low-melting polymorph compared to the locking together of molecules in the high-melting form.

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XV. *N,N′*-Ditosyl-2,3,5,6-tetrahydro-1*H*,7*H*-4,1,7-benzoxadiazonine

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Abstract. $C_{24}H_{26}N_2O_5S_2$, $M_r = 486{\cdot}6$, monoclinic, $C2$, $a = 27{\cdot}869\ (5)$, $b = 8{\cdot}374\ (2)$, $c = 23{\cdot}058\ (6)$ Å, $\beta = 119{\cdot}14\ (3)°$, $V = 4700\ (2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}375\ (1)$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 22{\cdot}54$ cm^{-1}, $F(000) = 2048$, room temperature, $R = 0{\cdot}0455$ for 4141 reflections with $I > 3\sigma(I)$. There are two different molecules in the asymmetric unit. Molecule 1 exists in a chair-like conformation; molecule 2 occurs in two forms: a chair-like and a twist-chair conformation in a ratio of 0·63:0·37, respectively.

Introduction. Based on the expected pharmacological properties and complex-formation ability, a new class of benzoxadiazonine derivatives has been synthesized (Glinka, Mikiciuk-Olasik & Kotełko, 1977; Mikiciuk-Olasik & Kotełko, 1984). The possibility of using them as complexing agents for ^{99m}Tc, employed in medical diagnostics (Kapuściński, Liniecki, Durski & Mikiciuk-Olasik, 1986), seemed to be of special interest. The title compound was obtained by a condensation of *N,N′*-ditosyl-*o*-phenylenediamine with bis(2-chloroethyl) ether in presence of potassium *tert*-butanolate. The formula was confirmed by IR, ^{1}H NMR and MS analyses. The present X-ray structure determination was undertaken to provide the evidence for the structure of the molecule.

Experimental. Colourless thick-prismatic crystals of size 0·2 × 0·3 × 0·5 mm from ethanol at room temperature. Diffraction data measured on a CAD-4 diffractometer using the θ–2θ scan technique,

0108-2701/92/010094-04$03.00

Table 1. *Positional parameters with e.s.d.'s*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	*y*	*z*	B_{eq}(Å^2)
O1	0·5627 (1)	0·671	0·1987 (2)	5·5 (1)
C2	0·5907 (3)	0·6202 (8)	0·2667 (3)	5·6 (2)
C3	0·6182 (2)	0·4616 (8)	0·2726 (3)	5·4 (2)
N4	0·6595 (2)	0·4719 (5)	0·2493 (2)	4·3 (1)
C5	0·6945 (2)	0·6097 (6)	0·2669 (2)	4·0 (1)
S1	0·68560 (5)	0·2959 (2)	0·24716 (6)	4·21 (3)
O11	0·7254 (1)	0·3232 (4)	0·2267 (2)	4·8 (1)
O12	0·6395 (1)	0·1931 (5)	0·2085 (2)	5·5 (1)
C11	0·7197 (2)	0·2227 (6)	0·3298 (2)	4·1 (1)
C12	0·7736 (2)	0·2571 (7)	0·3716 (3)	4·6 (2)
C13	0·7986 (2)	0·2047 (8)	0·4363 (3)	5·7 (2)
C14	0·7699 (2)	0·1123 (8)	0·4598 (3)	5·9 (2)
C15	0·7163 (3)	0·0809 (8)	0·4168 (3)	5·8 (2)
C16	0·6902 (2)	0·1328 (7)	0·3521 (3)	4·6 (2)
C141	0·7980 (4)	0·052 (2)	0·5302 (4)	10·0 (4)
C51	0·7348 (2)	0·6345 (8)	0·3314 (3)	5·1 (2)
C52	0·7695 (2)	0·7661 (9)	0·3495 (3)	6·1 (2)
C6	0·6881 (2)	0·7198 (6)	0·2184 (2)	4·0 (1)
N7	0·6437 (1)	0·7066 (5)	0·1531 (2)	4·1 (1)
C8	0·5963 (2)	0·8129 (8)	0·1353 (3)	4·9 (2)
C9	0·5796 (2)	0·8223 (7)	0·1882 (3)	5·0 (2)
S2	0·65868 (5)	0·6804 (2)	0·09226 (6)	4·89 (4)
O21	0·7128 (1)	0·6190 (5)	0·1220 (2)	5·8 (1)
O22	0·6454 (2)	0·8234 (5)	0·0524 (2)	7·1 (2)
C21	0·6114 (2)	0·5349 (7)	0·0413 (2)	4·5 (2)
C22	0·5640 (3)	0·5748 (9)	−0·0143 (3)	5·7 (2)
C23	0·5292 (3)	0·460 (1)	−0·0553 (3)	6·5 (2)
C24	0·5416 (2)	0·298 (1)	−0·0417 (3)	6·2 (2)
C25	0·5893 (3)	0·2596 (8)	0·0149 (4)	6·0 (2)
C26	0·6252 (2)	0·3735 (8)	0·0570 (3)	5·1 (2)
C241	0·5036 (4)	0·172 (1)	−0·0885 (5)	9·0 (3)
C53	0·7638 (2)	0·8739 (8)	0·3009 (3)	5·5 (2)
C54	0·7232 (2)	0·8533 (7)	0·2366 (3)	4·8 (2)
O1*	0·3764 (2)	−0·0234 (6)	0·3338 (3)	7·4 (2)
C2*	0·3394 (3)	0·0256 (9)	0·2706 (4)	6·4 (2)
C3*	0·3507 (2)	0·1921 (9)	0·2545 (3)	5·9 (2)
N4*	0·4041 (2)	0·1973 (5)	0·2540 (2)	4·6 (1)
C5*	0·4131 (2)	0·0753 (6)	0·2166 (2)	4·5 (2)
S3	0·42439 (5)	0·3812 (2)	0·25149 (7)	5·06 (4)
O31	0·4698 (1)	0·3670 (5)	0·2401 (2)	6·2 (1)
O32	0·4308 (2)	0·4584 (5)	0·3106 (2)	6·8 (1)
C31	0·3710 (2)	0·4805 (6)	0·1833 (2)	4·5 (1)
C32	0·3689 (2)	0·4783 (7)	0·1229 (3)	5·2 (2)
C33	0·3264 (2)	0·5577 (8)	0·0694 (3)	5·7 (2)
C34	0·2869 (2)	0·6412 (8)	0·0776 (3)	6·2 (2)
C35	0·2895 (3)	0·6396 (9)	0·1390 (3)	6·4 (2)
C36	0·3318 (2)	0·5627 (8)	0·1923 (3)	5·7 (2)
C341	0·2437 (5)	0·735 (2)	0·0211 (5)	10·4 (4)
C63	0·3909 (3)	−0·0543 (9)	0·1134 (4)	6·8 (3)
C64	0·3815 (3)	0·0664 (8)	0·1491 (3)	5·4 (2)
C6*	0·4539 (2)	−0·0391 (7)	0·2512 (3)	4·9 (2)
N7*	0·4843 (2)	−0·0408 (6)	0·3215 (2)	5·9 (1)
C8*	0·4640 (3)	−0·140 (1)	0·3593 (4)	7·1 (3)
C91*	0·4047 (4)	−0·159 (1)	0·3294 (5)	5·0 (4)
C92*	0·433 (1)	−0·033 (3)	0·379 (1)	8 (1)
S4	0·55072 (6)	−0·0137 (3)	0·3588 (1)	7·86 (6)
O41	0·5635 (2)	0·0428 (7)	0·3095 (3)	9·2 (2)
O42	0·5786 (2)	−0·1508 (7)	0·3981 (3)	11·9 (2)
C41	0·5621 (2)	0·136 (1)	0·4170 (3)	6·8 (2)
C42	0·5751 (4)	0·103 (1)	0·4818 (5)	9·7 (3)
C43	0·5796 (4)	0·222 (2)	0·5243 (5)	9·8 (3)
C44	0·5734 (3)	0·380 (1)	0·5047 (4)	7·9 (3)
C45	0·5627 (3)	0·413 (1)	0·4413 (4)	7·0 (2)
C46	0·5578 (2)	0·296 (1)	0·3977 (3)	6·6 (2)
C441	0·5770 (4)	0·516 (2)	0·5506 (6)	10·7 (5)
C61	0·4609 (3)	−0·1589 (8)	0·2139 (5)	6·6 (3)
C62	0·4311 (3)	−0·1622 (9)	0·1477 (4)	7·1 (3)

* Atoms in molecule 2.

graphite-monochromatized Cu $K\alpha$ radiation, not corrected for absorption. Lattice parameters by least squares using 25 reflections in the range 7·8–42·2°. Reflections measured to $(\sin\theta/\lambda)_{max} = 0{\cdot}63$ Å^{-1}, ranges of *hkl*: −34–30, −9–0, 0–28 respectively. From 4695 unique reflections, 4141 were accepted as observed by the criterion $I > 3\sigma(I)$, $R_{int} = 0{\cdot}0495$;

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

O1	C2	1·433 (7)		C53	C54	1·369 (7)	
O1	C9	1·415 (6)		O1*	C2*	1·377 (8)	
C2	C3	1·51 (1)		O1*	C91*	1·42 (1)	
C3	N4	1·491 (9)		O1*	C92*	1·41 (2)	
N4	C5	1·436 (6)		C2*	C3*	1·52 (1)	
N4	S1	1·656 (5)		C3*	N4*	1·493 (9)	
C5	C51	1·374 (6)		N4*	C5*	1·437 (8)	
C5	C6	1·393 (7)		N4*	S3	1·652 (5)	
S1	O11	1·419 (5)		C5*	C64	1·366 (7)	
S1	O12	1·438 (4)		C42	C43	1·36 (2)	
S1	C11	1·773 (5)		C43	C44	1·38 (2)	
C11	C12	1·363 (6)		C44	C45	1·37 (1)	
C11	C16	1·387 (9)		C5*	C6*	1·401 (7)	
C12	C13	1·376 (8)		S3	O31	1·419 (5)	
C13	C14	1·40 (1)		S3	O32	1·440 (5)	
C14	C15	1·358 (8)		S3	C31	1·760 (4)	
C14	C141	1·51 (1)		C31	C32	1·364 (9)	
C15	C16	1·372 (8)		C31	C36	1·39 (1)	
C51	C52	1·390 (9)		C32	C33	1·394 (7)	
C52	C53	1·39 (1)		C33	C34	1·39 (1)	
C6	N7	1·414 (5)		C34	C35	1·38 (1)	
C6	C54	1·409 (7)		C34	C341	1·49 (1)	
S4	C41	1·750 (8)		C35	C36	1·379 (8)	
C41	C42	1·38 (1)		C63	C64	1·41 (1)	
C41	C46	1·40 (1)		C63	C62	1·35 (1)	
N7	C8	1·475 (7)		C6*	N7*	1·418 (7)	
N7	S2	1·661 (5)		C6*	C61	1·39 (1)	
C8	C9	1·51 (1)		N7*	C8*	1·50 (1)	
S2	O21	1·415 (4)		N7*	S4	1·634 (4)	
S2	O22	1·444 (5)		C8*	C91*	1·46 (1)	
S2	C21	1·759 (5)		C8*	C92*	1·46 (3)	
C21	C22	1·361 (6)		C91*	C92*	1·48 (3)	
C21	C26	1·404 (8)		S4	O41	1·427 (7)	
C22	C23	1·367 (9)		S4	O42	1·434 (6)	
C23	C24	1·40 (1)		C44	C441	1·52 (2)	
C24	C25	1·372 (8)		C45	C46	1·36 (1)	
C24	C241	1·51 (1)		C61	C62	1·34 (1)	
C25	C26	1·381 (8)					

C2	O1	C9	113·7 (4)	C5*	C6*	C61	117·4 (5)
O1	C2	C3	109·8 (5)	N7*	C6*	C61	120·8 (5)
C2	C3	N4	111·4 (6)	C6*	N7*	C8*	119·0 (5)
C3	N4	C5	118·8 (5)	C6*	N7*	S4	119·5 (5)
C3	N4	S1	112·8 (4)	C8*	N7*	S4	115·0 (4)
C5	N4	S1	118·4 (3)	N7*	C8*	C91*	116·7 (7)
N4	C5	C51	120·9 (5)	N7*	C8*	C92*	107 (1)
N4	C5	C6	120·0 (3)	C91*	C8*	C92*	61 (1)
C51	C5	C6	119·1 (5)	O1*	C91*	C8*	114·8 (8)
N4	S1	O11	107·0 (2)	O1*	C91*	C92*	58 (1)
N4	S1	O12	106·2 (2)	C8*	C91*	C92*	60 (1)
N4	S1	C11	106·8 (2)	O1*	C92*	C8*	115 (2)
O11	S1	O12	119·7 (2)	O1*	C92*	C91*	58·6 (9)
O11	S1	C11	108·4 (2)	C8*	C92*	C91*	59 (1)
O12	S1	C11	108·0 (2)	N7*	S4	O41	106·6 (3)
S1	C11	C12	121·1 (5)	N7*	S4	O42	110·4 (3)
S1	C11	C16	118·9 (3)	N7*	S4	C41	103·8 (3)
C12	C11	C16	120·0 (5)	S2	C21	C22	121·8 (5)
C11	C12	C13	120·1 (6)	S2	C21	C26	118·2 (3)
C12	C13	C14	120·9 (5)	C22	C21	C26	119·9 (5)
C13	C14	C15	117·2 (5)	C21	C22	C23	121·0 (7)
C13	C14	C141	120·5 (6)	C22	C23	C24	120·9 (5)
C15	C14	C141	122·3 (8)	C23	C24	C25	117·2 (6)
C14	C15	C16	123·1 (7)	C23	C24	C241	120·4 (6)
C11	C16	C15	118·7 (5)	C25	C24	C241	122·3 (7)
C5	C51	C52	121·8 (6)	C24	C25	C26	122·9 (6)
C51	C52	C53	119·0 (5)	C21	C26	C25	118·0 (4)
C5	C6	N7	120·5 (4)	C52	C53	C54	120·1 (6)
C5	C6	C54	119·2 (4)	C6	C54	C53	120·7 (6)
N7	C6	C54	120·0 (4)	C2*	O1*	C91*	108·7 (7)
C6	N7	C8	116·8 (4)	C2*	O1*	C92*	143 (1)
C6	N7	S2	117·4 (4)	C91*	O1*	C92*	63 (1)
C8	N7	S2	113·8 (3)	O1*	C2*	C3*	113·3 (5)
N7	C8	C9	113·2 (4)	C2*	C3*	N4*	110·8 (6)
O1	C9	C8	110·5 (5)	C3*	N4*	C5*	116·7 (4)
N7	S2	O21	106·6 (2)	C3*	N4*	S3	112·8 (4)
N7	S2	O22	109·6 (3)	C5*	N4*	S3	118·6 (4)
N7	S2	C21	104·0 (3)	N4*	C5*	C64	121·3 (5)
O21	S2	O22	119·5 (3)	N4*	C5*	C6*	118·4 (4)
O21	S2	C21	110·3 (3)	C64	C5*	C6*	120·3 (6)
O22	S2	C21	105·8 (2)	N4*	S3	O31	106·3 (3)
N4*	S3	C31	108·5 (2)	N4*	S3	O32	105·8 (3)
O31	S3	O32	120·2 (2)	O41	S4	O42	119·9 (4)
O31	S3	C31	108·5 (3)	O41	S4	C41	110·5 (4)
O32	S3	C31	107·2 (3)	O42	S4	C41	104·5 (4)
S3	C31	C32	120·2 (5)	S4	C41	C42	122·6 (7)
S3	C31	C36	119·0 (4)	S4	C41	C46	119·3 (5)

Table 2 (*cont.*)

C32	C31	C36	120·8 (5)	C42	C41	C46	118·1 (8)
C31	C32	C33	119·6 (7)	C41	C42	C43	121 (1)
C32	C33	C34	120·4 (6)	C42	C43	C44	121 (1)
C33	C34	C35	118·8 (5)	C43	C44	C45	117·8 (9)
C33	C34	C341	120·0 (7)	C43	C44	C441	122 (1)
C35	C34	C341	121·1 (8)	C45	C44	C441	120 (1)
C34	C35	C36	120·8 (7)	C44	C45	C46	122·6 (9)
C31	C36	C35	119·5 (7)	C41	C46	C45	119·4 (7)
C64	C63	C62	118·4 (7)	C6*	C61	C62	121·5 (7)
C5*	C64	C63	120·4 (6)	C63	C62	C61	122·0 (9)
C5*	C6*	N7*	121·6 (6)				

* Atoms in molecule 2.

Table 3. *Selected torsion angles* (°)

O1—C2—C3—N4	60·5 (7)	O1*—C2*—C3*—N4*	65·2 (8)
C2—C3—N4—C5	41·3 (7)	C2*—C3*—N4*—C5*	48·8 (7)
C3—N4—C5—C6	−110·6 (6)	C3*—N4*—C5*—C6*	−112·2 (6)
N4—C5—C6—N7	6·8 (7)	N4*—C5*—C6*—N7*	2·8 (8)
C5—C6—N7—C8	100·6 (6)	C5*—C6*—N7*—C8*	91·8 (7)
C6—N7—C8—C9	−44·1 (7)	C6*—N7*—C8*—C91*	−31·6 (10)
		C6*—N7*—C8*—C92*	−96·5 (12)
N7—C8—C9—O1	−62·3 (6)	N7*—C8*—C91*—O1*	−75·0 (10)
		N7*—C8*—C92*—O1*	91·3 (17)
C8—C9—O1—C2	126·6 (5)	C8*—C91*—O1*—C2*	119·8 (9)
		C8*—C92*—O1*—C2*	−65·2 (27)
C9—O1—C2—C3	−123·2 (6)	C91*—O1*—C2*—C3*	−117·7 (8)
		C92*—O1*—C2*—C3*	−48·3 (21)
S1—N4—C3—C2	−173·6 (4)	S3*—N4*—C3*—C2*	−168·7 (5)
S1—N4—C5—C6	106·3 (5)	S3—N4*—C5*—C6*	107·6 (5)
C11—S1—N4—C5	83·3 (4)	C31—S3—N4*—C5*	87·7 (4)
C11—S1—N4—C3	−62·0 (5)	C31—S3—N4*—C3*	−54·0 (5)
S2—N7—C8—C9	174·1 (4)	S4—N7*—C8*—C91*	176·5 (6)
		S4—N7*—C8*—C92*	111·6 (12)
S2—N7—C6—C5	−119·0 (5)	S4—N7*—C6*—C5*	−117·6 (6)
C21—S2—N7—C8	−80·7 (5)	C41—S4—N7*—C8*	−78·8 (6)
C21—S2—N7—C6	137·7 (6)	C41—S4—N7*—C6*	129·5 (5)

* Atoms in molecule 2.

standard reflection 731, count variation 2·79%. Structure solved by direct methods using *SHELX*76 (Sheldrick, 1976), refinement carried out by full-matrix least squares using F magnitudes, 830 parameters (refined in two blocks, one for each molecule). All H atoms located on a difference map. Final R = 0·0455, unit weights, S = 1·6008. The absolute configuration of the molecules was established; the final R value for the alternative enantiomorph is 0·0577. Max. shift/e.s.d. = 0·488; the largest peaks on the final difference map were 0·33 and −0·4 e Å^{-3}. Complex atomic scattering factors from *SHELX*76.

Discussion. The final atomic coordinates are listed in Table 1,* bond lengths and bond angles in Table 2 and selected torsion angles in Table 3. The structures of molecules 1 and 2, with the atom-numbering scheme, are shown in Figs. 1(*a*) and 1(*b*).

There are two different molecules in the asymmetric unit. The main difference is that while molecule 1 exists as one conformer, molecule 2 occurs in two forms in a ratio of 0·63:0·37. The heterocyclic rings of molecule 1 and the predominant form in molecule 2 are in a chair-like conformation. They are approximately symmetrical with respect to a pseudo mirror plane passing through O1 and the middle of the C5—C6 bond: the asymmetry parameter (Duax & Norton, 1975) is Δ_m = 5·5 (6)° for molecule 1 and 14·2 (9)° for molecule 2. The heterocyclic ring of the other form of molecule 2 is in a twist-chair conformation.

The eight torsion-angle values about the S—N bond are in agreement with the data gathered by

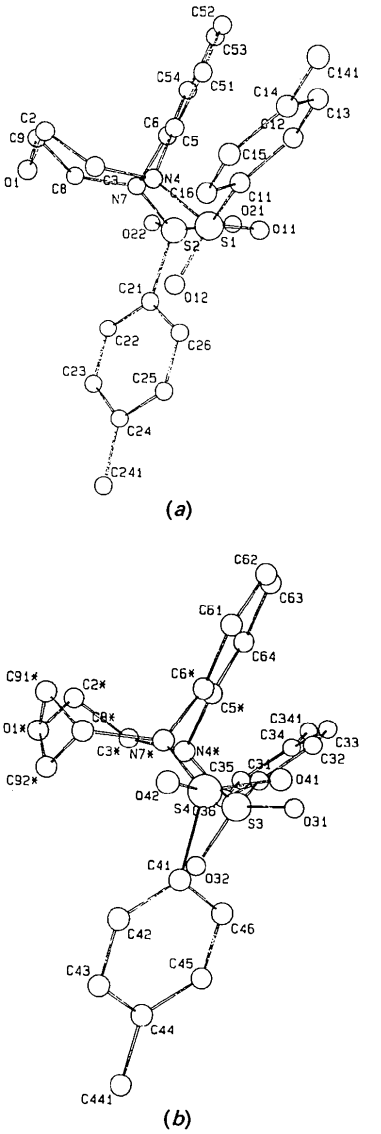

Fig. 1. The structure of (*a*) molecule 1 and (*b*) molecule 2 with the atom-numbering scheme.

* 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 54342 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Kálmán, Czugler & Argay (1981). The orientation of both tosyl substituents, with respect to the heterocyclic ring, is the same for molecules 1 and 2. A pronounced tendency to form pyramidal bonds is observed for N4 and N7 in both molecules; the sums of the bond angles about N fall in the range 348·1–353·5°.

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Structural Studies of Some Benzodiazepines. II. Structure of 3-(Isopropylamino)-4,5-dihydro-1*H*-2,4-benzodiazepine Hydroiodide (2)

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Abstract. $C_{12}H_{19}N_3^+.I^-$, $M_r = 332{\cdot}207$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}235\ (3)$, $b = 14{\cdot}671\ (3)$, $c = 7{\cdot}723\ (2)$ Å, $V = 1386{\cdot}28\ (57)$ Å^3, $Z = 4$, $D_x = 1{\cdot}5917\ (7)$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 181{\cdot}8$ cm^{-1}, $F(000) = 664$, diffractometer data at room temperature, $R = 5{\cdot}56\%$ for 2672 reflections with $I > 3\sigma(I)$. The benzodiazepine ring is in an envelope-type conformation, all N atoms are protonated and form hydrogen bonds with an I^- ion.

Introduction. This work is the second part in a series of crystal structure investigations of some benzodiazepine derivatives. The structure of 3-(benzylamino)-4,5-dihydro-1*H*-2,4-benzodiazepine hydrochloride (1) was reported earlier (Brzozowski, Stępień, Brzezińska, Glinka & Bavoux, 1989). Pharmacological experiments (E. Brzezińska & R. Glinka, unpublished results) have shown that (1) and (2) exhibit a depressive action on the central nervous system, reflected by the depression of the spontaneous locomotor activity. Both compounds also depressed the apomorphine-induced excitation and stereotypy in rats and induced hypothermia in mice. Thus, these compounds possess neuroleptic activity, which, however, is not very strong. (1) and (2) are weakly bound to the D_1 and D_2 receptors. Compound (1) also shows analgesic activity.

Experimental. Colourless crystals, equidimensional in habit (~0·6 mm), were obtained from ethanol at room temperature; the specimen used for the X-ray work, of size 0·2 × 0·2 × 0·2 mm, was cut from a larger crystal. Preliminary cell parameters were obtained from Weissenberg photographs. Diffraction data measured at room temperature on a CAD-4 diffractometer with graphite-monochromatized Cu $K\alpha$ radiation. Lattice parameters determined by least squares using setting angles of 25 reflections with $\theta_{max} = 50{\cdot}0°$. 3142 reflections (1/4 Ewald sphere)

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