C(3″) between the two compounds. It is difficult to imagine an electronic reason for the difference, and it is not obvious that intermolecular steric interactions are directly responsible since there are no particularly close approaches to atoms associated with that ring for either compound.

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Structure of (*Z*)-3-Methoxycarbonylamino-*N*,*N*-dimethyl-2-phenylpropenamide

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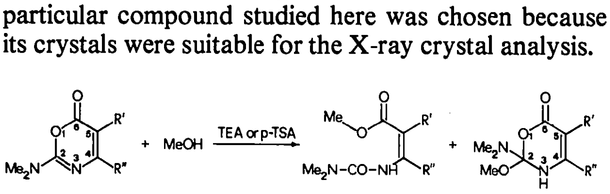
Abstract. $C_{13}H_{16}N_2O_3$, $M_r = 248{\cdot}3$, monoclinic, $P2_1/c$, $a = 10{\cdot}750\,(1)$, $b = 13{\cdot}039\,(1)$, $c = 10{\cdot}104\,(1)$ Å, $\beta = 113{\cdot}17\,(1)°$, $V = 1302{\cdot}0(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}267$ g cm^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å (graphite-crystal monochromator), $\mu = 0{\cdot}85$ cm^{-1}, $F(000) = 528$, room temperature, final $R = 0{\cdot}047$ for 2212 observed reflections. The dimethylamido group is tilted by about 70° with respect to the rest of the molecule; both dimethylamido and *N*-carboxymethyl-*N*-vinylamino groups are extensively conjugated. The molecules are strongly linked by an N—H···O hydrogen bond through a centre of symmetry, and the crystal packing is based on an H-bond network.

Introduction. Beccalli, Marchesini & Molinari (1986, hereafter BMM) studied the alcoholysis of several 2-dimethylamino-1,3-oxazin-6-ones (I). They always obtained a mixture of two products to which structures (II) and (III) were attributed, respectively. Later, BMM had some doubts on the nature of (III); for this reason, this work was carried out to check the structural formula of the second product of the reaction. The particular compound studied here was chosen because its crystals were suitable for the X-ray crystal analysis.



(I) (II) (III)

Experimental. A crystal measuring $0{\cdot}25 \times 0{\cdot}20 \times 0{\cdot}18$ mm was selected and mounted on a CAD-4 diffractometer. The cell parameters were obtained from least-squares fit of 25 reflections in the range $14 < \theta < 19°$; 2976 unique reflections [2212 with $I > \sigma(I)$] were measured up to $2\theta = 55°$, $h\ 0 \to 13$, $k\ 0 \to 16$, $l\ -13 \to 11$, with ω–2θ scan technique, and corrected for Lorentz and polarization effects, but not for absorption. Three standard reflections showed no significant intensity variation. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All H-atom positions were located from a difference Fourier synthesis and included isotropically in final anisotropic refinement based on *F*, with $w = 4F_o^2/[\sigma(F_o^2) + 0{\cdot}0004F_o^4]$. Final $R = 0{\cdot}047$, $wR = 0{\cdot}040$, $S = 1{\cdot}6$, max. residual density 0·2 e Å^{-3}, max. shift $0{\cdot}05\sigma$ on final cycle, extinction coefficient $g = 15(1) \times 10^{-6}$ [Larson (1967), equation (3)]. Scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used include *SDP* (Enraf–Nonius, 1979), *ORTEP*II (Johnson, 1976), *PARST* (Nardelli, 1983) and various in-house programs for refinement and geometrical analysis running on a Gould 32/97 computer.

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

As Fig. 1 shows, the title compound does not have the structure (III). Formally, the true structure is obtained by shifting the dimethylamino group from

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* 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 44168 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional coordinates and U_{eq} values*

$$U_{\mathrm{eq}} = (\textstyle\sum_i \sum_j \beta_{ij} \mathbf{a}_i.\mathbf{a}_j)/6\pi^2.$$

	x	y	z	U_{eq}(Å^2)
O(1)	0·3478 (1)	0·45758 (8)	0·4316 (1)	0·0430 (5)
O(2)	0·8216 (1)	0·5297 (1)	0·3382 (1)	0·0532 (5)
O(3)	0·7250 (1)	0·4315 (1)	0·1412 (1)	0·0598 (6)
N(1)	0·6083 (1)	0·4835 (1)	0·2742 (2)	0·0398 (6)
N(2)	0·3557 (1)	0·6024 (1)	0·3123 (1)	0·0376 (5)
C(1)	0·4898 (2)	0·4324 (1)	0·1917 (2)	0·0363 (6)
C(2)	0·3736 (2)	0·4362 (1)	0·2102 (2)	0·0322 (6)
C(3)	0·3592 (1)	0·4999 (1)	0·3270 (2)	0·0322 (6)
C(4)	0·2533 (2)	0·3757 (1)	0·1200 (2)	0·0330 (6)
C(5)	0·2610 (2)	0·2913 (1)	0·0387 (2)	0·0484 (8)
C(6)	0·1468 (2)	0·2379 (2)	−0·0463 (2)	0·0581 (9)
C(7)	0·0223 (2)	0·2662 (2)	−0·0511 (2)	0·0551 (9)
C(8)	0·0116 (2)	0·3477 (2)	0·0298 (2)	0·0513 (8)
C(9)	0·1259 (2)	0·4020 (1)	0·1138 (2)	0·0420 (7)
C(10)	0·7194 (2)	0·4777 (1)	0·2418 (2)	0·0382 (7)
C(11)	0·9427 (2)	0·5367 (3)	0·3102 (3)	0·063 (1)
C(12)	0·3713 (3)	0·6569 (2)	0·1945 (2)	0·0487 (9)
C(13)	0·3431 (3)	0·6656 (2)	0·4259 (2)	0·054 (1)

position 2 to 6 of the heterocycle (III) and by breaking the 1,6 bond. Structure (III) would really be an unstable intermediate in the reaction path from 2-dimethylamino-5-phenyl-1,3-oxazin-6-one to the title compound; this is in line with the retention of *Z* configuration at the C(2)—C(1) double bond. The title molecule is formed by two separate π systems. The smaller is the dimethylamido group, that is noticeably planar: the maximum deviation from the least-squares plane *A* through O(1), C(3), N(2), C(12) and C(13) is in fact 0·017 (3) Å for C(12). The π conjugation in this group is reflected in the short C(3)—N(2) single bond and in the long C(3)—O(1) double bond [1·343 (2) and 1·241 (2) Å respectively]; these values are usual in amide groups. The greater π system includes the rest of the molecule. Here both N(1)—C(1), 1·388 (2), and N(1)—C(10), 1·359 (3) Å, are much shorter than the pure single bond, such as N(2)—C(12) and N(2)—C(13) [1·452 (3) and 1·462 (3) Å, respectively]. Because of the torsion angles around the C(3)—C(2) bond [C(1)—C(2)—C(3)—O(1) −110·1 (2)°] no conjugation is present between the O(1)—C(3)—N(2) and the C(2)—C(1)—N(1) systems. The phenyl ring is tilted by 18·9 (1)° with respect to the C(1), C(2), C(3) and C(4) plane *B*, owing to H(1)···H(5) [2·20 (3) Å] interaction. The dihedral angle between planes *B* and *C* through N(1), C(10), O(2) and O(3) is only 5·3 (1)°. By contrast the angle between the planes *B* and *D* through C(2), C(3), O(1) and N(2) is 69·8 (1)°. While system *C* is strictly planar, *B* and *D* are not. Even the phenyl ring is not strictly planar, the total puckerig amplitude (Cremer & Pople, 1975) being Q_t = 0·017 (2) Å. The main intermolecular interaction is the strong hydrogen bond $\mathrm{H(N)\cdots O(1)}_{1-x,1-y,1-z}$ 2·08 (2) Å with N(1)···O(1) 2·923 (2) Å and N(1)—H(N)···O(1) 164 (1)°. The crystal packing is also determined by three weaker interactions: H(12*B*)···$\mathrm{O(3)}_{1-x,1-y,-z}$ 2·46 (2) Å, $\mathrm{H(6)\cdots O(1)}_{x,1/2-y,-1/2+z}$ 2·66 (2) and H(13*B*)···$\mathrm{O(1)}_{1-x,1-y,1-z}$ 2·80 (2) Å.

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

O(1)–C(3)	1·241 (2)	O(2)–C(10)	1·332 (2)
O(2)–C(11)	1·440 (3)	O(3)–C(10)	1·203 (2)
N(1)–C(1)	1·388 (2)	N(1)–C(10)	1·359 (3)
N(2)–C(3)	1·343 (2)	N(2)–C(12)	1·452 (3)
N(2)–C(13)	1·462 (3)	C(1)–C(2)	1·334 (3)
C(2)–C(3)	1·500 (2)	C(2)–C(4)	1·482 (2)
C(4)–C(5)	1·395 (2)	C(4)–C(9)	1·390 (3)
C(5)–C(6)	1·378 (2)	C(6)–C(7)	1·371 (3)
C(7)–C(8)	1·372 (3)	C(8)–C(9)	1·382 (2)
N(1)–H(N)	0·87 (2)	C(1)–H(1)	0·97 (2)
C(5)–H(5)	0·99 (2)	C(6)–H(6)	0·94 (2)
C(7)–H(7)	0·90 (2)	C(8)–H(8)	0·94 (2)
C(9)–H(9)	0·96 (2)	C(11)–H(11*A*)	0·93 (3)
C(11)–H(11*B*)	0·93 (3)	C(11)–H(11*C*)	0·94 (3)
C(12)–H(12*A*)	0·98 (2)	C(12)–H(12*B*)	0·95 (2)
C(12)–H(12*C*)	0·95 (2)	C(13)–H(13*A*)	0·98 (2)
C(13)–H(13*B*)	0·98 (2)	C(13)–H(13*C*)	0·92 (3)

C(10)–O(2)–C(11)	116·0 (2)	C(1)–N(1)–C(10)	121·0 (1)
C(12)–N(2)–C(13)	116·4 (1)	C(3)–N(2)–C(13)	118·8 (1)
C(3)–N(2)–C(12)	124·8 (1)	N(1)–C(1)–C(2)	125·9 (1)
C(1)–C(2)–C(4)	122·1 (1)	C(1)–C(2)–C(3)	121·5 (1)
C(3)–C(2)–C(4)	116·4 (1)	N(2)–C(3)–C(2)	118·1 (1)
O(1)–C(3)–C(2)	119·9 (1)	O(1)–C(3)–N(2)	121·9 (1)
C(2)–C(4)–C(9)	120·1 (1)	C(2)–C(4)–C(5)	122·9 (2)
C(5)–C(4)–C(9)	117·0 (2)	C(4)–C(5)–C(6)	121·4 (2)
C(5)–C(6)–C(7)	120·3 (2)	C(6)–C(7)–C(8)	119·8 (2)
C(7)–C(8)–C(9)	120·0 (2)	C(4)–C(9)–C(8)	121·6 (2)
O(3)–C(10)–N(1)	124·9 (2)	O(2)–C(10)–N(1)	110·2 (1)
O(2)–C(10)–O(3)	124·9 (2)		

C(1)–C(2)–C(3)–O(1)	−110·1 (2)	C(1)–C(2)–C(4)–C(5)	18·2 (3)
N(1)–C(1)C(2)–C(4)	−177·6 (2)	C(1)–C(2)–C(3)–N(2)	71·6 (2)
C(12)–N(2)–C(3)–O(1)	178·6 (2)	C(13)–N(2)–C(3)–C(2)	−179·4 (2)
C(1)–N(1)–C(10)–O(2)	−177·9 (1)	C(10)–N(1)–C(1)–C(2)	−177·1 (2)
C(11)–O(2)–C(10)–N(1)	−175·3 (2)		

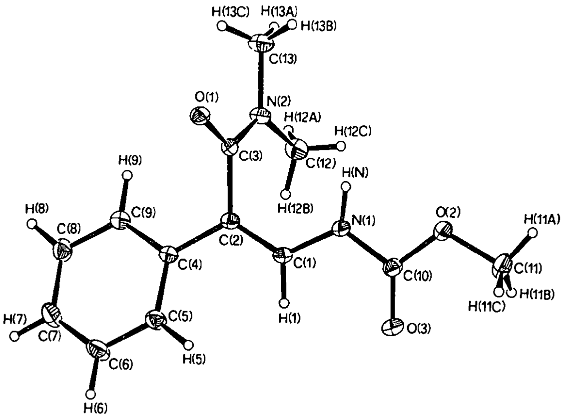

Fig. 1. *ORTEP*II plot (Johnson, 1976) of the molecule with the numbering scheme. Ellipsoids are drawn at the 20% probability level.

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2,9-Bis(phenylsulfonyl)-2,9-diaza[10](2,6)pyridinophane

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Abstract. $C_{25}H_{29}N_3O_4S_2$, $M_r = 499\cdot7$, monoclinic, $P2_1/c$, $a = 8\cdot168(3)$, $b = 21\cdot196(5)$, $c = 14\cdot969(4)$ Å, $\beta = 103\cdot58(2)°$, $V = 2519\cdot3(2)$ Å^3, $Z = 4$, $D_x = 1\cdot317$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 20\cdot96$ cm^{-1}, $F(000) = 1056$, room temperature, $R = 0\cdot047$ for 2628 reflections with $I > 3\sigma(I)$. The thirteen-membered ring has one half in a boat and the other half in a chair conformation. One of the sulfonyl substituents is equatorial, the other is axial. The benzene and pyridine rings are planar.

Introduction. The title compound was obtained by condensation of *N,N'*-(1,6-cyclohexylene)bis(benzenesulfonamide) with 2,6-bis(chloromethyl)pyridine in the presence of sodium butanolate at 403–413 K (Krakowiak, 1987). Pyridinophanes of this type have been the subject of interest because of their complex-forming properties with metals (Weber & Vögtle, 1976), their application as catalysts for ester aminolysis (Gandour, Walker, Nayak & Newkome, 1978) and as models for the pyridine nucleotide coenzymes (Dittner & Blidner, 1973). At present these compounds are under pharmacological investigation at the School of Medicine in Łódź.

Experimental. Colourless crystals from methanol, crystal size 0·2 × 0·2 × 0·4 mm, room temperature CAD-4 diffractometer using θ–2θ scan technique, unit cell from 25 reflections with $\theta < 16°$; 3144 independent reflections measured to $\theta = 70°$, 2628 non-zero reflections with $I > 3\sigma(I)$; data not corrected for absorption; graphite-monochromatized Cu $K\alpha$; maximum values of h,k,l 9, 23, 15, respectively; standard reflection 132, mean variation 0·27%; solution by direct methods using *SHELX76* (Sheldrick, 1976); non-H atoms found on E map, positions of H atoms from difference map; refinement by full-matrix least squares (F magnitudes, 398 parameters) to final $R = 0\cdot047$, unit weights; max. $\Delta/\sigma = 0\cdot43$; largest peak on final difference map 0·34 e Å^{-3}. Scattering factors as included in *SHELX76*.

Discussion. The final positional parameters are listed in Table 1,* bond lengths and valence angles in Table 2 and torsion angles in Table 3. The atomic numbering scheme is given in Fig. 1, the molecular structure and packing in the unit cell in Fig. 2. The molecule is not a symmetrical one. However, the 13-atom heterocycle, as a whole, may be considered to have a distorted crown conformation which can be divided into two parts: the first part (N3, C9, C8, N1, C1, C2, C3) being in a boat-like conformation while the other (C4, C5, C6, N2, C7, C13, N3) is in a chair-like conformation. The positions of the sulfonyl substituents, with respect to the

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* 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 44255 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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