New Potent Neuroleptic Drugs of Benzamide Derivatives. III.* Structures of YM-09151-1, N-[(2RS,3SR)-1-Benzyl-2-methyl-3-pyrrolidinyl]-5-chloro-2-methoxy-4-methylaminobenzamide Hydrochloride, and its Monohydrate

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Abstract. Anhydrous YM-09151-1: $C_{21}H_{27}ClN_3O_2^+$·Cl⁻, $M_r = 424.37$, orthorhombic, *Pbca*, $a = 29.741$ (5), $b = 12.827$ (3), $c = 11.513$ (2) Å, $V = 4392.0$ (15) Å³, $D_m = 1.27$, $D_x = 1.283$ g cm⁻³, $Z = 8$, $\lambda(\text{Cu}\,K\alpha) = 1.54178$ Å, $\mu = 28.65$ cm⁻¹, $F(000) = 1792$, room temperature, $R = 0.133$ for 3256 observed reflections. YM-09151-1 monohydrate: $C_{21}H_{27}ClN_3O_2^+$·Cl⁻·H₂O, $M_r = 442.39$, triclinic, $P\bar{1}$, $a = 10.451$ (2), $b = 17.082$ (3), $c = 7.206$ (1) Å, $\alpha = 95.79$ (2), $\beta = 100.34$ (1), $\gamma = 63.02$ (1)°, $V = 1127.5$ (3) Å³, $D_m = 1.29$, $D_x = 1.303$ g cm⁻³, $Z = 2$, $\lambda(\text{Mo}\,K\alpha) = 0.71073$ Å, $\mu = 3.120$ cm⁻¹, $F(000) = 468$, room temperature, $R = 0.120$ for 3981 observed reflections. The title compound, YM-09151-1, is a neuroleptic drug which has antipsychotic activity. Both crystals show disordering of the pyrrolidine ring; there is also disordering of the benzyl group in the monohydrate crystal. Four conformers are observed in the disordered crystals. An intramolecular hydrogen bond between the amidic N and the methoxy O is observed in both crystals. There is no significant difference in bond distances and angles among the four conformers which have an extended conformation as a whole. However, major differences between YM-09151-2 and the present molecules occur in the area of the tertiary N atom and in the overall conformation.

Introduction. The crystal structure determination of YM-09151-1 is undertaken as part of serial studies of new potent neuroleptic drugs. In a previous paper (Furuya, Iwanami, Takenaka & Sasada, 1982), we have described the crystal structure of the free base of the diastereoisomer (YM-09151-2) of YM-09151-1 and discussed the structural features in relation to neuroleptic activity. In particular, we emphasized the importance of the surface exposure of the N atom of the

pyrrolidine ring. In order to examine the structure–activity relationship of the benzamide neuroleptic, anhydrous and monohydrate crystals were prepared. Both crystals show disordering and four conformers are found. We report the three-dimensional crystal structures and discuss several structural features.
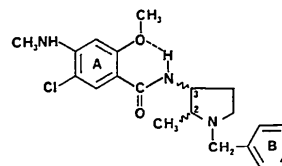
(1) YM-09151-1; (2R,3S) and (2S,3R)
(2) YM-09151-2; (2R,3R) and (2S,3S)

Experimental. YM-09151-1 was prepared by the method described by Iwanami, Takashima, Hirata, Hasegawa & Usuda (1981). The anhydrous and monohydrate crystals were obtained by recrystallization from acetone and aqueous ethanol solutions, respectively. Elemental analysis of the latter: found: C 57·05, H 6·59, N 9·36%; calc. C 57·02, H 6·61, N 9·50%.

Anhydrous crystal. Crystal 0·5 × 0·2 × 0·5 mm. Rigaku automated four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Accurate unit-cell constants from 40 high-angle reflections in the angular range $45° < 2\theta < 55°$. Density by flotation in a mixture of benzene and carbon tetrachloride. $5° < 2\theta < 120°$. $0 \le h \le 33$, $0 \le k \le 14$, $0 \le l \le 12$. θ–2θ scan mode, scanning rate 4°(2θ) min⁻¹. Stationary background counts for 10 s before and after each scan. Five reference reflections showed no significant intensity deterioration throughout the data collection. 3256 independent reflections, of which 945 weak reflections below background were considered zero reflections; the

observational threshold value, F_{lim}, was 3·15. Corrections for Lorentz and polarization factors, but not for absorption or secondary extinction. Standard deviations estimated by $\sigma^2(F_o) = \sigma_p^2(F_o) + qF_o^2$, where $\sigma_p(F_o)$ was evaluated by counting statistics and q (4·52 × 10^{-5}) was derived from the variations of the monitored reflections (McCandish, Stout & Andrews, 1975). Structure solved by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The refinement was sluggish and difference maps indicated the presence of scattering matter, which was interpreted as a disordered pyrrolidine ring. Constrained refinement then carried out with *SHELX76* (Sheldrick, 1976), in the pyrrolidine ring. Bond distances were loosely constrained to have the values of the corresponding ones of YM-09151-2. The site-occupancy factors of the disordered pyrrolidine ring were 0·664 and 0·336 for conformers (I) and (II), respectively. In the final refinement anisotropic temperature factors were used for the non-hydrogen atoms, except in the disordered parts.* The quantity minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2(F_o)$. In the refinement procedure, the zero reflections were assumed to have $F_o = F_{lim}$, but those with $|F_c| < F_{lim}$ were omitted. Final $R = 0·133$ ($wR = 0·091$), $\delta\rho_{max} = -0·54$ e Å^{-3}; $(\Delta/\sigma)_{max} = 0·3$ for C, 0·4 for N, 0·3 for O, 0·09 for Cl. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computations performed on a Hitachi HITAC M200 computer.

Monohydrate crystal. Crystal 0·3 × 0·3 × 0·5 mm. Mo $K\alpha$ radiation. 3981 reflections obtained in the range $2° < 2\theta < 50°$. $-12 \leq h \leq 12$, $-20 \leq k \leq 20$, $0 \leq l \leq 8$. 931 weak reflections considered as zero reflections. $F_{lim} = 1·72$. $q = 1·17 \times 10^{-5}$. Unit-cell dimensions with 40 reflections in the range $20° < 2\theta < 30°$. Both the pyrrolidine ring and the benzyl group showed disorder. As atomic overlapping of the disordered conformers (III) and (IV) is rather marked at the benzene ring of the benzyl group, the bond distances and angles in this part were fixed at 1·395 Å and 120°, respectively. The site-occupancy factors were 0·7 and 0·3 for the conformers (III) and (IV), respectively. Final $R = 0·120$ ($wR = 0·085$), $\delta\rho_{max} = 0·52$ e Å^{-3}; $(\Delta/\sigma)_{max} = 0·9$ for C, 0·7 for N, 0·6 for O, 0·1 for Cl. Other details are as for the anhydrous crystal.

Discussion. In the crystal structure, an intermolecular hydrogen-bond network is not constructed and the crystal seems to be stabilized by the stacking of the benzene rings of the benzyl group for the anhydrous crystal (Fig. 1) and of the benzamide group for the monohydrate crystal (Fig. 2). The structure of the

pyrrolidine ring is disordered in the anhydrous crystal; both the pyrrolidine ring and the benzyl group are disordered in the monohydrate.

Four conformers were formed as a result of the disordering of both structures. These are denoted as conformers (I) and (II) in the anhydrous crystal, and (III) and (IV) in the monohydrate crystal. Conformers (I) and (II) have a common benzamide moiety and benzyl group, whereas conformers (III) and (IV) have just a common benzamide moiety. Final atomic parameters for non-H atoms are listed in Tables 1 and 2 for the anhydrous and monohydrate crystals, respectively.* The molecular structures are presented in Figs. 3 and 4 with the numbering scheme of the atoms. Bond distances and angles are given in Tables 3 and 4.

Benzene ring A is almost planar for conformers (I) and (III) with maximum atomic deviations of 0·005 and 0·013 Å, respectively. The methoxy, N-methyl-amino, and carbamoyl groups attached to this ring also lie on the plane. An intramolecular hydrogen bond between the amide N(2) and the methoxy O(1) is observed in conformers (I) and (III), and forms a new
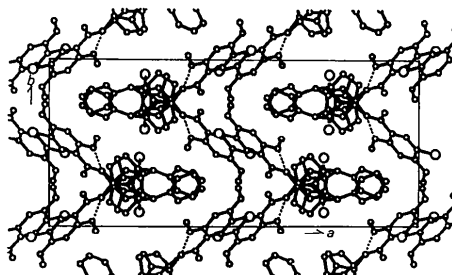
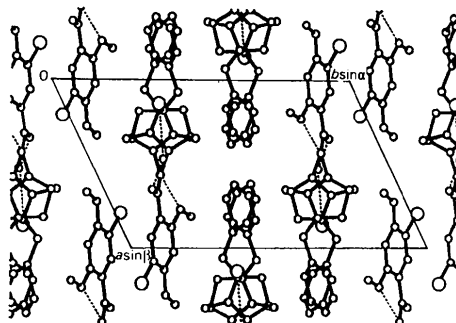Fig. 1. Crystal structure viewed down the c axis for the anhydrous crystal.

Fig. 2. Crystal structure viewed down the c axis for the monohydrate crystal.

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* As a result of the constrained refinement (for both crystals) there remained many small electron density peaks and H atoms were not clearly found on a difference map.

six-membered ring fused with the benzene ring A. This hydrogen-bond distance N(2)—O(1) is 2·678 (8) Å for (I) and 2·716 (9) Å for (III), which is almost the same as that of YM-09151-2, 2·680 Å. Benzene ring B is also almost planar for (I) with maximum atomic deviation 0·007 Å. The dihedral angle between this ring and benzene ring A, 57·8 (3)°, is relatively larger than the corresponding angle of YM-09151-2, 19·5°.

In both crystals, the Cl⁻ anion is hydrogen-bonded to the protonated N(3), Cl⁻—N(3) distances being 3·13 (1), 2·95 (1), 3·08 (1) and 3·02 (3) Å in conformers (I) to (IV), respectively. Furthermore, in the monohydrate crystal the Cl⁻ anion is bridged to carbonyl O(2) by the water molecule through the two hydrogen bonds, Cl⁻—O(W) and O(W)—O(2) being 3·238 (6) and 2·783 (8) Å respectively.

Table 1. *Fractional coordinates and equivalent isotropic temperature factors for the anhydrous crystal*

Here and in Table 2 the B values accompanied by $\langle \rangle$ are the equivalent isotropic temperature factors calculated from the anisotropic thermal parameters using the equation $B = 8\pi^2(U_1 + U_2 + U_3)/3$, where U_1, U_2, and U_3 are the principal components of the mean-square displacement matrix U. Values in $\langle \rangle$ are the anisotropicity defined by $[\sum(B-8\pi^2U_j)^2/3]^{1/2}$. The e.s.d.'s in parentheses refer to the last decimal places.

	x	y	z	$B(Å^2)$
Cl(1)	0·54280 (9)	0·0618 (2)	0·1788 (2)	7·3 ⟨25⟩
Cl(2)	0·25621 (8)	−0·4147 (2)	0·0462 (2)	5·6 ⟨10⟩
O(1)	0·3819 (2)	−0·0307 (5)	0·4576 (5)	6·4 ⟨26⟩
O(2)	0·4066 (2)	−0·1900 (5)	0·1469 (6)	8·9 ⟨50⟩
N(1)	0·5189 (2)	0·1679 (6)	0·4008 (7)	6·8 ⟨32⟩
N(2)	0·3587 (2)	−0·1711 (5)	0·2963 (6)	5·6 ⟨12⟩
N(3)	0·2533 (3)	−0·2905 (7)	0·2803 (8)	4·9 (2)
N(3')*	0·2605 (5)	−0·345 (1)	0·290 (1)	3·5 (4)
C(1)	0·4895 (3)	0·0923 (7)	0·3687 (7)	5·6 ⟨8⟩
C(2)	0·4944 (3)	0·0336 (6)	0·2643 (8)	5·4 ⟨21⟩
C(3)	0·4659 (3)	−0·0425 (7)	0·2216 (8)	5·8 ⟨24⟩
C(4)	0·4273 (3)	−0·0651 (7)	0·2886 (7)	4·9 ⟨6⟩
C(5)	0·4209 (3)	−0·0103 (7)	0·3931 (7)	5·2 ⟨14⟩
C(6)	0·4504 (3)	0·0684 (7)	0·4372 (8)	5·9 ⟨25⟩
C(7)	0·5115 (3)	0·2288 (7)	0·5088 (8)	6·7 ⟨26⟩
C(8)	0·3725 (3)	0·0273 (7)	0·5649 (6)	6·1 ⟨37⟩
C(9)	0·3970 (3)	−0·1463 (6)	0·2391 (8)	5·3 ⟨15⟩
C(10)	0·3260 (3)	−0·2367 (9)	0·234 (1)	4·0 (3)
C(11)	0·2992 (4)	−0·2917 (9)	0·326 (1)	5·2 (3)
C(12)	0·3147 (4)	−0·4031 (9)	0·350 (1)	4·7 (3)
C(13)	0·2468 (4)	−0·1853 (9)	0·231 (1)	5·2 (3)
C(14)	0·2929 (4)	−0·166 (1)	0·168 (1)	6·0 (3)
C(10')	0·3282 (7)	−0·260 (2)	0·279 (2)	4·3 (5)
C(11')	0·2788 (7)	−0·240 (2)	0·293 (2)	5·2 (6)
C(12')	0·2578 (6)	−0·166 (1)	0·204 (2)	2·5 (4)
C(13')	0·2927 (7)	−0·422 (2)	0·335 (2)	3·4 (5)
C(14')	0·3372 (6)	−0·358 (2)	0·352 (2)	2·8 (4)
C(15)	0·2192 (3)	−0·3358 (8)	0·3594 (8)	6·9 ⟨26⟩
C(16)	0·1729 (3)	−0·2979 (6)	0·3232 (7)	5·0 ⟨19⟩
C(17)	0·1518 (3)	−0·3465 (7)	0·2289 (8)	5·9 ⟨20⟩
C(18)	0·1079 (3)	−0·3097 (8)	0·1923 (9)	8·4 ⟨39⟩
C(19)	0·0881 (3)	−0·2236 (9)	0·255 (1)	9·8 ⟨61⟩
C(20)	0·1125 (4)	−0·1775 (8)	0·3491 (9)	9·7 ⟨55⟩
C(21)	0·1539 (3)	−0·2140 (7)	0·3824 (8)	6·7 ⟨26⟩

* Primed atoms refer to conformer (II).

Table 2. *Fractional coordinates and equivalent isotropic temperature factors for the monohydrate crystal*

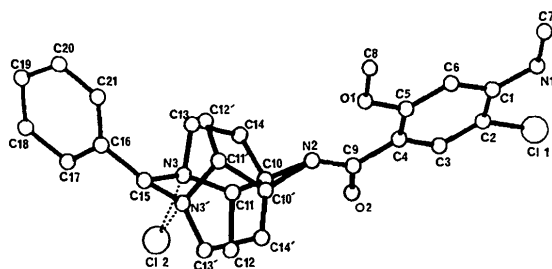|	x	y	z	$B(Å^2)$
Cl(1)	1·2099 (2)	−0·0202 (1)	0·2244 (3)	5·3 ⟨10⟩
Cl(2)	0·1382 (2)	0·3197 (2)	−0·1211 (3)	6·1 ⟨26⟩
O(1)	0·7701 (5)	0·2260 (3)	0·6795 (7)	4·8 ⟨10⟩
O(2)	0·6526 (5)	0·1617 (4)	0·1262 (7)	6·5 ⟨37⟩
O(W)	0·4698 (6)	0·2462 (4)	−0·1974 (7)	8·5 ⟨56⟩
N(1)	1·2780 (6)	0·0417 (4)	0·6108 (9)	4·9 ⟨17⟩
N(2)	0·5669 (7)	0·2113 (5)	0·4044 (9)	7·3 ⟨59⟩
N(3)	0·180 (2)	0·324 (1)	0·314 (2)	4·1 (5)
N(3')*	0·171 (3)	0·313 (2)	0·302 (4)	1·3 (7)
C(1)	1·1306 (9)	0·0766 (5)	0·548 (1)	4·0 ⟨13⟩
C(2)	1·0794 (8)	0·0539 (4)	0·362 (1)	3·8 ⟨10⟩
C(3)	0·9363 (8)	0·0843 (5)	0·286 (1)	3·9 ⟨7⟩
C(4)	0·8302 (7)	0·1426 (5)	0·394 (1)	3·7 ⟨13⟩
C(5)	0·8767 (8)	0·1685 (5)	0·580 (1)	3·8 ⟨9⟩
C(6)	1·0234 (8)	0·1368 (5)	0·654 (1)	3·9 ⟨8⟩
C(7)	1·3314 (8)	0·0602 (5)	0·807 (1)	5·9 ⟨25⟩
C(8)	0·8115 (8)	0·2504 (6)	0·872 (1)	6·0 ⟨33⟩
C(9)	0·6764 (9)	0·1727 (6)	0·300 (1)	5·2 ⟨19⟩
C(10)	0·420 (1)	0·2262 (8)	0·309 (1)	4·2 (2)
C(11)	0·326 (1)	0·3192 (8)	0·364 (2)	4·2 (2)
C(12)	0·346 (1)	0·3880 (7)	0·271 (2)	5·4 (3)
C(13)	0·185 (1)	0·2412 (8)	0·361 (2)	5·7 (3)
C(14)	0·349 (1)	0·1734 (9)	0·371 (2)	5·2 (3)
C(15)	0·070 (1)	0·4022 (7)	0·399 (1)	4·4 (2)
C(16)	−0·085 (1)	0·4126 (8)	0·341 (2)	4·4 (3)
C(17)	−0·148 (2)	0·381 (1)	0·450 (3)	5·5 (5)
C(18)	−0·293 (2)	0·397 (1)	0·397 (2)	6·6 (4)
C(19)	−0·374 (2)	0·445 (1)	0·236 (3)	8·8 (4)
C(20)	−0·311 (3)	0·477 (2)	0·127 (3)	9·5 (8)
C(21)	−0·166 (2)	0·460 (1)	0·180 (2)	7·4 (6)
C(10')	0·420 (2)	0·279 (2)	0·334 (3)	2·3 (4)
C(11')	0·317 (2)	0·243 (2)	0·357 (3)	2·7 (4)
C(12')	0·351 (2)	0·155 (1)	0·253 (2)	2·8 (5)
C(13')	0·175 (2)	0·393 (2)	0·395 (3)	3·1 (5)
C(14')	0·339 (2)	0·373 (1)	0·410 (3)	2·5 (4)
C(15')	0·057 (2)	0·293 (2)	0·348 (3)	3·5 (5)
C(16')	−0·097 (3)	0·367 (2)	0·319 (4)	3·2 (5)
C(17')	−0·186 (5)	0·373 (3)	0·448 (6)	5·0 (10)
C(18')	−0·328 (4)	0·441 (2)	0·434 (5)	6·2 (9)
C(19')	−0·380 (3)	0·502 (2)	0·290 (4)	4·7 (6)
C(20')	−0·291 (6)	0·495 (3)	0·161 (7)	6·2 (12)
C(21')	−0·149 (4)	0·428 (3)	0·175 (6)	5·7 (11)

* Primed atoms refer to conformer (IV).

Fig. 3. Molecular structure and atom numbering of the anhydrous crystal. Atoms with a prime refer to conformer (II).
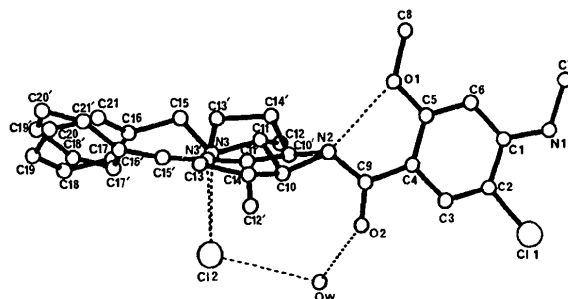

Fig. 4. Molecular structure and atom numbering of the monohydrate crystal. Atoms with a prime refer to conformer (IV).

In a previous paper (Furuya *et al.*, 1982), we proposed, as parameters for structure–activity correlation, the surface area *s* (Lee & Richard, 1971) of the tertiary amino N atom N(3) in addition to the other two parameters, *l* and *d*; *l* is the distance between N(3) and the centre of benzene ring *A*, and *d* is the deviation of N(3) from the mean plane of ring *A*. Table 5 summarizes these parameters and torsion angle τ [N(2)–C(10)–C(11)–N(3)] for the present four conformers and YM-09151-2. There is no significant difference in these parameters among the four conformers which have an extended conformation as a whole and are alike except for the pyrrolidine ring. However, there seems to be some relation between the *s*, *l* and τ values. The larger the *s* values are the longer are the *l* values. Furthermore, the *l* values of the *trans* arrangement between N(2) and N(3) are longer than that of the *gauche* (YM-09151-2). On the other hand, a major difference between the four conformers and YM-09151-2 is that the former has an extended conformation and exposes its tertiary amino N atom (areas $2 \cdot 7$–$3 \cdot 7$ Å^2), whereas the latter has a folded conformation and does not expose it. These results indicate that the parameters are cooperatively altered by the small change of the pyrrolidine-ring conformation so as to expose the N atom. Though it is not clear which is the most favourable for interacting with receptors and whether the other active conformers might or might not exist, some structural features of these four conformers would be important to establish the molecular mechanisms for interaction of benzamide neuroleptics with receptors.

Table 3. *Bond distances (Å) and angles (°) for the anhydrous crystal, with standard deviations in parentheses*

C(1)–C(2)	1·426 (13)	C(1)–C(6)	1·438 (13)
C(2)–C(3)	1·384 (13)	C(3)–C(4)	1·413 (12)
C(4)–C(5)	1·407 (12)	C(5)–C(6)	1·429 (12)
C(1)–N(1)	1·359 (12)	N(1)–C(7)	1·485 (12)
C(2)–Cl	1·780 (9)	C(5)–O(1)	1·402 (11)
O(1)–C(8)	1·469 (11)	C(4)–C(9)	1·489 (12)
C(9)–O(2)	1·233 (11)	C(9)–N(2)	1·354 (9)
C(15)–C(16)	1·518 (8)	C(16)–C(17)	1·400 (12)
C(16)–C(21)	1·393 (13)	C(17)–C(18)	1·452 (14)
C(18)–C(19)	1·444 (15)	C(19)–C(20)	1·431 (16)
C(20)–C(21)	1·374 (15)		

Disordered part: conformers (I) and (II)

N(2)–C(10)	1·473	1·471	C(11)–C(12)	1·527	1·527
C(10)–C(11)	1·502	1·499	N(3)–C(13)	1·478	1·472
C(10)–C(14)	1·541	1·541	C(13)–C(14)	1·572	1·571
C(11)–N(3)	1·464	1·457	N(3)–C(15)	1·482	1·472

C(2)–C(1)–C(6)	115·7 (8)	C(2)–C(1)–N(1)	122·6 (8)
C(6)–C(1)–N(1)	121·7 (8)	C(1)–C(2)–C(3)	127·5 (8)
C(1)–C(2)–Cl	116·3 (7)	C(3)–C(2)–Cl	116·2 (7)
C(2)–C(3)–C(4)	116·7 (8)	C(3)–C(4)–C(5)	118·3 (8)
C(3)–C(4)–C(9)	115·2 (7)	C(5)–C(4)–C(9)	126·5 (8)
C(4)–C(5)–C(6)	125·0 (8)	C(4)–C(5)–O(1)	118·1 (7)
C(6)–C(5)–O(1)	116·8 (7)	C(1)–C(6)–C(5)	116·9 (8)
C(1)–N(1)–C(7)	120·5 (8)	C(5)–O(1)–C(8)	120·6 (6)
C(4)–C(9)–O(2)	120·5 (8)	C(4)–C(9)–N(2)	119·2 (7)
O(2)–C(9)–N(2)	120·3 (7)	C(15)–C(16)–C(17)	118·4
C(15)–C(16)–C(21)	118·7	C(17)–C(16)–C(21)	122·9
C(16)–C(17)–C(18)	118·8	C(17)–C(18)–C(19)	118·1
C(18)–C(19)–C(20)	119·2	C(19)–C(20)–C(21)	121·7
C(16)–C(21)–C(20)	119·3		

Disordered part: conformers (I) and (II)

C(9)–N(2)–C(10)	117·1	129·4	C(11)–N(3)–C(13)	105·7	111·8
N(2)–C(10)–C(11)	106·0	117·2	C(11)–N(3)–C(15)	114·4	103·0
N(2)–C(10)–C(14)	109·1	116·5	C(13)–N(3)–C(15)	120·4	113·8
C(11)–C(10)–C(14)	106·8	104·5	N(3)–C(13)–C(14)	102·1	103·9
C(10)–C(11)–C(12)	114·1	115·7	C(10)–C(14)–C(13)	103·5	102·3
C(10)–C(11)–N(3)	103·6	101·7	N(3)–C(15)–C(16)	109·1	129·4
C(12)–C(11)–N(3)	110·9	113·7			

Table 4. *Bond distances (Å) and angles (°) for the monohydrate crystal, with standard deviations in parentheses*

C(1)–C(2)	1·437 (10)	C(1)–C(6)	1·415 (10)
C(2)–C(3)	1·364 (10)	C(3)–C(4)	1·404 (10)
C(4)–C(5)	1·438 (10)	C(5)–C(6)	1·394 (10)
C(1)–N(1)	1·379 (9)	N(1)–C(7)	1·482 (10)
C(2)–Cl	1·763 (7)	C(5)–O(1)	1·370 (8)
O(1)–C(8)	1·455 (9)	C(4)–C(9)	1·494 (10)
C(9)–O(2)	1·246 (9)	C(9)–N(2)	1·354 (9)

Disordered part: conformers (III) and (IV)

N(2)–C(10)	1·477	1·476	N(3)–C(13)	1·473	1·469
C(10)–C(11)	1·500	1·499	C(13)–C(14)	1·573	1·573
C(10)–C(14)	1·541	1·542	N(3)–C(15)	1·474	1·472
C(11)–N(3)	1·463	1·458	C(15)–C(16)	1·523	1·519
C(11)–C(12)	1·524	1·524			

C(2)–C(1)–C(6)	116·4 (6)	C(2)–C(1)–N(1)	119·5 (6)
C(6)–C(1)–N(1)	124·1 (6)	C(1)–C(2)–C(3)	124·3 (6)
C(1)–C(2)–Cl	117·8 (5)	C(3)–C(2)–Cl	118·0 (5)
C(2)–C(3)–C(4)	119·1 (6)	C(3)–C(4)–C(5)	118·5 (6)
C(3)–C(4)–C(9)	115·6 (6)	C(5)–C(4)–C(9)	125·9 (6)
C(4)–C(5)–C(6)	121·5 (6)	C(4)–C(5)–O(1)	116·9 (6)
C(6)–C(5)–O(1)	121·6 (6)	C(1)–C(6)–C(5)	120·1 (6)
C(1)–N(1)–C(7)	119·8 (6)	C(5)–O(1)–C(8)	118·9 (5)
C(4)–C(9)–O(2)	118·5 (7)	C(4)–C(9)–N(2)	119·9 (6)
O(2)–C(9)–N(2)	121·6 (6)		

Disordered part: conformers (III) and (IV)

C(9)–N(2)–C(10)	116·9	124·3	C(11)–N(3)–C(15)	112·4	112·5
N(2)–C(10)–C(11)	103·6	106·5	C(13)–N(3)–C(15)	113·2	114·8
N(2)–C(10)–C(14)	118·9	128·2	N(3)–C(13)–C(14)	104·0	103·4
C(11)–C(10)–C(14)	103·9	99·7	C(10)–C(14)–C(13)	104·1	106·8
C(10)–C(11)–C(12)	114·3	112·0	N(3)–C(15)–C(16)	113·9	116·4
C(10)–C(11)–N(3)	102·3	106·3	C(15)–C(16)–C(17)	117·8	123·5
C(12)–C(11)–N(3)	114·0	115·9	C(15)–C(16)–C(21)	122·1	116·5
C(11)–N(3)–C(13)	109·1	104·7			

Bond distances and angles of benzene ring *B* are fixed at 1·395 Å and 120°, respectively.

Table 5. *The surface areas of the tertiary amino N atom and some geometric parameters*

Compound	Surface area (s, Å²)	Distance (l, Å)	Deviation (d, Å)	Torsion angle (τ,°)
Conformer (I)	2·7	7·25	−0·7	−140·6
Conformer (II)	3·5	7·5	0·0	170·4
Conformer (III)	3·1	7·4	−0·3	165·0
Conformer (IV)	3·7	7·5	−0·4	−170·0
YM-09151-2 (free base)	0·0	6·26	−0·9	75·9

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Structure of N-(2-Nitrophenylthio)-S,S-diphenylsulfoximide. Intramolecular S···O Interaction

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Abstract. $C_{18}H_{14}N_2O_3S_2$, $M_r = 370 \cdot 44$, monoclinic, $C2/c$, $a = 27 \cdot 067$ (4), $b = 8 \cdot 483$ (1), $c = 14 \cdot 755$ (2) Å, $\beta = 92 \cdot 20$ (1)°, $V = 3385 \cdot 3$ (8) Å^3, $Z = 8$, $D_x = 1 \cdot 454$ Mg m^{-3}, Cu $K\alpha_1$, $\lambda = 1 \cdot 54051$ Å, $\mu = 2 \cdot 93$ mm^{-1}, $F(000) = 1536$, $T = 296$ K, $R = 0 \cdot 039$ for 2669 observed reflexions. The dimensions of the S-sulfoximido group are similar to those of other N-sulfoximides. The bonds about the tetracoordinated sulfur S^{VI} form a distorted tetrahedron with an NSO angle of $122 \cdot 7$ (1)°. The 2-nitrophenylthio group lies almost in one plane and associated with this planarity a short intramolecular S···O contact of $2 \cdot 532$ (2) Å is observed. The angle O···S—N is $175 \cdot 0$ (1)°.

Introduction. As a part of the systematic investigation of sulfoximides the title compound was synthesized by Akutagawa, Furukawa & Oae (1984). This is the first case in which the imido nitrogen is attached to sulfur; all other sulfoximides so far reported by X-ray analysis are those with the imido nitrogen attached to N. In order to investigate the structural character of this novel compound an X-ray analysis was undertaken.

Experimental. Orange plates, $0 \cdot 30 \times 0 \cdot 40 \times 0 \cdot 10$ mm, unit-cell parameters by least squares from 25 reflexions ($65 < 2\theta < 73°$), Rigaku AFC4 diffractometer, graphite monochromator, $2 < 2\theta < 130°$, $h = 0$–31, $k = 0$–9, $l = -17$–17, ω–2θ scan, scan range $\Delta\omega = 1 \cdot 0° + 0 \cdot 5°$ tanθ, scan speed 4° min^{-1} in 2θ, three reflexions monitored (006, 12,0,0, 333) every 50 reflexions, variations within $0 \cdot 5\%$. 2915 measured, 2853 unique, 2669 observed [$|F_o| \geq 3\sigma(F)$] reflexions.

Structure solved by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). All non-H atoms obtained from E map. H atoms found from difference map. Block-diagonal least squares with anisotropic temperature factors for non-H and isotropic for H. $\sum w(|F_o| - k^{-1}|F_o|)^2$ minimized. $w = 0 \cdot 5$ if $|F_o| < 7 \cdot 0$, $w = 1 \cdot 0$ if $7 \cdot 0 \leq |F_o| \leq 28 \cdot 0$, $w = (28/|F|)^2$ if $|F_o| > 28 \cdot 0$. Absorption correction applied numerically. Extinction parameter $g = 4 \times 10^{-7}$. $\Delta\rho(\text{max.}) = 0 \cdot 20$ e Å^{-3} [between S(1) and C(1)], $\Delta/\sigma(\text{max.}) = 0 \cdot 14$. $R = 0 \cdot 039$, $wR = 0 \cdot 050$, $S = 1 \cdot 098$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on HITAC M180 computer of the Data Processing Center of the University with programs *UNICS*III (Sakurai & Kobayashi, 1979), *MULTAN78* and *ORTEP*II (Johnson, 1971). The final atomic parameters are given in Table 1.*

Discussion. The molecular structure with the atom numbering is shown in Fig. 1. Bond distances and angles are listed in Table 2. All sulfoximides so far reported by X-ray analysis are those with the imido nitrogen attached to N (Andreetti, Bocelli & Sgarabotto, 1975, 1977; Andreetti, Bocelli, Coghi & Sgarabotto, 1974, 1975; Cabezuelo, Foces-Foces,

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