

C(13)	0.3554 (5)	0.1292 (6)	0.8237 (8)	0.060 (11)
C(14)	0.0559 (3)	-0.1769 (4)	0.3761 (7)	0.038 (7)
C(15)	-0.0006 (3)	-0.2195 (5)	0.4606 (7)	0.046 (8)
C(16)	0.0233 (4)	-0.2526 (5)	0.6043 (8)	0.059 (9)
C(17)	0.0989 (4)	-0.2407 (5)	0.6604 (7)	0.058 (8)
C(18)	0.1552 (3)	-0.1997 (4)	0.5758 (7)	0.044 (7)
C(19)	0.1327 (3)	-0.1694 (4)	0.4332 (7)	0.041 (7)
C(20)	0.2388 (3)	-0.1914 (4)	0.6285 (8)	0.049 (8)
C(21)	0.2620 (3)	-0.1860 (4)	0.7918 (7)	0.045 (8)
C(22)	0.3347 (4)	-0.2216 (5)	0.8393 (8)	0.059 (9)
C(23)	0.3583 (4)	-0.2164 (5)	0.9902 (10)	0.070 (11)
C(24)	0.3122 (5)	-0.1765 (6)	1.0907 (9)	0.068 (12)
C(25)	0.2415 (5)	-0.1404 (6)	1.0448 (8)	0.071 (11)
C(26)	0.2161 (4)	-0.1464 (5)	0.8952 (8)	0.058 (9)
O(2)	0.2891 (2)	-0.1870 (3)	0.5362 (5)	0.066 (6)
N(2)	-0.0748 (3)	-0.2307 (4)	0.4047 (7)	0.059 (8)

Table 2. Geometric parameters (Å, °)

Se(1)—Se(2)	2.341 (2)	C(11)—C(12)	1.358 (15)
Se(1)—C(1)	1.910 (5)	C(12)—C(13)	1.374 (13)
Se(2)—C(14)	1.898 (6)	C(14)—C(15)	1.404 (8)
O(1)—C(7)	1.207 (8)	C(14)—C(19)	1.384 (7)
C(1)—C(2)	1.376 (8)	C(15)—C(16)	1.410 (10)
C(1)—C(6)	1.382 (8)	C(16)—C(17)	1.369 (10)
C(2)—C(3)	1.418 (9)	C(17)—C(18)	1.390 (9)
C(3)—C(4)	1.371 (9)	C(18)—C(19)	1.381 (9)
C(4)—C(5)	1.389 (8)	C(18)—C(20)	1.482 (7)
C(5)—C(6)	1.392 (7)	C(20)—C(21)	1.485 (9)
C(5)—C(7)	1.480 (8)	C(25)—C(26)	1.378 (10)
C(7)—C(8)	1.485 (10)	C(21)—C(22)	1.395 (9)
C(8)—C(9)	1.363 (11)	C(21)—C(26)	1.370 (9)
C(8)—C(13)	1.390 (11)	C(22)—C(23)	1.381 (11)
C(9)—C(10)	1.390 (11)	C(23)—C(24)	1.356 (12)
C(10)—C(11)	1.375 (16)	C(24)—C(25)	1.364 (12)
Se(2)—Se(1)—C(1)	97.2 (2)	C(8)—C(13)—C(12)	119.8 (8)
Se(1)—Se(2)—C(14)	99.2 (2)	Se(2)—C(14)—C(15)	121.4 (4)
Se(1)—C(1)—C(2)	121.0 (4)	Se(2)—C(14)—C(19)	118.5 (4)
Se(1)—C(1)—C(6)	118.1 (4)	C(15)—C(14)—C(19)	120.2 (5)
C(2)—C(1)—C(6)	120.9 (5)	C(14)—C(15)—C(16)	118.0 (6)
C(1)—C(2)—C(3)	118.2 (5)	C(15)—C(16)—C(17)	120.4 (6)
C(2)—C(3)—C(4)	120.1 (6)	C(16)—C(17)—C(18)	121.5 (6)
C(3)—C(4)—C(5)	121.7 (5)	C(17)—C(18)—C(19)	118.3 (6)
C(4)—C(5)—C(6)	117.6 (5)	C(17)—C(18)—C(20)	123.0 (5)
C(4)—C(5)—C(7)	123.9 (5)	C(19)—C(18)—C(20)	118.6 (5)
C(6)—C(5)—C(7)	118.3 (5)	C(14)—C(19)—C(18)	121.5 (5)
C(1)—C(6)—C(5)	121.4 (5)	C(18)—C(20)—C(21)	121.1 (5)
O(1)—C(7)—C(5)	120.5 (6)	C(18)—C(20)—O(2)	119.4 (5)
O(1)—C(7)—C(8)	119.2 (6)	C(21)—C(20)—O(2)	119.6 (5)
C(5)—C(7)—C(8)	120.3 (6)	C(20)—C(21)—C(22)	117.5 (5)
C(7)—C(8)—C(9)	123.4 (6)	C(20)—C(21)—C(26)	123.1 (6)
C(7)—C(8)—C(13)	117.0 (6)	C(22)—C(21)—C(26)	119.4 (6)
C(9)—C(8)—C(13)	119.6 (7)	C(21)—C(22)—C(23)	118.8 (6)
C(8)—C(9)—C(10)	120.1 (8)	C(22)—C(23)—C(24)	120.8 (7)
C(9)—C(10)—C(11)	119.6 (9)	C(23)—C(24)—C(25)	120.7 (8)
C(10)—C(11)—C(12)	120.4 (10)	C(24)—C(25)—C(26)	119.4 (8)
C(11)—C(12)—C(13)	120.4 (10)	C(21)—C(26)—C(25)	120.8 (7)

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and dihedral angles between planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71235 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1036]

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## Structure of the Naphthylurethane of *trans*- $\beta$ -Ionol, a Severely Nonplanar 1,3-Diene

R. L. BEDDOES, A. A. GORMAN\* AND D. J. UNETT

*Chemistry Department, University of Manchester, Manchester M13 9PL, England*

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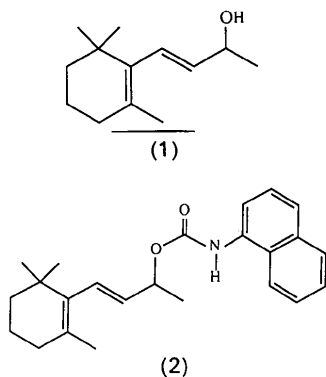
## Abstract

4-(2,6,6-Trimethyl-1-cyclohexen-1-yl)-3-buten-2-yl 1-naphthalenecarbamate, the 1-naphthylurethane of *trans*- $\beta$ -ionol, possesses a 1,3-diene  $\pi$  system which is grossly nonplanar as a consequence of sterically enforced torsion about the central single bond, the torsion angle between the formal double bonds being 54°. This result is in excellent agreement with conclusions concerning the 'nonvertical' behaviour of the parent *trans*- $\beta$ -ionol with respect to triplet-excitation transfer.

## Comment

It has been accepted generally that nonclassical or nonvertical triplet-excitation transfer to conjugated and flexible  $\pi$  systems is a consequence of double-bond torsion, either prior to or during the electronic energy transfer event (Saltiel, Marchand, Kirkor-

Kiminska, Smothers, Mueller & Charlton, 1984, and references therein). It has been proposed, however, that significant nonvertical behaviour is restricted to those molecules possessing a conjugated  $\pi$  system that is significantly nonplanar (Gorman, Hamblett, Irvine, Raby, Standen & Yeates, 1985) and that the key to such behaviour is torsion about formal single bonds in the ground state (Gorman, Beddoes, Hamblett, McNeeney, Prescott & Unett, 1991). As part of our research programme in this area it has been shown (Gorman, 1990) that the simple liquid 1,3-diene *trans*- $\beta$ -ionol (1), exhibits very pronounced nonvertical behaviour indicating that its  $\pi$  system must be *extremely* nonplanar with respect to torsion about the central single bond. These results contrast with previous work concerning both the geometry and triplet energy-accepting characteristics of (1) (Ramamurthy & Liu, 1976). We report here the X-ray crystallographic determination of the structure of a derivative of (1), the 1-naphthylurethane (2). It shows that the 1,3-diene unit is in fact strongly distorted from planarity with respect to central single-bond torsion, a result in total accord with the aforementioned extreme nonvertical triplet-excitation behaviour of (1).



Crystalline (2) was prepared by standard reaction of (1) (Mousseron-Canet, Mousseron & Lengendre, 1964) with 1-naphthyl isocyanate. Despite many attempts, it was not possible to obtain good quality crystals. The results given here are from a crystal of indifferent quality and there are indications of disorder over two conformations in the region of C22 and C23. However, the torsion angles of interest are established. Fig. 1 shows a *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule together with the numbering system used. Double-bond torsion angles are 6 (3) [C3—C2=C1—C27] and 176 (1)° [C2—C3=C4—C5], clearly indicating their essential planarity. In contrast, the single-bond torsion angle [C1=C2—C3=C4] is 54 (3)°. It is quite apparent from the structure that this large angle reflects interactions involving the H atoms attached to C3, C4, C25, C26 and C27, and is not influenced by the

urethane moiety. This conclusion is also supported by molecular-mechanics calculations on (1) which predict a very similar single-bond torsion angle, 57.5°. Bond lengths and angles do not differ significantly from expected values.

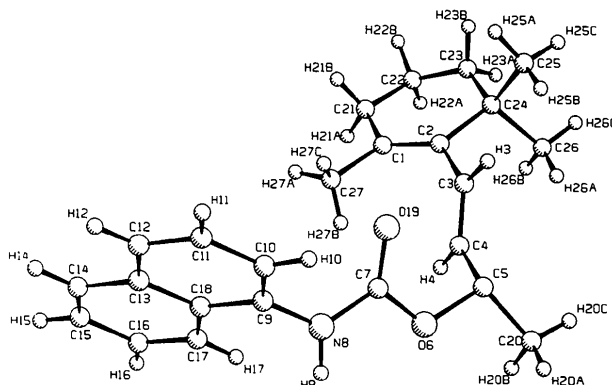


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing of (2) showing the atomic numbering scheme.

## Experimental

### Crystal data

$C_{24}H_{28}NO_2$   
 $M_r = 362.49$   
 Orthorhombic  
 $Pca2_1$   
 $a = 27.961$  (1) Å  
 $b = 8.154$  (4) Å  
 $c = 9.340$  (2) Å  
 $V = 2129$  (2) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.13$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069$  Å  
 Cell parameters from 25 reflections  
 $\theta = 10.03$ – $10.81^\circ$   
 $\mu = 0.07$  mm<sup>-1</sup>  
 $T = 294$  (1) K  
 Tabular  
 $0.37 \times 0.14 \times 0.05$  mm  
 Colourless  
 Crystal source: recrystallized from 95% ethanol

### Data collection

Enraf-Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)  
 $T_{\min} = 0.61$ ,  $T_{\max} = 1.14$   
 1984 measured reflections  
 1984 independent reflections

791 observed reflections  
 $[I > 2\sigma(I)]$   
 $\theta_{\max} = 25^\circ$   
 $h = 0 \rightarrow 29$   
 $k = 0 \rightarrow 9$   
 $l = 0 \rightarrow 11$   
 2 standard reflections  
 frequency: 150 min  
 intensity variation: none

### Refinement

Refinement on  $F$   
 Final  $R = 0.083$   
 $wR = 0.081$   
 $S = 2.05$   
 790 reflections  
 153 parameters  
 H-atom parameters not refined

$(\Delta/\sigma)_{\max} = 0.07$   
 $\Delta\rho_{\max} = 0.23$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.22$  e Å<sup>-3</sup>  
 Atomic scattering factors from Cromer & Waber (1974)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )
$$B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$B_{\text{eq}}$
C1	0.4199 (6)	-0.006 (2)	0.1352	4.8 (4)
C2	0.4275 (6)	0.149 (2)	0.114 (3)	4.4 (3)
C3	0.3954 (6)	0.282 (2)	0.173 (2)	4.5 (4)
C4	0.3829 (6)	0.302 (2)	0.305 (2)	4.0 (3)
C5	0.3474 (5)	0.435 (2)	0.356 (2)	3.9 (3)
O6	0.3076 (3)	0.349 (1)	0.428 (2)	4.3 (2)
C7	0.2774 (5)	0.269 (2)	0.344 (2)	3.5 (3)
N8	0.2444 (4)	0.182 (1)	0.420 (2)	3.5 (2)
C9	0.2085 (5)	0.088 (2)	0.359 (2)	3.4 (3)
C10	0.1787 (5)	0.149 (2)	0.258 (2)	4.0 (3)
C11	0.1431 (6)	0.051 (2)	0.196 (2)	5.2 (4)
C12	0.1380 (6)	-0.108 (2)	0.233 (2)	4.8 (4)
C13	0.1695 (5)	-0.178 (2)	0.336 (2)	3.3 (3)
C14	0.1651 (6)	-0.345 (2)	0.376 (2)	4.9 (4)
C15	0.1947 (6)	-0.409 (2)	0.470 (3)	5.9 (4)
C16	0.2303 (6)	-0.316 (2)	0.534 (3)	5.8 (5)
C17	0.2366 (5)	-0.155 (2)	0.501 (2)	4.0 (4)
C18	0.2057 (5)	-0.082 (2)	0.399 (2)	3.8 (3)
O19	0.2783 (3)	0.280 (1)	0.212 (2)	4.1 (5)
C20	0.3678 (6)	0.546 (2)	0.470 (2)	4.9 (8)
C21	0.4532 (9)	-0.127 (3)	0.079 (3)	8 (1)
C22	0.496 (1)	-0.067 (5)	0.016 (4)	13 (3)
C23	0.501 (1)	0.091 (6)	-0.028 (5)	15 (3)
C24	0.4688 (7)	0.223 (3)	0.030 (3)	7 (1)
C25	0.4520 (7)	0.335 (3)	-0.086 (3)	9 (1)
C26	0.5010 (8)	0.331 (3)	0.130 (4)	10 (2)
C27	0.3779 (6)	-0.076 (2)	0.205 (3)	6 (1)

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

C1—C2	1.30 (2)	C5—C20	1.51 (2)
C1—C21	1.45 (3)	O6—C7	1.33 (2)
C1—C27	1.46 (2)	C7—N8	1.37 (2)
C2—C3	1.51 (2)	C7—O19	1.24 (2)
C2—C24	1.52 (2)	N8—C9	1.39 (2)
C3—C4	1.30 (2)	C21—C22	1.43 (4)
C4—C5	1.55 (2)	C22—C23	1.36 (4)
C5—O6	1.48 (2)	C23—C24	1.50 (4)
C2—C1—C21	120 (2)	C5—O6—C7	116 (1)
C2—C1—C27	125 (2)	O6—C7—N8	112 (1)
C21—C1—C27	114 (2)	O6—C7—O19	123 (1)
C1—C2—C3	123 (2)	N8—C7—O19	125 (1)
C1—C2—C24	126 (2)	C7—N8—C9	124 (1)
C3—C2—C24	111 (2)	N8—C9—C10	122 (1)
C2—C3—C4	127 (2)	N8—C9—C18	118 (1)
C3—C4—C5	124 (1)	C10—C9—C18	120 (1)
C4—C5—O6	107 (1)	C1—C21—C22	117 (3)
C4—C5—C20	113 (1)	C21—C22—C23	122 (3)
O6—C5—C20	104 (1)	C22—C23—C24	121 (3)

Anisotropic vibration parameters were refined for the atoms O19 and C20–C27; all other non-H atoms were refined isotropically. Weights were applied according to the default scheme in *TEXSAN* (Molecular Structure Corporation, 1985). Program used to solve structure: *SHELXS86* (Sheldrick, 1985). Program used to refine structure: *TEXSAN*.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71318 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1043]

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## Structure du 2,5-Diphényl-3-furyl 2,5-Diphényl-3-thiényl Sulfoxyde

DARIA GINDEROW ET JEAN-PAUL MORNON

Laboratoire de Minéralogie et Cristallographie,  
URA 09 CNRS, Université Pierre et Marie Curie,  
T16, 4 place Jussieu, 75252 Paris CEDEX 05,  
France

IRENE ERDELMEIER, PATRICK DANSETTE  
ET DANIEL MANSUY

Laboratoire de Chimie et Biochimie Pharmacologiques  
et Toxicologiques, URA 400 CNRS, Université  
René Descartes, 45 rue des Saints-Pères,  
75270 Paris CEDEX 06, France

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## Abstract

2,5-Diphenyl-3-furyl 2,5-diphenyl-3-thienyl sulfoxide consists of a sulfoxide linked to a thienyl and a furyl group, which are each linked to two phenyl groups.