Table 7. *Comparison of selected angles of parent 1,4-dihydropyridine compounds and their decomposition products*

Compound	Torsion angle SUM (°)	Deviation from bisection (°)	References
Nifedipine	52.1	13.5	*a*
A	11.9	0.6 (5)	*b*
B	90.2	2.2	*c*
(I)	12.6	31.6 (5)	*d*
(II)	67.9	9.6 (7)	*d*
(III)	5.19	35.6 (8)	*d*

References: (*a*) Triggle, Schefter & Triggle (1980); (*b*) Rowan & Holt (1995) [compound *A*: dimethyl 2,6-dimethyl-4-(2-nitrophenyl)pyridine-3,5-dicarboxylate($CuCl_2$)]; (*c*) Fossheim, Svarteng, Mostad, Romming, Shefter & Triggle (1982) [compound *B*: dimethyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate]; (*d*) this work.

For each compound, the scan width was 0.6° above $K_{\alpha 1}$ and 0.6° below $K_{\alpha 2}$, with a variable scan rate and background counts on each side of the scan.

For all compounds, data collection: *XSCANS* (Siemens, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structures: *SHELXS*86 for (I) and (II); *SHELXL*93 (Sheldrick, 1993) for (III). For all compounds, molecular graphics: *XP* (Siemens, 1990)

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1105). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Tosyliminoiodo)benzene at 298 K

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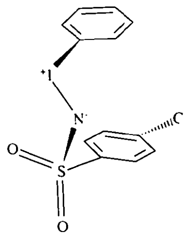
Abstract

The structure of (tosyliminoiodo)benzene (PhINTs), $C_{13}H_{12}INO_2S$, has been determined at 298 K and is compared with the structure previously determined at 130 K.

Comment

We are currently studying the primary nitrene transfer reagents ArINTs (Ts = *para*-toluenesulfonyl) (Cicero, Zhao & Protasiewicz, 1996) and have determined the structure of (tosyliminoiodo)benzene, PhINTs, (I), at room temperature. During the course of our work, the structure of the same compound at 130 K was reported by Power and co-workers (Mishra, Olmstead, Ellison & Power, 1995).



(I)

The compound at both temperatures exists as a zigzag polymer in the solid state utilizing N···I contacts to bridge the monomers in an asymmetric fashion. Intramolecular bond distances in (I) have nearly the same values as those obtained by Power and co-workers at 130 K. For example, the I—N bond distance is 2.027 (3) at 298 K *versus* 2.039 (2) Å at 130 K. Aromatic C—C distances range from 1.368 (8) to 1.390 (7) Å with an observed average value of 1.379 (7) Å. H atoms were located and their positions refined [C—H = 0.79 (6)–

0.98 (5) Å]. Intramolecular bond angles are for the most part in agreement, with the largest difference observed for the N—I—C angle in (I), which increases from 95.8 (1) at 130 K to 97.2 (1)° at 298 K. The different temperatures produce the greatest differences for the intermolecular contacts between the monomers of (I) in the polymer. The I···N secondary bonds increase from 2.482 (2) at 130 K to 2.549 (3) Å at 298 K (2.6%). Likewise, close I···I contacts increase from 3.853 (1) to 3.9114 (4) Å (1.5%). The unit-cell volume for (I) increases by 2.8% while the *b*-axis length (along which the polymer chain and I atoms are aligned) increases by 1.4% over this temperature range, correlating with the changes in the intermolecular I···N and I···I distances, respectively. The I···N bonds in both structures are much shorter than the I···N contacts of 3.140 (2) Å observed for the triclinic form of (tosyliminoiodo)-*o*-toluene (Cicero, Zhao & Protasiewicz. 1996), which displays a very similar intramolecular I—N bond length of 2.011 (2) Å and an N—I—C angle of 100.3 (1)°.

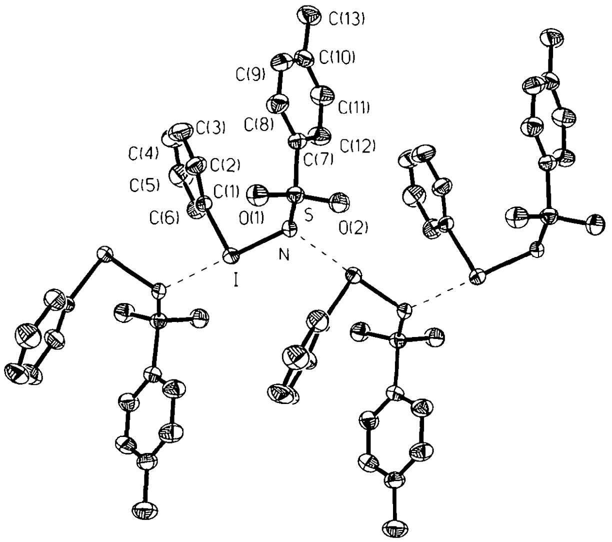


Fig. 1. Polymeric structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

Compound (I) was prepared by the literature method of Yamada, Yamamoto & Okawara (1975). Crystals were grown from an aqueous methanol solution at 273 K.

Crystal data

$C_{13}H_{12}INO_2S$
$M_r = 373.20$
Monoclinic
$P2_1/c$
$a = 11.6086$ (15) Å
$b = 7.7966$ (9) Å
$c = 15.2545$ (14) Å
$\beta = 104.520$ (7)°
$V = 1336.6$ (3) $Å^3$
$Z = 4$
$D_x = 1.855$ Mg m^{-3}
$D_m = 1.85$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 40 reflections
$\theta = 9.0$–27.2°
$\mu = 2.545$ mm^{-1}
$T = 298$ (2) K
Needle
0.40 × 0.08 × 0.08 mm
Yellow

Data collection

Siemens *P*4 diffractometer
θ–2θ scans
Absorption correction: ψ scan (North, Phillips & Matthews, 1968)
$T_{min} = 0.75$, $T_{max} = 0.82$
3155 measured reflections
2352 independent reflections
2043 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.026$
$\theta_{max} = 25.0°$
$h = -1 \rightarrow 13$
$k = -1 \rightarrow 9$
$l = -18 \rightarrow 17$
3 standard reflections monitored every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.106$
$S = 1.11$
2351 reflections
199 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0215P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.055$
$\Delta\rho_{max} = 0.60$ e $Å^{-3}$
$\Delta\rho_{min} = -0.37$ e $Å^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
I	0.98578 (2)	0.02332 (3)	0.74795 (2)	0.02925 (10)
N	0.8965 (3)	−0.1998 (4)	0.7489 (2)	0.0283 (7)
S	0.75881 (9)	−0.20367 (13)	0.69143 (7)	0.0299 (2)
O1	0.7259 (3)	−0.0545 (4)	0.6342 (2)	0.0407 (7)
O2	0.7390 (3)	−0.3691 (4)	0.6484 (2)	0.0446 (8)
C1	0.9262 (4)	0.1480 (5)	0.8514 (3)	0.0326 (9)
C2	0.8079 (4)	0.1901 (6)	0.8341 (3)	0.0418 (11)
C3	0.7673 (5)	0.2643 (7)	0.9036 (4)	0.0588 (15)
C4	0.8438 (5)	0.2962 (7)	0.9863 (4)	0.0557 (14)
C5	0.9616 (6)	0.2550 (7)	1.0007 (3)	0.0554 (14)
C6	1.0054 (4)	0.1765 (6)	0.9340 (3)	0.0418 (11)
C7	0.6725 (3)	−0.1990 (5)	0.7723 (3)	0.0299 (9)
C8	0.5673 (4)	−0.1083 (6)	0.7552 (3)	0.0424 (11)
C9	0.4979 (4)	−0.1138 (7)	0.8167 (4)	0.0478 (12)
C10	0.5317 (4)	−0.2078 (6)	0.8957 (3)	0.0402 (11)
C11	0.6377 (4)	−0.2962 (7)	0.9119 (3)	0.0445 (12)
C12	0.7081 (4)	−0.2936 (7)	0.8508 (3)	0.0426 (11)
C13	0.4556 (6)	−0.2165 (8)	0.9627 (4)	0.0561 (15)

Table 2. *Selected geometric parameters (Å, °)*

I—N	2.027 (3)	S—O2	1.439 (3)
I—C1	2.113 (4)	S—O1	1.447 (3)
I···N^i	2.549 (3)	S—C7	1.775 (4)
I···I^{ii}	3.9114 (4)	C10—C13	1.511 (6)
N—S	1.619 (3)		
N—I—C1	97.22 (14)	O2—S—N	106.7 (2)
N—I···N^i	178.09 (6)	O1—S—N	112.8 (2)
S—N—I	116.7 (2)	O2—S—C7	106.4 (2)
I—N···I^{ii}	117.03 (13)	O1—S—C7	107.1 (2)
O2—S—O1	117.2 (2)		

Symmetry codes: (i) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$.

Data collection: *XSCANS* (Siemens 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXTL-Plus*.

The author thanks the CWRU Chemistry Department for support of this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1198). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Clemizole

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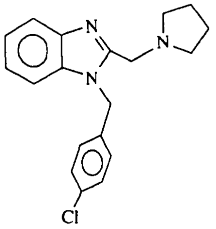
Abstract

The crystal structure of clemizole [1-(4-chlorobenzyl)-2-(1-pyrrolidinylmethyl)-1*H*-benzimidazole, $C_{19}H_{20}ClN_3$] is composed of independent molecules of the free base having normal molecular dimensions and no unusual contacts shorter than van der Waals distances.

Comment

The crystal structure of clemizole hydrochloride has been reported previously (Parvez, 1996). We have now separated the free base from its hydrochloride salt and have grown crystals suitable for data collection by the X-ray diffraction method. In the present paper, we report the crystal structure of the free base, clemizole, (I), which is an anti-allergic drug effective on H1 receptors.



(I)

An *ORTEP*II (Johnson, 1976) drawing of clemizole with the atomic numbering scheme is shown in Fig. 1. The molecular dimensions are unexceptional with distances C_{sp^2}—Cl 1.755 (5), C=N 1.328 (5) and C_{sp^3}—C_{sp^2} 1.492 (6) Å, and mean N—C_{sp^3} 1.462 (12), N—C_{sp^2} 1.384 (12), C_{sp^3}—C_{sp^3} 1.517 (7) and C—$C_{aromatic}$ 1.380 (14) Å.

The benzimidazole system and phenyl ring are essentially individually planar with maximum deviations from the respective least-squares planes of 0.061 (5) and 0.012 (6) Å; these planes are inclined at an angle of

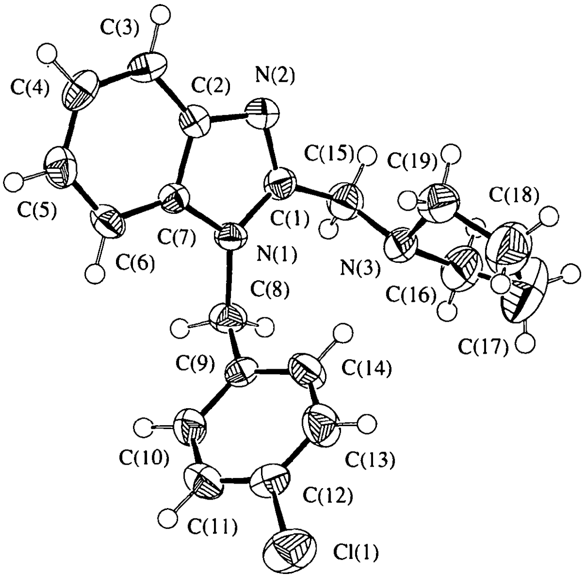


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.