

Fig. 2. Stereoscopic view of the molecular packing in the cell. The *a* axis points from left to right, the *b* axis onto the plane of the paper, and the *c* axis downwards. H atoms attached to C atoms are omitted.

O(1)—H(O1)···O(5) 157 (3), H(O1)···O(5)—C(17) 108.3 (9)°; O(2)···O(3) 2.888 (2), O(2)—H(O2) 0.82 (2), H(O2)···O(3) 2.10 (2) Å, O(2)—H(O2)···O(3) 164 (2), H(O2)···O(3)—C(10) 104.7 (6)°. This finding explains shifts observed in the IR and ¹³C NMR spectra for the 5- and 8-acetyl groups: 1695 cm⁻¹ (KBr) in IR and 215.5 and 215.7 p.p.m. (CDCl₃, Me₄Si) in ¹³C NMR in comparison with 1710 cm⁻¹ and 208.7 p.p.m. for the 13-acetyl group.

Molecular packing in the crystal is shown in Fig. 2. The molecules are held together by van der Waals interactions with normal intermolecular contacts.

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Structures of an Antithrombotic Agent and its Dimethyl Derivative: 4-Methoxy-*N,N'*-bis(3-pyridylmethyl)isophthalamide Monohydrate (I) and 4-Methoxy-*N,N'*-dimethyl-*N,N'*-bis(3-pyridylmethyl)isophthalamide Hemihydrate (II)

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Abstract. (I): C₂₁H₂₀N₄O₃·H₂O, *M_r* = 394.37, monoclinic, *P*2₁/*c*, *a* = 6.354 (2), *b* = 14.043 (1), *c* = 21.418 (3) Å, β = 92.33 (2)°, *V* = 1909.9 (6) Å³, *Z* = 4, *D_x* = 1.37 Mg m⁻³, Mo *Kα*, λ = 0.7107 Å, μ = 0.058 mm⁻¹, *F*(000) = 832, *T* = 293 K, *R* = 0.066 for 1830 observed reflections. (II): C₂₃H₂₄N₄O₃·½H₂O, *M_r* = 413.41, monoclinic, *P*2₁/*n*, *a* = 31.649 (4), *b* = 11.753 (2), *c* = 11.501 (2) Å, β = 93.11 (2)°, *V* =

4272 (1) Å³, *Z* = 8, *D_x* = 1.28 Mg m⁻³, Mo *Kα*, λ = 0.7107 Å, μ = 0.05 mm⁻¹, *F*(000) = 1752, *T* = 293 K, *R* = 0.065 for 5369 observed reflections. A considerable change in the shape of the basic molecular skeleton is caused by the *N*-methylation in (II), an inactive compound, to be compared with the planar shape observed in (I), an antithrombotic agent (picotamide). The two independent molecules in (II) are substantially

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References

- ASHIDA, T. (1973). *HBL5-V*. The Universal Crystallographic Computing System, Osaka. The Computation Center, Osaka Univ., Japan.
- BECKWITH, A. L. J., RODGERS, J. R. & WAGNER, R. D. (1982). *Aust. J. Chem.* **35**, 989–996.
- FUJII, S. (1979). *MOLCON*. The Universal Crystallographic Computing System, Osaka. The Computation Center, Osaka Univ., Japan.
- HENDRICKSON, J. B. (1961). *J. Am. Chem. Soc.* **83**, 4537–4547.
- HENKE, H. & KEUL, H. (1975). *Cryst. Struct. Commun.* **4**, 451–456.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
- LÉGER, J.-M., COLLETER, J.-C. & CARPY, A. (1982). *Cryst. Struct. Commun.* **11**, 1363–1368.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

related by a pseudocentre of symmetry. The resulting planarity of the molecular fragment present in (I) is justified in terms of conjugation effects in connection with hydrogen-bonding effects through extensive references to the existing literature.

Introduction. The present work is part of a research programme on structure–activity correlation in anti-thrombotic agents. Some derivatives of 4-OH-IPHT (IPHT = isophthalic acid) have been shown (De Cunto, Berrettini, Parise, De Regis, Orzalesi & Nenci, 1983, and references therein) to inhibit *in vitro* human platelet aggregation, an activity commonly correlated to the kind and number of substituents in the 4-OH-IPHT and 4-OCH₃-IPHT moieties, *per se* proaggregating agents. Among the bis(pyridylmethyl)amide derivatives compound (I) shows the largest effect, while compound (II) is not significantly active. The crystal structure analysis was undertaken to explain the hypothesis of a competitive inhibition-type mechanism of action. A preliminary communication on compound (I) was presented at the VIth European Crystallographic Meeting, 1980, Barcelona, Spain.

Experimental. (I) Colourless crystals from ethyl acetate, size 0.20 × 0.28 × 0.48 mm; unit-cell parameters from 23 reflections on a CAD-4 diffractometer with $7^\circ \leq \theta \leq 12^\circ$; *hkl* values respectively -7 to 7 , 0 to 16 , 0 to 24 ; $\omega/2\theta$ scan, $2\theta_{\max} = 50^\circ$; three standard reflections ($\bar{1}54$, 039 , 240), no significant intensity variation; systematic absences *h*0*l* for *l* odd and $0k0$ for *k* odd; no absorption or extinction correction applied; 4097 reflections measured, 2879 unique ($R_{\text{int}} = 0.139$), 1049 of which, having $I < 2\sigma(I)$, were considered unobserved; number of parameters refined 265. (II) Colourless prismatic crystals from ethyl acetate, size 0.08 × 0.20 × 0.25 mm; unit-cell parameters from 24 reflections on a Philips PW 1100 diffractometer, with $7^\circ \leq \theta \leq 10^\circ$; *hkl* values respectively -37 to 37 , 0 to 14 , 0 to 14 ; $\omega/2\theta$ scan, $2\theta_{\max} = 52^\circ$; two standard reflections (631 and 613), no significant intensity variation; systematic absences *h*0*l* for *h* + *l* odd and $0k0$ for *k* odd; no absorption correction applied; 9068 reflections measured, 8306 unique ($R_{\text{int}} = 0.123$), 2937 of which, having $I < 2.5\sigma(I)$, were considered unobserved; number of parameters refined 490. Both structures solved by the *SHELX76* system of programs (Sheldrick, 1976); the H atoms were calculated geometrically when they were not found in the ΔF syntheses. It is relevant for the discussion to underline here that the H atoms bound to N(9) and N(21) were found experimentally. Full-matrix anisotropic refinement (on *F*) of all heavy atoms for compound (I) and, except for C(1) to C(6) and C(10) plus C(22), for compound (II), led respectively to $R = 0.066$, $wR = 0.064$ and $R = 0.065$, $wR = 0.070$, with $w = 2.5288/[\sigma^2(F_o) + 0.000898F_o^2]$ and $3.86/[\sigma^2(F_o) + 0.000358F_o^2]$. The H

atoms were not refined. The atomic scattering factors were taken from *SHELX76*. Final difference Fourier map excursions (I) 0.3 to -0.24 and (II) 0.34 to -0.39 e Å⁻³. Max $\Delta/\sigma = 0.11$ (I) and 0.37 (II). Programs *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell, 1976) used for geometrical calculations.

Discussion. To our knowledge, this is the first crystal structure determination for the isophthalamide moiety. Some features are compared with those found in benzamides and anisoles.

The atomic coordinates for the non-H atoms with isotropic (or equivalent isotropic) temperature factors and details of the bond geometry are given in Tables 1 and 2, respectively.* Fig. 1 is a projection of the molecules of both compounds showing the atomic numbering and Fig. 2 represents the molecular packing. The main structural difference between (I) and (II) is the pronounced rotation of the amide groups in (II) when the N atom undergoes methylation. This effect is summarized in Table 3.

The twist angles observed in (I) are low if compared with most benzamides (no phthalamides were found in the bibliographic file of the Cambridge Crystallographic Database). The rich literature on the large deviations from planarity commonly found in benzamides has been discussed extensively by Di Rienzo, Domenicano & Foresti Serantoni (1977) who found a planar *p*-nitrobenzamide. More recent contributions have added credibility to the influence of the packing forces (Leiserowitz & Tuval, 1978; Taniguchi, Nakata, Takaki & Sakurai, 1978; Herbstein, Kaftory, Kapon & Saenger, 1981; Kato & Sakurai, 1982; Sakurai, 1982). However, the *o*-methoxybenzamide derivatives tend to planarity through an intramolecular N–H...O bond, which would introduce a stabilization in the system [Blaton, Peeters, De Ranter, Denisoff & Molle (hereafter BPDDM), 1980], the observed (or calculated by us) values for the dihedral angles defined in Table 3 ranging from 3.5 to 17° (BPDDM, 1980, 1981, 1982; Houttemane, Boivin, Nowogrocki, Thomas & Bonte, 1981; Ma, Camerman & Camerman, 1982). In compound (I) we observe a short intramolecular H bond, N(21)–H(21)...O(17) 2.646 (4) Å, while the relative interatomic angle is 140 (2)°. In the same molecule a planar arrangement is maintained by the amide group attached to C(3) (see Table 3, *n* = 3): here neither intramolecular H bonds, nor planar stabilization are foreseeable. The ‘enol term’ hypothesis formulated by Halfpenny & Small (1980*a,b*) is not applicable here

* Lists of structure factors, anisotropic temperature factors and unrefined H coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42530 (48 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atom coordinates ($\times 10^4$) and isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

For atoms marked with an asterisk $U_{eq} = \frac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ matrix})$.

Compound (I)	x	y	z	U or U_{eq}
C(1)	1016 (6)	82 (3)	2326 (2)	38*
C(2)	2410 (6)	-434 (3)	2706 (2)	35*
C(3)	4253 (6)	-54 (3)	2960 (2)	34*
C(4)	4691 (6)	912 (3)	2838 (2)	35*
C(5)	3313 (7)	1436 (3)	2458 (2)	44*
C(6)	1474 (7)	1033 (3)	2204 (2)	44*
C(7)	-865 (7)	-428 (3)	2062 (2)	42*
O(8)	-1168 (5)	-1282 (2)	2158 (1)	59*
N(9)	-2242 (5)	68 (2)	1687 (2)	45*
C(10)	-4081 (6)	-374 (3)	1392 (2)	47*
C(11)	-3663 (6)	-793 (3)	754 (2)	40*
C(12)	-1684 (7)	-869 (4)	513 (2)	62*
C(13)	-1457 (8)	-1249 (4)	-72 (2)	65*
C(14)	-3217 (9)	-1549 (3)	-400 (2)	60*
N(15)	-5152 (7)	-1483 (3)	-190 (2)	61*
C(16)	-5325 (7)	-1115 (3)	387 (2)	49*
O(17)	6487 (4)	1284 (2)	3100 (1)	49*
C(18)	6974 (8)	2265 (3)	3024 (3)	72*
C(19)	5656 (6)	-720 (3)	3339 (2)	38*
O(20)	5166 (5)	-1567 (2)	3385 (1)	50*
N(21)	7408 (5)	-382 (2)	3620 (1)	41*
C(22)	8974 (6)	-995 (3)	3932 (2)	42*
C(23)	8692 (6)	-1121 (3)	4622 (2)	39*
C(24)	6753 (7)	-1082 (3)	4894 (2)	53*
C(25)	6643 (8)	-1261 (4)	5528 (2)	62*
C(26)	8448 (9)	-1453 (4)	5860 (2)	68*
N(27)	10367 (7)	-1473 (3)	5623 (2)	70*
C(28)	10430 (7)	-1312 (3)	5011 (2)	50*
O(29)	-7749 (5)	-2048 (2)	-1224 (1)	57*

Compound (II)	x	y	z	U or U_{eq}
C(1)A	3258 (1)	1060 (3)	868 (3)	40
C(2)A	2869 (1)	594 (2)	491 (3)	38
C(3)A	2496 (1)	1091 (2)	809 (2)	37
C(4)A	2510 (1)	2050 (2)	1535 (2)	39
C(5)A	2896 (1)	2505 (3)	1943 (3)	44
C(6)A	3267 (1)	2017 (3)	1590 (3)	46
C(7)A	3668 (1)	503 (3)	605 (3)	41*
O(8)A	3781 (1)	-365 (2)	1141 (2)	62*
N(9)A	3909 (1)	999 (2)	-173 (2)	51*
C(10)A	4343 (1)	596 (3)	-298 (3)	59
C(11)A	4406 (1)	35 (3)	-1461 (3)	49*
C(12)A	4207 (1)	-963 (3)	-1780 (4)	70*
C(13)A	4280 (2)	-1441 (4)	-2825 (5)	93*
C(14)A	4552 (2)	-909 (6)	-3541 (5)	103*
N(15)A	4748 (1)	64 (4)	-3262 (3)	94*
C(16)A	4672 (1)	513 (4)	-2226 (3)	69*
C(17)A	2126 (1)	2474 (2)	1809 (2)	49*
C(18)A	2122 (1)	3504 (3)	2487 (3)	54*
C(19)A	2077 (1)	543 (2)	440 (3)	38*
C(20)A	1976 (1)	-355 (2)	897 (2)	57*
N(21)A	1833 (1)	1060 (2)	-394 (2)	40*
C(22)A	1421 (1)	574 (3)	-738 (3)	44
C(23)A	1058 (1)	1112 (2)	-119 (2)	41*
C(24)A	641 (1)	851 (3)	-467 (3)	52*
C(25)A	316 (1)	1316 (3)	150 (3)	64*
C(26)A	412 (1)	2006 (3)	1080 (3)	67*
N(27)A	810 (1)	2277 (3)	1416 (2)	64*
C(28)A	1120 (1)	1834 (3)	824 (3)	51*
C(29)A	3773 (1)	1997 (3)	-865 (3)	67*
C(30)A	1945 (1)	2122 (3)	-961 (3)	55*
C(1)B	1741 (1)	6226 (2)	-917 (2)	41
C(2)B	2115 (1)	6744 (2)	-541 (2)	39
C(3)B	2501 (1)	6315 (2)	-863 (2)	37
C(4)B	2506 (1)	5365 (2)	-1592 (2)	39
C(5)B	2131 (1)	4861 (3)	-2007 (3)	46
C(6)B	1751 (1)	5286 (3)	-1649 (2)	47
C(7)B	1321 (1)	6725 (3)	-600 (2)	41*
O(8)B	1197 (1)	7616 (2)	-1080 (2)	61*
N(9)B	1102 (1)	6176 (2)	191 (2)	44*
C(10)B	685 (1)	6603 (3)	474 (3)	52
C(11)B	707 (1)	7399 (2)	1505 (3)	41*
C(12)B	882 (1)	8480 (3)	1438 (3)	62*
C(13)B	886 (1)	9191 (3)	2393 (3)	67*
C(14)B	720 (1)	8793 (3)	3397 (3)	60*
N(15)B	555 (1)	7758 (3)	3485 (2)	63*
C(16)B	551 (1)	7090 (3)	2554 (3)	52*
O(17)B	2899 (1)	4997 (2)	-1857 (2)	50*
C(18)B	2927 (1)	3951 (3)	-2495 (3)	61*
C(19)B	2902 (1)	6916 (2)	-470 (2)	38*

Table 1 (cont.)

	x	y	z	U or U_{eq}
O(20)B	2993 (1)	7830 (2)	920 (2)	56*
N(21)B	3148 (1)	6436 (2)	388 (2)	39*
C(22)B	3539 (1)	7003 (3)	785 (3)	44
C(23)B	3927 (1)	6595 (2)	194 (2)	40*
C(24)B	4323 (1)	6961 (3)	607 (3)	54*
C(25)B	4673 (1)	6612 (3)	46 (3)	66*
C(26)B	4619 (1)	5898 (3)	907 (3)	66*
N(27)B	4244 (1)	5530 (2)	1305 (2)	61*
C(28)B	3905 (1)	5883 (3)	761 (3)	49*
C(29)B	1266 (1)	5192 (3)	857 (3)	66*
C(30)B	3055 (1)	5348 (3)	932 (3)	57*
O(61)	502 (1)	8492 (2)	-2450 (2)	83*

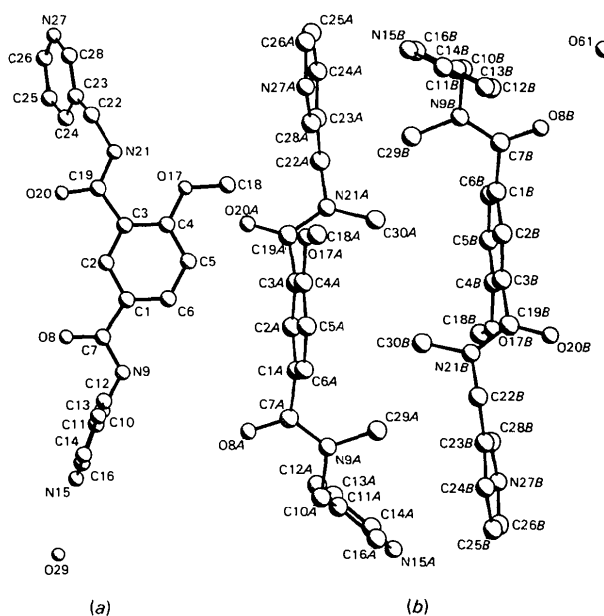


Fig. 1. Perspective views of the molecules of compounds (I) and (II) showing the atom-numbering schemes.

given the location of the H atoms by the structural analysis and the quite common values observed for the C=O lengths. The three remaining H bonds in (I) and all H bonds in (II) are related to the water molecules. On the other hand, when examining *p*-substituted anisoles, conjugation effects between the phenyl ring and the O of the methoxy group and their consequent tendency to be coplanar result in some shortening of the C(ar.)-O(ether) bond as well as an evident distortion from the sp^2 value of the C(ar.)-C(ar.)-O angles (Di Rienzo, Domenicano, Portalone & Vaciago, 1976, and references therein; Domiano, Nardelli, Balsamo, Macchia & Macchia, 1979). Fig. 3 shows a fragment of the molecule (I) we believe important to describe the geometry of these compounds. Through an extensive examination of the above literature concerning *o*-methoxybenzamide, benzamide and anisole derivatives, we noted some systematic features which are shown in Table 4. Values, when not found, have been calculated by us (for benzamides, only the exemplifying case of benzamide itself is reported). In comparison with

benzamide, χ values are significantly higher while ψ are, to a certain extent, similar; compared to anisoles, φ angles are constantly a little higher. Compound (II) shows only a slight deformation in φ value.

The τ_1 , τ_2 and τ_3 torsion angles also reflect the geometry of the system.

It is interesting to note that, when both amide and methoxy groups are present together on the phenyl ring, the observed φ and ψ angles are found to be closer to sp^2 values than those reported for the single substituents.

Table 2. Bond lengths (Å) and bond angles (°)

Compound (I)		Compound (II)	
		Molecule A	Molecule B
C(1) C(2)	1.382 (5)	1.394 (4)	1.379 (4)
C(1) C(6)	1.394 (6)	1.397 (4)	1.390 (4)
C(1) C(7)	1.483 (6)	1.499 (4)	1.516 (4)
C(2) C(3)	1.378 (5)	1.385 (4)	1.391 (4)
C(3) C(4)	1.411 (5)	1.403 (4)	1.396 (4)
C(3) C(19)	1.506 (5)	1.513 (4)	1.502 (4)
C(4) C(5)	1.382 (6)	1.392 (4)	1.390 (4)
C(4)-O(17)	1.356 (5)	1.364 (4)	1.365 (4)
C(5) C(6)	1.389 (6)	1.386 (5)	1.385 (4)
C(7)-O(8)	1.233 (5)	1.235 (4)	1.237 (4)
C(7) N(9)	1.356 (5)	1.340 (4)	1.339 (4)
N(9) C(10)	1.445 (5)	1.468 (5)	1.466 (4)
N(9) C(29)		1.469 (5)	1.466 (4)
C(10) C(11)	1.521 (6)	1.514 (5)	1.509 (4)
C(11) C(12)	1.381 (6)	1.372 (5)	1.389 (4)
C(11) C(16)	1.366 (6)	1.371 (5)	1.376 (4)
C(12) C(13)	1.375 (7)	1.357 (7)	1.379 (5)
C(13) C(14)	1.361 (7)	1.376 (8)	1.377 (5)
C(14) N(15)	1.329 (7)	1.332 (8)	1.331 (5)
N(15) C(16)	1.347 (6)	1.337 (6)	1.328 (4)
O(17) C(18)	1.423 (5)	1.440 (4)	1.437 (4)
C(19)-O(20)	1.234 (5)	1.230 (4)	1.232 (3)
C(19) N(21)	1.330 (5)	1.343 (4)	1.346 (4)
N(21) C(22)	1.457 (5)	1.460 (4)	1.459 (4)
N(21) C(30)		1.460 (4)	1.459 (4)
C(22) C(23)	1.506 (6)	1.521 (4)	1.512 (4)
C(23) C(24)	1.384 (6)	1.393 (4)	1.385 (4)
C(23) C(28)	1.381 (6)	1.383 (4)	1.379 (4)
C(24) C(25)	1.387 (7)	1.392 (5)	1.376 (5)
C(25) C(26)	1.351 (7)	1.363 (5)	1.384 (5)
C(26) N(27)	1.340 (7)	1.334 (5)	1.321 (5)
N(27) C(28)	1.332 (6)	1.331 (4)	1.336 (4)
C(2) C(1) C(6)	118.6 (4)	119.3 (3)	119.5 (3)
C(2) C(1) C(7)	117.2 (3)	121.6 (3)	120.2 (3)
C(6) C(1) C(7)	124.2 (4)	118.9 (3)	120.2 (3)
C(1) C(2) C(3)	122.9 (4)	120.2 (3)	120.5 (3)
C(2) C(3) C(4)	118.0 (3)	119.8 (3)	119.3 (3)
C(2) C(3) C(19)	116.5 (3)	119.6 (3)	119.4 (2)
C(4) C(3) C(19)	125.5 (3)	120.4 (3)	121.3 (2)
C(3) C(4) C(5)	119.8 (4)	120.5 (3)	120.6 (3)
C(3) C(4) O(17)	117.5 (3)	115.6 (3)	115.4 (2)
C(5) C(4) O(17)	122.7 (3)	123.9 (3)	124.0 (3)
C(4) C(5) C(6)	121.0 (4)	118.9 (3)	119.0 (3)
C(1) C(6) C(5)	119.8 (4)	121.2 (3)	121.0 (3)
C(1) C(6) O(17)	122.3 (4)	119.4 (3)	118.7 (3)
C(1) C(7) N(9)	117.9 (4)	118.4 (3)	118.0 (3)
O(8) C(7) N(9)	119.7 (4)	122.1 (3)	123.4 (3)
C(7) N(9) C(10)	122.0 (3)	119.7 (3)	119.4 (2)
C(7) N(9) C(29)		123.2 (3)	123.7 (3)
C(10) N(9) C(29)		117.0 (3)	116.9 (3)
N(9) C(10) C(11)	113.1 (3)	113.2 (3)	112.6 (2)
C(10) C(11) C(12)	124.0 (4)	121.9 (3)	121.7 (3)
C(10) C(11) C(16)	119.0 (4)	120.2 (3)	121.4 (3)
C(12) C(11) C(16)	117.0 (4)	117.9 (3)	116.9 (3)
C(11) C(12) C(13)	120.0 (4)	119.5 (4)	119.8 (3)
C(12) C(13) C(14)	118.4 (5)	119.0 (5)	118.3 (3)
C(13) C(14) N(15)	123.7 (5)	123.0 (5)	123.0 (3)
C(14) N(15) C(16)	116.6 (4)	116.7 (4)	117.7 (3)
C(11) C(16) N(15)	124.3 (4)	123.8 (4)	124.3 (3)
C(4) O(17) C(18)	120.6 (3)	117.8 (2)	117.9 (2)
C(3) C(19) O(20)	119.7 (3)	119.4 (3)	119.8 (2)
C(3) C(19) N(21)	119.2 (3)	117.9 (3)	117.9 (2)
C(20) C(19) N(21)	121.1 (4)	122.7 (3)	122.3 (3)
C(19) N(21) C(22)	122.6 (3)	119.2 (2)	119.0 (2)
C(19) N(21) C(30)		124.2 (3)	124.1 (2)
C(22) N(21) C(30)		116.6 (2)	116.9 (2)
N(21) C(22) C(23)	114.4 (3)	113.4 (2)	114.2 (2)
C(22) C(23) C(24)	123.4 (4)	120.1 (3)	119.2 (3)
C(22) C(23) C(28)	119.4 (4)	122.9 (3)	123.0 (3)
C(24) C(23) C(28)	117.2 (4)	116.9 (3)	117.8 (3)
C(23) C(24) C(25)	119.1 (4)	118.7 (3)	119.0 (3)
C(24) C(25) C(26)	118.4 (5)	119.7 (3)	118.9 (3)
C(25) C(26) N(27)	124.8 (5)	122.4 (3)	123.0 (3)
C(26) N(27) C(28)	115.7 (4)	117.9 (3)	117.7 (3)
C(23) C(28) N(27)	124.8 (4)	124.3 (3)	123.7 (3)

Table 3. Angles (°) between normals to the planes formed by the central ring [from C(1) to C(6)] and by the fragment $\text{C}(n)-\text{C}(=\text{O})-\text{N}<$

	Compound (I)		Compound (II)	
			Molecule A	Molecule B
$n = 1$	2.5 (2)		76.0 (1)	73.9 (1)
$n = 3$	3.9 (1)		74.8 (1)	75.8 (1)

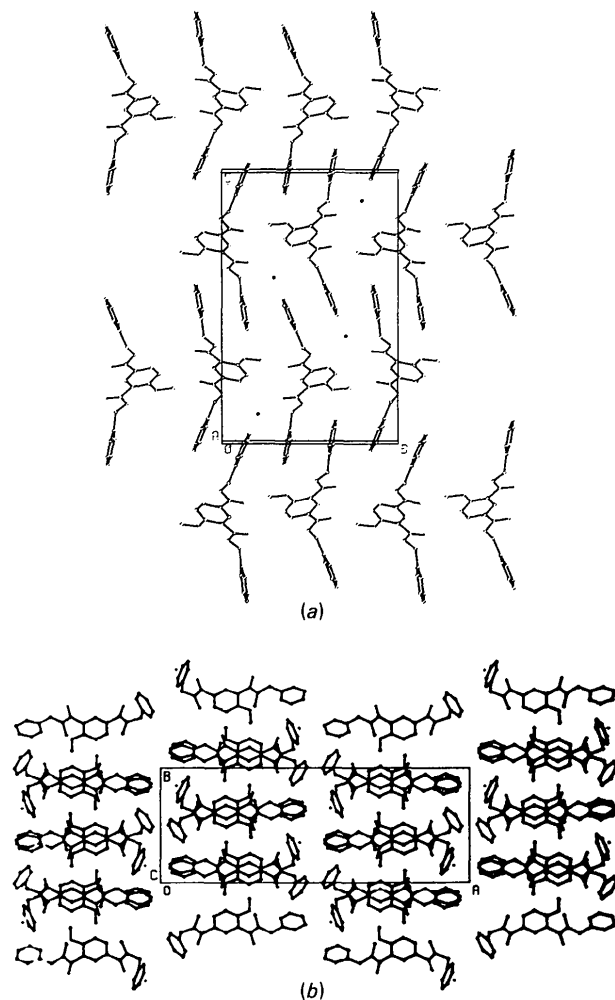


Fig. 2. Molecular packing viewed (a) down the x axis for (I) and (b) down the z axis for (II).

Table 4. Systematic deformation of sp^2 angles in the *O*-methoxybenzamide fragment shown in Fig. 3, listed together with relevant torsion angles; *p*-substituted anisoles (average value) and benzamide values are reported for comparison (e.s.d.'s, when available, 0.6°)

ψ	χ	ψ	τ_1	τ_2	τ_3	Reference
117.0°	126.7°	119.4°	-177.6°	-13.7°	-6.1°	(1)
116.7	126.2	119.2	-175.4	-4.8	0.9	(2)
116.7	126.2	119.4	174.4	-16.9	-3.5	(3)
117	125	119	169.1	13.0	*	(4)
117	127	118	*	*	*	(5)
117	128	116	176.5	11.1/-13.9	*	(6)
				[molecules (1)/(2)]		
117.5	125.5	119.2	-176.7	4.1	6.8	(7)
117.8	126.0	117.7	-177.1	-6.3	0.0	(8)
115	—	—	176.5	—	—	(9)
—	121.7	117.5	—	-27.8	-17.8, -173.4	(10)
				(two H atoms)		

References: (1) BPDDM (1980): primperan. (2) BPDDM (1981): sulpiride hydrochloride. (3) BPDDM (1982): tiapride hydrochloride. (4) Houttemane, Boivin, Nowogrocki, Thomas & Bonte (1981): racemic sulpiride. (5) Ma, Camerman & Camerman (1982): racemic sulpiride. (6) Ma, Camerman & Camerman (1982): *S*(-)-sulpiride average [molecules (1) and (2)]. (7) This paper. (8) Foresti, Riva di Sanseverino & Sabatino, unpublished. (9) Di Rienzo, Domenicano, Portalone & Vaciano (1976): *p*-substituted anisoles. (10) Blake & Small (1972): benzamide.

* Coordinates unavailable for calculations.

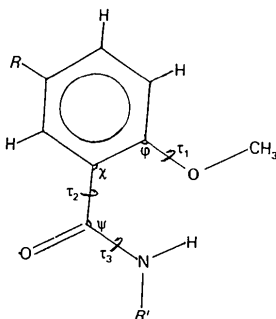


Fig. 3. The *o*-methoxybenzamide fragment with relevant angles labelled as in Table 4.

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References

- BLAKE, C. C. F. & SMALL, R. W. H. (1972). *Acta Cryst.* **B28**, 2201-2206.
- BLATON, N. M., PEETERS, O. M., DE RANTER, C. J., DENISOFF, O. & MOLLE, L. (1980). *Cryst. Struct. Commun.* **9**, 857-864.
- BLATON, N. M., PEETERS, O. M., DE RANTER, C. J., DENISOFF, O. & MOLLE, L. (1981). *Cryst. Struct. Commun.* **10**, 833-838.
- BLATON, N. M., PEETERS, O. M., DE RANTER, C. J., DENISOFF, O. & MOLLE, L. (1982). *Cryst. Struct. Commun.* **11**, 1357-1362.
- DE CUNTO, M., BERRETTINI, M., PARISE, P., DE REGIS, M., ORZALESI, G. & NENCI, G. G. (1983). *Boll. Soc. Ital. Biol. Sper.* **59**, 302-308.
- DI RIENZO, F., DOMENICANO, A. & FORESTI SERANTONI, E. (1977). *Acta Cryst.* **B33**, 3854-3858.
- DI RIENZO, F., DOMENICANO, A., PORTALONE, G. & VACIANO, A. (1976). *Proc. Yugosl. Cent. Crystallogr.* **11**, A102-A104.
- DOMIANO, P., NARDELLI, M., BALSAMO, A., MACCHIA, B. & MACCHIA, F. (1979). *Acta Cryst.* **B35**, 1363-1372.
- HALFPENNY, J. & SMALL, R. W. H. (1980a). *Acta Cryst.* **B36**, 1194-1196.
- HALFPENNY, J. & SMALL, R. W. H. (1980b). *Acta Cryst.* **B36**, 2786-2788.
- HERBSTEIN, F. H., KAFTORY, M., KAPON, M. & SAENGER, W. (1981). *Z. Kristallogr.* **154**, 11-30.
- HOUTTEMANE, C., BOIVIN, J. C., NOWOGROCKI, G., THOMAS, D. J. & BONTE, J. P. (1981). *Acta Cryst.* **B37**, 981-984.
- KATO, Y. & SAKURAI, K. (1982). *Bull. Chem. Soc. Jpn*, **55**, 1643-1644.
- LEISEROWITZ, L. & TUVAL, M. (1978). *Acta Cryst.* **B34**, 1230-1247.
- MA, L. Y. Y., CAMERMAN, N. & CAMERMAN, A. (1982). *Acta Cryst.* **B38**, 2861-2865.
- MOTHERWELL, W. D. S. (1976). *PLUTO*. A program for drawing crystal and molecular structures. Univ. of Cambridge, England.
- ROBERTS, P. J. & SHELDRIK, G. M. (1975). *XANADU*. A program for crystallographic calculations. Univ. of Cambridge, England.
- SAKURAI, K. (1982). *Mem. Osaka Kyoiku Univ.* **30**, 119-136.
- SHELDRIK, G. M. (1976). *SHELX76*. A program for crystal structure determination. Univ. of Cambridge, England.
- TANIGUCHI, T., NAKATA, K., TAKAKI, Y. & SAKURAI, K. (1978). *Acta Cryst.* **B34**, 2574-2578.

Acta Cryst. (1986). **C42**, 224-227

Structure of 5,11-Methano-2,8-dimethyl-5,6,11,12-tetrahydrobenzo[*b,f*][1,5]diazocine (Tröger's Base) at 163 K

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Abstract. $C_{17}H_{18}N_2$, $M_r = 250.34$, orthorhombic, *Pccn*, $a = 12.774$ (2), $b = 30.290$ (5), $c = 10.386$ (2) Å, $V = 4018.5$ (13) Å³, $Z = 12$, $D_x = 1.241$, $D_m =$

1.20 (1) g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.685$ cm⁻¹, $F(000) = 1608$, $T = 163$ K, $R = 0.0668$ for 1738 reflections, ($F \geq 4\sigma_F$). There are 1.5 molecules per

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