

- Karthe, P., Sadasivan, C. & Gautham, N. (1993). *Acta Cryst.* **B49**, 1069–1071.
- Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1994). *Structure Correlation*, Vol. 2, edited by H.-B. Bürgi & J. D. Dunitz, Appendix A. Weinheim, Germany: VCH Publishers.
- Sheldrick, G. M. (1997a). *SHELXS97. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Spek, A. L. (1997a). *PLATON. Molecular Geometry Program*. Version of May 1997. University of Utrecht, The Netherlands.
- Spek, A. L. (1997b). *PLUTON. Molecular Graphics Program*. Version of May 1997. University of Utrecht, The Netherlands.

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## Conformational Variations in Vinylsulfoximines

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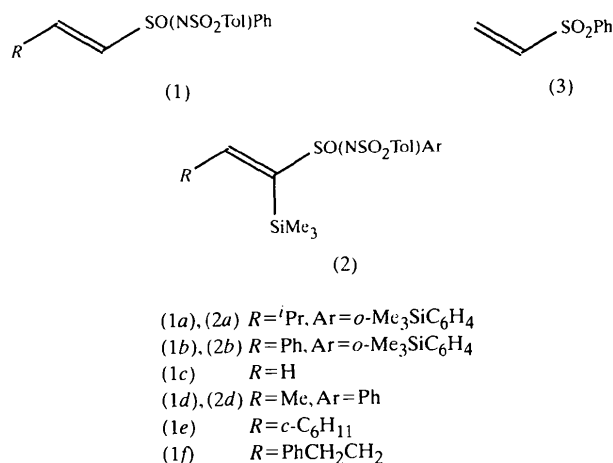
### Abstract

In a series of vinylsulfoximines {*S*-ethenyl-*S*-phenyl-*N*-(*p*-tolylsulfonyl)sulfoximide [(1*c*); C<sub>15</sub>H<sub>15</sub>NO<sub>3</sub>S<sub>2</sub>], (*E*)-*S*-phenyl-*S*-(prop-1-enyl)-*N*-(*p*-tolylsulfonyl)sulfoximide [(1*d*); C<sub>16</sub>H<sub>17</sub>NO<sub>3</sub>S<sub>2</sub>], (*E*)-*S*-(2-cyclohexylethenyl)-*S*-phenyl-*N*-(*p*-tolylsulfonyl)sulfoximide [(1*e*); C<sub>21</sub>H<sub>25</sub>NO<sub>3</sub>S<sub>2</sub>], (*E*)-*S*-phenyl-*S*-(4-phenylbut-1-enyl)-*N*-(*p*-tolylsulfonyl)sulfoximide [(1*f*); C<sub>23</sub>H<sub>23</sub>NO<sub>3</sub>S<sub>2</sub>], (*E*)-*S*-(3-methyl-1-trimethylsilylbut-1-enyl)-*N*-(*p*-tolylsulfonyl)-*S*-(2-trimethylsilylphenyl)sulfoximide [(2*a*); C<sub>24</sub>H<sub>37</sub>NO<sub>3</sub>S<sub>2</sub>-Si<sub>2</sub>], (*E*)-*S*-(2-phenyl-1-trimethylsilylethenyl)-*N*-(*p*-tolylsulfonyl)-*S*-(2-trimethylsilylphenyl)sulfoximide [(2*b*); C<sub>27</sub>H<sub>35</sub>NO<sub>3</sub>S<sub>2</sub>Si<sub>2</sub>], (*E*)-*S*-phenyl-*N*-(*p*-tolylsulfonyl)-*S*-(1-trimethylsilylprop-1-enyl)sulfoximide [(2*d*); C<sub>19</sub>H<sub>25</sub>NO<sub>3</sub>-S<sub>2</sub>Si]} and in phenyl vinyl sulfone [(3); C<sub>8</sub>H<sub>8</sub>O<sub>2</sub>S], the vinyl double bond is found to be approximately *syn*-coplanar with either the S=O or the S=N bond, the preference between these two being correlated in most cases with the size of the terminal vinyl substituent. Addition of trimethylsilyl groups at the  $\alpha$  position of the vinyl group, and in the *ortho* position of the phenyl substituent on S, introduces further steric constraints, so that the substituted phenyl group becomes *syn*-coplanar with the vinyl double bond. The observed

conformations may also be preferred in solution, as they are consistent with the observed diastereoselectivity of addition reactions of vinylsulfoximines.

### Comment

The sulfoximine group has been used as a chiral equivalent of the sulfone group in order to control the stereochemical outcome of reactions (Pyne, 1992, and references therein). Specifically, the role of the sulfoximine group in controlling the diastereoselectivity of addition reactions to vinylsulfoximines has been explored, since these compounds are chiral equivalents of the synthetically useful vinyl sulfones. The outcome of conjugate addition reactions (*e.g.* Pyne, 1986*a,b*; Bailey *et al.*, 1993; Jackson *et al.*, 1996) and cycloaddition reactions (Glass *et al.*, 1984; David *et al.*, 1995) of vinyl sulfones has been studied. While the stereochemical outcome of these reactions clearly depends on, amongst other factors, the conformational preferences of the vinylsulfoximine group in solution, studies of the solid-state conformations can yield useful data. Thus, we have already reported on the structure of the vinylsulfoximine (1*a*) (Bailey *et al.*, 1993), in which the C=C and S=N bonds are approximately *syn*-coplanar. This is in contrast to the analogous vinylsulfoximine (1*b*) (Dang *et al.*, 1993), in which the C=C and S=O bonds are essentially coplanar. We report here the single-crystal X-ray structure analysis of a series of four additional vinylsulfoximines, (1*c*)–(1*f*), and three  $\alpha$ -silylvinylsulfoximines, (2*a*), (2*b*) and (2*d*), with a view to establishing the conformational trends that exist. For the purpose of comparison, we also present the structure of phenyl vinyl sulfone, (3), as the archetypal vinyl sulfone.



The key structural feature of the vinyl sulfone, (3) (Fig. 1), is that the C=C and one of the S=O bonds are almost *syn*-coplanar [the C=C–S=O torsion angles are 4.85 (18) and –125.14 (16)°]. This may reflect the fact that the O atom is the smaller group at the S,

and may simply be a consequence of allylic strain. Inspection of the structure of the closely analogous vinylsulfoximine (1c) (Fig. 2) reveals that it adopts an almost identical conformation, with the single S=O bond *syn*-coplanar [torsion angle  $1.77(15)^\circ$ ]. These structures are closely reminiscent of the structure of (1b) (Dang *et al.*, 1993). The vinylsulfoximine (1d), carrying a methyl substituent, is also found in a very similar conformation (Fig. 3). Of special note in the case of this structure is the striking  $\pi$ - $\pi$  stacking of the two aromatic rings within the molecule (Hunter & Sanders, 1990). However, on moving to the vinylsulfoximines (1e) and (1f), a completely different conformation is observed, in which it is the S=N bond that is approximately *syn*-coplanar with the C=C bond (Figs. 4 and 5). This is very similar to what we previously observed in the case of the isopropyl derivative, (1a). Thus, the solid-state conformations of vinylsulfoximines fall into two categories, apparently dependent upon the size of the

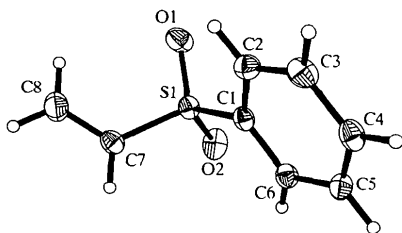


Fig. 1. The molecular structure of compound (3), showing the atom labels and 50% probability ellipsoids for the non-H atoms.

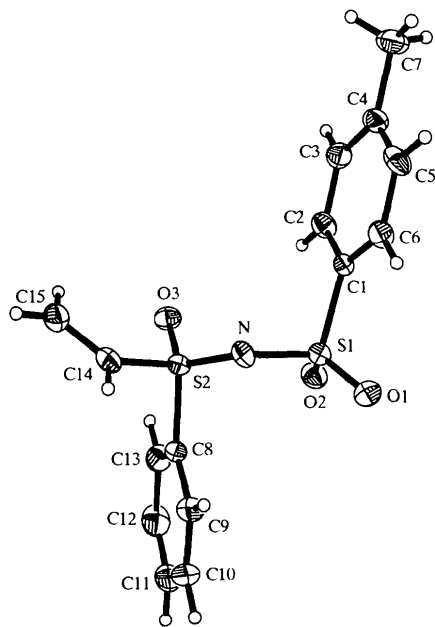


Fig. 2. The molecular structure of compound (1c), showing the atom labels and 50% probability ellipsoids for the non-H atoms.

substituent (Table 1). The apparent anomaly in the case of the phenyl derivative, (1b), is rather difficult to explain.

The structures of the  $\alpha$ -silylvinylsulfoximines, (2a), (2b) and (2d), also fall into two types. The methyl derivative, (2d), exists in a conformation in which the arrangement of the alkene and sulfoximine groups is almost indistinguishable from that of its unsilylated precursor, (1d), but the  $\pi$ - $\pi$  stacking present in (1d) does not occur in (2d) (Fig. 6). However, the phenyl derivative, (2b), which is additionally substituted with an *ortho*-silyl group on the phenyl ring, exists in a conformation in which the S-phenyl bond is approximately coplanar with the C=C bond, rather than with either of the double bonds at the S atom

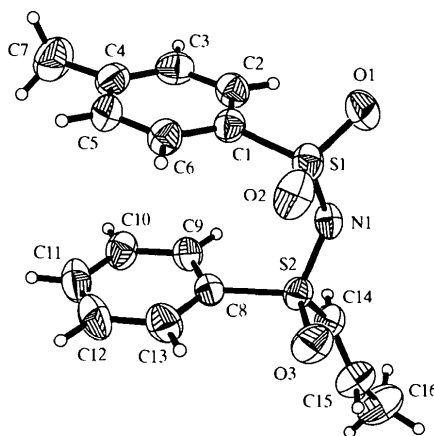


Fig. 3. The molecular structure of compound (1d), showing the atom labels and 50% probability ellipsoids for the non-H atoms.

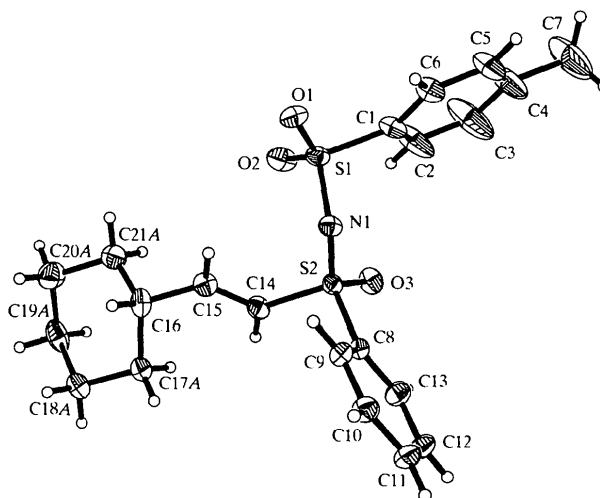


Fig. 4. The molecular structure of compound (1e), showing the atom labels and 50% probability ellipsoids for the non-H atoms; only one disorder component is shown.

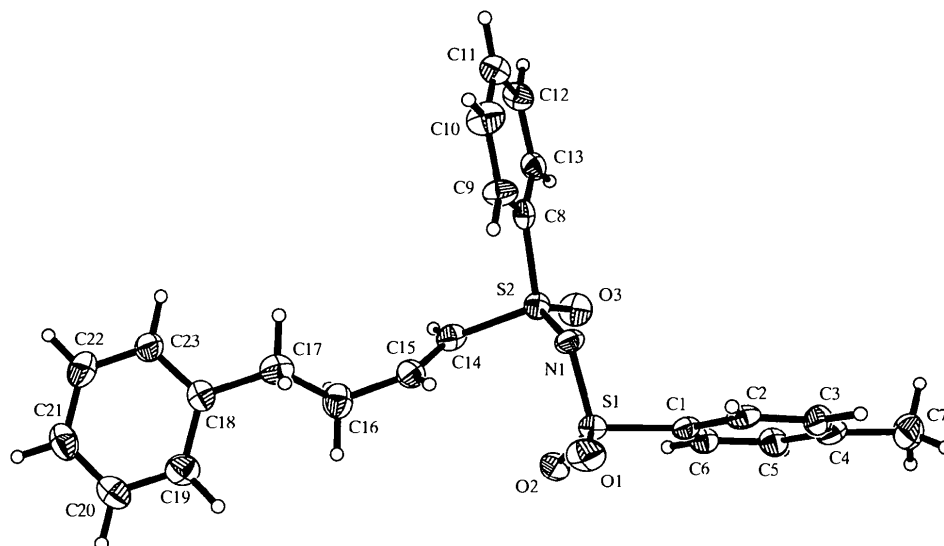


Fig. 5. The molecular structure of compound (1f), showing the atom labels and 50% probability ellipsoids for the non-H atoms.

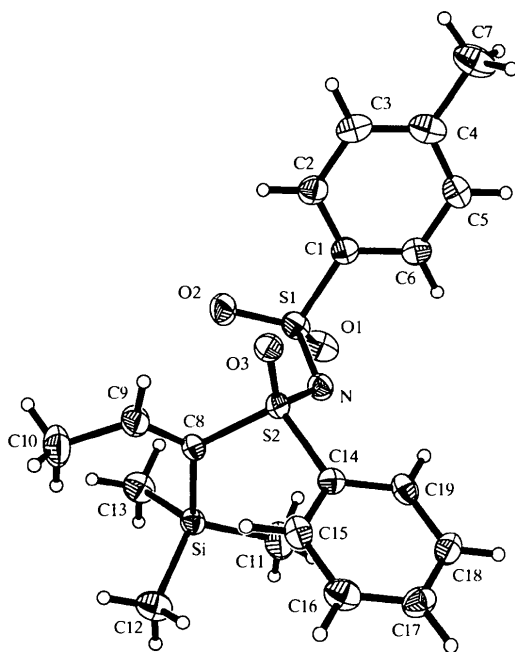


Fig. 6. The molecular structure of compound (2d), showing the atom labels and 50% probability ellipsoids for the non-H atoms.

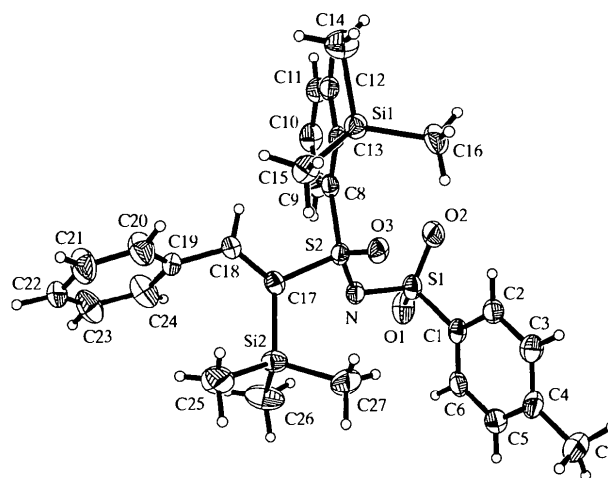


Fig. 7. The molecular structure of compound (2b), showing the atom labels and 50% probability ellipsoids for the non-H atoms; only one disorder component is shown.

(Fig. 7). It is also noteworthy that the presence of the trimethylsilyl group forces the phenyl group on the double bond out of the plane, resulting in an almost complete loss of conjugation between these groups. In the corresponding isopropyl compound, (2a) (Fig. 8), the overall conformation of both crystallographically independent molecules is similar to that of (2b), with the substituted phenyl group and the vinyl double bond approximately coplanar (Table 2).

It is interesting to note that we have observed substantially higher stereoselectivity in conjugate addition reactions of alkyl lithium reagents to compound (4) [an analogue of (2a), without the additional trimethylsilyl substituent on the aromatic ring] compared with additions to (2d) (Jackson *et al.*, 1996). The observed major stereoisomer in the addition of alkyl lithium reagents to (4) may result from a reactive conformation similar to that found for (2a), with addition of alkyl lithium to the less hindered face, perhaps assisted by coordination to the sulfoximine oxygen. The observed stereoselectivity in these reactions is consistent with the retention of the solid-state conformation as a preferred conformation of the various molecules in solution.

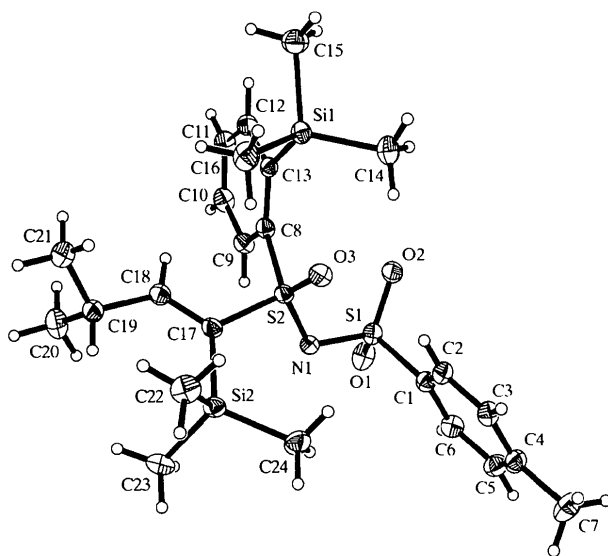


Fig. 8. The structure of one of the crystallographically independent molecules of compound (2a), showing the atom labels and 50% probability ellipsoids for the non-H atoms.

## Experimental

Compound (1c) was prepared by a literature method (David *et al.*, 1995) based on an unpublished procedure (Gibson & Wierzchlejski, 1993). Compounds (1d), (1e), (1f), (2a), (2b) and (2d) were prepared according to literature procedures (Jackson *et al.*, 1996). Compound (3) was obtained from the Aldrich Chemical Company. In each case, crystals were obtained by vapour diffusion of petroleum ether (b.p. 333–353 K) into an ethyl acetate solution of the compound.

### Compound (1c)

#### Crystal data

$C_{15}H_{15}NO_3S_2$

$M_r = 321.40$

Monoclinic

$P2_1/a$

$a = 15.8782$  (11) Å

$b = 5.7840$  (9) Å

$c = 17.1481$  (9) Å

$\beta = 107.535$  (4)°

$V = 1501.7$  (3) Å<sup>3</sup>

$Z = 4$

$D_x = 1.422$  Mg m<sup>-3</sup>

$D_m$  not measured

#### Data collection

Rigaku AFC-7R diffractometer

$\omega/2\theta$  scans

Absorption correction:

$\psi$  scan (North *et al.*, 1968)

$T_{\min} = 0.946$ ,  $T_{\max} = 1.000$

3271 measured reflections

3271 independent reflections

Mo  $K\alpha$  radiation

$\lambda = 0.71069$  Å

Cell parameters from 25

reflections

$\theta = 17.50$ – $19.45^\circ$

$\mu = 0.363$  mm<sup>-1</sup>

$T = 293$  (2) K

Plate

$0.40 \times 0.35 \times 0.05$  mm

Colourless

2818 reflections with

$I > 2\sigma(I)$

$\theta_{\max} = 27^\circ$

$h = 0 \rightarrow 20$

$k = 0 \rightarrow 7$

$l = -21 \rightarrow 20$

3 standard reflections

every 150 reflections

intensity decay: none

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.081$

$S = 1.081$

3271 reflections

192 parameters

H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2 + 0.7403P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.333$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.286$  e Å<sup>-3</sup>

Extinction correction:

SHELXTL

Extinction coefficient:

0.0004 (5)

Scattering factors from

International Tables for Crystallography (Vol. C)

### Compound (1d)

#### Crystal data

$C_{16}H_{17}NO_3S_2$

$M_r = 335.43$

Monoclinic

$C2/c$

$a = 35.6712$  (18) Å

$b = 5.8847$  (19) Å

$c = 15.4068$  (15) Å

$\beta = 90.737$  (7)°

$V = 3233.8$  (11) Å<sup>3</sup>

$Z = 8$

$D_x = 1.378$  Mg m<sup>-3</sup>

$D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda = 0.71069$  Å

Cell parameters from 25

reflections

$\theta = 17.05$ – $20.24^\circ$

$\mu = 0.340$  mm<sup>-1</sup>

$T = 293$  (2) K

Plate

$0.35 \times 0.30 \times 0.15$  mm

Colourless

### Data collection

Rigaku AFC-7R diffractometer

$\omega/2\theta$  scans

Absorption correction:

$\psi$  scan (North *et al.*, 1968)

$T_{\min} = 0.962$ ,  $T_{\max} = 1.000$

3532 measured reflections

3532 independent reflections

2710 reflections with

$I > 2\sigma(I)$

$\theta_{\max} = 27^\circ$

$h = 0 \rightarrow 45$

$k = 0 \rightarrow 7$

$l = -19 \rightarrow 19$

3 standard reflections

every 150 reflections

intensity decay: none

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.111$

$S = 1.030$

3532 reflections

202 parameters

H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 2.4959P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.024$

$\Delta\rho_{\max} = 0.277$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.306$  e Å<sup>-3</sup>

Extinction correction:

SHELXTL

Extinction coefficient:

0.00016 (19)

Scattering factors from

International Tables for Crystallography (Vol. C)

### Compound (1e)

#### Crystal data

$C_{21}H_{25}NO_3S_2$

$M_r = 403.54$

Mo  $K\alpha$  radiation

$\lambda = 0.71069$  Å

**Monoclinic** $P2_1/n$ 

$a = 10.370$  (2) Å  
 $b = 9.9267$  (18) Å  
 $c = 19.9997$  (16) Å  
 $\beta = 94.285$  (10)°  
 $V = 2053.0$  (6) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.306$  Mg m<sup>-3</sup>  
 $D_m$  not measured

**Data collection**

Rigaku AFC-7R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 4492 measured reflections  
 4492 independent reflections  
 3479 reflections with  
 $I > 2\sigma(I)$

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.104$   
 $S = 1.026$   
 4492 reflections  
 292 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 1.11P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

**Compound (1f)****Crystal data**

$C_{23}H_{23}NO_3S_2$   
 $M_r = 425.54$   
 Orthorhombic  
 $P2_12_12_1$   
 $a = 9.968$  (3) Å  
 $b = 11.6851$  (14) Å  
 $c = 18.509$  (4) Å  
 $V = 2155.9$  (8) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.311$  Mg m<sup>-3</sup>  
 $D_m$  not measured

**Data collection**

Stoe-Siemens diffractometer with Cryostream cooler (Cosier & Glazer, 1986)  
 $\omega/\theta$  scans  
 Absorption correction: none  
 2243 measured reflections  
 1987 independent reflections  
 1342 reflections with  
 $I > 2\sigma(I)$

Cell parameters from 20 reflections  
 $\theta = 9.60$ – $10.50$ °  
 $\mu = 0.280$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism  
 $0.25 \times 0.25 \times 0.10$  mm  
 Colourless

$\theta_{\max} = 27$ °  
 $h = 0 \rightarrow 13$   
 $k = 0 \rightarrow 12$   
 $l = -25 \rightarrow 25$   
 3 standard reflections every 150 reflections  
 intensity decay: none

$(\Delta/\sigma)_{\max} = 0.055$   
 $\Delta\rho_{\max} = 0.358$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.343$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXTL*  
 Extinction coefficient: 0.0018 (6)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å  
 Cell parameters from 43 reflections  
 $\theta = 9.79$ – $10.95$ °  
 $\mu = 0.271$  mm<sup>-1</sup>  
 $T = 160$  (2) K  
 Needle  
 $0.39 \times 0.12 \times 0.08$  mm  
 Colourless

$R_{\text{int}} = 0.031$   
 $\theta_{\max} = 22.51$ °  
 $h = -2 \rightarrow 10$   
 $k = -12 \rightarrow 12$   
 $l = -19 \rightarrow 19$   
 5 standard reflections frequency: 60 min  
 intensity decay: 1%

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.095$   
 $S = 1.024$   
 1987 reflections  
 264 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + (0.0498P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.003$   
 $\Delta\rho_{\max} = 0.271$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.298$  e Å<sup>-3</sup>

**Compound (2a)****Crystal data**

$C_{24}H_{37}NO_3S_2Si_2$   
 $M_r = 507.85$   
 Triclinic  
 $P\bar{1}$   
 $a = 13.876$  (7) Å  
 $b = 21.926$  (8) Å  
 $c = 9.363$  (2) Å  
 $\alpha = 97.05$  (3)°  
 $\beta = 91.78$  (3)°  
 $\gamma = 99.83$  (3)°  
 $V = 2781.7$  (18) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.213$  Mg m<sup>-3</sup>  
 $D_m$  not measured

**Data collection**

Rigaku AFC-7R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: none  
 12 102 measured reflections  
 12 102 independent reflections  
 9505 reflections with  
 $I > 2\sigma(I)$

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.109$   
 $S = 1.044$   
 12 102 reflections  
 596 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + (0.0582P)^2 + 0.8605P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

**Compound (2b)****Crystal data**

$C_{27}H_{35}NO_3S_2Si_2$   
 $M_r = 541.86$

Extinction correction: *SHELXTL*  
 Extinction coefficient: 0.0011 (5)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)  
 Absolute structure: Flack (1983)  
 Flack parameter = 0.39 (14)

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069$  Å  
 Cell parameters from 25 reflections  
 $\theta = 17.10$ – $19.60$ °  
 $\mu = 0.302$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block  
 $0.4 \times 0.4 \times 0.2$  mm  
 Colourless

$\theta_{\max} = 27.02$ °  
 $h = 0 \rightarrow 17$   
 $k = -27 \rightarrow 27$   
 $l = -11 \rightarrow 11$   
 3 standard reflections every 150 reflections  
 intensity decay: 0.8%

$(\Delta/\sigma)_{\max} = 0.006$   
 $\Delta\rho_{\max} = 0.406$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.496$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXTL*  
 Extinction coefficient: 0.0019 (3)  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å

## Monoclinic

$P2_1/c$   
 $a = 11.142 (3) \text{ \AA}$   
 $b = 17.128 (4) \text{ \AA}$   
 $c = 15.541 (4) \text{ \AA}$   
 $\beta = 100.35 (3)^\circ$   
 $V = 2917.6 (13) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.234 \text{ Mg m}^{-3}$   
 $D_m$  not measured

## Data collection

Stoe–Siemens diffractometer  
 with Cryostream cooler  
 (Cosier & Glazer, 1986)  
 $\omega/\theta$  scans with on-line  
 profile fitting (Clegg,  
 1981)  
 Absorption correction: none  
 5466 measured reflections  
 5100 independent reflections

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.068$   
 $wR(F^2) = 0.227$   
 $S = 1.013$   
 5100 reflections  
 336 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + 25.4072P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

## Compound (2d)

## Crystal data

$C_{19}H_{25}NO_3S_2Si$   
 $M_r = 407.61$   
 Monoclinic  
 $P2_1/n$   
 $a = 11.019 (2) \text{ \AA}$   
 $b = 17.093 (4) \text{ \AA}$   
 $c = 11.044 (3) \text{ \AA}$   
 $\beta = 99.73 (3)^\circ$   
 $V = 2050.1 (8) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.321 \text{ Mg m}^{-3}$   
 $D_m$  not measured

## Data collection

Stoe–Siemens diffractometer  
 with Cryostream cooler  
 (Cosier & Glazer, 1986)  
 $\omega/\theta$  scans with on-line  
 profile fitting (Clegg,  
 1981)  
 Absorption correction: none  
 3442 measured reflections  
 2638 independent reflections

## Cell parameters from 32 reflections

$\theta = 11.34\text{--}12.44^\circ$   
 $\mu = 0.292 \text{ mm}^{-1}$   
 $T = 160 (2) \text{ K}$   
 Block  
 $0.35 \times 0.34 \times 0.27 \text{ mm}$   
 Colourless

3599 reflections with  
 $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.114$   
 $\theta_{\text{max}} = 24.97^\circ$   
 $h = -13 \rightarrow 13$   
 $k = 0 \rightarrow 20$   
 $l = -18 \rightarrow 18$   
 4 standard reflections  
 frequency: 60 min  
 intensity decay: 2.4%

$(\Delta/\sigma)_{\text{max}} = 0.050$   
 $\Delta\rho_{\text{max}} = 0.477 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.413 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 34  
 reflections  
 $\theta = 10.25\text{--}12.16^\circ$   
 $\mu = 0.337 \text{ mm}^{-1}$   
 $T = 160 (2) \text{ K}$   
 Block  
 $0.29 \times 0.27 \times 0.23 \text{ mm}$   
 Colourless

1989 reflections with  
 $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.054$   
 $\theta_{\text{max}} = 22.49^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -4 \rightarrow 18$   
 $l = -11 \rightarrow 11$   
 5 standard reflections  
 frequency: 60 min  
 intensity decay: none

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.047$   
 $wR(F^2) = 0.149$   
 $S = 1.197$   
 2638 reflections  
 240 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + (0.0252P)^2 + 7.8529P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

## Compound (3)

## Crystal data

$C_8H_8O_2S$   
 $M_r = 168.20$   
 Orthorhombic  
*Pbcn*  
 $a = 9.747 (2) \text{ \AA}$   
 $b = 10.648 (2) \text{ \AA}$   
 $c = 15.418 (3) \text{ \AA}$   
 $V = 1600.2 (5) \text{ \AA}^3$   
 $Z = 8$   
 $D_x = 1.396 \text{ Mg m}^{-3}$   
 $D_m$  not measured

## Data collection

Stoe–Siemens diffractometer  
 with Cryostream cooler  
 (Cosier & Glazer, 1986)  
 $\omega/\theta$  scans with on-line  
 profile fitting (Clegg,  
 1981)  
 Absorption correction: none  
 3547 measured reflections  
 1407 independent reflections

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.087$   
 $S = 1.054$   
 1407 reflections  
 100 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.8206P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.361 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.347 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 32  
 reflections  
 $\theta = 11.40\text{--}12.09^\circ$   
 $\mu = 0.347 \text{ mm}^{-1}$   
 $T = 160 (2) \text{ K}$   
 Block  
 $0.34 \times 0.26 \times 0.26 \text{ mm}$   
 Colourless

1212 reflections with  
 $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\text{max}} = 24.95^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -12 \rightarrow 12$   
 $l = -18 \rightarrow 18$   
 5 standard reflections  
 frequency: 60 min  
 intensity decay: none

$(\Delta/\sigma)_{\text{max}} = 0.003$   
 $\Delta\rho_{\text{max}} = 0.329 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.508 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

Table 1. Selected torsion angles ( $^\circ$ ) for the vinylsulfoximines in series (1)

Compound	R	C=C—S=O	C=C—S=N
(1a)†	<sup>t</sup> Pr	-145.6	-11.6
(1b)†	Ph	-9.1	-132.0
(1c)	H	1.77 (15)	130.43 (13)
(1d)	Me	5.6 (2)	133.3 (2)
(1e)	<i>c</i> -C <sub>6</sub> H <sub>11</sub>	-141.47 (17)	-7.1 (2)
(1f)	PhCH <sub>2</sub> CH <sub>2</sub>	146.6 (5)	13.1 (5)

† Bailey *et al.* (1993). ‡ Dang *et al.* (1993).

Table 2. Selected torsion angles ( $^{\circ}$ ) for the  $\alpha$ -silylvinylsulfoximines in series (2)

Compound	R	C=C—S=O	C=C—S=N
(2a)†	<sup>t</sup> Pr	109.98 (15)	-123.66 (15)
(2a)†	<sup>t</sup> Pr	-115.94 (15)	117.76 (15)
(2b)	Ph	108.9 (5)	-125.1 (5)
(2d)	Me	-0.8 (5)	-134.7 (4)

† Two crystallographically independent molecules.

For compounds (1f) and (2d), data collection was restricted to a  $\theta_{\max}$  of  $22.5^{\circ}$ , because higher-angle intensities were very weak. H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds), and with  $U_{\text{iso}}$  constrained to be 1.2 (1.5 for methyl groups) times  $U_{\text{eq}}$  of the carrier atom. For compound (1e), twofold disorder of the cyclohexyl group was resolved and refined with essentially equal occupancies [50.6(6):49.4(6)%], without the need for any constraints or restraints.

For compounds (1c), (1d), (1e) and (2a); data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSCIAFC Diffractometer Control Software*; data reduction: *MSCIAFC Diffractometer Control Software*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs. For compounds (1f), (2b), (2d) and (3); data collection: *DIF4* (Stoe & Cie, 1988); cell refinement: *DIF4*; data reduction: local programs; program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1428). Services for accessing these data are described at the back of the journal.

## References

- Bailey, P. L., Clegg, W., Jackson, R. F. W. & Meth-Cohn, O. (1993). *J. Chem. Soc. Perkin Trans. 1*, pp. 343–350.
- Clegg, W. (1981). *Acta Cryst. A37*, 22–28.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Dang, Z., Pyne, S. G., Skelton, B. W. & White, A. H. (1993). *Aust. J. Chem.* **46**, 143–147.
- David, D. M., Bakavoli, M., Pyne, S. G., Skelton, B. W. & White, A. H. (1995). *Tetrahedron*, **51**, 12393–12402.
- Flack, H. D. (1983). *Acta Cryst. A39*, 876–881.
- Gibson (née Thomas), S. E. & Wierzchlejski, A. (1993). Personal communication.
- Glass, R. S., Reineke, K. & Shanklin, M. (1984). *J. Org. Chem.* **49**, 1527–1533.
- Hunter, C. A. & Sanders, J. K. M. (1990). *J. Am. Chem. Soc.* **112**, 5525–5534.
- Jackson, R. F. W., Briggs, A. D., Brown, P. A., Clegg, W., Elsegood, M. R. J. & Frampton, C. S. (1996). *J. Chem. Soc. Perkin Trans. 1*, pp. 1673–1682.
- Molecular Structure Corporation (1992). *MSCIAFC Diffractometer Control Software*. Version 4.3.0. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst. A24*, 351–359.

Pyne, S. G. (1986a). *J. Org. Chem.* **51**, 81–87.

Pyne, S. G. (1986b). *Tetrahedron Lett.* **27**, 1691–1694.

Pyne, S. G. (1992). *Sulfur Rep.* **12**, 57–93.

Sheldrick, G. M. (1994). *SHELXTL. Structure Determination Programs*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Stoe & Cie (1988). *DIF4. Diffractometer Control Program*. Version 7.04. Stoe & Cie, Darmstadt, Germany.

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## A Hexacyclic Estrone Derivative

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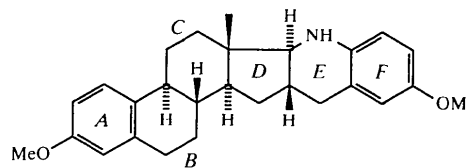
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### Abstract

The structure of 3,6'-dimethoxy-1',2' $\alpha$ ,3' $\beta$ ,4'-tetrahydroquinolino[3',2':16,17]estra-1,3,5(10)-triene, C<sub>27</sub>H<sub>33</sub>NO<sub>2</sub>, is reported.

### Comment

The synthesis of the title compound, (I), via a boron trifluoride ethyl etherate-initiated cyclization reaction of the corresponding D-secoestrone imine derivative, will



(I)

be published elsewhere (Frank *et al.*, 1998). The structure was investigated to determine the relative configuration of the newly formed stereogenic centres (C16 and C17), which could not be established unambiguously by NMR spectroscopy. The B/C, C/D and D/E ring fusions are all *trans*. Rings A and F are both planar, rings B and E adopt distorted half-chair conformations, ring C a chair conformation and ring D an envelope