

## References

- Altomare, A., Cascarano, G., Giacobuzzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Fletcher, R. J., Tsai, C. & Hughes, R. E. (1971). *J. Phys. Chem.* **75**, 918–922.
- Görbitz, C. H. & Backe, P. (1996). In preparation.
- Görbitz, C. H. & Etter, M. C. (1992). *Int. J. Pept. Protein Res.* **39**, 93–110.
- Görbitz, C. H. & Gundersen, E. (1996a). *Acta Chem. Scand.* In the press.
- Görbitz, C. H. & Gundersen, E. (1996b). In preparation.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Kempster, C. J. E. & Lipson, H. (1972). *Acta Cryst.* **B28**, 3674.
- Mitra, S. N. & Subramanian, E. (1994). *Biopolymers*, **34**, 1139–1143.
- Pattabhi, V., Venkatesan, K. & Hall, S. R. (1974). *J. Chem. Soc. Perkin Trans 2*, pp. 1722–1727.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Stenkamp, R. E. & Jensen, L. H. (1975). *Acta Cryst.* **B31**, 857–861.
- Suresh, C. G. & Vijayan, M. (1985). *Int. J. Pept. Protein Res.* **26**, 311–328.

*Acta Cryst.* (1996). **C52**, 1767–1770

## Intermediates in the Synthesis of (+)-Grandisol. I

J. ZUKERMAN-SCHPECTOR<sup>a</sup> AND HUGO J. MONTEIRO<sup>b</sup>

<sup>a</sup>Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676, 13565-905 - São Carlos, SP, Brazil, and <sup>b</sup>Departamento de Química, Universidade de Brasília, 70910-900 - Brasília, DF, Brazil. E-mail: julio@ifqsc.sc.usp.br

(Received 31 January 1996; accepted 26 March 1996)

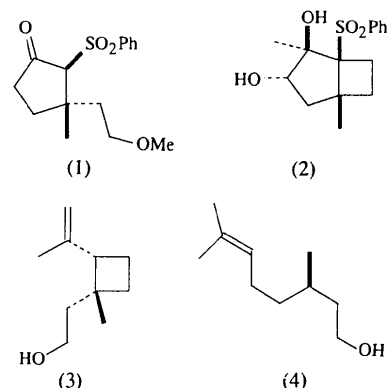
### Abstract

In both (2*R*,3*S*)-3-(2-methoxyethyl)-3-methyl-2-(phenylsulfonyl)cyclopentanone, C<sub>15</sub>H<sub>20</sub>O<sub>4</sub>S (1), and (1*S*,2*R*,3*S*,5*R*)-2,5-dimethyl-1-(phenylsulfonyl)bicyclo[3.2.0]heptane-2,3-diol, C<sub>15</sub>H<sub>20</sub>O<sub>4</sub>S (2), the five-membered ring is in an envelope conformation. In compound (1), the phenylsulfonyl and the methyl groups are *cis* to each other and the cyclization to the bicycloheptane, (2), occurs with retention of this stereochemistry. The hydroxyl groups in (2) are *trans* to each other.

### Comment

The terpene, (+)-*cis*-2-isopropenyl-1-methylcyclobutaneethanol (3), named (+)-grandisol (Tumlinson *et al.*, 1971) is the principal component in the aggregation

pheromone produced by the male of the cotton boll weevil, *Anthonomus grandis* Boheman (Franke *et al.*, 1989), which is a serious pest in Brazilian cotton fields. Its potential use in traps for monitoring crop infestation in integrated pest management makes this terpene, especially the more active (+)-enantiomer (Dickens & Mori, 1989), a target for the synthetic organic chemist. As the success of a synthetic route aiming at the synthesis of (+)-grandisol, starting with the easily available (+)-citronellol (4), depends on the generation of intermediates with the correct functionality and stereochemistry, the unambiguous stereostructure determination of them is required. We report here the crystal structure determination of two of them, (2*R*,3*S*)-3-(2-methoxyethyl)-3-methyl-2-(phenylsulfonyl)cyclopentanone, (1), and (1*S*,2*R*,3*S*,5*R*)-2,5-dimethyl-1-(phenylsulfonyl)bicyclo[3.2.0]heptan-2,3-diol, (2).



In both compounds, the S atom is tetrahedrally bonded to two C and two O atoms with tetrahedral angles ranging from 108.1(2) to 109.5(3)° in (1) and from 106.59(10) to 110.88(10)° in (2), with the exception of the O—S—O angle which is 117.9(3)° and 116.82(10)° in (1) and (2), respectively. In compound (1), the phenylsulfonyl and the methyl groups are *cis* to each other and cyclization to bicycloheptane, (2), occurs with retention of this configuration, the rings being, therefore, *cis*-fused. Cremer & Pople's (1975) puckering parameters show that, in both compounds, the five-membered ring is in an envelope conformation,  $q_2 = 0.390(6) \text{ \AA}$ ,  $\varphi_2 = 75.1(9)^\circ$  ( $E_{C3}$ ) for (1) and  $q_2 = 0.361(3) \text{ \AA}$ ,  $\varphi_2 = 65.3(5)^\circ$  ( $E_{C3}$ ) for (2). The four-membered ring in compound (2) is planar within experimental accuracy, making a dihedral angle of 106.1(1)° with the best least-squares plane through the five-membered ring. The phenyl ring makes a dihedral angle of 58.7(3)° with the cyclopentanone ring in (1) and dihedral angles of 117.7(1) and 15.2(2)° with the five- and four-membered rings, respectively, in (2).

The molecules in (1) are joined through a C—H...O interaction: C4...O3<sup>i</sup> = 3.334(8), HC4...O3<sup>i</sup> = 2.47(8) Å, C4—HC4...O3<sup>i</sup> = 142(5)° [symmetry op-

eration: (i) =  $-x, 0.5+y, -1.5-z$ ]. The molecules in (2) are joined through hydrogen bonds:  $O3 \cdots O1^{ii} = 2.931(2)$ ,  $HO3 \cdots O1^{ii} = 2.152(31) \text{ \AA}$ ,  $O3-HO3 \cdots O1^{ii} = 163(3)^\circ$ ;  $O4 \cdots O2^{iii} = 2.927(2)$ ,  $HO4 \cdots O2^{iii} = 2.122(32) \text{ \AA}$ ,  $O4-HO4 \cdots O2^{iii} = 172(3)^\circ$  [symmetry operations: (ii) =  $1-x, 2-y, 1-z$ ; (iii) =  $-1+x, y, z$ ].

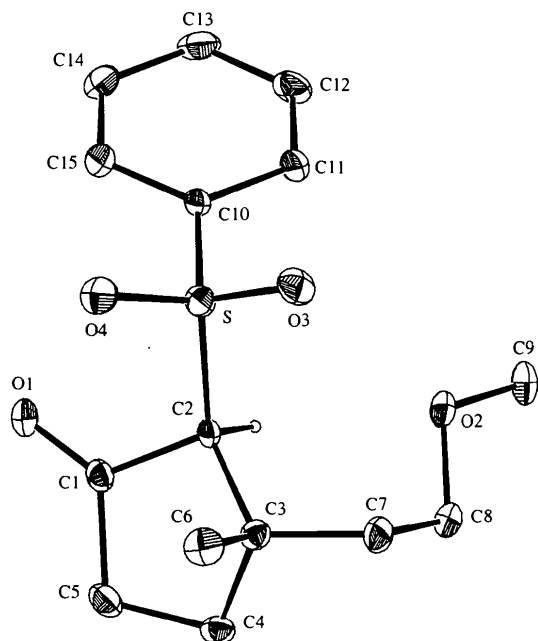


Fig. 1. The molecular structure of (1) showing the atom labelling. 50% probability displacement ellipsoids are shown.

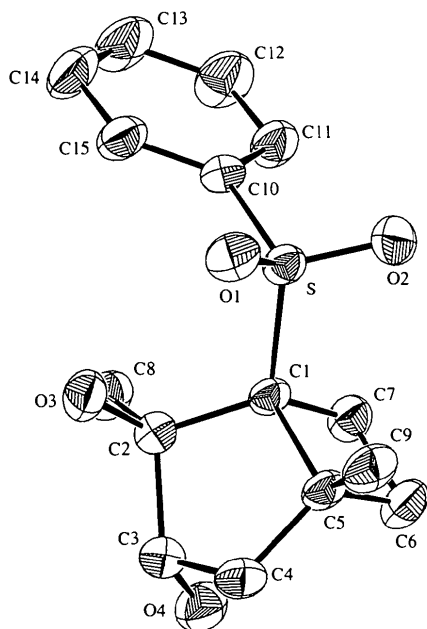


Fig. 2. The molecular structure of (2) showing the atom labelling. 50% probability displacement ellipsoids are shown.

## Experimental

Crystals of compound (1) were obtained by slow evaporation from diethyl ether at 269 K. Crystals of compound (2) were obtained by slow evaporation from a dichloromethane/diethyl ether mixture.

### Compound (1)

#### Crystal data

C<sub>15</sub>H<sub>20</sub>O<sub>4</sub>S  
*M<sub>r</sub>* = 296.381  
 Orthorhombic  
*P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 10.004 (1) Å  
*b* = 10.273 (1) Å  
*c* = 14.719 (1) Å  
*V* = 1512.69 (23) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.3014 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
*λ* = 0.71073 Å  
 Cell parameters from 25 reflections  
*θ* = 9.49–16.79°  
*μ* = 0.224 mm<sup>-1</sup>  
*T* = 293 K  
 Irregular  
 0.50 × 0.38 × 0.30 mm  
 Colourless

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
*ω*/*2θ* scans  
 Absorption correction: none  
 2237 measured reflections  
 2211 independent reflections  
 1486 observed reflections  
 [*I* > 2σ(*I*)]

*R*<sub>int</sub> = 0.0093  
*θ*<sub>max</sub> = 27.98°  
*h* = 0 → 13  
*k* = 0 → 13  
*l* = -1 → 19  
 2 standard reflections  
 frequency: 30 min  
 intensity decay: -1.1%

#### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.0681  
*wR*(*F*<sup>2</sup>) = 0.1613  
*S* = 1.110  
 2211 reflections  
 243 parameters  
 Only coordinates of H atoms refined and one common *U*  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.1120*P*)<sup>2</sup> + 0.4682*P*]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> = -0.041  
 Δρ<sub>max</sub> = 0.495 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.625 e Å<sup>-3</sup>

Extinction correction: *SHELXL93* (Sheldrick, 1993)  
 Extinction coefficient: 0.0125 (44)  
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)  
 Absolute configuration: Flack (1983)  
 Flack parameter = -0.08 (22)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>) for (1)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
S	-0.17381 (13)	-0.03800 (13)	-0.83449 (10)	0.0574 (4)
O1	-0.1200 (5)	0.2141 (5)	-0.9439 (3)	0.0811 (14)
O2	0.0309 (4)	0.0240 (5)	-0.6346 (3)	0.0700 (11)
O3	-0.1492 (5)	-0.1467 (4)	-0.7786 (4)	0.0802 (13)
O4	-0.2147 (5)	-0.0615 (5)	-0.9270 (3)	0.0840 (15)
C1	-0.0248 (6)	0.1659 (5)	-0.9091 (4)	0.0559 (12)
C2	-0.0277 (4)	0.0623 (5)	-0.8338 (3)	0.0457 (10)
C3	0.1096 (5)	-0.0065 (5)	-0.8414 (4)	0.0550 (12)

C4	0.1985 (6)	0.1091 (7)	-0.8668 (4)	0.0628 (15)
C5	0.1193 (7)	0.1920 (8)	-0.9312 (5)	0.073 (2)
C6	0.1114 (8)	-0.1054 (8)	-0.9179 (6)	0.078 (2)
C7	0.1578 (7)	-0.0745 (6)	-0.7538 (5)	0.069 (2)
C8	0.1609 (6)	0.0024 (7)	-0.6666 (5)	0.071 (2)
C9	0.0276 (10)	0.0670 (9)	-0.5439 (4)	0.081 (2)
C10	-0.2950 (5)	0.0597 (6)	-0.7811 (3)	0.0561 (13)
C11	-0.3028 (6)	0.0605 (8)	-0.6864 (4)	0.070 (2)
C12	-0.3981 (8)	0.1339 (10)	-0.6457 (5)	0.090 (3)
C13	-0.4843 (7)	0.2095 (9)	-0.6935 (6)	0.091 (2)
C14	-0.4762 (7)	0.2088 (8)	-0.7876 (6)	0.082 (2)
C15	-0.3822 (6)	0.1343 (7)	-0.8319 (5)	0.0681 (15)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (1)

S—O3	1.409 (5)	C3—C4	1.530 (8)
S—O4	1.442 (4)	C3—C7	1.544 (8)
S—C10	1.759 (6)	C4—C5	1.500 (10)
S—C2	1.788 (4)	C7—C8	1.507 (10)
O1—C1	1.189 (7)	C10—C15	1.381 (8)
O2—C8	1.400 (8)	C10—C11	1.397 (7)
O2—C9	1.407 (8)	C11—C12	1.355 (11)
C1—C5	1.502 (9)	C12—C13	1.357 (12)
C1—C2	1.537 (7)	C13—C14	1.387 (11)
C2—C3	1.548 (7)	C14—C15	1.377 (10)
C3—C6	1.516 (9)		
O3—S—O4	117.9 (3)	C6—C3—C2	111.7 (5)
O3—S—C10	108.2 (3)	C4—C3—C2	100.3 (4)
O4—S—C10	108.8 (3)	C7—C3—C2	115.0 (5)
O3—S—C2	108.1 (2)	C5—C4—C3	106.7 (5)
O4—S—C2	109.5 (3)	C4—C5—C1	105.7 (5)
C10—S—C2	103.4 (2)	C8—C7—C3	118.8 (5)
C8—O2—C9	113.0 (6)	O2—C8—C7	110.5 (5)
O1—C1—C5	127.0 (6)	C15—C10—C11	120.1 (6)
O1—C1—C2	125.7 (5)	C15—C10—S	120.7 (5)
C5—C1—C2	107.3 (5)	C11—C10—S	119.2 (5)
C1—C2—C3	104.3 (4)	C12—C11—C10	118.9 (7)
C1—C2—S	114.2 (3)	C11—C12—C13	122.4 (7)
C3—C2—S	117.5 (3)	C12—C13—C14	118.5 (7)
C6—C3—C4	109.4 (5)	C15—C14—C13	121.1 (7)
C6—C3—C7	108.2 (5)	C14—C15—C10	118.9 (7)
C4—C3—C7	111.9 (5)		

## Compound (2)

## Crystal data

C<sub>15</sub>H<sub>20</sub>O<sub>4</sub>S $M_r = 296.381$ 

Triclinic

 $P\bar{1}$  $a = 7.6400 (10) \text{\AA}$  $b = 9.8420 (10) \text{\AA}$  $c = 11.0590 (10) \text{\AA}$  $\alpha = 116.160 (10)^\circ$  $\beta = 100.760 (10)^\circ$  $\gamma = 90.260 (10)^\circ$  $V = 729.69 (16) \text{\AA}^3$  $Z = 2$  $D_x = 1.3489 \text{ Mg m}^{-3}$  $D_m$  not measured

## Data collection

Enraf-Nonius CAD-4

diffractometer

 $\omega/2\theta$  scans

Absorption correction:

none

2770 measured reflections

2563 independent reflections

2119 observed reflections

 $[I > 2\sigma(I)]$ Mo  $K\alpha$  radiation $\lambda = 0.71073 \text{\AA}$ 

Cell parameters from 25

reflections

 $\theta = 9.01\text{--}19.00^\circ$  $\mu = 0.232 \text{ mm}^{-1}$  $T = 293 \text{ K}$ 

Irregular

 $0.40 \times 0.30 \times 0.20 \text{ mm}$ 

Colourless

 $R_{\text{int}} = 0.0116$  $\theta_{\text{max}} = 24.97^\circ$  $h = 0 \rightarrow 9$  $k = -11 \rightarrow 11$  $l = -13 \rightarrow 12$ 

2 standard reflections

frequency: 30 min

intensity decay:  $-0.9\%$ 

## Refinement

Refinement on  $F^2$  $R(F) = 0.0448$  $wR(F^2) = 0.1152$  $S = 1.063$ 

2563 reflections

243 parameters

Only coordinates of H atoms

refined and one common

 $U$  $w = 1/[\sigma^2(F_o^2) + (0.0823P)^2 + 0.2415P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} < 0.001$  $\Delta\rho_{\text{max}} = 0.363 \text{ e \AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.744 \text{ e \AA}^{-3}$ 

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.0263 (55)

Atomic scattering factors

from *International Tables*for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (2)

	x	y	z	$U_{\text{eq}}$
S	0.46777 (7)	0.78014 (6)	0.23036 (5)	0.0306 (2)
O1	0.5526 (2)	0.9171 (2)	0.3475 (2)	0.0418 (4)
O2	0.5791 (2)	0.6583 (2)	0.1764 (2)	0.0402 (4)
O3	0.1949 (2)	0.9623 (2)	0.3859 (2)	0.0395 (4)
O4	-0.0754 (2)	0.6178 (2)	0.3171 (2)	0.0472 (5)
C1	0.2831 (3)	0.7026 (2)	0.2675 (2)	0.0281 (5)
C2	0.1287 (3)	0.8065 (2)	0.3053 (2)	0.0316 (5)
C3	0.0453 (3)	0.7518 (3)	0.3960 (2)	0.0383 (5)
C4	0.2039 (3)	0.7153 (3)	0.4781 (2)	0.0410 (6)
C5	0.3394 (3)	0.6489 (2)	0.3853 (2)	0.0333 (5)
C6	0.2876 (4)	0.4832 (3)	0.2744 (3)	0.0417 (6)
C7	0.2245 (3)	0.5332 (3)	0.1619 (2)	0.0345 (5)
C8	-0.0092 (3)	0.8020 (3)	0.1853 (3)	0.0412 (6)
C9	0.5289 (4)	0.6826 (3)	0.4704 (3)	0.0445 (6)
C10	0.3764 (3)	0.8250 (3)	0.0952 (2)	0.0330 (5)
C11	0.3331 (4)	0.7104 (3)	-0.0379 (2)	0.0431 (6)
C12	0.2519 (4)	0.7439 (3)	-0.1427 (3)	0.0566 (7)
C13	0.2185 (5)	0.8916 (3)	-0.1134 (3)	0.0588 (8)
C14	0.2640 (4)	1.0055 (3)	0.0183 (3)	0.0537 (7)
C15	0.3438 (4)	0.9741 (3)	0.1254 (3)	0.0424 (6)

Table 4. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (2)

S—O1	1.433 (2)	C3—C4	1.517 (3)
S—O2	1.444 (2)	C4—C5	1.538 (3)
S—C10	1.764 (2)	C5—C9	1.518 (3)
S—C1	1.795 (2)	C5—C6	1.539 (3)
O3—C2	1.422 (3)	C6—C7	1.530 (3)
O4—C3	1.423 (3)	C10—C11	1.378 (3)
C1—C7	1.558 (3)	C10—C15	1.389 (3)
C1—C2	1.558 (3)	C11—C12	1.381 (4)
C1—C5	1.600 (3)	C12—C13	1.379 (4)
C2—C8	1.516 (3)	C13—C14	1.366 (4)
C2—C3	1.553 (3)	C14—C15	1.387 (4)
O1—S—O2	116.82 (10)	O4—C3—C2	112.9 (2)
O1—S—C10	108.57 (10)	C4—C3—C2	104.5 (2)
O2—S—C10	107.17 (10)	C3—C4—C5	107.9 (2)
O1—S—C1	110.88 (10)	C9—C5—C4	110.9 (2)
O2—S—C1	106.59 (10)	C9—C5—C6	115.3 (2)
C10—S—C1	106.28 (10)	C4—C5—C6	114.8 (2)
C7—C1—C2	115.9 (2)	C9—C5—C1	121.7 (2)
C7—C1—C5	88.7 (2)	C4—C5—C1	103.6 (2)
C2—C1—C5	107.4 (2)	C6—C5—C1	88.7 (2)
C7—C1—S	112.00 (14)	C7—C6—C5	91.9 (2)
C2—C1—S	115.72 (14)	C6—C7—C1	90.6 (2)
C5—C1—S	114.11 (14)	C11—C10—C15	121.2 (2)
O3—C2—C8	105.1 (2)	C11—C10—S	119.3 (2)
O3—C2—C3	108.6 (2)	C15—C10—S	119.4 (2)
C8—C2—C3	112.3 (2)	C10—C11—C12	119.3 (2)
O3—C2—C1	111.8 (2)	C13—C12—C11	119.7 (3)
C8—C2—C1	116.2 (2)	C14—C13—C12	120.9 (3)
C3—C2—C1	102.9 (2)	C13—C14—C15	120.4 (3)
O4—C3—C4	108.5 (2)	C14—C15—C10	118.4 (2)

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<sup>2</sup> 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.

## References

- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.  
 Dickens, J. C. & Mori, K. (1989). *J. Chem. Ecol.* **15**, 517–528.  
 Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Franke, W., Bartels, J., Krohn, S., Schultz, S., Baader, E., Tengo, J. & Schneider, D. (1989). *Pure Appl. Chem.* **61**, 539–542.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Tumlinson, J. H., Gueldner, R. C., Hardee, D. D., Thompson, A. C., Hedin, P. A. & Minyard, J. P. (1971). *J. Org. Chem.* **36**, 2616–2621.  
 Zsolnai, L. (1995). *ZORTEP. An Interactive Molecular Graphics Program*. University of Heidelberg, Germany.

*Acta Cryst.* (1996). **C52**, 1770–1772

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

ÖMER ERGİN,<sup>a</sup> REIJO SILLANPÄÄ<sup>b</sup> AND AKGÜL YEŞİLADA<sup>c</sup>

<sup>a</sup>Balıkesir Üniversitesi, Necatibey Eğitim Fakültesi Fizik Eğitimi Bölümü, Balıkesir, Turkey, <sup>b</sup>Department of Chemistry, University of Turku, SF-20500 Turku, Finland, and <sup>c</sup>Hacettepe Üniversitesi, Eczacılık Fakültesi, Farmasotik Kimya Bölümü, Ankara, Turkey

(Received 21 July 1995; accepted 20 November 1995)

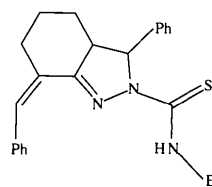
### Abstract

The title compound, C<sub>23</sub>H<sub>25</sub>N<sub>3</sub>S, 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, <sup>1</sup>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 thiocarbonyl 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-Benzylidene-8-ethylthiocarbonyl-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