C(4a)], the maximum torsion angle [37·4 (6)°] corresponding to the N(7)—N(12) bond. Finally, ring *A* has an envelope conformation [φ = 72 (11)°, θ = 50 (9)° and q = 0·03 (3) Å, origin at N(7)], C(9) being at the flap with a deviation of −0·043 (6) Å from the least-squares plane defined by the five remaining atoms. O(1), O(2) and Cl deviate 0·063 (5), −0·048 (4) and 0·031 (2) Å respectively from this plane. The least-squares planes for rings *A* and *B* form an angle of 13·6 (6)°.

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Structure of *N*-{2-[2-Methyl-3-(*p*-nitrobenzyl)oxy-1-isothioureido]ethyl}-*p*-toluenesulfonamide

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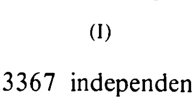
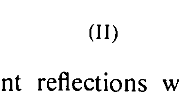
Abstract. $C_{18}H_{22}N_4O_5S_2$, M_r = 438·53, triclinic, $P\bar{1}$, a = 8·127 (2), b = 7·128 (2), c = 18·040 (3) Å, α = 86·50 (2), β = 97·13 (2), γ = 94·30 (2)°; V = 1032·6 Å^3; Z = 2, d_c = 1·43 Mg m^{-3}, $F(000)$ = 460; μ(Mo $K\alpha$) = 0·247 mm^{-1}. The structure was solved by direct methods and refined by full-matrix least squares to R = 0·043 for 2973 independent reflections. The molecule has an open-chain conformation with an intramolecular hydrogen bond of −NH···O type involving the amidine system and benzyloxy O atom. Steric effects of substituents at the C=N double bond, the conjugation in the amidine system and probably also the packing forces cause a curved conformation of the chain. A short van der Waals N···N contact [2·947 (6) Å] is found in the molecule.

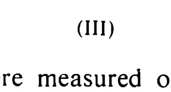
Introduction. The total compound was synthesized (Wolski, 1980) in the course of research on new analogs of sulfamides having a hypoglycemic activity. The analogs generally show a hypoglycemic activity lower than that of the commercially available drugs. The investigations of these analogs by chemical and other methods (IR, ^{1}H-NMR, ^{13}C-NMR, MS) gave no clear indication as to their molecular structure. Among the three possible structures, those with an open chain (II or III) were more likely; it was impossible, however, to exclude completely the cyclic form (I) with the chiral C atom bonded to four heteroatoms. Analysis by X-ray diffraction was undertaken to solve the problem adequately.



(I)



(II)



(III)

3367 independent reflections were measured on a four-circle automatic CAD-4 diffractometer (Jagiellonian University, SLAF & BS, Kraków) by the $\omega/2\theta$ technique up to $2\theta_{max}$ = 48° with graphite-monochromatized Mo $K\alpha$ radiation (λ = 0·71069 Å). 2973 of these having $I > 2\sigma_I$ were considered as observed and after Lp correction were used in the refinement process.

The structure was solved by direct methods using the *SHELX* system (Sheldrick, 1976). The atomic parameters were refined with isotropic and then anisotropic thermal parameters (XRAY system,

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Stewart, Kundell & Baldwin, 1970). The atomic scattering factors for non-H atoms were those of Cromer & Waber (1965) and for H those of Stewart, Davidson & Simpson (1965). H-atom positions obtained from the difference Fourier synthesis were refined for three cycles with B_{iso} held invariant (B_{iso} values one unit higher than B_{eq} of the adjacent atoms). The refinement of non-H parameters was then repeated. The refinement process was terminated when the average shift/error value was below 0·01. R was then 0·043 (unit weights). Use of statistical weights ($1/\sigma_F^2$) resulted in an increase in R of 0·014 and no improvement in molecular geometry. The positional parameters are listed in Table 1.*

Table 1. *Fractional coordinates* ($\times 10^4$, *for* H $\times 10^3$) *and isotropic thermal parameters with their e.s.d.'s in parentheses*

	x	y	z	B_{eq} (Å^2)*
C(1)	1629 (9)	2186 (12)	4699 (4)	5·8 (3)
C(2)	3234 (6)	2337 (7)	5206 (3)	4·1 (3)
C(3)	4738 (8)	2496 (9)	4920 (3)	4·9 (3)
C(4)	6219 (7)	2662 (8)	5383 (3)	4·6 (3)
C(5)	6198 (6)	2673 (7)	6147 (3)	3·5 (2)
C(6)	4709 (6)	2495 (8)	6449 (3)	4·0 (2)
C(7)	3245 (6)	2321 (8)	5974 (3)	4·2 (3)
S(8)	8076 (2)	2986 (2)	6745 (1)	3·81 (6)
O(9)	9374 (4)	2503 (6)	6342 (2)	5·3 (2)
O(10)	8177 (5)	4806 (5)	7050 (2)	5·1 (2)
N(11)	7967 (6)	1490 (7)	7443 (3)	4·1 (2)
C(12)	7782 (7)	−531 (8)	7319 (3)	4·3 (3)
C(13)	7209 (7)	−1632 (8)	7994 (3)	4·4 (3)
N(14)	5633 (5)	−1109 (6)	8197 (3)	4·3 (2)
C(15)	4139 (6)	−1464 (7)	7791 (3)	3·9 (2)
S(16)	4007 (2)	−3284 (2)	7162 (1)	4·91 (7)
C(17)	2001 (8)	−2978 (11)	6640 (4)	5·7 (3)
N(18)	2803 (5)	−646 (6)	7850 (3)	4·5 (2)
O(19)	3216 (4)	755 (5)	8398 (2)	5·1 (2)
C(20)	1911 (8)	1911 (10)	8298 (4)	5·3 (4)
C(21)	2144 (6)	3318 (7)	8928 (3)	4·3 (3)
C(22)	2942 (7)	2812 (8)	9627 (3)	4·7 (3)
C(23)	3118 (8)	4031 (9)	10201 (3)	5·0 (3)
C(24)	2453 (8)	5748 (8)	10060 (3)	4·8 (3)
C(25)	1663 (9)	6295 (9)	9375 (4)	5·8 (4)
C(26)	1513 (9)	5073 (9)	8808 (4)	5·5 (3)
N(27)	2655 (8)	7056 (8)	10675 (4)	6·5 (3)
O(28)	3272 (7)	6486 (8)	11295 (3)	7·9 (3)
O(29)	2179 (10)	8617 (8)	10541 (3)	9·4 (4)
H(11)	170 (8)	310 (9)	430 (4)	†
H(12)	134 (8)	100 (10)	452 (4)	
H(13)	79 (9)	230 (10)	493 (4)	
H(31)	477 (7)	263 (8)	438 (4)	
H(41)	721 (7)	293 (8)	517 (3)	
H(61)	476 (7)	234 (8)	701 (3)	
H(71)	227 (7)	204 (8)	619 (3)	
H(111)	751 (8)	177 (9)	771 (3)	
H(121)	689 (7)	−75 (8)	683 (3)	
H(122)	884 (7)	−77 (8)	722 (3)	
H(131)	799 (7)	−146 (8)	842 (3)	
H(132)	706 (7)	−317 (8)	795 (3)	
H(141)	561 (7)	−30 (8)	853 (3)	
H(171)	182 (8)	−392 (9)	627 (4)	
H(172)	194 (8)	−161 (9)	641 (4)	
H(173)	121 (8)	−340 (9)	702 (4)	
H(201)	80 (8)	104 (9)	839 (4)	
H(202)	184 (8)	276 (9)	779 (4)	
H(221)	343 (7)	147 (8)	974 (3)	
H(231)	378 (7)	375 (8)	1071 (4)	
H(251)	123 (8)	755 (9)	928 (4)	
H(261)	96 (8)	539 (9)	833 (4)	

* Calculated from anisotropic thermal parameters as $8\pi^2(U_1U_2U_3)^{1/3}$.

† Set constant (B_{eq} of adjacent atom + 1).

Discussion. Tables 2 and 3 give the calculated bond lengths and bond angles respectively. Fig. 1 shows an *ORTEP* diagram (Johnson, 1965) of the molecule with the crystallographic numbering of atoms. The molecule shows the open-chain structure (II). The bond lengths and bond angles in the aryl-sulfonamide group agree with the values typical for S^{VI} atoms in *N*-sulfonylsulfilimines (Kálmán, Párkányi & Kucsman, 1980). The sulfonic S atom has approximately tetrahedral coordination with an enlarged O—S—O angle, 119·1 (2)°, as expected. In the C(5)—S(8)—O and N(11)—S(8)—O pairs of angles, one angle is slightly and the other a few degrees below the tetrahedral value; *e.g.* C(5)—S(8)—O(10) = 109·3 (2) and C(5)—S(8)—O(9) = 106·9 (2)°. The shortening of all

Table 2. *Bond lengths* (Å) *with their e.s.d.'s in parentheses*

C(1)—C(2)	1·498 (9)	C(15)—S(16)	1·764 (5)
C(2)—C(3)	1·379 (8)	C(15)—N(18)	1·289 (7)
C(2)—C(7)	1·384 (8)	S(16)—C(17)	1·800 (7)
C(3)—C(4)	1·379 (8)	N(18)—O(19)	1·441 (6)
C(4)—C(5)	1·380 (8)	O(19)—C(20)	1·417 (8)
C(5)—C(6)	1·382 (7)	C(20)—C(21)	1·507 (9)
C(5)—S(8)	1·765 (5)	C(21)—C(22)	1·384 (8)
C(6)—C(7)	1·379 (7)	C(21)—C(26)	1·382 (9)
S(8)—O(9)	1·427 (4)	C(22)—C(23)	1·379 (9)
S(8)—O(10)	1·433 (4)	C(23)—C(24)	1·371 (9)
S(8)—N(11)	1·605 (5)	C(24)—C(25)	1·370 (9)
N(11)—C(12)	1·465 (7)	C(24)—N(27)	1·477 (9)
C(12)—C(13)	1·511 (8)	C(25)—C(26)	1·371 (10)
C(13)—N(14)	1·455 (8)	N(27)—O(28)	1·226 (8)
N(14)—C(15)	1·352 (6)	N(27)—O(29)	1·206 (9)
C(1)—H(11)	0·951 (67)	C(13)—H(132)	1·096 (58)
C(1)—H(12)	0·928 (68)	N(14)—H(141)	0·862 (61)
C(1)—H(13)	0·851 (75)	C(17)—H(171)	0·962 (68)
C(3)—H(31)	0·981 (64)	C(17)—H(172)	1·040 (66)
C(4)—H(41)	0·940 (61)	C(17)—H(173)	1·010 (69)
C(6)—H(61)	1·003 (59)	C(20)—H(201)	1·107 (61)
C(7)—H(71)	0·927 (59)	C(20)—H(202)	1·033 (64)
N(11)—H(111)	0·692 (67)	C(22)—H(221)	1·066 (58)
C(12)—H(121)	1·076 (55)	C(23)—H(231)	1·023 (59)
C(12)—H(122)	0·934 (62)	C(25)—H(251)	0·987 (67)
C(13)—H(131)	0·946 (56)	C(26)—H(261)	0·941 (64)

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* Lists of structure factors, anisotropic thermal parameters, bond angles involving H atoms and torsional angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36396 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Valence angles (°) with their e.s.d.'s in parentheses*

C(1)–C(2)–C(3)	120·9 (6)	N(14)–C(15)–S(16)	116·5 (4)
C(1)–C(2)–C(7)	120·9 (5)	N(14)–C(15)–N(18)	125·6 (5)
C(2)–C(3)–C(4)	121·2 (6)	C(15)–S(16)–C(17)	101·3 (3)
C(3)–C(2)–C(7)	118·2 (5)	S(16)–C(15)–N(18)	117·8 (4)
C(2)–C(7)–C(6)	121·7 (5)	C(15)–N(18)–O(19)	107·5 (4)
C(3)–C(4)–C(5)	119·5 (5)	N(18)–O(19)–C(20)	106·6 (4)
C(4)–C(5)–C(6)	120·5 (5)	O(19)–C(20)–C(21)	109·0 (5)
C(4)–C(5)–S(8)	120·1 (4)	C(20)–C(21)–C(22)	121·8 (5)
C(5)–C(6)–C(7)	118·9 (5)	C(20)–C(21)–C(26)	119·0 (5)
C(6)–C(5)–S(8)	119·3 (4)	C(21)–C(22)–C(23)	121·1 (5)
C(5)–S(8)–O(9)	106·9 (2)	C(22)–C(21)–C(26)	119·2 (5)
C(5)–S(8)–O(10)	109·3 (2)	C(21)–C(26)–C(25)	120·5 (6)
C(5)–S(8)–N(11)	107·0 (2)	C(22)–C(23)–C(24)	117·9 (5)
O(9)–S(8)–O(10)	119·1 (2)	C(23)–C(24)–C(25)	122·5 (6)
O(9)–S(8)–N(11)	108·1 (3)	C(23)–C(24)–N(27)	117·7 (5)
O(10)–S(8)–N(11)	105·8 (3)	C(24)–C(25)–C(26)	118·8 (6)
S(8)–N(11)–C(12)	119·9 (4)	C(25)–C(24)–N(27)	119·7 (5)
N(11)–C(12)–C(13)	110·7 (5)	C(24)–N(27)–O(28)	118·3 (6)
C(12)–C(13)–N(14)	114·5 (5)	C(24)–N(27)–O(29)	118·1 (6)
C(13)–N(14)–C(15)	124·7 (5)	O(28)–N(27)–O(29)	123·6 (7)

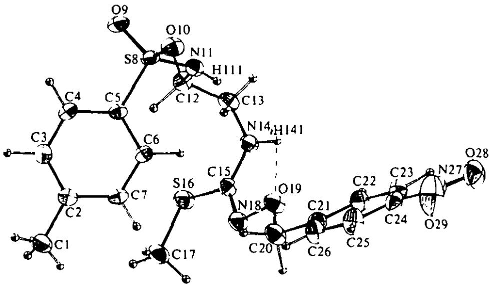

Fig. 1. *ORTEP* (Johnson, 1965) diagram of the molecule (crystallographic numbering of atoms). Thermal-motion ellipsoids are set at 40% probability level. H atoms are shown as spheres of arbitrary radius.

bond lengths around the S atom suggests that 3*d* orbitals are used in π-bond formation (Cruickshank, 1961). The average S—O bond length is 1·43 Å which corresponds to a π-bond order of about 0·67, according to the graph given by Cruickshank (1961). The S—N bond length of 1·605 (8) Å is comparable with the value 1·61 (1) Å for sulfamide and corresponds to a π-bond order of 0·25 (Cruickshank, 1961). The C(sp^2)—S bond length, 1·764 (5) Å, is typical for sulfonamides (Kálmán, Duffin & Kucsman, 1971). The sum of the valence angles around N(14), close to 360°, and the rather short N(14)—C(15) distance [1·352 (6) Å] suggest that there is some conjugation between the C=N bond and the lone electron pair on N(14). This situation is similar to the conjugation between the bonds in amidine systems; *e.g.* in formamidoxime (Hall, 1965) or in amidinopenicillanic acid (Krajewski, Urbańczyk-Lipkowska, Gluziński, Lebioda, Buśko-Oszczapowicz & Cieślak, 1981). The H(141) atom forms an intramolecular hydrogen bond with O(19). The five-membered chelate ring thus formed has an $A\cdots H$ distance of 2·12 (6) Å and a $D—H\cdots A$ angle of 108 (2)°. The C(15)—N(18)—O(19) angle [107·5 (4)°] is smaller than expected (120°) which may be a consequence of the hydrogen bonding. Fig. 2 presents another view of the molecule. The clearly visible curving of the chain may be caused by steric hindrances involving the chain atoms and/or by the hydrogen bonding. There is another short intramolecular van der Waals contact between N(11) and N(14) [2·947 (6) Å]. The existence of a hydrogen bond here must, however, be excluded owing to unsuitable diverting of the N(11)—H bond. There are relatively short intermolecular distances involving N(11) and N(14) atoms and O atoms in the nitro group of the neighboring molecule $(1-x, 1-y, 2-z)$ [O(28)$\cdots$N(11) = 3·078 (8), O(29)$\cdots$N(14) = 3·243 (8) Å].

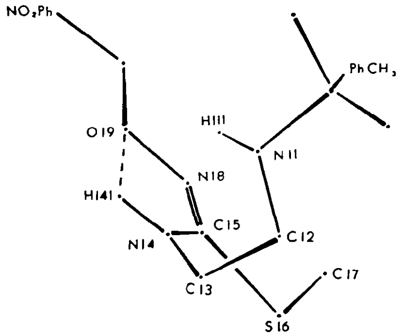

Fig. 2. Conformation of the chain part of the molecule (Cartesian coordinates, projection onto *XZ* plane).

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