C1—N7—C8	126.6 (2)	127.40 (13)	-
N7—C8—N10$^-$	113.4 (2)	111.28 (13)	116.0 (4)
C8—N10$^-$—N7	120.7 (2)	115.15 (13)	115.1 (4)

(*b*)

	1-Carbamoyl-guanidine (neutral)	Bis(phenyl)-1-carbamoyl-guanidinium (cation)	1-Carbamoyl-guanidinium (cation)
C13—N19	-	1.409 (2)	-
N19—C20	1.358 (4)	1.344 (2)	1.326 (3)
C20—O21	1.251 (4)	1.216 (2)	1.224 (3)
C20—N22	1.355 (4)	1.402 (2)	1.392 (3)
N22—C23	1.348 (4)	1.349 (2)	1.358 (3)
C23—N24	1.324 (4)	1.311 (2)	1.314 (3)
C23—N25	1.338 (4)	1.325 (2)	1.319 (3)
N25—C26	-	1.432 (2)	-
O21···H24*A*	1.98 (4)	2.02 (2)	2.04 (3)
C13—N19—C20	-	128.72 (14)	-
N19—C20—N22	113.4 (3)	110.64 (13)	113.6 (2)
C20—N22—C23	119.8 (3)	126.91 (13)	125.9 (2)
N22—C23—N25	116.2 (3)	116.64 (13)	117.1 (2)
C23—N25—C26	-	125.78 (13)	-
O21—C20—N22	127.1 (3)	122.76 (14)	122.4 (2)
N22—C23—N24	126.3 (3)	120.96 (14)	121.0 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

Mean *d*(N—H) = 0.88 Å; the e.s.d. of an individual measurement is 0.02 Å, and this is also the e.s.d. of the sample of six values. Note that the e.s.d.'s given in Table 3 of YHCT96 did not properly take into account the hydrogen e.s.d.'s.

N—H···O/N—C	*d*(N···O/N)	*d*(H···ON)	Angle †	Angle ‡
Intramolecular				
N24—H24*A*···O21═C20§	2.712 (2)	2.02 (2)	134 (2)	98.3 (6)
Cation-to-anion				
N19—H19···N12≡C11	2.888 (2)	2.01 (2)	166 (2)	134.7 (5)
N22—H22···N12≡C11	2.943 (2)	2.16 (2)	157 (2)	103.6 (5)
N24—H24*B*···N12^{i}≡C11^{i}	2.979 (2)	2.20 (2)	147 (2)	114.6 (5)
N24-H24*A*···O21^{ii}═C20^{ii}§	3.037 (2)	2.38 (2)	134 (2)	152.2 (5)
N25—H25···O9═C8	2.813 (2)	1.96 (2)	163 (2)	144.9 (5)
Anion-to-anion				
N7—H7···N10^{iii}—C8^{iii}	3.063 (2)	2.26 (2)	177 (2)	134.9 (5)

† N—H···O/N. ‡ H···O/N—C. § Bifurcated hydrogen bond formed by N24—H24*A*.

Symmetry codes: (i) $1+x, y, z$; (ii) $1-x, -y, -z$; (iii) $-x, 1-y, 1-z$.

Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

The authors are grateful to Professor H. J. E. Loewenthal and Dr Mark Botoshansky for their help.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1255). Services for accessing these data are described at the back of the journal.

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trans-Methyl 2,9-Bis(phenylsulfonyl)-1,2,3,4-tetrahydro-4-β-carbolinylidene-acetate†

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Abstract

The title compound, $C_{26}H_{22}N_2O_6S_2$, consists of a β-carboline moiety, two phenylsulfonyl groups and one methoxycarbonylmethylene group. The six-membered heterocyclic ring adopts a half-chair conformation. The substituent at the indole N atom is equatorial while that at the second N atom is axial. The methoxycarbonylmethylene group is approximately coplanar to the β-carboline moiety. The dihedral angle between the two phenyl rings is 78.3 (1)°.

Comment

Derivatives of β-carbolines have been used as inverse agonist pharmacophores for the benzodiazepine receptor. While 3-substituted β-carbolines have been shown to interact at benzodiazepine receptors, a number of other related β-carbolines have proven to be effective non-competitive inhibitors of the indolamine 2,3-dioxygenase (IDO) enzyme system (Cox & Cook, 1995). The tetrahydro-β-carboline unit is central to an enormous range of indole alkaloids, and stereospecific syntheses of derivatives are widely sought (Everett *et al.*, 1990).

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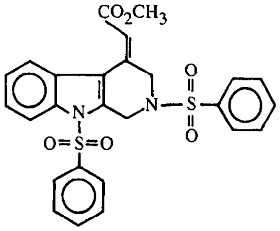
† DCB contribution No. 877.

Fig. 1 shows the displacement ellipsoid plot of the title molecule, (I), with the numbering scheme. The geometry at the S atoms (S1 and S2) is distorted from the ideal tetrahedral configuration. The C—N distances in the *C* ring [N2—C9 = 1.474 (5), N2—C10 = 1.463 (5) Å] are significantly higher than in the *B* ring [N1—C1 = 1.419 (5), N1—C11 = 1.404 Å] in agreement with the electron-withdrawing character of the phenylsulfonyl group. The S—N bond distances [S1—N1 = 1.670 (3), S2—N2 = 1.634 (4) Å] lie within the observed range of 1.63–1.69 Å (Kálmán, Czugler & Argay, 1981). The mean S—O, S—C and S—N distances of 1.430 (3), 1.759 (4) and 1.652 (4) Å, respectively, conform to those found in *N*-(4-nitrophenyl)-4-phenylsulfonamide (Gomes *et al.*, 1993). The dimensions of the three phenyl rings (*A*, *D* and *E*) are normal. The phenylsulfonyl group bonded to N1 is in an equatorial position, while the substituent at N2 is in an axial position.



(I)

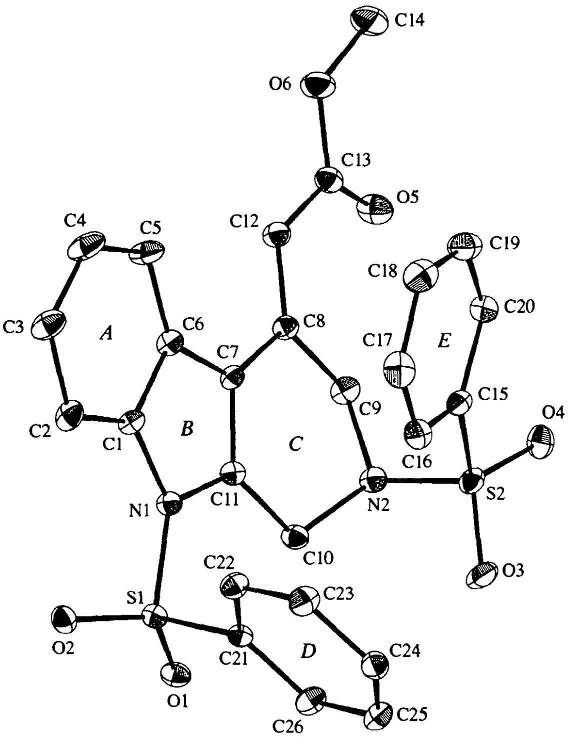


Fig. 1. 30% probability displacement ellipsoid plot of the molecule with the numbering scheme.

The rings *A*, *B*, *D* and *E* are planar, while *A* and *B* are also coplanar. The six-membered heterocyclic ring *C* is in a half-chair conformation. The dihedral angle between rings *D* and *E* is 78.3 (1)°. A large number of C—H···O-type interactions appear to contribute to crystal packing and stability.

Experimental

The compound was synthesized by a reductive condensation process (Mohanakrishnan, 1995) in the Department of Organic Chemistry, University of Madras, India. The crystals were grown from acetone at room temperature.

Crystal data

$C_{26}H_{22}N_2O_6S_2$
$M_r = 522.59$
Triclinic
$P\bar{1}$
$a = 10.956\ (1)$ Å
$b = 14.626\ (1)$ Å
$c = 7.606\ (1)$ Å
$\alpha = 93.66\ (1)°$
$\beta = 92.06\ (1)°$
$\gamma = 92.59\ (1)°$
$V = 1214.2\ (2)$ Å^3
$Z = 2$
$D_x = 1.429$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 15–25°$
$\mu = 0.265$ mm^{-1}
$T = 293\ (2)$ K
Transparent parallelepiped
$0.25 \times 0.2 \times 0.15$ mm
Pale yellow

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4260 measured reflections
4058 independent reflections
2771 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.0177$

$\theta_{\rm max} = 25.00°$
$h = 0 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -9 \rightarrow 8$
3 standard reflections every 100 reflections frequency: 60 min intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.166$
$S = 1.074$
4058 reflections
404 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 2.1063P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.024$
$\Delta\rho_{\rm max} = 0.366$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.416$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0039 (12)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—O2	1.426 (3)	N1—C11	1.404 (5)
S1—O1	1.430 (3)	N1—C1	1.419 (5)
S1—N1	1.670 (3)	N2—C10	1.463 (5)

S1—C21	1.758 (4)	N2—C9	1.474 (5)
S2—O4	1.433 (3)	O5—C13	1.193 (6)
S2—O3	1.432 (3)	O6—C13	1.346 (6)
S2—N2	1.634 (4)	O6—C14	1.444 (6)
S2—C15	1.761 (4)		
C11—N1—C1	107.9 (3)	C2—C1—N1	129.6 (4)
C11—N1—S1	125.2 (3)	N2—C9—C8	113.8 (3)
C1—N1—S1	126.2 (3)	N2—C10—C11	108.5 (3)
C10—N2—C9	113.7 (3)	C7—C11—N1	109.5 (3)
C10—N2—S2	119.5 (3)	N1—C11—C10	126.3 (3)
C9—N2—S2	117.4 (3)	O5—C13—O6	123.3 (4)
C13—O6—C14	116.0 (4)	O5—C13—C12	128.0 (4)
C6—C1—N1	107.7 (4)	O6—C13—C12	108.7 (4)
S1—N1—C1—C2	−6.1 (7)	S2—N2—C9—C8	−90.3 (4)

The H atoms of C14 were fixed by a riding model. All the other H atoms were refined isotropically.

Data collection: Rigaku *AFC-7R* diffractometer. Cell refinement: *MSC/AFC Diffraction Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*92 (Vickovic, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983, 1995).

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N,*N*′-Dicyclohexylurea†

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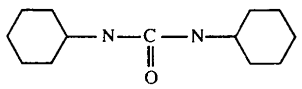
Abstract

The title molecule, $C_{13}H_{24}N_2O$, possesses twofold symmetry, with the C═O group lying on a crystallographic twofold axis in the unit cell. The cyclohexane rings adopt a chair conformation. Successive molecules along the twofold axis are related by the short *b* translation and interact through two identical diad-related N—H···O hydrogen bonds [2.962 (5) Å] in which the O atom lying on the twofold axis acts as a common acceptor for the diad-related amino protons.

Comment

An interesting aspect of the title structure, (I), is that the asymmetric unit consists of mainly the *N*-cyclohexyl group and half of the C═O moiety. The C═O group lies on a crystallographic twofold axis and thus the title molecule possesses a twofold symmetry relating the *N*-cyclohexyl groups. The bond lengths and angles observed in the cyclohexane ring, as well as their variations around the ring, are found to be within the observed range of values reported for related structures, such as *N*,*N*′-dicyclohexyl-*N*-4-phenylbutyrylurea (Ball, Brown & Bennet, 1990) and 3-cyclohexyl-1-[2-(1*H*-indol-3-yl)ethyl]urea (Ishida, Yamashita, Takai & Inoue, 1983).



(I)

An *ORTEP* diagram (Johnson, 1965; Vickovic, 1994) of the title molecule is shown in Fig. 1. For the cyclohexane ring, the average value of the bond angle, 112.8°, and that of the absolute torsion angle, 50.7°, as well as the distribution of the torsion angles around the ring, agree closely with the theoretically predicted values of 111.5 and 54.7° for chair conformations (Bixon & Lifson, 1967). As stated in the abstract, the molecules interact through two identical intermolecular N—H···O hydrogen bonds. This interaction gives rise to an infinite sheet-like structure, similar to the parallel

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† DCB contribution No. 878.