The H atoms were found in difference Fourier maps and refined with an overall isotropic displacement parameter that converged to U = 0.068 (2) and 0.054 (2) Å^2 for compounds (1) and (2), respectively.

For both compounds, data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structures: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ZORTEP* (Zsolnai, 1995).

This work has received partial support from FAPESP (Proc. 94/1213-5), CNPq (Proc. 300003/89-7), CAPES and FINEP.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1145). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 1770–1772

5-Benzylidene-8-ethylthiocarbamoyl-9-phenyl-7,8-diazabicyclo[4.3.0]non-6-ene

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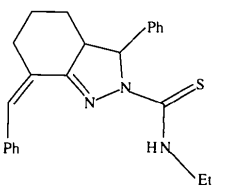
Abstract

The title compound, $C_{23}H_{25}N_3S$, was obtained by the reaction of dibenzylidenecyclohexanone with hydrazine hydrate, followed by the treatment of the resulting pyrazoline compound with ethyl isothiocyanate. The structure of the title compound was elucidated by IR, ^{1}H NMR spectroscopy and elementary analysis, and the stereochemical properties were investigated by X-ray analysis. The cyclohexane ring has a slightly distorted chair conformation while the 2-pyrazoline system is highly flattened from an ideal envelope conformation. One phenyl ring and thiocarbamoyl group are linked to the 2-pyrazoline system in axial and in equatorial positions, respectively. The other phenyl ring is bonded to the exocyclic double bond.

Comment

5-Benzylidine-8-ethylthiocarbomyl-9-phenyl-7,8-diazabicyclo[4.3.0]non-6-ene, (I), which consists of a pyrazoline structure condensed with a cyclohexane ring was shown to have potent antidepressant activity during our previous study (Bilgin, Yeşilada, Palaska & Sunal, 1992). These types of compounds may be formed as 1*H*,9*H*-*cis*,*trans* isomers or as a mixture in the reaction media (Hassner & Michelson, 1992; Lóránd *et al.*, 1985). On the other hand, the stereochemistry of biologically active compounds plays an important role as far as drug–receptor interactions are concerned (Foye, 1989).



(I)

In this study the molecular structure of the title compound, (I), was investigated in order to clarify the configuration of the isomeric form together with the conformation of the ring systems, which we think should aid our future research on the structure–activity relationship of such compounds. All bond lengths and bond angles are in fair agreement with the literature values (Ergin, Sillanpää & Ezer, 1993; Lóránd *et al.*, 1985). It is concluded from the torsion angles (Table 2) and also from the sums of bond angles at C(2) and C(5), 358.5 and 359.8°, respectively, that the aliphatic six-membered ring has a slightly distorted chair conformation. Taking into account the fact that the C(2)—C(3) bond is also part of the 2-pyrazoline system and that both C(5) and C(2) are involved in exocyclic double bonding, the deviation from an ideal chair conformation is surprisingly small. As the absolute values of the torsion angles of 2-pyrazoline system are 3.5 (2) and −12.6 (2)°, the ring is highly flattened from an ideal envelope conformation with C(3) as the 'flap' atom. This is also confirmed by the sum of bond angles (538.1°) in the five-membered ring. The torsion

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angles also show that the phenyl ring and thiocarbamoyl group are linked to the 2-pyrazoline system in axial and equatorial positions, respectively, and the other phenyl ring is equatorially bonded to the 2-pyrazoline system by means of the exocyclic C(5)=C(17) double bond (Fig. 1). The shortest intermolecular contacts are S(1)···H(24^{i}) = 3.048 (1), N(2)···H(19^{ii}) = 2.758 (2), N(3)···H(17^{iii}) = 2.827 (2), C(22)···H(8^{iv}) = 2.75 (3) Å [symmetry codes: (i) $x + 1, y + 1, z$; (ii) $x, y, z + 1$; (iii) $-x + 1, -y, -z$; (iv) $-x, -y, -z$].

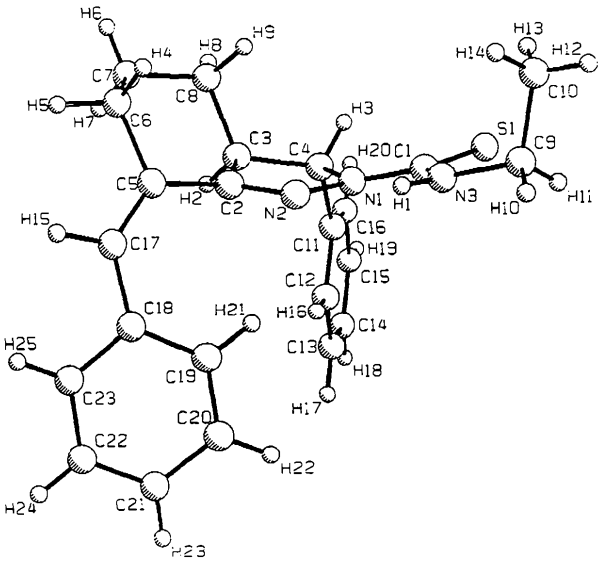

Fig. 1. A view of the molecule showing the labelling of the atoms.

Experimental

The title compound was obtained by the reaction of dibenzylidenecyclohexanone with hydrazine hydrate, followed by treatment of the resulting pyrazoline compound with ethyl isothiocyanate.

Crystal data

$C_{23}H_{25}N_3S$
M_r = 375.53
Triclinic
$P\bar{1}$
a = 10.492 (2) Å
b = 11.436 (2) Å
c = 9.334 (1) Å
α = 92.04 (1)°
β = 97.31 (2)°
γ = 110.68 (2)°
V = 1035.4 (5) Å^3
Z = 2
D_x = 1.204 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 15.15–18.85°
μ = 0.160 mm^{-1}
T = 294 K
Prism
0.360 × 0.320 × 0.240 mm
Colourless

Data collection

Rigaku AFC-5S diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3865 measured reflections
3647 independent reflections
2417 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.012
θ_{max} = 25°
$h = 0 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -11 \rightarrow 11$
3 standard reflections monitored every 150 reflections
intensity decay: −1.50%

Refinement

Refinement on F
R = 0.037
wR = 0.044
S = 1.46
2417 reflections
280 parameters
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max}$ = 0.01

$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
S(1)	0.65597 (7)	0.41236 (6)	0.11466 (7)	4.85 (3)
N(1)	0.4258 (2)	0.2435 (2)	0.1714 (2)	3.32 (6)
N(2)	0.3446 (2)	0.1674 (2)	0.2657 (2)	3.28 (7)
N(3)	0.6082 (2)	0.2894 (2)	0.3516 (2)	3.86 (8)
C(1)	0.5607 (2)	0.3117 (2)	0.2189 (2)	3.28 (8)
C(2)	0.2177 (2)	0.1397 (2)	0.2113 (2)	3.01 (8)
C(3)	0.1966 (2)	0.2055 (2)	0.0788 (2)	3.29 (8)
C(4)	0.3429 (2)	0.2609 (2)	0.0386 (2)	3.26 (8)
C(5)	0.0991 (2)	0.0747 (2)	0.2851 (2)	3.45 (8)
C(6)	0.0243 (3)	0.1627 (3)	0.3161 (3)	4.7 (1)
C(7)	−0.0010 (3)	0.2313 (3)	0.1839 (3)	5.3 (1)
C(8)	0.1312 (3)	0.2990 (3)	0.1229 (3)	4.6 (1)
C(9)	0.7469 (3)	0.3578 (3)	0.4279 (3)	4.8 (1)
C(10)	0.7593 (4)	0.4802 (3)	0.5006 (3)	7.4 (1)
C(11)	0.3597 (2)	0.1938 (2)	−0.0955 (2)	3.39 (8)
C(12)	0.3642 (3)	0.0745 (2)	−0.0946 (3)	4.9 (1)
C(13)	0.3677 (3)	0.0109 (3)	−0.2227 (4)	6.4 (1)
C(14)	0.3658 (3)	0.0663 (4)	−0.3510 (4)	7.2 (2)
C(15)	0.3627 (3)	0.1838 (4)	−0.3520 (3)	6.4 (1)
C(16)	0.3603 (2)	0.2483 (3)	−0.2252 (2)	4.6 (1)
C(17)	0.0527 (2)	−0.0462 (2)	0.3112 (2)	3.65 (9)
C(18)	0.0980 (2)	−0.1492 (2)	0.2730 (2)	3.35 (8)
C(19)	0.2283 (2)	−0.1353 (2)	0.2393 (3)	4.2 (1)
C(20)	0.2570 (2)	−0.2362 (2)	0.1891 (3)	4.6 (1)
C(21)	0.1578 (3)	−0.3554 (2)	0.1749 (3)	4.7 (1)
C(22)	0.0319 (3)	−0.3722 (2)	0.2152 (3)	4.9 (1)
C(23)	0.0017 (2)	−0.2714 (2)	0.2634 (3)	4.1 (1)

Table 2. *Selected torsion angles (°)*

Aliphatic six-membered ring	
C(2)—C(3)—C(8)—C(7)	−56.1 (3)
C(3)—C(8)—C(7)—C(6)	58.9 (3)
C(3)—C(2)—C(5)—C(6)	−51.5 (3)
C(2)—C(5)—C(6)—C(7)	47.1 (3)
C(5)—C(2)—C(3)—C(8)	55.8 (3)
C(5)—C(6)—C(7)—C(8)	−53.7 (3)
Thiocarbonyl group	
N(2)—N(1)—C(1)—N(3)	6.4 (3)
S(1)—C(1)—N(1)—N(2)	−174.5 (2)
N(1)—C(1)—N(3)—C(9)	−175.7 (2)
2-Pyrazoline system	
C(2)—N(2)—N(1)—C(4)	−3.5 (2)
N(2)—C(2)—C(3)—C(4)	12.5 (2)

N(2)—N(1)—C(4)—C(3)	10.6 (2)
N(1)—C(4)—C(3)—C(2)	−12.6 (2)
N(1)—N(2)—C(2)—C(3)	−6.0 (2)
Phenyl rings	
C(3)—C(4)—C(11)—C(12)	−72.0 (3)
C(3)—C(4)—C(11)—C(16)	103.1 (2)
C(5)—C(17)—C(18)—C(23)	−155.5 (2)
C(5)—C(17)—C(18)—C(19)	21.7 (4)

The structure was solved by direct methods (Gilmore, 1984) and successive Fourier syntheses. Refinement was performed by full-matrix least-squares methods. Non-H atoms were refined anisotropically. All H atoms were found from difference maps, atoms H(1)–H(11) and H(15) (total of 12 atoms) were refined with fixed isotropic displacement parameters (1.2 $\times B_{eq}$ of the parent atom), but the other H atoms were not refined.

Data collection: Rigaku *AFC-5S* software (Rigaku Corporation, 1988). Cell refinement: *TEXSAN* (Molecular Structure Corporation, 1989). Data reduction: *TEXSAN*. Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *WINDOWS* 3.1.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1081). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Benzoyl-3-(4-methoxyphenyl)thiourea

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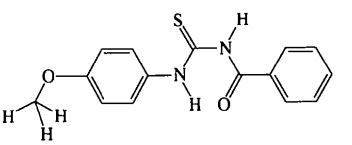
Abstract

In the title molecule, $C_{15}H_{14}N_2O_2S$, there is an intramolecular N—H···O hydrogen bond of 2.618 (4) Å between an amide N atom and the benzoyl O atom, which completes a nearly planar six-membered ring in the central part of the molecule. The benzene rings of the benzoyl and methoxyphenyl groups make dihedral angles of 28.8 (4) and 44.2 (4)°, respectively, with this plane. In the crystal, the molecules are packed in a centrosymmetric manner through weak N—H···S interactions.

Comment

During our systematic search for non-linear optical organic crystals having short cut-off wavelengths, we isolated the title compound, 1-benzoyl-3-(4-methoxyphenyl)thiourea, (I).



(I)

Since we have no access to the Cambridge Structural Database (Allen *et al.*, 1979), a search of Chemical Abstracts was carried out on compounds of type R–C_6H_4–CO–NH–CS–NH–C_6H_4–R'. One similar determination, with R = Cl and R' = H, was found (Simonov, Pobedimskaya, Martin & Masia, 1988), and there are no significant geometrical differences between that and the present determination. An intramolecular hydrogen bond [N1—H7···O1 2.618 (4) Å] completes an almost planar six-membered ring with the C1, N2 and C8 atoms; the maximum deviation from the best plane through the five non-H atoms is 0.014 (6) Å and the distance of the S atom from this plane is 0.0905 (2) Å. Weak intermolecular interactions were also found; N2—H13···S^{i} 3.507 (3), C7—H5···S^{i} 3.363 (3), C15—H1···O1^{ii} 3.387 (6) and C11—H12···O1^{iii} 3.424 (4) Å [symmetry codes: (i) $1-x$, $-y$, $1-z$; (ii) $1-x$, $1-y$, $-z$; (iii) $1-x$, $1-y$, $1-z$]. All these interactions play a role in the centrosymmetric packing. From the last

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