$C_{13}H_{16}N_2O_3$

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2,9-Bis(phenylsulfonyl)-2,9-diaza[10](2,6)pyridinophane

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Abstract. $C_{25}H_{29}N_3O_4S_2$, $M_r = 499.7$, monoclinic, $P2_1/c$, $a = 8.168(3)$, $b = 21.196(5)$, $c = 14.969(4)$ Å, $\beta = 103.58(2)°$, $V = 2519.3(2)$ Å^3, $Z = 4$, $D_x = 1.317$ g cm^{-3}, $\lambda(Cu\,K\alpha) = 1.54178$ Å, $\mu = 20.96$ cm^{-1}, $F(000) = 1056$, room temperature, $R = 0.047$ for 2628 reflections with $I > 3\sigma(I)$. The thirteen-membered ring has one half in a boat and the other half in a chair conformation. One of the sulfonyl substituents is equatorial, the other is axial. The benzene and pyridine rings are planar.

Introduction. The title compound was obtained by condensation of N,N'-(1,6-cyclohexylene)bis(benzene-sulfonamide) with 2,6-bis(chloromethyl)pyridine in the presence of sodium butanolate at 403–413 K (Krakowiak, 1987). Pyridinophanes of this type have been the subject of interest because of their complex-forming properties with metals (Weber & Vögtle, 1976), their application as catalysts for ester aminolysis (Gandour, Walker, Nayak & Newkome, 1978) and as models for the pyridine nucleotide coenzymes (Dittner & Blidner, 1973). At present these compounds are under pharmacological investigation at the School of Medicine in Łódź.

Experimental. Colourless crystals from methanol, crystal size 0·2 × 0·2 × 0·4 mm, room temperature CAD-4 diffractometer using θ–2θ scan technique, unit cell from 25 reflections with $\theta < 16°$; 3144 independent reflections measured to $\theta = 70°$, 2628 non-zero reflec-

tions with $I > 3\sigma(I)$; data not corrected for absorption; graphite-monochromatized Cu $K\alpha$; maximum values of h,k,l 9, 23, 15, respectively; standard reflection 132, mean variation 0·27%; solution by direct methods using SHELX76 (Sheldrick, 1976); non-H atoms found on E map, positions of H atoms from difference map; refinement by full-matrix least squares (F magnitudes, 398 parameters) to final $R = 0.047$, unit weights; max. $\Delta/\sigma = 0.43$; largest peak on final difference map 0·34 e Å^{-3}. Scattering factors as included in SHELX76.

Discussion. The final positional parameters are listed in Table 1,* bond lengths and valence angles in Table 2 and torsion angles in Table 3. The atomic numbering scheme is given in Fig. 1, the molecular structure and packing in the unit cell in Fig. 2. The molecule is not a symmetrical one. However, the 13-atom heterocycle, as a whole, may be considered to have a distorted crown conformation which can be divided into two parts: the first part (N3, C9, C8, N1, C1, C2, C3) being in a boat-like conformation while the other (C4, C5, C6, N2, C7, C13, N3) is in a chair-like conformation. The positions of the sulfonyl substituents, with respect to the

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* 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 44255 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

thirteen-atom ring, are different. S1—N1 adopts the axial position while S2—N2 is equatorial. The benzene and pyridine rings are planar, the maximum deviation of the torsional angle being 2·8 (7)°. The molecules are linked by van der Waals forces only.

Table 1. *Final positional parameters* $(\times 10^4)$ *and isotropic temperature factors* $(\text{Å}^2 \times 10^4)$ *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33} + U_{13}\cos\beta).$$

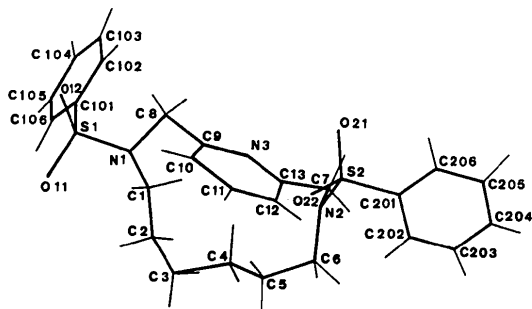|	x	y	z	U_{eq}
C101	8179 (5)	2140 (2)	5810 (3)	666 (28)
C102	8461 (6)	2223 (3)	6753 (3)	815 (36)
C103	8798 (7)	1699 (3)	7324 (3)	860 (39)
C104	8821 (7)	1117 (3)	6953 (4)	910 (43)
C105	8543 (7)	1029 (3)	6016 (4)	869 (39)
C106	8237 (6)	1548 (3)	5451 (3)	792 (35)
S1	7701 (2)	2797 (1)	5057 (1)	840 (9)
O11	8329 (5)	2640 (2)	4265 (2)	1176 (30)
O12	8286 (5)	3357 (2)	5578 (3)	1131 (30)
N1	5676 (5)	2847 (2)	4728 (2)	658 (23)
C1	4742 (7)	2359 (2)	4103 (3)	737 (32)
C2	3634 (6)	2641 (2)	3235 (3)	710 (32)
C3	4669 (7)	2881 (2)	2592 (4)	704 (33)
C4	3653 (8)	3271 (3)	1724 (4)	812 (38)
C5	3146 (6)	3910 (3)	2008 (3)	646 (30)
C6	4622 (6)	4360 (2)	2295 (3)	621 (28)
C7	4511 (7)	5088 (2)	3600 (3)	676 (30)
C8	4750 (7)	3166 (2)	5330 (3)	778 (35)
C9	3685 (6)	3705 (2)	4868 (3)	635 (28)
C10	2020 (7)	3766 (3)	4904 (3)	774 (35)
C11	1110 (8)	4283 (3)	4510 (4)	883 (40)
C12	1913 (7)	4731 (3)	4097 (3)	771 (36)
C13	3565 (6)	4633 (2)	4070 (3)	602 (27)
N3	4463 (4)	4137 (2)	4456 (2)	589 (21)
N2	4108 (4)	4974 (2)	2598 (2)	582 (21)
S2	4026 (1)	5565 (1)	1898 (1)	621 (7)
O21	3484 (4)	6108 (1)	2324 (2)	804 (22)
O22	3073 (4)	5359 (2)	1020 (2)	819 (22)
C201	6089 (5)	5718 (2)	1783 (3)	598 (26)
C202	6496 (7)	5645 (3)	949 (4)	868 (38)
C203	8091 (10)	5798 (3)	870 (5)	1169 (55)
C204	9276 (8)	5999 (3)	1617 (6)	1105 (54)
C205	8908 (8)	6057 (3)	2447 (5)	904 (45)
C206	7284 (6)	5923 (2)	2538 (3)	724 (32)

Table 2. *Bond lengths* (Å) *and valence angles* (°)

C101—C102	1·388 (6)	N2—S2	1·624 (3)
C102—C103	1·389 (7)	C7—C13	1·510 (6)
C103—C104	1·355 (8)	C8—C9	1·503 (6)
C104—C105	1·380 (7)	C9—C10	1·379 (6)
C105—C106	1·376 (7)	C10—C11	1·377 (7)
C106—C101	1·370 (6)	C11—C12	1·381 (7)
C101—S1	1·774 (5)	C12—C13	1·375 (6)
S1—O11	1·437 (3)	C13—N3	1·333 (5)
S1—O12	1·439 (4)	C9—N3	1·344 (5)
S1—N1	1·615 (4)	S2—O21	1·434 (3)
N1—C1	1·481 (5)	S2—O22	1·430 (3)
N1—C8	1·471 (6)	S2—C201	1·763 (4)
C1—C2	1·522 (6)	C201—C202	1·374 (6)
C2—C3	1·509 (7)	C202—C203	1·374 (8)
C3—C4	1·599 (6)	C203—C204	1·365 (9)
C4—C5	1·505 (7)	C204—C205	1·350 (8)
C5—C6	1·518 (6)	C205—C206	1·394 (7)
C6—N2	1·472 (5)	C206—C201	1·379 (6)
N2—C7	1·479 (5)		
C106—C101—C102	120·1 (5)	N3—C9—C8	116·4 (4)
C103—C102—C101	119·1 (5)	N3—C9—C10	122·1 (4)
C104—C103—C102	119·7 (5)	C11—C10—C9	119·7 (5)
C105—C104—C103	121·7 (5)	C12—C11—C10	118·2 (5)
C106—C105—C104	118·6 (6)	C13—C12—C11	118·8 (5)
C105—C106—C101	120·7 (5)	C12—C13—C7	122·1 (4)
S1—C101—C102	120·4 (4)	N3—C13—C7	114·6 (4)
S1—C101—C106	119·5 (3)	N3—C13—C12	123·4 (4)
O11—S1—C101	106·0 (2)	C13—N3—C9	117·7 (4)
O12—S1—C101	107·9 (2)	C7—N2—C6	116·4 (3)
O11—S1—O12	120·0 (2)	S2—N2—C6	117·1 (3)
N1—S1—C101	107·6 (2)	S2—N2—C7	119·6 (3)
N1—S1—O11	107·6 (2)	O21—S2—N2	107·5 (2)
N1—S1—O12	107·2 (2)	O22—S2—N2	106·7 (2)
C1—N1—S1	119·0 (3)	O22—S2—O21	119·7 (2)
C8—N1—S1	118·7 (3)	C201—S2—N2	108·0 (2)
C8—N1—C1	116·5 (4)	C201—S2—O21	107·4 (2)
C2—C1—N1	112·3 (4)	C201—S2—O22	107·1 (2)
C3—C2—C1	111·5 (4)	C202—C201—S2	120·6 (4)
C4—C3—C2	115·5 (4)	C206—C201—S2	119·1 (3)
C5—C4—C3	111·2 (4)	C206—C201—C202	120·3 (4)
C6—C5—C4	113·1 (4)	C203—C202—C201	119·2 (5)
N2—C6—C5	112·2 (4)	C204—C203—C202	120·5 (6)
N2—C7—C13	110·6 (3)	C205—C204—C203	121·0 (6)
C9—C8—N1	113·1 (4)	C206—C205—C204	119·6 (6)
C10—C9—C8	121·4 (4)	C205—C206—C201	119·4 (5)

Fig. 1. View of the molecular structure showing the atomic numbering scheme.
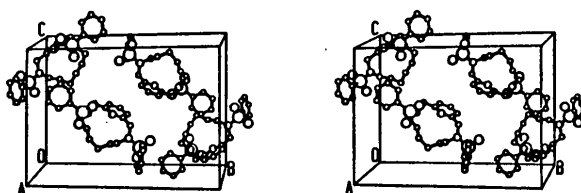
Fig. 2. Stereoview of the packing in the unit cell.

Table 3. *The torsion angles* (°) *of the thirteen-membered ring*

N1—C1—C2—C3	72·8 (5)	C13—N3—C9—C8	−177·5 (4)
C1—C2—C3—C4	−171·9 (4)	N3—C9—C8—N1	−51·8 (5)
C2—C3—C4—C5	71·7 (6)	C9—C8—N1—C1	−83·6 (5)
C3—C4—C5—C6	71·5 (6)	C8—N1—C1—C2	82·0 (5)
C4—C5—C6—N2	−178·1 (4)	S1—N1—C1—C2	−125·0 (4)
C5—C6—N2—C7	103·6 (4)	S1—N1—C8—C9	123·3 (4)
C6—N2—C7—C13	−64·2 (5)	S2—N2—C6—C5	−105·5 (4)
N2—C7—C13—N3	99·0 (4)	S2—N2—C7—C13	145·6 (3)
C7—C13—N3—C9	−178·5 (4)		

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