

significantly from linearity. This angle is $171.5(3)^\circ$ in the title compound and its value compares very well with the average of $172(1)^\circ$ with a range of 170.2 – 176.2° for the ten structures contained in the Cambridge Crystallographic Database. The deviation is in-plane with the central part of the molecule. In the literature this deviation of linearity has been ascribed to packing effects (Bullough & Wheatley, 1957; Naqvi & Wheatley, 1970; Lambrecht *et al.*, 1981). However, the fact that this non-linearity was observed in all reported ketenimine structures that all have different crystallographic environments warrants the view that it is an intrinsic property of the ketenimine moiety. This view is supported by results of SCF calculations mentioned above in which optimization of the $C=C=N$ angle led to an energy minimum with an angle of 175° and a twist angle of the *N*-phenyl ring of 25° . This minimum is 1.8 kJ mol^{-1} lower than the minimum obtained with the model in which this angle was constrained to 180° . From the X-ray observations and the results of the SCF calculations it may be concluded that the packing effects mainly affect the orientation of the *N*-phenyl ring with respect to the ketenimine π system, rather than the linearity of the $C=C=N$ chain.

The stacking of the molecules in the unit cell is shown in Fig. 2. The twists of the terminal phenyl rings, mentioned before, are necessary to prevent severe non-bonded repulsions between the aromatic rings.

Nevertheless, some intramolecular contacts are short: $H(C9)\cdots C14$, $H(C13)\cdots C6$, $H(C13)\cdots C1$ and $H(C5)\cdots C14$ which are $2.55(1)$, $2.71(1)$, $2.73(1)$ and $2.74(1) \text{ \AA}$ respectively.

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XI. 5,6,7,8-Tetrahydro-*N,N'*-ditosyl-4*H*-dibenzo[*b,i*][1,5,7]oxadiazecine. XII. 4,5,6,7,8,9,10,11-Octahydro-*N,N'*-ditosyl-1-oxa-5,10-diazadibenzo[*b,l*]cyclotridecene

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Abstract. (XI) $C_{29}H_{28}N_2O_5S_2$, $M_r = 548.68$, monoclinic, $P2_1/c$, $a = 13.917(4)$, $b = 9.970(4)$, $c = 19.711(6) \text{ \AA}$, $\beta = 101.15(2)^\circ$, $V = 2683.33 \text{ \AA}^3$, $Z =$

4, $\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu = 0.24 \text{ mm}^{-1}$, $D_x = 1.358 \text{ g cm}^{-3}$, $F(000) = 1152$. Diffractometer data at room temperature, $R = 0.0368$ for 2735 reflections with $I > 2.5\sigma(I)$. The heterocycle is in a boat-like conformation. The tosyl groups adopt an *exo, exo* conformation. The heterocycle system and the tosyl

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Table 1. Fractional atomic coordinates and thermal parameters (Å²) for (XI)

$U_{eq} = \frac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$

	x	y	z	U_{eq}
S1	0.75469 (5)	0.04687 (8)	0.88691 (4)	0.0483 (4)
S2	0.78723 (6)	-0.00454 (9)	0.66713 (4)	0.0596 (5)
O1	0.7025 (2)	0.0355 (2)	0.9426 (1)	0.061 (1)
O2	0.8320 (1)	-0.0462 (2)	0.8838 (1)	0.060 (1)
O3	0.8550 (2)	0.0486 (3)	0.7245 (1)	0.083 (2)
O4	0.7667 (2)	0.0670 (2)	0.6030 (1)	0.081 (2)
O5	0.4605 (1)	-0.0802 (2)	0.7204 (1)	0.052 (1)
N1	0.6772 (2)	0.0351 (2)	0.8131 (1)	0.045 (1)
N2	0.6825 (2)	-0.0213 (2)	0.6928 (1)	0.050 (1)
C1	0.8017 (2)	0.2116 (3)	0.8911 (1)	0.048 (2)
C2	0.8608 (2)	0.2521 (3)	0.8462 (2)	0.072 (2)
C3	0.9024 (3)	0.3778 (4)	0.8533 (2)	0.075 (2)
C4	0.8865 (2)	0.4655 (3)	0.9038 (2)	0.060 (2)
C5	0.8278 (3)	0.4233 (4)	0.9479 (2)	0.080 (3)
C6	0.7849 (3)	0.2969 (4)	0.9424 (2)	0.073 (2)
C7	0.9330 (3)	0.6041 (4)	0.9107 (2)	0.089 (3)
C8	0.6863 (2)	-0.0713 (3)	0.7640 (1)	0.047 (2)
C9	0.8277 (2)	-0.1656 (3)	0.6508 (1)	0.053 (2)
C10	0.8068 (2)	-0.2184 (3)	0.5850 (2)	0.061 (2)
C11	0.8387 (3)	-0.3464 (4)	0.5738 (2)	0.077 (2)
C12	0.8904 (2)	-0.4234 (4)	0.6265 (3)	0.079 (3)
C13	0.9106 (3)	-0.3690 (5)	0.6922 (2)	0.086 (3)
C14	0.8799 (2)	-0.2415 (4)	0.7049 (2)	0.075 (2)
C15	0.9238 (3)	-0.5648 (5)	0.6122 (4)	0.124 (4)
C16	0.5853 (2)	0.1107 (3)	0.8064 (1)	0.043 (1)
C17	0.4999 (2)	0.0392 (3)	0.8287 (1)	0.046 (1)
C18	0.4776 (2)	0.0621 (3)	0.8939 (1)	0.056 (2)
C19	0.4009 (2)	-0.0016 (4)	0.9151 (2)	0.069 (2)
C20	0.3429 (2)	-0.0908 (4)	0.8709 (2)	0.074 (2)
C21	0.3615 (2)	-0.1150 (3)	0.8052 (2)	0.066 (2)
C22	0.4396 (2)	-0.0492 (3)	0.7852 (2)	0.049 (2)
C23	0.4446 (2)	0.0172 (3)	0.6696 (1)	0.048 (2)
C24	0.3640 (2)	0.1009 (3)	0.6601 (2)	0.060 (2)
C25	0.3521 (3)	0.1944 (4)	0.6075 (2)	0.073 (2)
C26	0.4197 (3)	0.2033 (4)	0.5646 (2)	0.076 (2)
C27	0.4996 (3)	0.1185 (4)	0.5735 (2)	0.068 (2)
C28	0.5127 (2)	0.0214 (3)	0.6265 (1)	0.051 (2)
C29	0.5996 (2)	-0.0708 (3)	0.6379 (1)	0.055 (2)

Table 2. Fractional atomic coordinates and thermal parameters (Å²) for (XII)

$U_{eq} = \frac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$

	x	y	z	U_{iso} or U_{eq}
S1	-0.0056 (2)	0.6839 (2)	0.1042 (1)	0.048 (1)*
S2	0.5424 (2)	1.0930 (2)	0.2270 (1)	0.056 (2)*
O1	0.2419 (4)	0.9093 (4)	0.0351 (3)	0.053 (4)*
O2	-0.1255 (4)	0.7028 (4)	0.0641 (3)	0.059 (4)*
O3	0.0059 (5)	0.6274 (4)	0.1675 (3)	0.065 (4)*
O4	0.5742 (5)	1.0579 (4)	0.2981 (3)	0.074 (4)*
O5	0.6359 (5)	1.1299 (5)	0.1905 (3)	0.079 (4)*
N1	0.0542 (5)	0.7896 (5)	0.1245 (3)	0.045 (4)*
N2	0.4789 (6)	1.0031 (5)	0.1800 (3)	0.050 (5)*
C7	0.3111 (8)	0.4935 (7)	-0.0854 (5)	0.074 (7)*
C8	0.1796 (7)	0.7876 (6)	0.1715 (4)	0.062 (6)*
C9	0.1992 (8)	0.8667 (7)	0.2263 (4)	0.068 (6)*
C10	0.2720 (7)	0.9552 (6)	0.2078 (4)	0.056 (6)*
C11	0.4066 (7)	0.9301 (7)	0.2123 (4)	0.059 (6)*
C18	0.1592 (9)	1.4058 (8)	0.2309 (6)	0.097 (9)*
C19	0.4453 (7)	1.0166 (6)	0.1022 (4)	0.047 (5)*
C32	0.0122 (6)	0.8737 (6)	0.0800 (4)	0.046 (5)*
C1	0.0821 (6)	0.6274 (5)	0.0475 (4)	0.042 (2)
C2	0.1949 (7)	0.5828 (6)	0.0780 (4)	0.050 (2)
C3	0.2668 (7)	0.5409 (6)	0.0342 (4)	0.052 (2)
C4	0.2299 (7)	0.5391 (6)	-0.0383 (4)	0.056 (2)
C5	0.1196 (7)	0.5817 (6)	-0.0668 (4)	0.057 (2)
C6	0.0459 (7)	0.6246 (6)	-0.0249 (4)	0.049 (2)
C12	0.4309 (6)	1.1843 (6)	0.2276 (4)	0.043 (2)
C13	0.4042 (7)	1.2461 (6)	0.1704 (4)	0.053 (2)
C14	0.3142 (7)	1.3163 (6)	0.1715 (4)	0.061 (2)
C15	0.2531 (8)	1.3271 (6)	0.2294 (4)	0.058 (2)
C16	0.2831 (7)	1.2631 (6)	0.2839 (4)	0.059 (2)
C17	0.3698 (7)	1.1938 (6)	0.2843 (4)	0.056 (2)
C20	0.4575 (6)	0.9234 (6)	0.0652 (4)	0.040 (2)
C21	0.5729 (7)	0.8814 (6)	0.0640 (4)	0.050 (2)
C22	0.5873 (7)	0.7939 (6)	0.0335 (4)	0.055 (2)
C23	0.4855 (7)	0.7412 (6)	0.0032 (4)	0.057 (2)
C24	0.3677 (7)	0.7807 (6)	0.0026 (4)	0.051 (2)
C25	0.3551 (7)	0.8687 (6)	0.0318 (4)	0.040 (2)
C26	0.1418 (7)	0.8863 (5)	-0.0188 (4)	0.044 (2)
C27	0.1552 (8)	0.8879 (5)	-0.0885 (4)	0.053 (2)
C28	0.0497 (8)	0.8722 (6)	-0.1404 (5)	0.062 (2)
C29	-0.0602 (8)	0.8565 (6)	-0.1198 (4)	0.054 (2)
C30	-0.0730 (7)	0.8580 (6)	-0.0507 (4)	0.053 (2)
C31	0.0302 (6)	0.8724 (6)	0.0041 (4)	0.043 (2)

* U_{eq} values.

groups are arranged in alternate layers parallel to (001). (XII) C₃₂H₃₄N₂O₅S₂, $M_r = 590.76$, monoclinic, $P2_1/c$, $a = 11.055$ (6), $b = 13.835$ (5), $c = 19.227$ (5) Å, $\beta = 99.73$ (3)°, $V = 2898.39$ Å³, $Z = 4$, $\lambda(\text{Mo } K\alpha) = 0.071069$ Å, $\mu = 0.23$ mm⁻¹, $D_x = 1.353$ g cm⁻³, $F(000) = 1248$. Diffractometer data at room temperature, $R = 0.0487$ for 1437 reflections with $I > 2.5\sigma(I)$. The heterocycle may be considered to consist of two parts, both having a boat-like conformation. The tosyl groups adopt an *exo, exo* conformation. Pairs of centrosymmetric N1-tosyl groups approach to a distance of 3.65 Å between their rings.

Introduction. In the course of a study on the structure-pharmacological activity relationship of heterocyclic systems we recently reported new results for nine-membered moieties having two fused aromatic rings (Glinka, 1986; Stępień, Wajsman, Grabowski, Glinka & Perrin, 1987; Olszak, Stępień, Wajsman, Grabowski, Glinka & Lecocq, 1987). The title compounds (XI) and (XII) show comparatively low neuroleptic action. Namely, the reaction with muscarinic and dopamine D₂ receptors could be observed.

Compound (XI) was obtained by reaction of 2,2'-bis(tosylaminomethyl)diphenyl ether with methylene bromide using sodium butoxide as a catalyst, while

(XII) was obtained by reaction of 2,2'-(tosylaminomethyl)diphenyl ether with 1,4-ditosylbutane in DMF as a solvent. Formulae for both compounds were confirmed by MS, IR and NMR spectra. The present paper reports on the X-ray crystal structures.

Experimental. Colourless crystals from ethanol at room temperature; prismatic crystals of size 0.2 × 0.5 × 2 mm (cut to 0.2 × 0.3 × 0.3 mm) for (XI) and thick tabular crystals of (XII) of size 0.2 × 0.3 × 0.3 mm. Diffraction data measured on an Enraf-Nonius CAD-4 diffractometer using $\omega-2\theta$ scan technique, graphite-monochromatized Mo $K\alpha$, not corrected for absorption. Data collected to $(\sin\theta/\lambda)_{\max} = 0.63$ Å⁻¹, ranges of h, k, l -16 to 16, 0 to 11, 0 to 22 for (XI) and -12 to 12, 0 to 15, 0 to 22 for (XII). Lattice parameters by least-squares methods using 25 reflections in the θ range 7–13°, three standard reflections, no intensity variation, $R_{\text{int}} = 0.0146$ and 0.0242 for (XI) and (XII) respectively. From a total of 4069 and 3711 unique reflections, 2735 and 1437 with $I > 2.5\sigma(I)$ were considered observed for (XI) and (XII) respectively.

Table 3. Bond lengths (Å) and angles (°) for (XI)

S1-N1	1.639 (2)	S2-N2	1.642 (2)
S1-O1	1.434 (2)	S2-O4	1.431 (2)
S1-O2	1.431 (2)	S2-O3	1.426 (2)
S1-C1	1.764 (3)	S2-C9	1.752 (3)
C1-C6	1.374 (4)	C9-C10	1.377 (4)
C6-C5	1.389 (5)	C10-C11	1.382 (5)
C5-C4	1.370 (5)	C11-C12	1.378 (5)
C4-C3	1.375 (5)	C12-C13	1.383 (6)
C3-C2	1.375 (4)	C13-C14	1.379 (6)
C2-C1	1.380 (4)	C14-C9	1.392 (4)
C4-C7	1.521 (5)	C12-C15	1.528 (6)
O5-C22	1.397 (3)	O5-C23	1.382 (3)
C22-C17	1.393 (4)	C23-C28	1.391 (4)
C17-C16	1.522 (4)	C28-C29	1.501 (4)
C16-N1	1.468 (3)	C29-N2	1.504 (4)
N1-C8	1.456 (3)	N2-C8	1.481 (3)
C17-C18	1.398 (4)	C28-C27	1.410 (4)
C18-C19	1.374 (4)	C27-C26	1.382 (5)
C19-C20	1.389 (5)	C26-C25	1.385 (5)
C20-C21	1.389 (5)	C25-C24	1.379 (5)
C21-C22	1.390 (4)	C24-C23	1.381 (4)
S1-N1-C16	117.1 (2)	S2-N2-C29	114.5 (2)
S1-N1-C8	121.0 (2)	S2-N2-C8	117.1 (2)
N1-S1-O1	109.3 (1)	N2-S2-O4	106.6 (1)
N1-S1-O2	106.6 (1)	N2-S2-O3	106.5 (1)
N1-S1-C1	106.5 (1)	N2-S2-C9	107.4 (2)
C1-S1-O1	106.0 (1)	C9-S2-O4	108.1 (1)
C1-S1-O2	109.3 (1)	C9-S2-O3	107.4 (1)
O1-S1-O2	118.6 (1)	O4-S2-O3	120.2 (2)
S1-C1-C6	119.8 (2)	S2-C9-C10	120.5 (2)
S1-C1-C2	120.2 (2)	S2-C9-C14	119.8 (2)
C1-C2-C3	119.6 (3)	C9-C14-C13	119.8 (3)
C2-C3-C4	121.9 (3)	C14-C13-C12	121.1 (4)
C3-C4-C5	117.6 (3)	C13-C12-C11	118.1 (4)
C4-C5-C6	122.0 (3)	C12-C11-C10	122.0 (4)
C5-C6-C1	119.1 (3)	C11-C10-C9	119.2 (3)
C6-C1-C2	119.8 (3)	C10-C9-C14	119.8 (3)
C7-C4-C3	121.2 (3)	C15-C12-C13	121.3 (4)
C7-C4-C5	121.2 (3)	C15-C12-C11	120.6 (4)
C22-O5-C23	118.3 (2)	N1-C8-N2	113.1 (2)
C8-N1-C16	120.0 (2)	C8-N2-C29	117.0 (2)
N1-C16-C17	116.7 (2)	N2-C29-C28	113.0 (2)
C16-C17-C22	122.0 (2)	C29-C28-C23	121.0 (3)
C17-C22-O5	119.7 (2)	C28-C23-O5	115.5 (3)
C16-C17-C18	120.9 (3)	C27-C28-C29	121.1 (3)
C17-C22-C21	122.3 (3)	C28-C23-C24	122.0 (3)
C22-C21-C20	118.7 (3)	C23-C24-C25	119.2 (3)
C21-C20-C19	120.3 (3)	C24-C25-C26	120.3 (3)
C20-C19-C18	119.8 (3)	C25-C26-C27	120.7 (3)
C19-C18-C17	121.8 (3)	C26-C27-C28	119.9 (3)
C18-C17-C22	117.0 (3)	C27-C28-C23	117.9 (3)
O5-C22-C21	117.9 (3)	O5-C23-C24	122.4 (3)

The structures were solved by direct methods using *SHELX86* (Sheldrick, 1986) and the refinement carried out by full-matrix least squares using *F* magnitudes and 352 [for (XI)] and 259 [for (XII)] parameters. All H atoms were located by difference maps and refined 'riding' on their corresponding C atom; three common values of thermal parameters were refined for phenyl, methylene and methyl H atoms respectively. *SHELX76* (Sheldrick, 1976) used for refinement. Final *R* factors 0.0368 for (XI) and 0.0487 for (XII); *wR* 0.0435 and 0.0493, with $w = 1/\sigma^2(F_o) + 0.00266F_o^2$, $S = 1.0382$ and $w = 1/\sigma^2(F_o) + 0.000813F_o^2$, $S = 1.8567$, for (XI) and (XII) respectively. Max. Δ/σ 0.417 and 0.136, largest peak in the ΔF maps 0.21 and 0.26 e Å⁻³, for (XI) and (XII) respectively. Atomic scattering factors from *SHELX76*. Thermal parameters were treated anisotropically for all non-H atoms of compound (XI) and all but the phenyl ring atoms of compound (XII). The geometries of the molecules were calculated using *FFE3* (Busing, Martin

Table 4. Bond lengths (Å) and angles (°) for (XII)

S1-N1	1.626 (6)	S2-N2	1.625 (6)
S1-O2	1.440 (5)	S1-O5	1.438 (6)
S1-O3	1.433 (5)	S2-O4	1.436 (6)
S1-C1	1.760 (8)	S2-C12	1.766 (8)
C1-C2	1.426 (9)	C12-C13	1.385 (10)
C2-C3	1.380 (10)	C13-C14	1.393 (10)
C3-C4	1.384 (10)	C14-C15	1.404 (10)
C4-C5	1.381 (10)	C15-C16	1.369 (10)
C5-C6	1.374 (10)	C16-C17	1.354 (10)
C6-C1	1.382 (9)	C17-C12	1.383 (9)
C7-C4	1.516 (11)	C18-C15	1.508 (11)
C31-C30	1.429 (9)	C20-C21	1.405 (10)
C30-C29	1.360 (10)	C21-C22	1.367 (10)
C29-C28	1.358 (10)	C22-C23	1.385 (10)
C28-C27	1.417 (10)	C23-C24	1.411 (10)
C27-C26	1.374 (10)	C24-C25	1.358 (10)
C26-C31	1.391 (10)	C25-C20	1.421 (9)
O1-C26	1.418 (8)	O1-C25	1.383 (8)
C31-C32	1.507 (10)	C20-C19	1.490 (10)
N1-C32	1.471 (9)	N2-C19	1.491 (9)
N1-C8	1.521 (9)	N2-C11	1.487 (10)
C8-C9	1.511 (11)	C9-C10	1.540 (11)
C10-C11	1.516 (10)		
N1-S1-O2	105.3 (3)	N2-S2-O5	106.1 (4)
N1-S1-O3	108.2 (3)	N2-S2-O4	106.5 (4)
N1-S1-C1	107.6 (3)	N2-S2-C12	108.3 (3)
C1-S1-O2	107.6 (4)	C12-S2-O5	108.0 (4)
C1-S1-O3	107.8 (4)	C12-S2-O4	107.2 (4)
O2-S1-O3	119.7 (3)	O4-S2-O5	120.3 (4)
S1-N1-C32	119.8 (5)	S2-N2-C19	118.2 (5)
S1-N1-C8	114.7 (5)	S2-N2-C11	119.9 (5)
S1-C1-C2	118.2 (6)	S2-C12-C13	119.5 (6)
S1-C1-C6	122.7 (6)	S2-C12-C17	120.5 (6)
C6-C1-C2	119.1 (7)	C17-C12-C13	120.0 (7)
C1-C2-C3	118.9 (7)	C12-C13-C14	118.4 (8)
C2-C3-C4	121.4 (8)	C13-C14-C15	121.8 (8)
C3-C4-C5	118.8 (8)	C14-C15-C16	116.8 (8)
C4-C5-C6	121.6 (8)	C15-C16-C17	122.8 (9)
C5-C6-C1	120.1 (7)	C16-C17-C12	120.2 (8)
C7-C4-C3	120.4 (8)	C18-C15-C14	121.1 (9)
C7-C4-C5	120.8 (8)	C18-C15-C16	122.1 (9)
C31-C30-C29	121.3 (8)	C20-C21-C22	123.0 (8)
C30-C29-C28	121.9 (9)	C21-C22-C23	120.1 (8)
C29-C28-C27	119.3 (8)	C22-C23-C24	118.9 (8)
C28-C27-C26	118.2 (8)	C23-C24-C25	120.2 (8)
C27-C26-C31	123.9 (7)	C24-C25-C20	122.3 (7)
C26-C31-C30	115.3 (7)	C25-C20-C21	115.4 (7)
C30-C31-C32	119.7 (7)	C19-C20-C21	121.4 (7)
O1-C26-C27	120.7 (7)	O1-C25-C24	122.7 (7)
O1-C26-C31	115.1 (6)	O1-C25-C20	114.9 (7)
C26-C31-C32	125.0 (7)	C25-C20-C19	123.1 (7)
C31-C32-N1	118.2 (6)	C20-C19-N2	110.1 (6)
C32-N1-C8	121.7 (6)	C19-N2-C11	116.3 (6)
N1-C8-C9	114.3 (7)	N2-C11-C10	114.4 (7)
C25-O1-C26	118.4 (5)	C11-C10-C9	110.5 (7)
		C10-C9-C8	115.7 (7)

& Levy, 1971). The drawings were prepared using the program *SCHAKAL* (Keller, 1984).

Discussion. The final positional parameters are listed in Tables 1 and 2, the interatomic distances and angles in Tables 3 and 4.* Figs. 1, 2, 3 and 4 show the molecular structure and the specific arrangements of molecules in (XI), and the molecular structure and the packing pattern of (XII), respectively.

In the compound (XI), structural differences are observed around N1 and N2, the latter showing a

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51501 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

tendency to form pyramidal bonds: the sum of its angles is $348.6(3)^\circ$. For N1, instead, the values add up to $358.1(3)^\circ$, in agreement with our previous results (Stępień, Wajsman, Grabowski, Krakowiak & Perrin, 1987; Stępień, Wajsman, Grabowski, Glinka & Perrin, 1987; Derewenda, Stępień, Olszak, Krakowiak & Perrin, 1987; Olszak, Stępień, Wajsman, Grabowski, Glinka & Lecocq, 1987). The resulting 'lone pair' at N2 is antiparallel with the C9–S2 bond. No such differences are noticeable in the compound (XII).

Both tosyl groups in either compound are oriented outwards from the heterocycle, showing an *exo, exo* conformation. The eight torsion angle values about S–N in both compounds range between 60 and 80° , in agreement with the data gathered by Kálmán, Czugler

& Argay (1981), with the exception of C1–S1–N1–C8 in compound (XI). All the geometric values for bond distances and angles found for the tosyl groups lie within the limits established by the same authors.

In the compound (XI), the ten-membered heterocycle has a boat-like conformation with approximately inverted sides and O5, C8 as edge-up atoms. Using the torsion angle criteria of Duax, Weeks & Rohrer (1976), however, even the uniquely possible pseudo-symmetry plane or axis passing through these two atoms is excluded by the quite high values of ΔC_s or ΔC_2 , respectively.

In order to describe the conformation of the 13-membered ring in (XII), we find it easier to divide it into two parts, the first (C9, C8, N1, C32, C31, C26, O1) being in a boat-like conformation and the second (O1, C25, C20, C19, N2, C11, C10) in a boat conformation.

The rings in both compounds do not show any significant non-bonded contact. In compound (XI) the

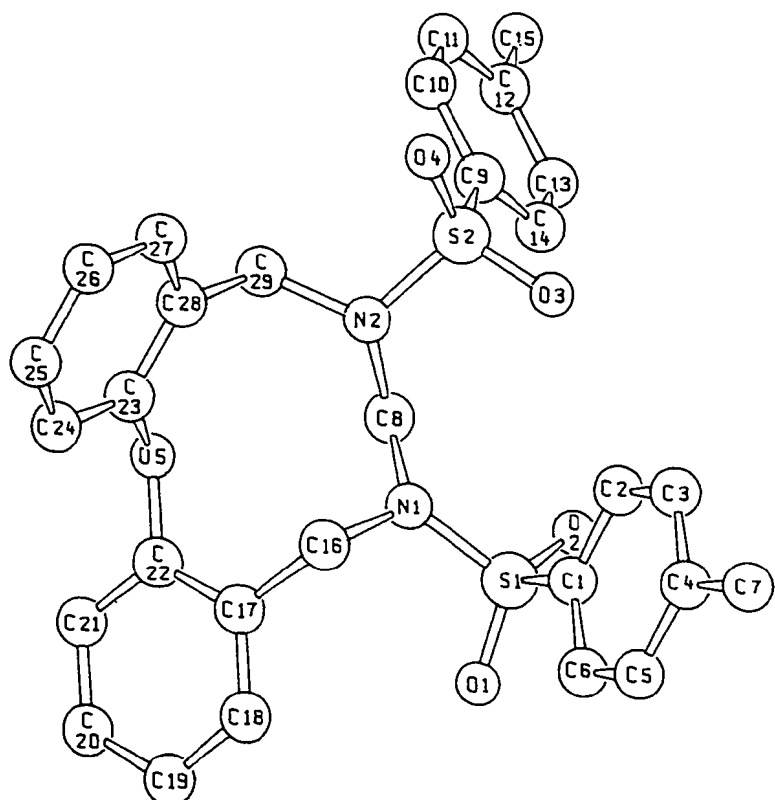


Fig. 1. Molecular structure and atom-numbering scheme for (XI).

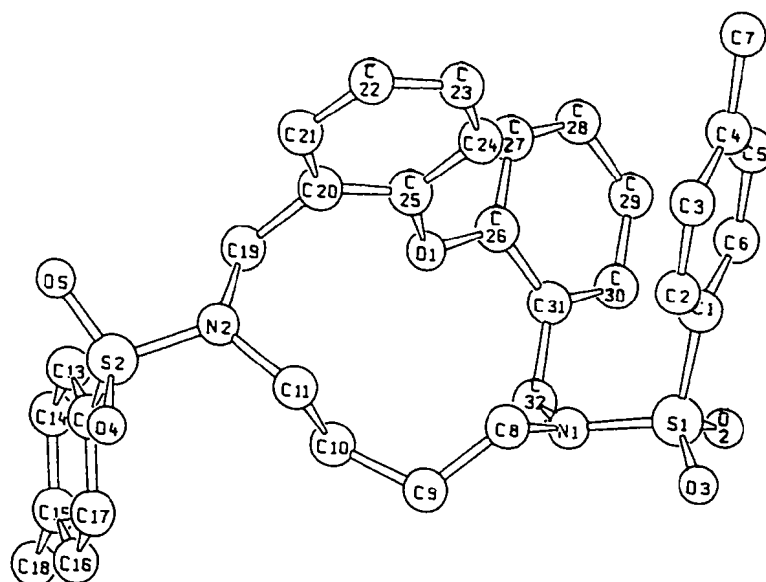


Fig. 3. Molecular structure and atom-numbering scheme for (XII).

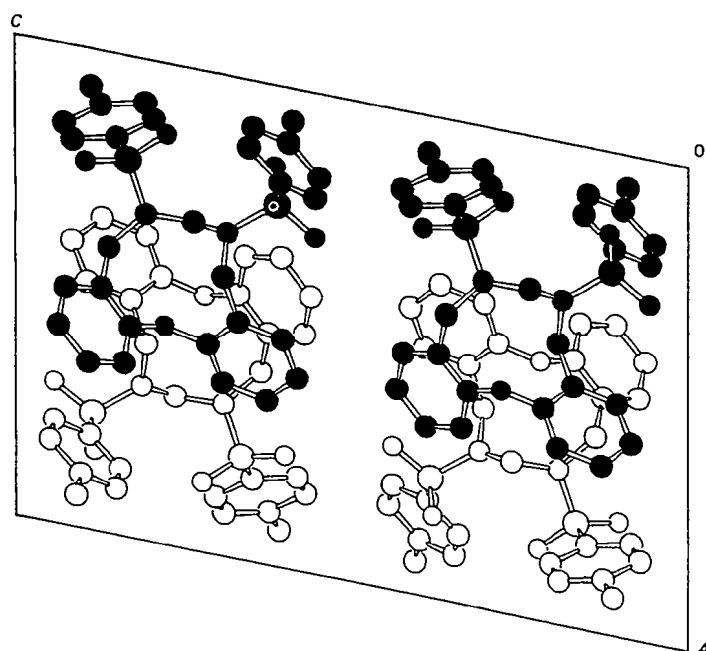


Fig. 2. Arrangement of molecules, viewed along the *y* axis, for (XI).

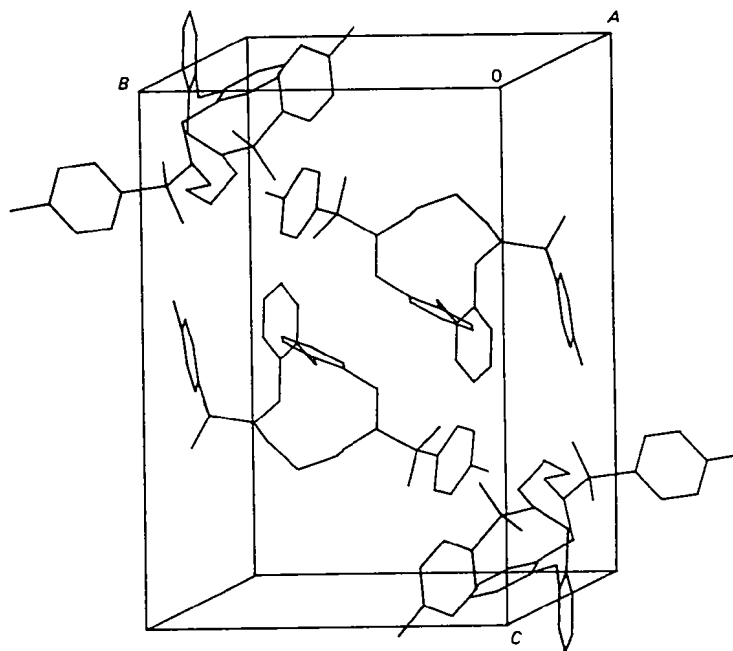


Fig. 4. Packing diagram for (XII) (viewed along the *x* axis).

ring and the tosyl groups are arranged (Fig. 2) as sets of alternate layers parallel to (100): the packing is probably responsible for the mentioned deviations from the normal molecular geometry. In compound (XII) (Fig. 4) the aromatic rings of neighbouring molecules are facing each other astride the centre of inversion: their relative distance is 3.65 Å.

There are only van der Waals intermolecular contacts in both compounds.

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Structure du Nitro-7 Benzothiasélénole-2,1 One-3

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Abstract. C₇H₃NO₃SSe, *M_r* = 260.13, orthorhombic, *P*2₁2₁2₁, *a* = 5.508 (4), *b* = 11.331 (9), *c* = 13.244 (9) Å, *V* = 826.60 (5) Å³, *Z* = 4, *D_x* = 2.090 g cm⁻³, λ(Mo Kα) = 0.7107 Å, μ = 50.9 cm⁻¹, *T* = 290 K, m.p. 417–418 K, *F*(000) = 504. Final *R* = 0.072 for 728 observed reflexions. Structure solved by direct method. There is ring closure through an Se–S bond equal to 2.222 (5) Å. An Se...O bonding interaction with an O atom of the nitro group is also observed: 2.51 (1) Å. The cohesion of the crystal is the result of van der Waals interactions.

Introduction. L'ebsele (phényl-2 2*H*-benzisosélénazole-1,2 one-3) (Fig. 1*a*) est une molécule organosélénée qui présente, outre des propriétés anti-inflammatoires marquées, une activité catalytique destructrice d'hydroperoxydes (Fisher & Dereu, 1987) similaire au glutathionperoxydase.

La synthèse du dérivé portant un groupement nitré en 7 (Lambert, Christiaens, Renson & Dereu, 1988),

susceptible de stabiliser par un effet de champ le sélénium cationoïde (Reich, Willis & Wollowitz, 1982; Kice & Chiou, 1986), a conduit avec bon rendement,

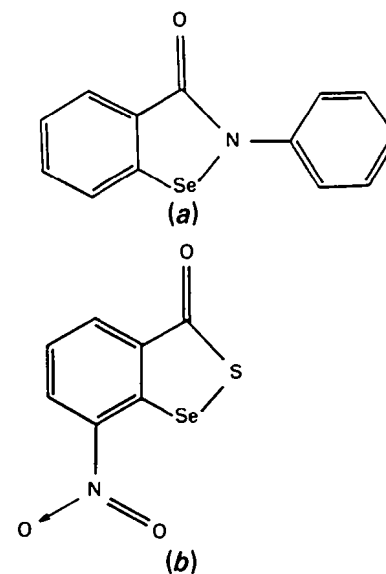


Fig. 1. (a) Formule de l'ebsele; (b) formule du composé étudié.