

are different, an appreciable conjugation within the  $N^1-C=N^2$  system can be expected. Therefore the rotation around the  $N^1-C$  bond is hindered and, in the case of unsymmetrically substituted  $N^1$ , two isomeric forms are possible. One can therefore conclude that the bulky methyl group can cause greater steric hindrance in a crystal structure than a flat phenyl ring.

Molecular packing is shown in Fig. 2. No intermolecular contacts significantly shorter than the sum of the van der Waals radii are observed.

This work was supported by Project RP.II.10.6.1.5.(MNIŠW).

#### References

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

- JASKÓLSKI, M. (1982). *Fourth Symposium on Organic Crystal Chemistry*, Poznań, September 1982, edited by Z. KALUSKI, pp. 70–71. A. Mickiewicz Univ.
- JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
- LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* **A30**, 580–584.
- 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.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- NORRESTAM, R., MERTZ, S. & CROSSLAND, I. (1983). *Acta Cryst.* **C39**, 1554–1556.
- OSZCZAPOWICZ, J., RACZYŃSKA, E. & ORLIŃSKI, R. (1981). *Pol. J. Chem.* **55**, 2031–2037.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- TYKARSKA, E., JASKÓLSKI, M. & KOSTURKIEWICZ, Z. (1986). *Acta Cryst.* **C42**, 740–743.

*Acta Cryst.* (1986). **C42**, 1818–1821

## Structure of 1-[[*(*4-Carbamoylpyridinio)methoxy]methyl]-2-[(hydroxyimino)methyl]pyridinium Dichloride (*A*) and Diiodide (*B*) Monohydrates

BY BORIS KAMENAR, IVAN VICKOVIĆ AND MILENKO BRUVO

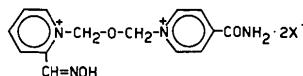
*Laboratory of General and Inorganic Chemistry, Faculty of Science, The University, PO Box 153, 41001 Zagreb, Yugoslavia*

(Received 7 April 1986; accepted 8 July 1986)

**Abstract.** (*A*):  $C_{14}H_{16}N_4O_3^{2+} \cdot 2Cl^- \cdot H_2O$ ,  $M_r = 377.23$ , monoclinic,  $P2_1/n$ ,  $a = 13.348$  (9),  $b = 8.982$  (6),  $c = 15.214$  (9) Å,  $\beta = 106.09$  (5)°,  $V = 1752.6$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.430$  Mg m<sup>-3</sup>,  $\lambda(Cu K\alpha) = 1.5418$  Å,  $\mu(Cu K\alpha) = 3.583$  mm<sup>-1</sup>,  $F(000) = 784$ ,  $T = 298$  K, final  $R = 0.054$  for 1846 observed reflections. (*B*):  $C_{14}H_{16}N_4O_3^{2+} \cdot 2I^- \cdot H_2O$ ,  $M_r = 560.13$ , triclinic,  $P\bar{1}$ ,  $a = 18.448$  (7),  $b = 7.964$  (4),  $c = 8.032$  (4) Å,  $\alpha = 119.85$  (4),  $\beta = 95.46$  (3),  $\gamma = 93.48$  (3)°,  $V = 1010.9$  (9) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.840$  Mg m<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.7107$  Å,  $\mu(Mo K\alpha) = 3.174$  mm<sup>-1</sup>,  $F(000) = 536$ ,  $T = 298$  K, final  $R = 0.075$  for 4368 observed reflections. The molecules of both compounds have similar but not identical shapes: the angle between the best planes through the two pyridinium rings amounts to 20.2 (1)° in (*A*) and 45.2 (4)° in (*B*). The interatomic distances between N from pyridine ring (i) and its oxime O and between N from pyridine ring (ii) and its carbonyl O are 4.638 (5) and 4.910 (6) Å in (*A*), and 4.605 (15) and 4.947 (14) Å in (*B*), respectively, and are very similar to such distances in nicotine and in the

nicotinic conformation of acetylcholine. This could be the reason for their antisoman activity.

**Introduction.** In recent years considerable attention has been given to the study of a bispyridinium oxime known under the code name HI-6 as a very efficient antidote against soman poisoning (Bošković, 1981). In order to contribute to the elucidation of the structure–activity relationship as well as to the explanation of the possible mechanism of the antidotal activity of this class of compounds we have undertaken the X-ray structure analysis of compound HI-6 in the form of its chloride and iodide (Kamenar & Vicković, 1984; Binenfeld, Deljac, Kamenar & Vicković, 1984).



**Experimental.** (*A*) Prism-shaped specimen 0.15 × 0.04 × 0.44 mm. Unit cell from 18 reflections (12 ≤

$2\theta \leq 28^\circ$ ). Intensity data collected on a Philips PW 1100 diffractometer, graphite-monochromated Cu  $K\alpha$  radiation,  $\theta$ - $2\theta$  scan,  $8 \leq 2\theta \leq 140^\circ$  ( $h: -16 \rightarrow 15$ ,  $k: 0 \rightarrow 10$ ,  $l: 0 \rightarrow 18$ ), scan rate  $0.03^\circ \text{ s}^{-1}$ , scan width  $1.2^\circ$ . Three standard reflections monitored every 2 h: no significant intensity variations. 1888 unique reflections, seven (400, 311,  $\bar{4}02$ ,  $\bar{2}02$ , 202, 312, 212) rejected because of too great a difference between  $F_o$  and  $F_c$ , 35 with  $I \leq 3\sigma(I)$ . Lorentz, polarization and absorption corrections applied. Structure solved by direct methods using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) with 296 largest  $|E|$  values in the range  $3.52 > |E| > 1.34$ . Refinement on  $F$  by full-matrix least-squares method using *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976); anisotropic thermal parameters for all non-hydrogen atoms. All hydrogen atoms found in a difference Fourier map, except the H atom attached to C(6), which was placed at calculated position. Unit weight for all observations. Final  $R = 0.054$  ( $wR = 0.063$ ),  $S = 1.8$ , 282 parameters refined. Max. and min. heights in final difference Fourier synthesis:  $0.47$  and  $0.99 \text{ e } \text{Å}^{-3}$ .  $(\Delta/\sigma)_{\text{max}} = 0.23$  for non-hydrogen atoms. Scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed on a UNIVAC 1110 computer. (B) Crystal  $0.25 \times 0.35 \times 0.48 \text{ mm}$ . Unit cell from 16 reflections ( $10 \leq 2\theta \leq 14^\circ$ ). Philips PW 1100 diffractometer, graphite-monochromated Mo  $K\alpha$  radiation,  $\theta$ - $2\theta$  scan,  $6 \leq 2\theta \leq 60^\circ$  ( $h: -25 \rightarrow 25$ ,  $k: -11 \rightarrow 9$ ,  $l: 0 \rightarrow 11$ ), scan rate  $0.05^\circ \text{ s}^{-1}$ , scan width  $1.1^\circ$ . No intensity variation of

standard reflections. Corrections for Lorentz, polarization and absorption effects (Harkema, 1979). 4534 independent reflections measured, 4368 observed [ $I \geq 3\sigma(I)$ ]. Direct methods (*MULTAN80*) with 346 largest  $|E|$  values in the range  $2.60 > |E| > 1.67$ . All H atoms belonging to the bispyridinium monooxime cation found in a difference Fourier map, but not H atoms from water molecule. Full-matrix least-squares anisotropic refinement of all non-H atoms.  $R = 0.074$  ( $wR = 0.087$ ),  $S = 11.8$ , 217 parameters refined, unit weight assumed for all observations,  $(\Delta/\sigma)_{\text{max}} = 1.0$  for non-H atoms. Relatively high value of  $R$  owing to poor quality of the crystals. Max. and min. heights in final Fourier map:  $4.7$  and  $1.6 \text{ e } \text{Å}^{-3}$ . Other details as for (A).

**Discussion.** Final atomic coordinates are given in Table 1,\* bond lengths, angles and some selected torsion angles in Table 2. Structures of both chloride and iodide derivatives are shown in Fig. 1.

The structures of both compounds (A) and (B) consist of bispyridinium cations, chloride or iodide anions and water molecules. The bond lengths and angles in the cations are within expected values. The configurations of the antidote cations (A) and (B) are

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43212 (55 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{Å}^2 \times 10^3$ ) for compounds (A) and (B)

$$U_{\text{eq}} = \frac{1}{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{13}aca^*c^*\cos\beta + 2U_{23}bcb^*c^*\cos\alpha + 2U_{12}aba^*b^*\cos\gamma].$$

	Compound (A)				Compound (B)			
	$x$	$y$	$z$	$U_{\text{eq}}$	$x$	$y$	$z$	$U_{\text{eq}}$
Cl(1) or I(1)	3148 (1)	-1205 (2)	3486 (1)	47 (0.4)	1813 (1)	9253 (2)	4172 (2)	68 (0.3)
Cl(2) or I(2)	6275 (1)	993 (1)	2593 (1)	42 (0.4)	3575 (1)	3877 (1)	10082 (1)	50 (0.2)
O(w)	4093 (3)	-559 (5)	1837 (3)	66 (1)	3016 (8)	9209 (21)	9825 (20)	112 (4)
O(1)	6254 (3)	1595 (4)	-167 (2)	47 (1)	-292 (6)	2058 (17)	1343 (16)	66 (3)
O(2)	4963 (3)	4607 (4)	1966 (2)	37 (1)	2131 (5)	4451 (13)	4551 (13)	44 (2)
O(3)	3920 (3)	2815 (4)	6192 (2)	52 (1)	5858 (5)	9162 (15)	6804 (15)	55 (3)
N(1)	6217 (3)	5929 (4)	1481 (2)	27 (1)	1389 (6)	4589 (14)	6890 (15)	38 (2)
N(2)	6248 (3)	3129 (4)	-172 (3)	37 (1)	-128 (7)	2222 (17)	3118 (17)	52 (3)
N(3)	4201 (3)	4468 (4)	3193 (3)	30 (1)	3417 (5)	5476 (14)	5534 (14)	35 (2)
N(4)	3764 (3)	612 (5)	5451 (3)	37 (1)	5139 (7)	11584 (17)	7864 (18)	54 (3)
C(1)	6329 (4)	5272 (5)	700 (3)	28 (1)	743 (7)	3481 (18)	5892 (19)	40 (3)
C(2)	6498 (4)	6201 (6)	24 (3)	34 (1)	322 (8)	2658 (21)	6658 (23)	55 (4)
C(3)	6513 (4)	7723 (6)	122 (4)	42 (1)	580 (11)	2939 (26)	8490 (26)	71 (5)
C(4)	6407 (5)	8335 (6)	925 (4)	44 (1)	1235 (11)	4050 (28)	9456 (24)	72 (5)
C(5)	6270 (4)	7427 (6)	1592 (4)	37 (1)	1641 (9)	4855 (21)	8657 (21)	54 (4)
C(6)	6288 (4)	3649 (6)	611 (4)	35 (1)	514 (7)	3153 (20)	3944 (20)	45 (3)
C(7)	6023 (4)	5013 (6)	2226 (3)	32 (1)	1818 (7)	5683 (19)	6182 (20)	45 (3)
C(8)	4307 (4)	5373 (6)	2384 (3)	37 (1)	2803 (7)	3896 (18)	4950 (21)	45 (3)
C(9)	3785 (4)	5145 (6)	3798 (4)	43 (1)	4053 (7)	5372 (19)	6402 (20)	44 (3)
C(10)	3667 (4)	4361 (6)	4542 (4)	42 (1)	4648 (7)	6717 (20)	6908 (19)	43 (3)
C(11)	3942 (4)	2872 (5)	4650 (3)	30 (1)	4581 (7)	8270 (17)	6578 (16)	34 (3)
C(12)	4342 (5)	2219 (5)	3999 (4)	44 (2)	3921 (7)	8360 (18)	5712 (18)	38 (3)
C(13)	4464 (5)	3042 (6)	3274 (4)	49 (2)	3341 (7)	6928 (19)	5161 (19)	39 (3)
C(14)	3863 (4)	2084 (6)	5496 (3)	35 (1)	5259 (7)	9706 (19)	7115 (18)	41 (3)

similar but not identical, most probably owing to different hydrogen bonding between neighbouring ions and water molecules. The angles between the least-squares best planes through the pyridinium rings (i) and (ii) are 20.2 (1) and 45.2 (4)° in (A) and (B), respectively. Whereas in (A) the N atoms from carbamoyl groups, chloride ions, water O and oxime O atoms participate in the three-dimensional intermolecular hydrogen bonds [N(4)···Cl(1) 3.304 (5),

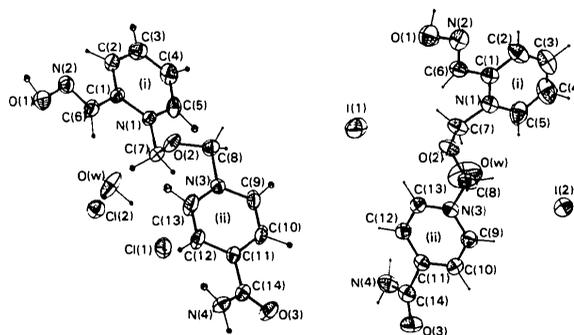


Fig. 1. Perspective view of the structures of the compounds (A) (left) and (B) (right) with atom labelling scheme.

Table 2. Bond lengths (Å), bond angles (°) and selected torsion angles (°) for compounds (A) and (B)

	(A)	(B)
O(1)—N(2)	1.378 (5)	1.367 (20)
O(2)—C(7)	1.408 (6)	1.395 (16)
O(2)—C(8)	1.398 (7)	1.402 (17)
O(3)—C(14)	1.230 (6)	1.214 (16)
N(1)—C(1)	1.371 (6)	1.352 (15)
N(1)—C(5)	1.355 (7)	1.356 (21)
N(1)—C(7)	1.480 (6)	1.485 (22)
N(2)—C(6)	1.267 (8)	1.283 (17)
N(3)—C(8)	1.514 (7)	1.492 (16)
N(3)—C(9)	1.345 (8)	1.335 (18)
N(3)—C(13)	1.325 (7)	1.343 (22)
N(4)—C(14)	1.329 (7)	1.348 (19)
C(1)—C(2)	1.390 (7)	1.357 (26)
C(1)—C(6)	1.464 (7)	1.466 (23)
C(2)—C(3)	1.375 (8)	1.403 (29)
C(3)—C(4)	1.382 (9)	1.361 (25)
C(4)—C(5)	1.353 (9)	1.351 (31)
C(9)—C(10)	1.379 (9)	1.363 (19)
C(10)—C(11)	1.385 (7)	1.398 (24)
C(11)—C(12)	1.380 (8)	1.368 (19)
C(11)—C(14)	1.498 (7)	1.514 (18)
C(12)—C(13)	1.374 (9)	1.379 (18)
C(7)—O(2)—C(8)	116.9 (4)	114.9 (11)
C(1)—N(1)—C(5)	121.4 (4)	120.6 (13)
C(1)—N(1)—C(7)	120.6 (4)	121.8 (12)
C(5)—N(1)—C(7)	118.1 (4)	117.4 (12)
O(1)—N(2)—C(6)	111.4 (4)	111.0 (13)
C(8)—N(3)—C(9)	117.6 (4)	117.6 (11)
C(8)—N(3)—C(13)	120.8 (4)	121.4 (11)
C(9)—N(3)—C(13)	121.5 (5)	121.0 (12)
N(1)—C(1)—C(2)	117.5 (4)	120.9 (14)
N(1)—C(1)—C(6)	119.9 (4)	118.5 (13)
C(2)—C(1)—C(6)	122.6 (5)	120.6 (14)
C(1)—C(2)—C(3)	121.1 (5)	118.6 (16)
C(2)—C(3)—C(4)	119.3 (5)	119.3 (18)
C(3)—C(4)—C(5)	119.4 (6)	120.7 (19)
N(1)—C(5)—C(4)	121.2 (5)	119.9 (16)
N(2)—C(6)—C(5)	116.4 (5)	116.1 (13)
O(2)—C(7)—N(1)	107.7 (4)	112.2 (11)
O(2)—C(8)—N(3)	108.5 (4)	110.9 (11)
N(3)—C(9)—C(10)	119.6 (5)	121.1 (13)
C(9)—C(10)—C(11)	120.3 (5)	119.3 (13)
C(10)—C(11)—C(12)	117.9 (5)	118.6 (13)
C(10)—C(11)—C(14)	118.6 (4)	117.7 (12)
C(12)—C(11)—C(14)	123.3 (5)	123.7 (12)
C(11)—C(12)—C(13)	120.2 (5)	120.2 (13)
N(3)—C(13)—C(12)	120.5 (5)	119.9 (13)
O(3)—C(14)—N(4)	123.9 (5)	123.4 (14)
O(3)—C(14)—C(11)	119.0 (4)	121.3 (14)
N(4)—C(14)—C(11)	117.1 (4)	115.2 (12)
O(1)—N(2)—C(6)—C(1)	177.6 (4)	178.0 (12)
O(2)—C(7)—N(1)—C(1)	-78.2 (5)	-70.6 (16)
O(2)—C(7)—N(1)—C(5)	101.2 (5)	114.2 (14)
O(2)—C(8)—N(3)—C(9)	166.3 (4)	164.1 (12)
O(2)—C(8)—N(3)—C(13)	-16.8 (6)	-17.5 (17)
O(3)—C(14)—C(11)—C(10)	-25.2 (7)	40.3 (20)
O(3)—C(14)—C(11)—C(12)	150.3 (5)	-136.6 (15)
N(1)—C(1)—C(6)—N(2)	170.3 (5)	-174.7 (13)
N(1)—C(7)—O(2)—C(8)	-103.7 (4)	-84.0 (14)
N(2)—C(6)—C(1)—C(2)	-10.9 (8)	6.5 (22)
N(3)—C(8)—O(2)—C(7)	-95.5 (5)	-79.5 (14)
N(4)—C(14)—C(11)—C(10)	156.7 (5)	-142.7 (14)
N(4)—C(14)—C(11)—C(12)	-27.9 (7)	40.4 (19)

N(4)···Cl(2<sup>i</sup>) 3.320 (5), O(w)···Cl(1) 3.158 (5), O(w)···Cl(2) 3.145 (4) and O(1)···O(w<sup>ii</sup>) 2.624 (6) Å [(i) 1 - x, - y, - z; (ii) 1 - x, 1 - y, - z], the only significant interactions in (B) are N(4)···I(2) of 3.64 (1) and N(4)···I(2<sup>iii</sup>) of 3.70 (1) Å [(iii) 1 - x, 2 - y, 2 - z] (Hamilton & Ibers, 1968).

The compound HI-6 with CONH<sub>2</sub> group in position 4 of the pyridinium ring (ii) is structurally similar to the analogous antidotes HS-6 and HGG-12 with either a carbamoyl or a benzoyl group in position 3. However, the interatomic distances between pyridinium N and oxime O [ring (i)] and between pyridinium N and carbonyl O [ring (ii)] which amount to 4.62 and 4.71 Å in HS-6, and 4.64 and 4.61 Å in HGG-12 are characteristic. Such values agree well with those found for the corresponding distances in acetylcholine with nicotinic conformation (4.93 Å) and in nicotine (4.76 Å) (Su, Tang, Ma, Shin, Liu & Wu, 1983). The present results show that this agreement is even better in the case of HI-6 in which the distances between N(1) and O(1) and N(3) and O(3) are 4.638 (5) and 4.910 (6) Å, and 4.605 (15) and 4.947 (14) Å in (A) and (B), respectively. This may suggest that the antisoman activity of the bispyridinium monooxime carbonyl derivatives does not depend upon the position (3 or 4) of the carbonyl substituent.

The authors thank Professor Z. Binenfeld for suggesting the problem, Dr V. Deljac for preparing the compounds, and the Foundation for Scientific Research of SR Croatia, Zagreb, for financial support.

#### References

- BINENFELD, Z., DELJAC, V., KAMENAR, B. & VICKOVIĆ, I. (1984). *Acta Pharm. Jugosl.* **34**, 195–199.  
BOŠKOVIĆ, B. (1981). *Fund. Appl. Toxicol.* **1**, 203–213.

- HAMILTON, W. C. & IBERS, J. A. (1968). *Hydrogen Bonding in Solids*, pp. 259–265. New York: Benjamin.
- HARKEMA, S. (1979). *Program to Perform Absorption Correction for X-ray Diffraction Data for Use in Connection with a PW 1100 Diffractometer*. Chemical Physics Laboratory, Twente, Univ. of Technology, The Netherlands.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- KAMENAR, B. & VICKOVIĆ, I. (1984). *God. Jugosl. Cent. Kristalogr.* **19**, 62–63.
- MAIN, P., FISKE, S., 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.
- STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- SU, C.-T., TANG, C.-P., MA, C., SHIN, Y.-S., LIU, C.-Y. & WU, M.-T. (1983). *Fund. Appl. Toxicol.* **3**, 271–277.

*Acta Cryst.* (1986). **C42**, 1821–1823

## 2,2'-Azobis(1-morpholino-1-phenylethylene)

BY TULLIO PILATI AND RICCARDO BIANCHI

Centro del CNR per lo Studio delle Relazioni fra Struttura e Reattività chimica, via Golgi 19, 20133 Milano, Italy

(Received 16 April 1986; accepted 8 July 1986)

**Abstract.**  $C_{24}H_{28}N_4O_2$ ,  $M_r = 404.5$ , triclinic,  $P\bar{1}$ ,  $a = 6.040$  (1),  $b = 13.466$  (4),  $c = 14.626$  (4) Å,  $\alpha = 111.03$  (3),  $\beta = 99.92$  (2),  $\gamma = 91.34$  (2)°,  $V = 1089.1$  (5) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.22$ ,  $D_x = 1.23$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.07$  mm<sup>-1</sup>,  $F(000) = 432$ , room temperature,  $R = 0.057$ ,  $wR = 0.042$  for 3036 observed reflections [ $I > 0.5\sigma(I)$ ]. The two molecules in the unit cell occupy sites of symmetry  $\bar{1}$ , and they have different conformations. The dihedral angle between the planes through the phenyl ring and the ethene system is 116.2 (2)° for molecule *A* and 125.6 (2)° for molecule *B*.

**Introduction.** The reaction of methyl azido(phenylhydrazono)acetate with enamines leads to different kinds of ring-closed products (Bruché, Garanti & Zecchi, 1985). However, the reaction with 1-morpholino-1-phenylethylene yields the title compound as the main product, whose structure has been determined by X-ray diffraction to confirm and complement the spectroscopic and analytical results. This study is connected with a line of research dealing with the synthetic potential of *C*-azidohydrazones.

**Experimental.**  $D_m$  by flotation in dilute  $K_2HgI_4$  solution; yellow-orange prism  $0.25 \times 0.20 \times 0.08$  mm. Intensity data collected with an Enraf–Nonius CAD-4 diffractometer, graphite-monochromated  $Mo\ K\alpha$  radiation,  $\theta/2\theta$  scan technique, variable rate  $2\text{--}10^\circ \text{ min}^{-1}$ . Corrections for Lorentz and polarization but not for absorption. Structure solved using direct-methods program *MULTAN* (Germain, Main & Woolfson, 1971), H atoms from difference Fourier synthesis.

Full-matrix least-squares refinement of scale factor, secondary-extinction parameter  $g$  (Larson, 1967, equation 3), anisotropic heavy atoms and isotropic H atoms; quantity minimized  $\sum w(|F_o| - |F_c|)^2$ . Scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used include *SDP* (Enraf–Nonius, 1979), *ORTEP* (Johnson, 1965) and various in-house programs for refinement and geometrical analysis running on a Gould 32/97 computer. Details of data collection and refinement listed in Table 1.

Table 1. *Details of data collection and structure refinement*

<i>(a)</i> Data collection	
Cell-parameter determination:	
No.; range of reflection (°)	25; 10–12
Range: $h$	0,7
$k$	–16,16
$l$	–17,17
$\theta$ (°)	0.0–25.0
Standard reflections	
No.; variation of intensity	3; 2%
No. of intensity measurements	3818
No. of unique reflections	3818
<i>(b)</i> Refinement	
Refined reflections:	
No.; criterion for use	3036; $I > 0.5\sigma(I)$
$R$ ; $wR$	0.057; 0.042
Goodness of fit, $S$	1.25
Weighting scheme; $a$	$w = 4F_o/[ \sigma^2(F_o^2) + aF_o^2 ]$ ; 0.0004
$(\Delta/\sigma)_{\max}$ in the last cycle	< 0.01
$ \Delta\rho _{\max}$ in final difference Fourier map ( $e\ \text{Å}^{-3}$ )	0.2
Extinction coefficient $g(\times 10^6)$	18 (2)