CROATICA CHEMICA ACTA CCACAA **56** (1) 87—95 (1983)

CCA-1357

YU ISSN 0011-1643
UDC 547.944.1
Original Scientific Paper

Crystal and Molecular Structure of Two (Phenylsulfonyl)-3-(hexahydroazepin-1-yl)-ureas: (A) 1-(4-Chlorophenylsulfonyl)-3-(hexahydro-1*H*-azepin-1-yl)-urea and (B) 1-(4-Methylphenylsulfonyl)-3-(hexahydro-1*H*-azepin-1-yl)-urea

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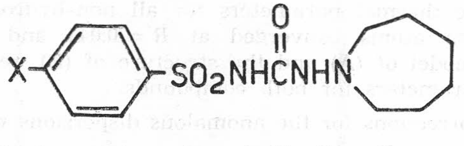
Received October 14, 1982

The crystal structure of the title compounds has been determined from diffractometer X-ray intensity data using direct methods and refined by the least-squares procedure to a conventional *R* index of 0.044 and 0.065 for (A) and (B), respectively. Crystals of both compounds are triclinic, space group $P\bar{1}$, with two molecules in the unit cells. Molecules (A) and (B) have a similar but not identical shape, the difference most probably being due to different hydrogen bonding between neighbouring molecules. The shape of individual molecules may be described in terms of three planes: hexahydroazepine rings, benzene rings and urea residues with the S atoms of the SO_2 groups. The seven-membered hexahydroazepine ring of (B) has a boat conformation while that of (A) is disordered with approximately half of the molecules in boat and half in chair form.

INTRODUCTION

The title compounds are known as substances of relatively small toxicity but with a high blood sugar decreasing effect.[1] Both were prepared by the reaction of the sodium salt of 4-chlorophenylsulfonylamide, or of the analogous 4-methylphenylsulfonamide, with 1-(hexahydro-1*H*-azepin-1-yl)-carbamoylbenzotriazole.[2] The structure of (A), which was solved first, showed one anomalously short carbon-carbon bond of 136 pm in the hexahydroazepine ring, indicating the possibility that the ring was not fully hydrogenated. Although the preparative procedure excludes such a possibility we decided to solve also the structure of the analogous compound (B). Since in (B) all distances within the seven-membered ring have the expected values it seems most probable that the apparent shortening of the particular carbon-carbon bond in the hexahydroazepine ring of (A) is due to



(A) X = Cl

(B) X = CH_3

disorder in the ring atom positions. A posteriori such a conclusion was supported also by C-13 NMR measurements.

EXPERIMENTAL

Both compounds were given to us by Professor I. Butula. They were recrystallized from ethylacetate.

Crystal Data

From rotation and Weissenberg photographs and from single crystal diffractometry:

(A) $C_{13}H_{18}O_3N_3SCl$, $M = 331.82$, triclinic, $a = 1.3423(6)$, $b = 0.9090(4)$, $c = 0.6475(3)$ nam, $\alpha = 85.98(4)$, $\beta = 86.14(4)$, $\gamma = 82.02(3)^{\circ}$, $V = 0.7783$ nm^3, $Z = 2$, $D_m = 1.40$ Mg · m^{-3} (by flotation), $D_c = 1.407$ Mg · m^{-3}, space group $P\bar{1}$ (No. 2) μ (Mo*Kα*) = 3.4 cm^{-1}, $\lambda = 71.07$ pm, specimen size $0.09 \times 0.24 \times 0.60$ mm^3.

(B) $C_{14}H_{21}O_3N_3S$, $M = 311.41$, triclinic, $a = 1.3528(3)$, $b = 0.9230(3)$, $c = 0.6360(6)$ nm, $\alpha = 85.80(3)$, $\beta = 87.21(3)$, $\gamma = 79.01(3)''$, $V = 0.7770$ nm^3, $Z = 2$, $D_m = 1.33$ Mg · m^{-3} (by flotation), $D_c = 1.331$ Mg · m^{-3}, space group $P\bar{1}$ (No. 2), μ (Mo*Kα*) = 1.8 cm^{-1}, $\lambda = 71.07$ pm, specimen size $0.27 \times 0.41 \times 0.69$ mm^3.

*Intensity Data, Structure Determinations and Refinement**

For each crystal, the intensity of every independent reflection up to $2\,\Theta = 60^{\circ}$ was measured on a fully automated Philips PW 1100 diffractometer using graphite-monochromated Mo*Kα* radiation ($\omega - 2\,\Theta$ scan technique, scan width 1.20° for (A) and 1.60° for (B), scan rate 0.04° s^{-1}). Reflections with $I > 3\,\sigma(I)$, where σ is the standard deviation based on simple counting statistics, were not included in the subsequent calculations, which were based on the remaining 2588 and 3228 independent reflections for (A) and (B), respectively. The intensities were corrected for Lorentz and polarization effects but not for absorption.

Both structures were solved using direct methods, MULTAN 78 and MULTAN 80 for (A) and (B), respectively. Refinement was performed using SHELX 76[3] by the least-squares procedure minimizing $\Sigma\, w\,(|F_o| - |F_c|)^2$ with $w = |\sigma^2(F_o) + 0.0354\,F_o^2|^{-1}$ for (B) while for (A) unit weight was assumed for all observations. The C(3), C(4) and C(5) atoms, but in particular C(4) atom in compound (A), showed larger thermal vibrations indicating the possibility of disorder in the seven-membered ring. Indeed, the positional disorder of the C(4) atom was proved by subsequent Fourier syntheses. The resulting F_o map was compatible with a mixture of the boat and chair form of the hexahydroazepine ring. The maximum belonging to the C(4) atom was clearly resolved into two [C(4a) and C(4b)] well separated single maxima 103 pm apart. In both structures hydrogen atoms were located either in the difference Fourier maps or generated at fixed positions. The refinement of (A) proceeded on the assumption that the crystal contains equal amount of each form with distinct sites for C(4a) and C(4b) and with 0.5 occupation for each position. The refinements with anisotropic thermal parameters for all non-hydrogen atoms and isotropic ones for hydrogen atoms converged at $R = 0.044$ and $R = 0.065$ ($R_w = 0.074$) for the disordered model of (A) and the structure of (B), respectively. Table I lists the final atomic parameters for both compounds.

The scattering factors and corrections for the anomalous dispersions were taken from ref. 4 and 5.

All calculations were carried out on the UNIVAC 1100 computer of the University Computing Centre. NMR spectra were recorded with a JEOL FX 100 spectrometer at 25.05 MHz.

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* The lists of observed and calculated structure factors are obtainable from the authors on request.

TABLE I

Atomic Coordinates (x 10^4, for H atoms x 10^3) and Equivalent Isotropic Thermal Parameters for the non-H atoms (x 10^4) with Estimated Standard Deviations in Parentheses.

$$B_{eq} = \frac{4}{3} \Sigma_i \Sigma_j b_{ij} a_i a_j$$

	Compound (A)				Compound (B)			
Atom	x/a	y/b	z/c	B_{eq} (pm^2)	x/a	y/b	z/c	B_{eq} (pm^2)
C(1)	2366(3)	1424(4)	1463(6)	4.35(10)	4016(2)	4091(3)	2502(5)	4.61(6)
C(2)	3105(3)	2364(5)	2171(8)	6.13(15)	3002(2)	3605(3)	2687(4)	4.18(6)
C(3)	3765(4)	1693(8)	3870(10)	8.57(23)	2480(2)	3719(4)	610(5)	5.53(8)
C(4)					3029(2)	3010(5)	—1245(5)	6.05(10)
C(4a)	3109(7)	1476(12)	5895(13)	5.43(28)				
C(4b)	3452(9)	465(12)	5385(22)	7.51(43)				
C(5)	2457(5)	458(9)	5954(8)	9.53(25)	3999(3)	1819(4)	—825(5)	5.52(9)
C(6)	1591(3)	393(4)	4751(6)	4.70(10)	4875(2)	2467(3)	—267(4)	4.81(6)
N(1)	1474(2)	1409(3)	2881(4)	3.13(7)	4897(1)	2994(2)	1852(3)	3.79(5)
N(2)	670(2)	1040(3)	1810(5)	4.19(8)	5090(1)	1789(2)	3369(4)	4.06(5)
C(7)	—167(2)	2001(3)	1556(5)	2.99(7)	5957(1)	1480(2)	4385(3)	3.30(4)
O(1)	—877(2)	1734(2)	612(4)	3.84(6)	6171(1)	414(2)	5669(3)	4.00(4)
N(3)	—165(2)	3318(3)	2519(4)	2.93(6)	6603(1)	2462(2)	3841(3)	3.85(4)
S	—1067(1)	4725(1)	2471(1)	2.88(2)	7739(1)	2280(1)	4791(1)	3.70(2)
O(2)	—663(2)	5881(2)	3409(4)	3.66(6)	8146(1)	3426(2)	3615(4)	5.24(5)
O(3)	—1429(2)	5007(3)	443(3)	3.90(6)	7655(2)	2268(2)	7026(3)	5.11(5)
C(8)	—2045(2)	4144(3)	4146(5)	2.87(7)	8390(1)	552(2)	4057(3)	3.27(4)
C(9)	—1851(2)	3699(4)	6196(5)	3.33(8)	8666(2)	344(3)	1960(4)	4.74(6)
C(10)	—2625(3)	3314(4)	7544(5)	3.84(9)	9216(2)	—1029(3)	1439(4)	4.91(7)
C(11)	—3577(3)	3393(4)	6819(6)	4.25(10)	9471(2)	—2169(3)	2917(4)	4.82(6)
C(12)	—3783(3)	3824(4)	4784(6)	4.46(11)	9193(2)	—1940(3)	5008(4)	4.78(6)
C(13)	—3001(2)	4205(4)	3428(6)	3.80(8)	8642(2)	—586(3)	5585(4)	4.48(6)
Cl	—4555(1)	2925(2)	8529(2)	7.13(4)				
C(14)					10051(2)	—3649(4)	2310(6)	6.17(9)
H(1a)	273	32	132		416	443	403	
H(1b)	212	190	—5		395	503	136	
H(2a)	267	334	278		312	247	330	
H(2b)	359	267	86		252	429	377	
H(3a)	446	121	306		227	488	15	
H(3b)	389	256	477		181	325	91	
H(3c)	412	57	343					
H(3d)	434	235	408					
H(4a)					251	247	—200	
H(4b)					324	387	—231	
H(4a1)	265	246	613					
H(4a2)	361	117	723					
H(4b1)	381	75	681					
H(4b2)	366	—31	471					
H(5a)	248	—57	701		420	123	—224	
H(5b)	228	139	677		384	105	45	
H(5c)	296	—55	561					
H(5d)	217	40	751					
H(6a)	163	—70	426		491	340	—138	
H(6b)	92	68	574		554	163	—46	
H(2)	76	19	123		471	115	360	
H(3)	40	341	314		641	316	282	
H(9)	—110	366	672		846	122	76	
H(10)	—249	295	914		945	—119	—19	
H(12)	—453	386	426		940	—282	620	
H(13)	—314	455	183		842	—42	722	
H(14a)					1018	—439	371	
H(14b)					1077	—353	157	
H(14c)					962	—410	122	

TABLE II

Interatomic Distances and Angles with Estimated Standard Deviations in Parentheses

Distances/pm	Compound (A)	Compound (B)
C(1)—C(2)	150.8(6)	151.9(4)
C(2)—C(3)	150.1(8)	151.6(4)
C(3)—C(4)		148.8(5)
C(3)—C(4a)	154.6(10)	
C(3)—C(4b)	151.8(14)	
C(4a)—C(4b)	103.4(15)	
C(4)—C(5)		156.3(5)
C(4a)—C(5)	135.5(14)	
C(4b)—C(5)	136.2(14)	
C(5)—C(6)	145.2(8)	149.3(5)
C(6)—N(1)	147.3(4)	146.9(3)
N(1)—N(2)	141.1(4)	141.2(3)
C(1)—N(1)	146.0(5)	147.3(3)
N(2)—C(7)	133.5(4)	133.9(2)
O(1)—C(7)	122.6(4)	123.1(2)
C(7)—N(3)	138.7(4)	139.0(2)
N(3)—S	163.4(3)	165.3(2)
S—O(2)	143.4(3)	143.1(2)
S—O(3)	142.5(2)	142.0(2)
S—C(8)	176.0(3)	175.2(2)
C(8)—C(9)	139.1(4)	138.4(3)
C(9)—C(10)	137.8(5)	139.5(4)
C(10)—C(11)	138.2(6)	136.3(4)
C(11)—Cl	173.9(4)	
C(11)—C(14)		150.7(4)
C(11)—C(12)	138.3(5)	138.4(4)
C(12)—C(13)	138.8(5)	139.2(4)
C(13)—C(8)	138.7(4)	138.2(3)
Distances/pm		
Hydrogen bonds		
N(2)—H(2) . . . O(1) (i)	303.0(4)	
N(2)—H(2) . . . O(1) (ii)		290.8(3)
N(3)—H(3) . . . O(2) (iii)	309.9(4)	
Symmetry code		
(i) = $-x, -y, -z$; (ii) = $1-x, -y, 1-z$; (iii) = $-x, 1-y, 1-z$		
Angles (°)		
C(1)—N(1)—C(6)	115.9(3)	115.9(2)
N(1)—C(1)—C(2)	112.5(3)	117.3(2)
C(1)—C(2)—C(3)	117.1(4)	113.8(2)
C(2)—C(3)—C(4)		119.6(2)
C(2)—C(3)—C(4a)	109.5(5)	
C(2)—C(3)—C(4b)	121.3(7)	
C(3)—C(4)—C(5)		117.6(3)
C(3)—C(4a)—C(5)	117.7(7)	
C(3)—C(4b)—C(5)	119.2(9)	
C(4)—C(5)—C(6)		113.1(3)
C(4a)—C(5)—C(6)	131.0(6)	
C(4b)—C(5)—C(6)	131.8(7)	
C(5)—C(6)—N(1)	117.3(4)	119.1(2)
C(6)—N(1)—N(2)	108.2(3)	110.5(2)
N(2)—N(1)—C(1)	109.4(3)	111.1(2)
N(1)—N(2)—C(7)	121.4(3)	121.0(2)
N(2)—C(7)—O(1)	123.5(3)	123.3(2)

TABLE II (continued)

	Compound (B)	Compound (A)
N(2)—C(7)—N(3)	113.2(3)	114.0(2)
O(1)—C(7)—N(3)	123.3(2)	122.8(2)
C(7)—N(3)—S	125.2(2)	123.4(1)
N(3)—S—O(2)	104.5(2)	102.8(1)
N(3)—S—O(3)	110.5(1)	109.2(1)
O(2)—S—O(3)	119.2(2)	119.8(2)
N(3)—S—C(8)	105.1(1)	105.4(1)
O(2)—S—C(8)	108.4(2)	109.5(1)
O(3)—S—C(8)	108.2(2)	109.1(1)
S—C(8)—C(9)	119.0(2)	119.9(2)
C(8)—C(9)—C(10)	119.4(3)	118.4(2)
C(9)—C(10)—C(11)	118.8(3)	122.2(2)
C(10)—C(11)—Cl	118.7(3)	
C(10)—C(11)—C(14)		121.2(3)
Cl—C(11)—C(12)	118.6(3)	
C(14)—C(11)—C(12)		120.2(2)
C(10)—C(11)—C(12)	122.6(4)	118.6(2)
C(11)—C(12)—C(13)	118.4(4)	120.7(2)
C(12)—C(13)—C(8)	119.4(3)	119.5(2)
C(13)—C(8)—C(9)	121.4(3)	120.5(2)
C(13)—C(8)—S	119.6(3)	119.7(2)

TABLE III

Torsion Angles/°

	Compound (A)	Compound (B)
N(1)—C(1)—C(2)—C(3)	78.3(5)	—83.6(3)
C(1)—C(2)—C(3)—C(4)		52.7(4)
C(1)—C(2)—C(3)—C(4a)	—66.1(7)	
C(1)—C(2)—C(3)—C(4b)	—24.1(9)	
C(2)—C(3)—C(4)—C(5)		16.7(5)
C(2)—C(3)—C(4a)—C(5)	67.1(9)	
C(2)—C(3)—C(4b)—C(5)	—33.3(14)	
C(3)—C(4)—C(5)—C(6)		—73.3(4)
C(3)—C(4a)—C(5)—C(6)	—62.9(11)	
C(3)—C(4b)—C(5)—C(6)	58.9(15)	
C(4)—C(5)—C(6)—N(1)		75.2(3)
C(4a)—C(5)—C(6)—N(1)	13.3(9)	
C(4b)—C(5)—C(6)—N(1)	—47.6(11)	
C(5)—C(6)—N(1)—C(1)	49.4(5)	—55.2(3)
C(5)—C(6)—N(1)—N(2)	172.7(4)	72.4(3)
C(6)—N(1)—C(1)—C(2)	—80.1(4)	63.1(3)
C(6)—N(1)—N(2)—C(7)	116.3(3)	112.0(2)
C(2)—C(1)—N(1)—N(2)	157.2(3)	—64.2(3)
C(1)—N(1)—N(2)—C(7)	—116.5(3)	—117.9(2)
N(1)—N(2)—C(7)—O(1)	178.9(3)	—177.6(2)
N(1)—N(2)—C(7)—N(3)	—2.8(4)	1.6(3)
N(2)—C(7)—N(3)—S	179.0(2)	—177.2(2)
O(1)—C(7)—N(3)—S	—2.7(4)	2.1(3)
C(7)—N(3)—S—O(2)	—172.0(2)	173.7(2)
C(7)—N(3)—S—O(3)	—42.5(3)	—58.1(2)
C(7)—N(3)—S—C(8)	74.0(3)	59.0(2)
N(3)—S—C(8)—C(9)	57.4(3)	70.1(2)
O(2)—S—C(8)—C(9)	—53.9(3)	—39.9(2)
O(3)—S—C(8)—C(9)	175.5(3)	—172.8(2)

DESCRIPTION OF THE STRUCTURES AND DISCUSSION

Interatomic distances and interbond angles with their standard deviations are given in Table II, the torsion angles in Table III. The atom numbering schemes and the structure of individual molecules of (A) and (B) are shown in Figures 1. and 2. Figures 3. and 4. show the packing and hydrogen bonding between adjacent molecules.

The bond lengths and angles in both compounds in general do not differ significantly from those previously observed in related compounds: the benzene rings are strictly planar, the SO_2 groups as well as the ureas have no remarkable features. In the structure of (B) the seven-membered ring has a boat conformation while the one in (A) clearly shows a disordered position of the C(4) atom indicating that statistically half of the molecules are in

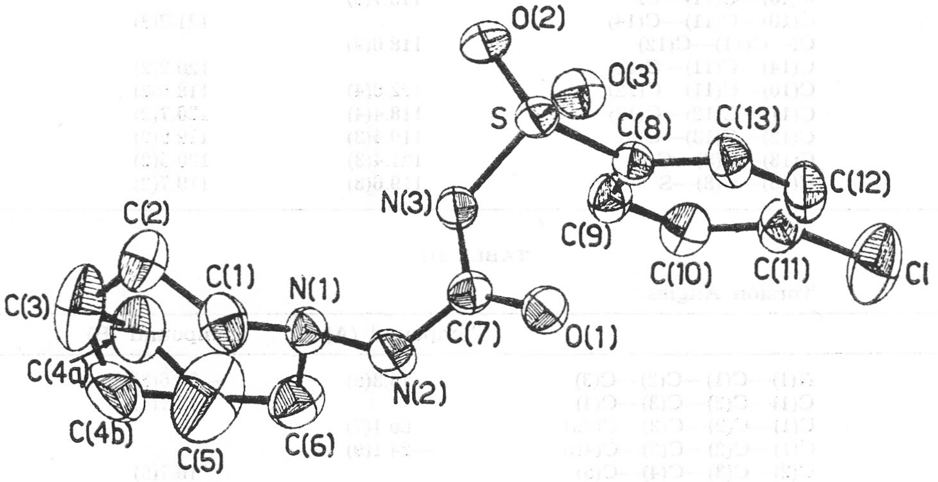

Figure 1. The molecule of 1-(4-chlorophenylsulfonyl)-3-hexahydro-1-*H*-azepin-1-yl)urea with atom numbering scheme. The hydrogen atoms are omitted for clarity.

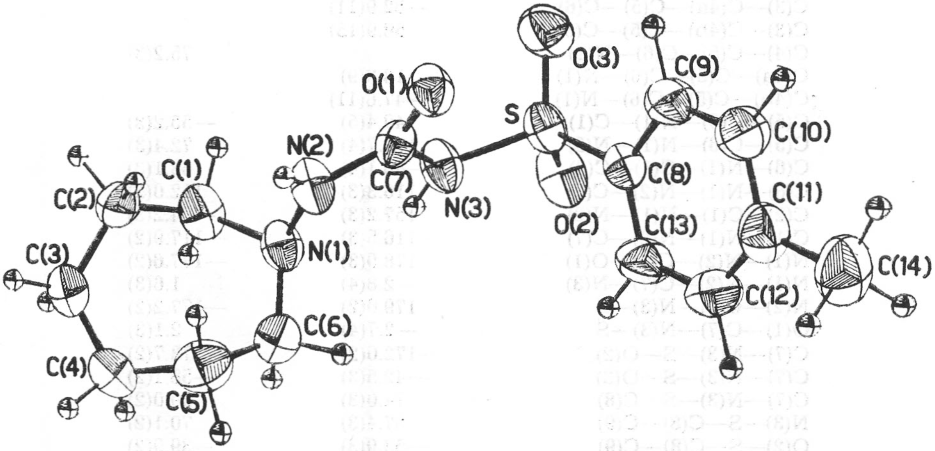

Figure 2. The molecule of 1-(4-methylphenylsulfonyl)-3-hexahydro-1-*H*-azepin-1-yl)urea with atom numbering scheme.

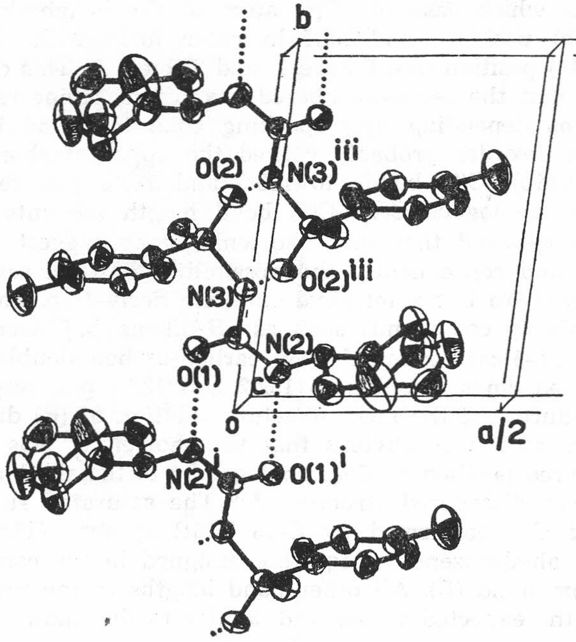

Figure 3. ORTEP drawing of the crystal structure of (A). The molecules form infinite chains linked by centrosymmetrically related N(2)—H(2) . . . O(1) and N(3)—H(3) . . . O(2) hydrogen bonds.

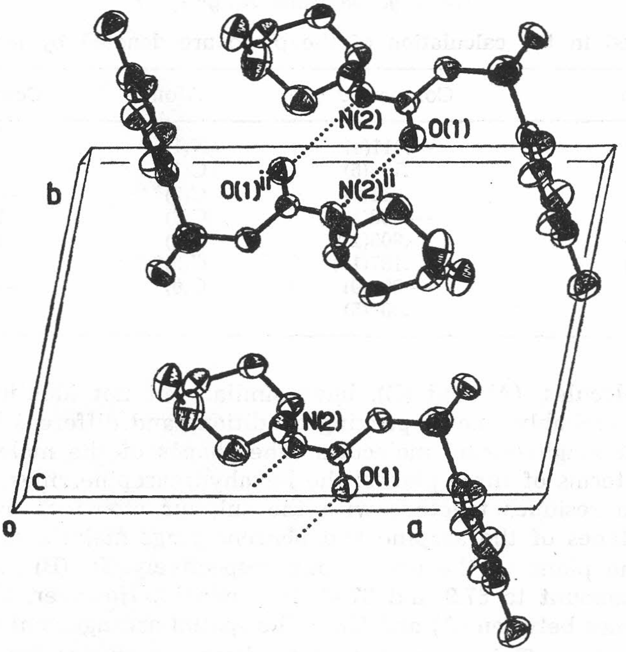

Figure 4. ORTEP drawing of the crystal structure of (B). The molecules are linked in pairs by centrosymmetrically related N(2)—H(2) . . . O(1) hydrogen bonds.

the boat form, in which case the C(4) atom of the hexahydroazepine ring occupies the C(4b) position, and half in chair form with the C(4) atom occupying the C(4a) position (see Figure 1. and Table IV). This conformational disorder suggests that the seven-membered hexahydroazepine ring may adopt both conformations depending upon packing conditions and intermolecular interactions. Such disorder probably caused the apparent shortening of the C(4a)—C(5) and C(4b)—C(5) bonds to 135.5 and 136.2 pm, respectively. In compound (B) the analogous C(4)—C(5) bond length amounts to 156.3 pm. Originally it was believed that such shortening may suggest a double carbon-carbon bond and consequently the possibility that the seven-membered ring is not a hexahydro but a tetrahydroazepine derivative. For example, in the azepine containing compounds such as 5*H*-dibenz | *b, f* | azepine[6] and 5*H*-dibenz | *b, f* | azepine-5-carboxamide[7,8] the carbon-carbon double bond within the seven-membered rings amounts to 132.2 and 133.1 pm, respectively. The preparative procedure and the later structure solution of (B) did not support such a conclusion, so it was obvious that the shortening was apparent and due to the disordered position of C(4), analogously to the similar bond shortening in some other disordered structures.[9,10] The saturated structure of the azepine ring was also confirmed by C-13 NMR spectra. (Three methylene groups of the hexahydroazepine ring were assigned in the compound (A) as well as in the compound (B). All other bond lengths in the seven-membered rings are within the expected values and similar to the known data (see e. g. ref. 11).

TABLE IV

Displacements of Atoms from the Least-squares Best Planes through the Hexahydroazepine Rings (pm)

Atoms included in the calculation of the plane are denoted by asterisks.

Atom	Compound (A)	Atom	Compound (B)
N(1)*	—.011(4)	N(1)	—.598(3)
C(1)	.754(5)	C(1)*	.045(4)
C(2)*	.011(7)	C(2)*	—.038(4)
C(3)*	—.008(10)	C(3)	1.024(5)
C(4a)	—.805(13)	C(4)	1.199(5)
C(4b)	.187(19)	C(5)*	.038(5)
C(5)	—.281(9)	C(6)*	—.046(4)
C(6)*	.008(5)		

Both molecules, (A) and (B), have similar but not identical shape. The difference is probably due to packing conditions and different hydrogen bonding between neighbouring molecules. The shapes of the molecules are best described in terms of three planes: the hexahydroazepine rings, benzene rings and the urea residues together with the sulphur atoms of the SO_2 groups. In (A) the planes of the azepine and benzene rings make angles of 83.6 and 84.4° with the plane of the urea group respectively. In (B) such angles are similar and amount to 87.9 and 87.4°, respectively. However, the most significant difference between (A) and (B) is the spatial arrangement of urea groups and benzene rings. This is most obvious from the values for the N(3)—S—C(8)—C(9) torsion angles, 57.4 and 70.1° for (A) and (B), respectively.

The molecules in the crystal structures are held together by hydrogen bonding and van der Waals interactions. The (A) and (B) compounds differ in hydrogen bondings. The molecules in (B) are linked together in pairs by hydrogen bonds of 290.8 pm realized between N(2) and O(1) atoms of centrosymmetrically related urea groups (Figure 4.). The molecules in (A), apart from being hydrogen bonded (303.0 pm) in the same way as those in (B), are also additionally linked through two centrosymmetrical hydrogen bonds of 309.9 pm between the urea nitrogen atom N(3) and the SO_2 oxygen atom O(2), realizing thus an infinite chain of molecules (Figure 3.).

Acknowledgement. — The authors thank Dr. S. Djokić, Director of the Research Institute of the Pharmaceutical and Chemical Works PLIVA, Zagreb, for suggesting the problem, Professor I. Butula from the Faculty of Pharmacy and Biochemistry, Zagreb, for supplying us with the compounds, Dr. Z. Veksli and Dr. Z. Meić from the Rudjer Bošković Institute, Zagreb, for NMR spectra, Mr. M. Bruvo, M. Sc., from this Laboratory, for collecting the diffractometer data, and the Foundation for Scientific Research of SR Croatia, Zagreb, for financial support.

REFERENCES

1. J. B. Wright, *German Patent* 1,186.865; C. A. 63 (1965) 14760.
2. I. Butula, V. Vela, and M. V. Proštenik, *Croat. Chem. Acta,* **52** (1979) 47.
3. G. M. Sheldrick, SHELX. A *Program for Crystal Structure Determination.* Univ. of Cambridge, England, 1976.
4. D. T. Cromer and J. B. Mann, *Acta Cryst.* **A24** (1968) 321.
5. D. T. Cromer and D. Liberman, *J. Chem. Phys.* **53** (1970) 1891.
6. J. P. Reboul, B. Cristau, J. C. Soyfer, and J. Estienne, *Acta Cryst.* **B36** (1980) 2683.
7. J. P. Reboul, B. Cristau, J. C. Soyfer, and J. P. Astier, *Acta Cryst.* **B37** (1981) 1844.
8. V. L. Himes, A. D. Mighell, and W. H. De Camp, *Acta Cryst.* **B37** (1981) 2242.
9. B. Kamenar, B. Korpar-Čolig, A. Hergold-Brundić, and Z. Popović, *Acta Cryst.* **B38** (1982) 1593.
10. B. Kamenar, B. Korpar-Čolig, and M. Penavić, To be published.
11. F. K. Winkler and J. D. Dunitz, *Acta Cryst.* **B31** (1975) 268.

SAŽETAK

Kristalna i molekulska struktura dviju (fenilsulfonil)-3-(heksahidroazepin-1-il)-urea: (A) 1-(4-klorfenilsulfonil)-3-(heksahidro-1*H*-azepin-1-il)-urea i (B) 1-(4-metilfenilsulfonil)-3-(heksahidro-1*H*-azepin-1-il)-urea

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Kristalna struktura spojeva u naslovu određena je na osnovi intenziteta refleksa prikupljenih na rendgenskom difraktometru i utočnjena metodom najmanjih kvadrata do faktora $R = 0.044$ za (A) i 0.065 za (B). Kristali oba spoja jesu triklinski, prostorne grupe $P\bar{1}$, i sadržavaju dvije molekule u elementarnim ćelijama. Molekule u spojevima (A) i (B) imaju sličan ali ne i identičan oblik, najvjerojatnije zbog različitih vodikovih veza između susjednih molekula. Oblik pojedinih molekula najbolje se može opisati s pomoću triju ravnina: heksahidroazepinski prstenovi, benzenski prstenovi i ravnine urea zajedno sa S atomima iz SO_2 skupina. Sedmeročlani heksahidroazepinski prsten u spoju (B) ima konformaciju kolijevke, dok je onaj u (A) neuređen s približno polovicom molekula u konformaciji kolijevke, a polovicom u konformaciji stolice.