

formed using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1997).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1997).

This work was supported in part by the National Science Foundation, through the Northwestern University Materials Research Center (grant No. DMR 9632472), and by the China National Planning Committee.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1397). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 996–1000

Regioselectivity in dibenzobarrelene photorearrangements: photoproducts derived from 9-substituted-dibenzobarrelenes

M. MUNEER,^a D. RAMAIAH,^a E. S. AJITKUMAR,^a M. C. SAJIMON,^a NIGAM P. RATH^b AND M. V. GEORGE^{a,c}

^aPhotochemistry Research Unit, Regional Research Laboratory (CSIR), Trivandrum 695 019, India, ^bDepartment of Chemistry, University of Missouri–St. Louis, 8001 Natural Bridge Road, St. Louis, MO 63121, USA, and ^cJawaharlal Nehru Centre for Advanced Scientific Research, Bangalore 560 064, India. E-mail: nigam_rath@umsl.edu

(Received 3 August 1998; accepted 11 January 1999)

Abstract

Irradiation of the 9-formyl-, 9-acetyl-, 9-nitro- and 9-*tert*-substituted dibenzobarrelenes gave the corresponding 4b-substituted dibenzosemibullvalenes, (2a) (8b, 8c-dibenzoyl-4b, 8b, 8c, 8d-tetrahydrodibenzo[*a,f*]cyclopropa[*cd*]pentalene-4b-carbaldehyde, C₃₁H₂₀O₃), (2b) (4b-acetyl-8b, 8c-dibenzoyl-4b, 8b, 8c, 8d-tetra-

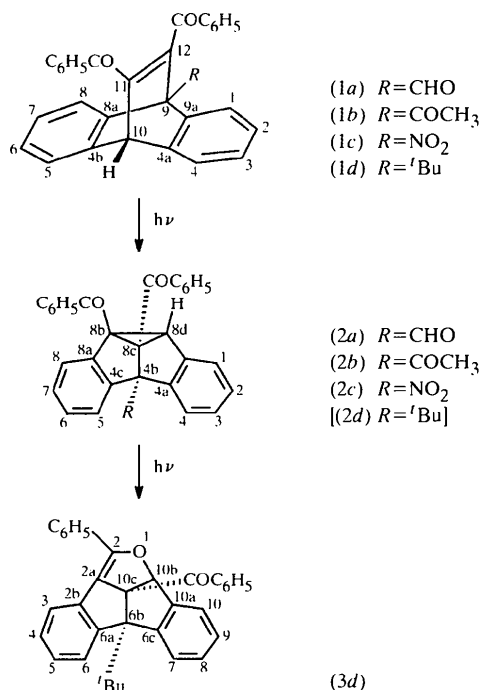
hydrodibenzo[*a,f*]cyclopropa[*cd*]pentalene, C₃₂H₂₂O₃), (2c) (8b, 8c-dibenzoyl-4b-nitro-4b, 8b, 8c, 8d-tetrahydrodibenzo[*a,f*]cyclopropa[*cd*]pentalene, C₃₀H₁₉NO₄) and (2d) (8b, 8c-dibenzoyl-4b-*tert*-butyl-4b, 8b, 8c, 8d-tetrahydrodibenzo[*a,f*]cyclopropa[*cd*]pentalene). The product (2d) gave the corresponding dibenzopentalenofuran derivative, (3d) (10c-benzoyl-6b-*tert*-butyl-2-phenyl-10b, 10c-dihydro-6b-*H*-dibenzo[*a,f*]furo[2,3,4-*cd*]pentalene, C₃₄H₂₈O₂). The structures of (2a), (2b), (2c) and (3d) have been established unequivocally *via* X-ray crystallographic analysis. The molecular structures of (2a), (2b), and (2c) incorporate the basic dibenzosemibullvalene skeleton, which contains the 6:5:5:6:3 fused ring system with normal bond lengths and bond angles. The crystal structures of (2a), (2b), and (2c) have intermolecular hydrogen-bonding interactions (< 2.60 Å) which are almost linear around the H atoms. The structure of (3d) shows a 6:5:5:6:5 fused ring system and does not indicate hydrogen-bonding-type intermolecular interactions.

Comment

Several aspects of the photorearrangement of dibenzobarrelenes containing the 1,2-dibenzoylalkene moiety are reported in the literature (Kumar *et al.*, 1984, 1993, 1996, 1997; Murty *et al.*, 1985; Pratapan *et al.*, 1987, 1990; Asokan *et al.*, 1991; Scheffer & Yang, 1995; Mathew *et al.*, 1996a,b; Ramaiah *et al.*, 1996; Muneer *et al.*, 1996). It has been found that the observed regioselectivity in these rearrangements depends on several factors, including the electronic and steric requirements of the bridgehead substituents. Thus, it has been observed that if an electron-withdrawing substituent such as the cyano group is present at the bridgehead position of the starting dibenzobarrelene, then the 8b-cyanosubstituted dibenzosemibullvalene is formed on irradiation (Murty *et al.*, 1985; Muneer *et al.*, 1996). On the other hand, the presence of substituents such as methyl, methoxy, formyl, acetoxy *etc.* at the bridgehead position of the starting dibenzobarrelenes results in the formation of the corresponding 4b-substituted dibenzosemibullvalenes upon irradiation (Murty *et al.*, 1985). In view of the importance of understanding the role of bridgehead substituents in the observed regioselectivity in the photorearrangements of dibenzobarrelenes, it became essential to determine the structures of the photoproducts unambiguously through X-ray crystallographic analysis.

In the present study, we have examined the structures of the photoproducts (2a), (2b), (2c) and (3d), derived from the dibenzobarrelenes, 11,12-dibenzoyl-9,10-dihydro-9-formyl-9,10-ethenoanthracene (1a), 9-acetyl-11,12-dibenzoyl-9,10-dihydro-9,10-ethenoanthracene (1b), 11,12-dibenzoyl-9,10-dihydro-9-nitro-9,10-ethenoanthracene (1c) and 9-*tert*-butyl-11,12-dibenzoyl-9,10-dihydro-9,10-ethenoanthracene (1d), respectively. It has been reported earlier, on the basis

of analytical results and spectral data, that the photolysis of the (1a) dibenzobarrelene leads to the corresponding 4b-substituted dibenzosemibullvalene, (2a) (Murty *et al.*, 1985). In a recent study, it has been shown that irradiation of the 9-nitro- and 9-*tert*-butyl-substituted dibenzobarrelenes, (1c) and (1d), gave the corresponding dibenzosemibullvalenes, (2c) (81%) and (2d) (95%), respectively. Irradiation of the 9-acetyl-substituted derivative, (1b), on the other hand, gave a mixture of the 4b-substituted [(2b), 70%] and 8b-substituted (20%) dibenzosemibullvalenes (Sajimon *et al.* 1999).



The projection views of the molecules (2a), (2b), (2c) and (3d) are shown in Figs. 1–4, respectively. The molecular structures of (2a), (2b) and (2c) incorporate the basic dibenzosemibullvalene skeleton, which contains the 6:5:5:6:3 fused ring system, with two benzoyl groups attached to the cyclopropane ring. The 9-substituents present in the starting dibenzobarrelenes, (1a), (1b) and (1c), have ended up in the 4b-position of the product dibenzosemibullvalenes. In the cases of (2a), (2b) and (2c), the cyclopropane rings remain intact. These photoproducts have normal bond lengths and angles typical of the folded dibenzosemibullvalene skeleton (Jones *et al.*, 1997). The cyclopropane ring is asymmetrical, with C8b—C8d being the longest of the three bonds and the angle C8b—C8c—C8d being the largest [C8b—C8d 1.545 (3), 1.559 (6) and 1.575 (2) Å, and C8b—C8c—C8d 61.0 (2), 61.8 (3) and 62.85 (9)°, respectively, for (2a), (2b) and (2c)]. No significant effect on the geometrical parameters due to the difference in R groups at C4b has been observed.

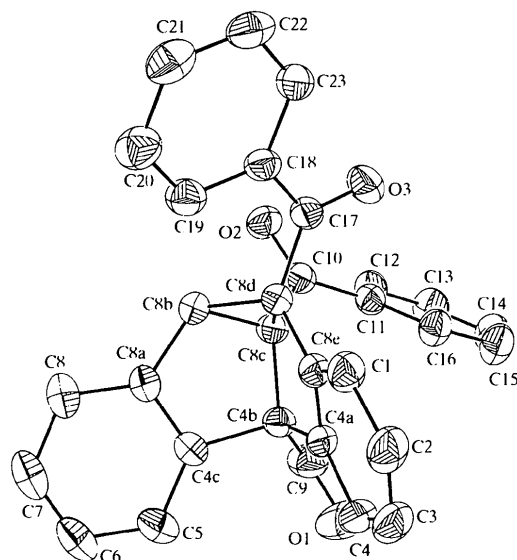


Fig. 1. View of molecule (2a) with 30% probability ellipsoids. H atoms are omitted for clarity.

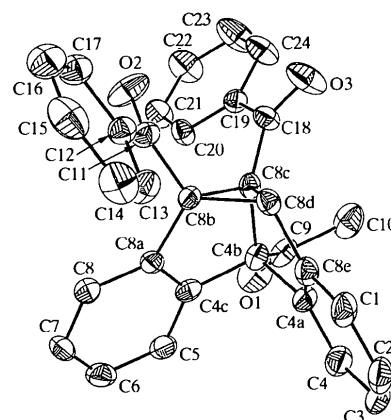


Fig. 2. View of molecule (2b) with 30% probability ellipsoids. H atoms are omitted for clarity.

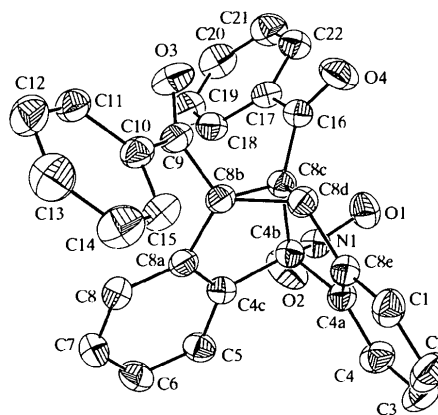


Fig. 3. View of molecule (2c) with 50% probability ellipsoids. H atoms are omitted for clarity.

The structures of the three dibenzosemibullvalenes reveal hydrogen-bonding interactions involving carbonyl-O atoms and aromatic-H or methyl-H atoms. Compound (2a) forms three linear hydrogen bonds, with H22A···O2($x-1, y, z$) 2.49, H3A···O3($\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$) 2.44 and H8A···O1($x-\frac{1}{2}, \frac{3}{2}-y, z-\frac{1}{2}$) 2.53 Å, and with angles around the H atoms of 142.6, 158.0 and 152.3°, respectively. Compound (2b) also exhibits three hydrogen bonds, one of which involves a methyl-H atom, H10B. The hydrogen-bonding distances and angles around the H atoms are H10B···O1($1-x, -\frac{1}{2}+y, \frac{1}{2}-z$) 2.51 Å and C10—H10B···O1 133.9°; H17A···O2($-x, 1-y, -z$) 2.58 Å and C17—H17A···O2 132.1°, and H15A···O3($-x, -y, -z$) 2.56 Å and C15—H15A···O3 139.0°. Compound (2c) exhibits two hydrogen-bonding interactions in the crystal lattice which are less than the van der Waals radii ($r_O + r_H = 2.6$ Å; Pauling, 1963). These two interactions have distances and angles of H8A···O1($-x, -\frac{1}{2}+y, \frac{1}{2}-z$) 2.55 Å and C8—H8A···O1 133.2°, and H11A···O3($-x, -y, -z$) 2.39 Å and C11—H11A···O3 159.9°. The other intermolecular interactions for this molecule involving the second O atom of NO₂ (O2) and the second carbonyl-O atom (O4; two interactions) are longer than 2.6 Å, in the range 2.62–2.71 Å.

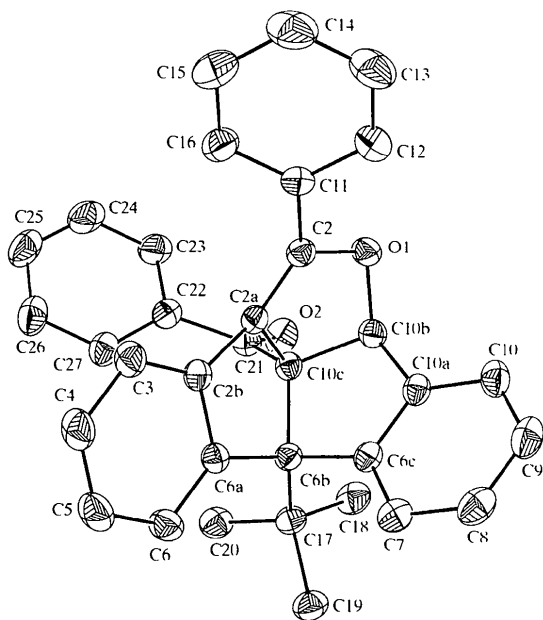


Fig. 4. View of molecule (3d) with 30% probability ellipsoids. H atoms are omitted for clarity.

The structure of compound (3d) reveals the presence of a 6:5:5:6:5 ring system, which is essentially a dibenzopentalenofuran derivative. It is assumed that the dibenzosemibullvalene, (2d), formed initially from (1d), undergoes a thermal ring enlargement to give (3d). Such ring-enlargement reactions of dibenzosemibullvalenes

have been reported earlier (Pratapan *et al.*, 1990). It appears that the presence of a bulky *tert*-butyl group at the 4b-position of the (2d) dibenzosemibullvalene may facilitate the cleavage of the cyclopropane ring and subsequent ring enlargement, resulting in the formation of (3d). The molecule has normal geometry and dimensions. The aromatic double bonds, C11=C16 and C11=C12, of the C11–C16 phenyl ring are not fully conjugated with the C2=C2a double bond [C12—C11—C2—C2a 143.32(4) and C16—C11—C2—C2a 143.26(5)°, $\cos^2(\text{angles}) = 0.64$]. The crystal packing of (3d) does not show hydrogen bonding, possibly due to the presence of the bulky *tert*-butyl group at the bridgehead position. Only a weak interaction of the carbonyl O2 atom with an aromatic H atom [H10A···O2($-x, -y, -z$) 2.82 Å] is indicated for this molecule.

The fact that the different substituents, such as CHO, COCH₃, NO₂ and C(CH₃)₃, which are present in the 9-position of the starting dibenzobarrelenes (1a)–(1d), end up in the 4b-position of the photoproducts reveals that the di- π -methane rearrangements of these dibenzobarrelenes proceed in a regioselective manner. Thus, it is evident that in these systems, the initial bridging on the di- π -methane rearrangement pathway is between atoms C4a and C11 of the dibenzobarrelenes, to give the corresponding biradical intermediates. These in turn proceed through known pathways to give the observed end products.

It is of interest to note that, irrespective of the electronic and steric requirements of the different substituents in the starting dibenzobarrelenes, (1a)–(1d), each primarily leads to the 4b-substituted dibenzosemibullvalene.

Experimental

Full experimental details, including photochemical studies, spectroscopic data and details of the syntheses of (2a), (2b), (2c) and (3d), will be published elsewhere (Sajimon *et al.*, 1999). Crystals of (2a) and (2c) suitable for X-ray diffraction were grown by slow evaporation of acetonitrile solutions. Crystals of (2b) were obtained from a 1:4 mixture of ethyl acetate and chloroform, and compound (3d) was crystallized from a 1:1 mixture of dichloromethane and methanol.

Compound (2a)

Crystal data

C₃₁H₂₀O₃
 $M_r = 440.47$
 Monoclinic
 $P2_1/n$
 $a = 8.7836(6)$ Å
 $b = 16.5431(13)$ Å
 $c = 15.5118(11)$ Å
 $\beta = 93.117(6)^\circ$
 $V = 2250.7(3)$ Å³
 $Z = 4$
 $D_x = 1.300$ Mg m⁻³
 D_m not measured

Cu K α radiation
 $\lambda = 1.54178$ Å
 Cell parameters from 33 reflections
 $\theta = 5.36$ – 27.12°
 $\mu = 0.660$ mm⁻¹
 $T = 298(2)$ K
 Rectangular
 $0.41 \times 0.33 \times 0.15$ mm
 Colorless

Data collection

Bruker P4 diffractometer
 $2\theta/\omega$ scans
 Absorption correction: none
 7774 measured reflections
 3677 independent reflections
 2593 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.07$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.175$
 $S = 1.073$
 3677 reflections
 308 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0857P)^2 + 0.8370P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$

Compound (2b)**Crystal data**

$\text{C}_{32}\text{H}_{22}\text{O}_3$
 $M_r = 454.50$
 Monoclinic
 $P2_1/c$
 $a = 9.0656$ (15) Å
 $b = 8.779$ (3) Å
 $c = 29.758$ (5) Å
 $\beta = 94.672$ (13)°
 $V = 2360.6$ (9) Å³
 $Z = 4$
 $D_x = 1.279$ Mg m⁻³
 D_m not measured

Data collection

Bruker P4 diffractometer
 $2\theta/\omega$ scans
 Absorption correction: none
 7761 measured reflections
 4136 independent reflections
 1606 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.077$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.077$
 $wR(F^2) = 0.184$
 $S = 1.040$
 4136 reflections
 316 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0490P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$\theta_{\text{max}} = 66.98^\circ$
 $h = -10 \rightarrow 10$
 $k = 0 \rightarrow 19$
 $l = 0 \rightarrow 16$
 3 standard reflections
 every 97 reflections
 intensity decay: 2.3%

$\Delta\rho_{\text{max}} = 0.178$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.210$ e Å⁻³
 Extinction correction:
 SHELXTL (Sheldrick,
 1998)
 Extinction coefficient:
 0.0064 (6)
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 29
 reflections
 $\theta = 5.30\text{--}6.81^\circ$
 $\mu = 0.081$ mm⁻¹
 $T = 298$ (2) K
 Rectangular plate
 $0.33 \times 0.30 \times 0.10$ mm
 Colorless

$(\Delta/\sigma)_{\text{max}} = 0.008$
 $\Delta\rho_{\text{max}} = 0.194$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.251$ e Å⁻³
 Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Compound (2c)**Crystal data**

$\text{C}_{30}\text{H}_{19}\text{NO}_4$
 $M_r = 457.46$
 Orthorhombic
 $Pbca$
 $a = 16.8684$ (1) Å
 $b = 15.3955$ (1) Å
 $c = 17.5200$ (2) Å
 $V = 4549.90$ (7) Å³
 $Z = 8$
 $D_x = 1.336$ Mg m⁻³
 D_m not measured

Data collection

Bruker CCD area-detector
 diffractometer
 φ and ω scans
 Absorption correction: none
 95 639 measured reflections
 4967 independent reflections

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.110$
 $S = 1.056$
 4967 reflections
 316 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 1.5858P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Compound (3d)**Crystal data**

$\text{C}_{34}\text{H}_{28}\text{O}_2$
 $M_r = 468.56$
 Monoclinic
 $P2_1/c$
 $a = 16.6439$ (6) Å
 $b = 17.9178$ (8) Å
 $c = 8.3487$ (3) Å
 $\beta = 94.963$ (5)°
 $V = 2480.43$ (17) Å³
 $Z = 4$
 $D_x = 1.255$ Mg m⁻³
 D_m not measured

Data collection

Bruker P4 diffractometer
 ω scans
 Absorption correction: none
 5695 measured reflections
 4159 independent reflections
 3375 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 8192
 reflections
 $\theta = 2\text{--}27^\circ$
 $\mu = 0.089$ mm⁻¹
 $T = 223$ (2) K
 Rectangular
 $0.46 \times 0.45 \times 0.20$ mm
 Yellow

3882 reflections with
 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.08$
 $\theta_{\text{max}} = 27^\circ$
 $h = 0 \rightarrow 21$
 $k = 0 \rightarrow 19$
 $l = 0 \rightarrow 22$

$(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.156$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.234$ e Å⁻³
 Extinction correction: none
 Scattering factors from
*International Tables for
 Crystallography* (Vol. C)

Cu $K\alpha$ radiation
 $\lambda = 1.54178$ Å
 Cell parameters from 57
 reflections
 $\theta = 4.93\text{--}47.84^\circ$
 $\mu = 0.594$ mm⁻¹
 $T = 298$ (2) K
 Rectangular
 $0.45 \times 0.40 \times 0.10$ mm
 Colorless

$\theta_{\text{max}} = 67^\circ$
 $h = -19 \rightarrow 19$
 $k = 0 \rightarrow 21$
 $l = 0 \rightarrow 9$
 3 standard reflections
 every 97 reflections
 intensity decay: 1.8%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.124$
 $S = 1.062$
 4159 reflections
 326 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.4764P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.189 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.173 \text{ e } \text{\AA}^{-3}$
 Extinction correction:
SHELXTL (Sheldrick, 1998)
 Extinction coefficient:
 0.0067 (4)
 Scattering factors from
International Tables for Crystallography (Vol. C)

Data collections were carried out on Bruker P4 or Bruker CCD X-ray diffractometers. In the case of the CCD instrument, data were collected by the double-pass method. The first 50 frames of data were recollected at the end of data collection to monitor crystal decay. Corrections to the data for systematic errors were applied using *SADABS* (Blessing, 1995). H atoms were treated using appropriate riding models (*AFIX* = *m3* in *SHELXTL*).

For (2a), (2b) and (3d), data collection: *XSCANS* (Siemens, 1994). For (2c), data collection: *SMART* (Siemens, 1997). For (2a), (2b) and (3d), cell refinement: *XSCANS*. For (2c), cell refinement: *SAINT*. For (2a), (2b) and (3d), data reduction: *XSCANS*. For (2c), data reduction: *SAINT*. For all compounds, program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the Council of Scientific and Industrial Research (Document No. RRLT-PRU-78 from the Regional Research Laboratory CSIR, Trivandrum, India), Government of India (MM, MCS and MVG), Jawaharlal Nehru Centre for Advanced Scientific Research, Bangalore, India (MVG), and NSF (CHE-9101834), Missouri Research Board, Center for Molecular Electronics, Department of Chemistry and Research Awards Grant of the University of Missouri–St. Louis (NPR), for financial support of this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1047). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 1000–1003

Intermolecular N—H···O and C—H···O interactions form a two-dimensional network in (2*S*,4*S*,5*R*)-(–)-3,4-dimethyl-5-phenyl-2-(pyrrol-2-yl)-1,3-oxazolidine

JOHN F. GALLAGHER AND LAVELLE M. FITZSIMONS

School of Chemical Sciences, Dublin City University, Dublin 9, Ireland. E-mail: gallagherjfg@dcu.ie

(Received 26 November 1998; accepted 4 February 1999)

Abstract

The title compound, C₁₅H₁₈N₂O, prepared from (1*R*,2*S*)-(–)-ephedrine, crystallizes in space group *P1* with two molecules in the asymmetric unit. The oxazolidine rings of the two molecules adopt an envelope conformation, with the N atom 0.609 (6) and 0.623 (6) Å from the plane of the other four oxazolidine ring atoms. Intermolecular N_{pyrrole}—H···O and C_{phenyl}—H···O interactions generate a two-dimensional hydrogen-bonded network with N···O and C···O distances of 3.004 (4) and 3.051 (4) Å, respectively, and 3.599 (5) and 3.632 (5) Å, respectively, for the two independent hydrogen-bonding systems.