393.