par centrosymétrie, les configurations des mêmes atomes sont donc respectivement *S* et *R*.

La Fig. 2 montre que les cations forment des couches qui se développent au voisinage des plans (011). Dans une même couche, près de l'axe [100], se trouvent deux noyaux benzimidazolyl qui se correspondent par un centre de symétrie. La distance entre leurs plans moyens est d'environ 3,47 Å, c'est-à-dire comparable à celle qui sépare deux couches consécutives d'atomes de carbone dans le graphite. En outre, un dessin représentant les deux noyaux, vus selon la normale à *P*(*AB*), permet de constater qu'ils se recouvrent partiellement, un atome d'azote de l'un d'eux se superposant au noyau benzénique de l'autre. Autrement dit, à l'état cristallisé, les cations tendent à former des dimères. Les distances les plus courtes entre les atomes des deux groupements benzimidazolyl sont C(3a)—C(3a^{vi}) [3,529 (2) Å], N(3)—C(4^{vi}) [3,557 (2) Å] et C(2)—C(4^{vi}) [3,534 (2) Å] [(vi): 1—*x*, 2—*y*, −*z*]. De nombreuses liaisons hydrogène participent aussi à la cohésion de la structure (Fig. 2).

Ainsi, les cations sont associés aux anions par les liaisons N(11)—H(11)···O(25^{i}), N(11)—H′(11)···O(28^{ii}), O(21)—H(21)···O(25^{ii}) et O(22)—H(22)···O(28^{iii}). Entre les molécules d'eau s'établissent les liaisons O(29)—H(29)···O(31^{v}), O(30)—H(30)···O(29) et O(31)—H(31)···O(29). Les molécules d'eau sont unies aux anions par O(30)—H′(30)···O(28^{iii}) et aux cations par O(29)—H(29)···N(1^{iv}) et par O(31)—H′(31)···O(22). Les longueurs et les angles de ces liaisons sont rapportés dans le Tableau 2. A une exception près, leurs longueurs sont comprises entre 2,714 (1) et 2,884 (2) Å et leurs angles entre 161 (2) et 178 (2)°. L'exception est constituée par O(31)—H(31)···O(29) [3,016 (3) Å, 154 (2)°].

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Structure of N^1,N^1-(3-Oxapentamethylene)-N^2-phenylacetamidine

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Abstract. *N*-(1-Morpholinoethylidene)aniline, $C_{12}H_{16}N_2O$, $M_r = 204{\cdot}27$, orthorhombic, *Pbca*, $a = 9{\cdot}970$ (1), $b = 33{\cdot}715$ (3), $c = 6{\cdot}687$ (1) Å, $V = 2247{\cdot}8$ (5) Å^3, $Z = 8$, $D_m = 1{\cdot}20$, $D_x = 1{\cdot}21$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 5{\cdot}43$ cm^{-1}, $F(000) = 880$, room temperature, $R = 0{\cdot}043$ for 1362 observed reflexions. The molecule has a *trans* [N^2 (*E*)] configuration. The N=C(—C)—N amidine group is nonplanar. The morpholine ring is in a chair conformation. The amidine bond lengths C—N^1 and C=N^2 are differentiated: 1·371 (2) and 1·283 (2) Å, respectively.

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Introduction. The present study is part of a series of structural works on amidine derivatives (Ciszak, Gdaniec, Jaskólski, Kosturkiewicz, Owsiański & Tykarska, 1989). The amidine system tends to be planar owing to the mesomeric effect. Earlier papers (Norrestam, Mertz & Crossland, 1983; Ciszak, Gdaniec & Kosturkiewicz, 1986; Ciszak, Gdaniec, Jaskólski & Kosturkiewicz, 1988) reported that the acetamidine fragment N=C(—C)—N is planar with both N atoms having sp^2 hybridization. It was of interest to investigate the changes in the amidine system brought about when N^1 belongs to a morpholine ring.

Experimental. Colourless plate-shaped crystals obtained from absolute ethanol, density by flotation, approximate unit-cell dimensions and space group from Weissenberg photographs. Crystal 0·5 × 0·5 × 0·4 mm, Syntex $P2_1$ diffractometer, Cu $K\alpha$ radiation, graphite monochromator, precise cell parameters from least-squares treatment of setting angles of 15 reflexions with $18 \le 2\theta \le 29°$. No absorption correction. 1503 unique reflexions with $2\theta \le 115°$ measured in the range h 0–10, k 0–36, l 0–7. No significant intensity variation (±2·4%) for two standard reflexions ($\bar{2}1\bar{1}$, $\bar{2}8\bar{1}$) monitored every 100 reflexions. Peak profile analysis according to Lehmann & Larsen (1974). 1362 observed reflexions with $I \ge 1{\cdot}96\sigma(I)$. Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All H atoms from $\Delta\rho$ map. Full-matrix least-squares minimization of $\sum w(\Delta F)^2$, anisotropic non-H atoms and isotropic H atoms. F_c values multiplied by $(1 - xF_c^2/\sin 2\theta)$ where x is the empirical isotropic extinction parameter refined to $10\,(4) \times 10^{-7}$; $R = 0{\cdot}043$, $wR = 0{\cdot}059$, $S = 3{\cdot}11$, $w = 1/[\sigma^2(F_o) + 0{\cdot}00025F_o^2]$, $(\Delta/\sigma)_{max}$ in last refinement cycle 0·04, $(\Delta\rho)_{max} = 0{\cdot}10$, $(\Delta\rho)_{min} = -0{\cdot}11$ e Å^{-3}. Computer programs: *MULTAN*80 (Main *et al.*, 1980), *SHELX*76 (Sheldrick, 1976) and local programs (Jaskólski, 1982). Molecular illustrations drawn using *PLUTO* (Motherwell & Clegg, 1978) and *ORTEP* (Johnson, 1976). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed in the Computer Centre of A. Mickiewicz University.

Discussion. Atomic coordinates are given in Table 1, bond distances, angles and selected torsion angles in Table 2.*

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_k U_{ik}a_i^*a_k^*(\mathbf{a}_i.\mathbf{a}_k).$$

	x	y	z	U_{eq}
C(1)	0·6438 (1)	0·12177 (4)	0·1416 (2)	0·0430 (5)
C(2)	0·6104 (2)	0·16280 (4)	0·4204 (2)	0·0429 (5)
C(3)	0·6916 (2)	0·19327 (5)	0·4888 (2)	0·0507 (6)
C(4)	0·6378 (2)	0·22487 (5)	0·5919 (3)	0·0578 (7)
C(5)	0·5023 (2)	0·22673 (5)	0·6307 (3)	0·0580 (7)
C(6)	0·4210 (2)	0·19659 (6)	0·5646 (3)	0·0590 (7)
C(7)	0·4735 (2)	0·16494 (6)	0·4602 (3)	0·0530 (6)
C(8)	0·6609 (2)	0·07088 (7)	−0·1273 (3)	0·0622 (7)
C(9)	0·7783 (2)	0·05313 (6)	−0·2369 (3)	0·0681 (7)
C(10)	0·8992 (2)	0·04609 (6)	0·0586 (3)	0·0639 (7)
C(11)	0·7876 (2)	0·06329 (6)	0·1796 (3)	0·0558 (6)
C(12)	0·5584 (2)	0·14679 (6)	0·0057 (3)	0·0574 (7)
N(1)	0·7067 (1)	0·08984 (4)	0·0559 (2)	0·0500 (5)
N(2)	0·6683 (1)	0·12950 (4)	0·3259 (2)	0·0473 (5)
O(1)	0·8519 (1)	0·02655 (4)	−0·1164 (2)	0·0713 (5)

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

C(1)—N(1)	1·371 (2)	C(5)—C(6)	1·373 (3)
C(1)—N(2)	1·283 (2)	C(6)—C(7)	1·378 (3)
C(1)—C(12)	1·504 (3)	C(8)—C(9)	1·505 (3)
C(2)—C(3)	1·385 (2)	C(8)—N(1)	1·455 (3)
C(2)—C(7)	1·393 (2)	C(9)—O(1)	1·411 (3)
C(2)—N(2)	1·412 (2)	C(10)—C(11)	1·493 (3)
C(3)—C(4)	1·378 (2)	C(10)—O(1)	1·424 (3)
C(4)—C(5)	1·377 (3)	C(11)—N(1)	1·462 (2)

C(12)—C(1)—N(1)	116·5 (1)	C(2)—C(7)—C(6)	120·6 (2)
C(12)—C(1)—N(2)	125·1 (1)	C(9)—C(8)—N(1)	109·9 (2)
N(1)—C(1)—N(2)	118·3 (1)	C(8)—C(9)—O(1)	112·3 (2)
C(3)—C(2)—C(7)	118·1 (1)	C(11)—C(10)—O(1)	112·2 (2)
C(3)—C(2)—N(2)	119·9 (1)	C(10)—C(11)—N(1)	110·0 (1)
C(7)—C(2)—N(2)	121·8 (1)	C(1)—N(1)—C(8)	123·6 (1)
C(2)—C(3)—C(4)	120·8 (1)	C(1)—N(1)—C(11)	119·8 (1)
C(3)—C(4)—C(5)	120·8 (2)	C(8)—N(1)—C(11)	112·4 (1)
C(4)—C(5)—C(6)	119·0 (2)	C(1)—N(2)—C(2)	120·9 (1)
C(5)—C(6)—C(7)	120·8 (2)	C(9)—O(1)—C(10)	110·4 (1)

C(12)—C(1)—N(1)—C(8)	−22·9 (2)	C(7)—C(2)—N(2)—C(1)	68·8 (2)
C(12)—C(1)—N(1)—C(11)	−177·8 (2)	N(1)—C(8)—C(9)—O(1)	55·4 (2)
N(2)—C(1)—N(1)—C(8)	161·4 (2)	C(9)—C(8)—N(1)—C(1)	150·7 (2)
N(2)—C(1)—N(1)—C(11)	6·4 (2)	C(9)—C(8)—N(1)—C(11)	−52·7 (2)
C(12)—C(1)—N(2)—C(2)	2·4 (2)	C(8)—C(9)—O(1)—C(10)	−58·1 (2)
N(1)—C(1)—N(2)—C(2)	177·7 (2)	O(1)—C(10)—C(11)—N(1)	−55·4 (2)
N(2)—C(2)—C(3)—C(4)	−176·1 (2)	C(11)—C(10)—O(1)—C(9)	58·2 (2)
N(2)—C(2)—C(7)—C(6)	175·6 (2)	C(10)—C(11)—N(1)—C(1)	−149·4 (2)
C(3)—C(2)—N(2)—C(1)	−115·7 (2)	C(10)—C(11)—N(1)—C(8)	53·0 (2)

An *ORTEP* (Johnson, 1976) view of the molecule with the atomic labelling scheme is shown in Fig. 1. The molecule has a *trans* [N^2 (*E*)] configuration. The N(2)C(1)C(12)N(1) amidine group is nonplanar ($\chi^2 = 327$). Deviations from the mean plane amount to 0·007 (1), −0·022 (2), 0·014 (2) and 0·006 (1) Å, respectively, for the above atoms. The Winkler & Dunitz (1971) parameters characterizing out-of-plane deformations of the amidine group with the α atoms at N(1), χ_C, χ_N and τ [4·3 (4), 25·1 (4) and 171·8 (4)°, respectively], indicate significant deviation from planarity, mostly due to the pyramidalization of the N(1) atom. The atoms around N(1) form a flat pyramid [the sum of angles at N(1) is 355·8 (2)°]. The distance of N(1) from the C(1)C(8)C(11) plane

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52248 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[0·171 (1) Å] also indicates a distortion from sp^2 hybridization at the amine N atom.

The bond lengths and angles in the morpholine ring are comparable with the values reported by Bellucci, Bertolasi, Ferretti & Gilli (1985). Endocyclic torsion angles of the morpholine ring show that it adopts a chair conformation. The mean value calculated for the absolute values of the endocyclic torsion angles is 55·5 (5)°. The phenyl substituent at N(2) is planar ($\chi^2 = 6{\cdot}7$). The angles between the mean plane of the amidine group and the C(8)N(1)-C(11) plane and the phenyl ring are 19·5 (1) and 67·5 (1)°, respectively.

The C(1)—N(1) and C(1)—N(2) bonds are significantly differentiated: 1·371 (2) and 1·283 (2) Å, respectively. From the semiempirical correlation of Norrestam *et al.* (1983) ($r = r_0 - 0{\cdot}18p$) between π-bond orders (p) and bond distances (r), where r_0 is a standard single-bond distance (1·458 Å for C—N), the following π-bond orders were obtained: 0·5 for C(1)—N(1) and 1·0 for C(1)—N(2). C(1)—N(1) is, therefore, intermediate between a single and a double bond, while C(1)—N(2) is a typical double bond. The C(1)—C(12) bond length of 1·504 (3) is shorter than the standard single-bond C—C distance (1·517 Å) (Norrestam *et al.*, 1983).

The N(1)—C(1)—N(2) angle has a typical value [118·3 (1)°] for the molecules of *trans* [N^2 (*E*)] acetamidines investigated so far (Gilli & Bertolasi, 1979; Ciszak *et al.*, 1989). In *cis* [N^2 (*Z*)] unsubstituted acetamidine (Norrestam *et al.*, 1983) this angle is much wider than 120°, owing to steric effects between the bulky methyl group at C(1) and other substituents attached to the N atoms. The endocyclic phenyl angle C(3)—C(2)—C(7) [118·1 (1)°], below 120°, may reveal electron-releasing properties of the —N=C(*R*)—N group and an inductive effect, rather than π conjugation between amidine and aromatic fragments.

The molecular packing is shown in Fig. 2. No unusually short intermolecular contacts are present in the crystal.

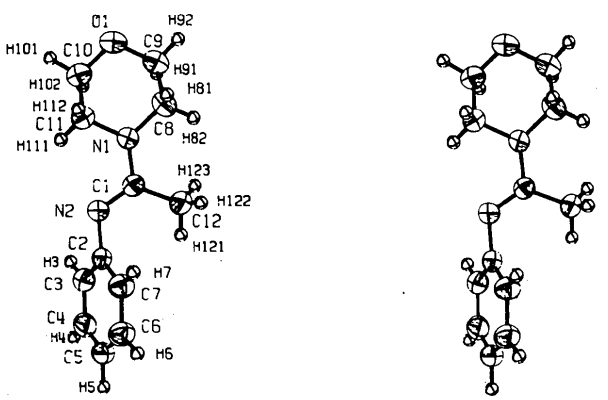

Fig. 1. *ORTEP* (Johnson, 1976) stereoscopic view of the molecule with the thermal ellipsoids at 50% probability.

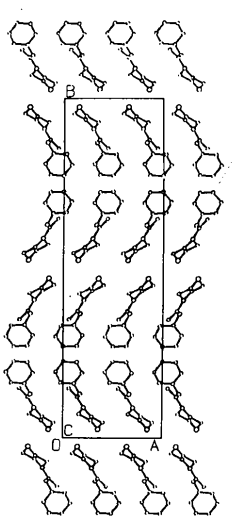

Fig. 2. Projection of the structure down **c**.

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