

Collection des données

Diffractomètre 4 cercles	$\theta_{\max} = 21^\circ$
Syntex Nicolet P2 ₁	$h = -12 \rightarrow 12$
Balayage θ -2 θ	$k = 0 \rightarrow 9$
Correction d'absorption: néant	$l = 0 \rightarrow 24$
2488 réflexions mesurées	3 réflexions de référence
2488 réflexions indépendantes	mesurées toutes les 50 réflexions
1155 réflexions observées	variation d'intensité: < 0,1%
$[I > 2\sigma(I)]$	

Affinement

Affinement basé sur les F	$(\Delta/\sigma)_{\max} = 0,48$
R = 0,056	$\Delta\rho_{\max} = 0,26 \text{ e } \text{\AA}^{-3}$
wR = 0,042	$\Delta\rho_{\min} = -0,25 \text{ e } \text{\AA}^{-3}$
S = 2,02	Correction d'extinction: néant
1155 réflexions	
175 paramètres	Facteurs de diffusion de
Les paramètres x, y, z des atomes d'hydrogène affinés	<i>International Tables for X-ray Crystallography</i> (1974, Tome IV)
$w = 1/\sigma^2(F)$	

Tableau 1. Coordonnées atomiques et facteurs d'agitation thermique isotropes équivalents (\AA^2)

$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$				
	x	y	z	B_{eq}
S	0,4375 (1)	0,1101 (2)	0,65617 (7)	4,02
N(1)	0,3873 (4)	-0,1499 (6)	0,5430 (2)	2,94
C(2)	0,3521 (5)	0,0305 (7)	0,5742 (2)	2,83
N(3)	0,2392 (4)	0,1369 (6)	0,5327 (2)	3,04
C(4)	0,1582 (5)	0,0769 (8)	0,4634 (2)	3,04
C(5)	0,1466 (5)	-0,1958 (8)	0,3603 (2)	3,01
C(6)	0,2029 (5)	-0,3751 (8)	0,3395 (3)	3,28
C(7)	0,3149 (5)	-0,4873 (8)	0,3866 (3)	3,22
O(8)	0,3663 (3)	-0,3974 (5)	0,4568 (2)	3,17
C(9)	0,3159 (5)	-0,2171 (7)	0,4758 (3)	2,67
C(10)	0,2046 (4)	-0,1136 (8)	0,4326 (2)	2,75
O(40)	0,0575 (4)	0,1860 (5)	0,4346 (2)	4,66
C(50)	0,0294 (6)	-0,086 (1)	0,3073 (3)	4,45
O(70)	0,3706 (4)	-0,6472 (5)	0,3743 (2)	4,12
C(100)	-0,1946 (7)	0,432 (1)	0,3389 (4)	5,25
O(110)	-0,1494 (4)	0,4929 (6)	0,4146 (2)	4,47

Tableau 2. Paramètres géométriques (\AA , $^\circ$)

S—C(2)	1,639 (5)	C(5)—C(10)	1,429 (6)
N(1)—C(2)	1,367 (5)	C(5)—C(50)	1,498 (7)
N(1)—C(9)	1,352 (5)	C(6)—C(7)	1,425 (6)
N(3)—C(2)	1,364 (5)	C(7)—O(8)	1,404 (5)
N(3)—C(4)	1,401 (5)	C(7)—O(70)	1,198 (5)
C(4)—O(40)	1,214 (5)	O(8)—C(9)	1,330 (5)
C(4)—C(10)	1,452 (6)	C(9)—C(10)	1,359 (5)
C(5)—C(6)	1,355 (6)	C(100)—O(110)	1,414 (7)
C(2)—N(1)—C(9)	122,4 (4)	C(5)—C(6)—C(7)	123,4 (5)
N(1)—C(2)—S	122,2 (3)	O(70)—C(7)—C(6)	128,7 (5)
N(3)—C(2)—S	122,8 (4)	O(70)—C(7)—O(8)	116,0 (4)
N(1)—C(2)—N(3)	114,9 (4)	C(6)—C(7)—O(8)	115,2 (4)
C(2)—N(3)—C(4)	126,3 (4)	C(9)—O(8)—C(7)	121,4 (4)
O(40)—C(4)—N(3)	118,2 (5)	O(8)—C(9)—N(1)	112,3 (4)
O(40)—C(4)—C(10)	126,1 (4)	O(8)—C(9)—C(10)	123,6 (4)
N(3)—C(4)—C(10)	115,7 (4)	N(1)—C(9)—C(10)	124,1 (5)
C(6)—C(5)—C(10)	118,2 (4)	C(9)—C(10)—C(5)	118,0 (5)
C(6)—C(5)—C(50)	120,2 (5)	C(9)—C(10)—C(4)	116,5 (4)
C(10)—C(5)—C(50)	121,6 (5)	C(5)—C(10)—C(4)	125,5 (4)

Enregistrement des intensités: toutes les intensités ont été corrigées des effets de Lorentz et de polarisation. La structure a

été résolue à l'aide des méthodes directes (*MULTAN88*; Debaerdemaeker *et al.*, 1988). L'affinement des paramètres x, y, z, B pour les atomes non-hydrogènes et x, y, z pour les atomes d'hydrogène, a été effectué à l'aide du programme *ORXFLS* (Busing, 1971). Les dessins de la structure ont été obtenus à l'aide du programme *ORTEPII* (Johnson, 1976). Les angles dièdres ont été calculés à l'aide du programme *BEST PLANES* (Ito & Sugawara, 1983).

Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances et angles des atomes d'hydrogène, des plans moyens et des angles de torsion ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 71241: 12 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: PA1040]

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Synthesis and Structure of *trans*-1,2,3-Tris(3-thenoyl)cyclopropane

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Abstract

The title cyclopropane derivative, C₁₈H₁₂O₃S₃, was synthesized and its structure investigated by single-crystal X-ray diffraction and spectroscopic methods. This structure analysis addresses the problem of spontaneously resolved helical stereoisomerism. The temperature-dependent ¹H NMR spectrum shows a relevant barrier to rotation about the CO–thiophene bonds, with $\Delta G_{423} \approx 89$ (21) kJ mol⁻¹.

Comment

Recently, a convenient synthesis of 1,2,3-tris(aryl)cyclopropanes (1) by the one-step cyclotrimerization of 2-bromoacetylarenes and heteroarenes was reported (Saba, 1990). The unequivocal steric course of the reaction, with regard to the 1,2-*trans* substitution in the cyclopropane ring of (1), has been derived by crystal structure analysis and confirmed by NMR analysis.



We report herein the synthesis and structure determination of *trans*-1,2,3-tris(3-thienyl)cyclopropane (2), which presents an unusual case of spontaneously resolved helical stereoisomerism. The configuration of one of the enantiomers is resolved by the present structure analysis. Moreover, the temperature-dependent ¹H NMR spectra in Me₂SO confirm the hindered rotation around the bonds between the carbonyl and thienyl groups at room temperature.

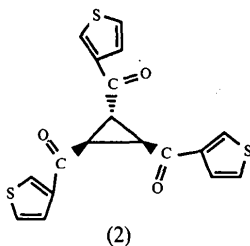


Fig. 1 shows a molecule of the title compound and its numbering scheme. The assumed enantiomer is that with the lowest *R* values, *R* = 0.054 and *wR* = 0.067, while the opposite enantiomer gives *R* = 0.058 and *wR* = 0.072. The orientations of the carbonyl groups with respect to cyclopropane are defined by the torsion angles O1—C5—C6—C8 19 (1), O2—C9—C7—C6 -17 (1) and O3—C14—C8—C7 -31 (1)°; the torsion angles about the CO-thiophene bonds are O1—C5—C4—C3 172.4 (9), O2—C9—C10—C13 -162.0 (9) and O3—C14—C15—C16 -179.5 (7)°. The S1, S2 and S3 heterocyclic rings are inclined at dihedral angles of 80.7 (5), 61.9 (6) and 54.8 (7)°, respectively, to the C6—C7—C8 plane. The dihedral angles between the carbonyl planes and the linked thiophene rings are 6.1 (3) and 1.3 (4)° for the rings containing S1, S2 and S3, respectively.

Fig. 2 shows the ¹H NMR (300 MHz) spectra of (2) in deuterated Me₂SO at various temperatures. As the sample was warmed, an upfield shift of two extra deshielded signals, due to single protons near sulfur in the thiophene rings, was observed. The signals exhibited by the neighbouring protons at 7.70 (*dd*), 7.57 (*m*) and 7.60 (*m*), 7.47 (*dd*) p.p.m. collapse to two singlets at 7.64 and

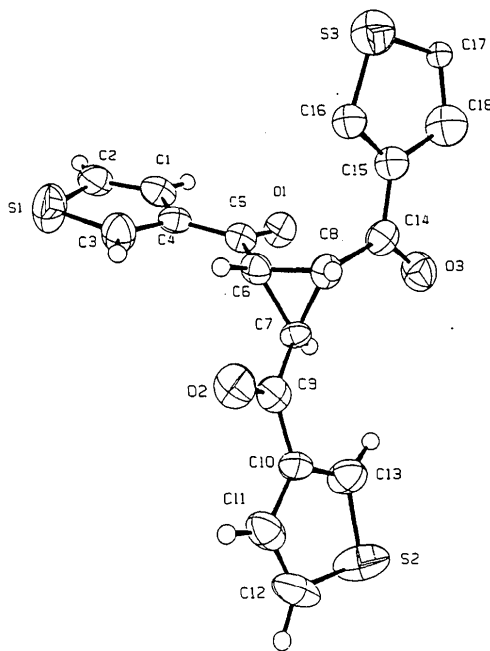


Fig. 1. ORTEP (Johnson, 1965) view of the molecule in its assumed absolute configuration showing the atomic numbering scheme. Ellipsoids are at the 50% probability level.

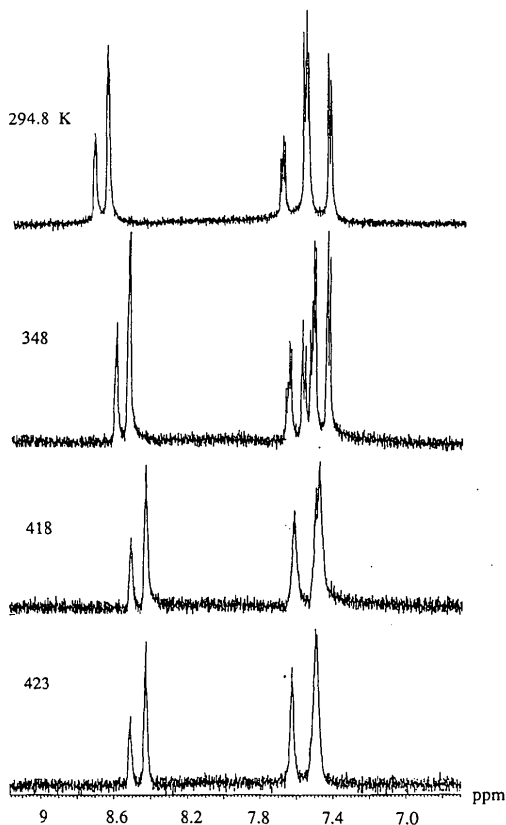


Fig. 2. Temperature-dependent ¹H (300 MHz) NMR spectrum of the aromatic protons.

7.52 p.p.m., respectively, at 528 K through a coalescence temperature of ca 423 K. The free-energy value at this temperature (ΔG_{423}) was calculated (Abraham, Fischer & Loftus, 1988) to be ca 88.8 (21.2) kJ mol⁻¹. With respect to the cyclopropane protons, no temperature dependence of the shift in signals was observed.

Experimental

Crystal data

C₁₈H₁₂O₃S₃

$M_r = 372.47$

Orthorhombic

*Pna*2₁

$a = 14.645$ (2) Å

$b = 20.891$ (5) Å

$c = 5.404$ (2) Å

$V = 1653.3$ (8) Å³

$Z = 4$

$D_x = 1.496$ Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.5418$ Å

Cell parameters from 10 reflections

$\theta = 23.2$ – 26.5°

$\mu = 4.221$ mm⁻¹

$T = 296$ K

Needles

$0.30 \times 0.05 \times 0.02$ mm

Colourless

Data collection

Rigaku AFC-5R diffractometer

ω - 2θ scans

Absorption correction:

empirical (azimuthal scan)

$T_{\min} = 0.84$, $T_{\max} = 1.00$

2852 measured reflections

1490 independent reflections

1032 observed reflections

$[I > 3\sigma(I)]$

$R_{\text{int}} = 0.051$

$\theta_{\text{max}} = 60.1^\circ$

$h = -16 \rightarrow 16$

$k = 0 \rightarrow 23$

$l = 0 \rightarrow 6$

3 standard reflections

monitored every 150

reflections

intensity variation: none

Refinement

Refinement on F^2

$R = 0.054$

$wR = 0.067$

$S = 1.96$

1032 reflections

190 parameters

All H-atom parameters

refined

$w = 4F_o^2/\sigma^2(F_o^2)$

$\Delta\rho_{\text{max}} = 0.41$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography* [1974, Vol. IV, Tables

2.2A and 2.3.1 (C, O, S)

and 2.2C (H)]

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

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

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
S1	0.5514 (2)	0.5878 (1)	0.7909	0.081 (1)
S2	0.1723 (2)	0.2283 (1)	1.1851 (9)	0.082 (1)
S3†	0.0616 (4)	0.7070 (2)	0.851 (1)	0.051 (2)
S23†	-0.0010 (10)	0.7044 (3)	1.162 (2)	0.061 (2)
O1	0.2744 (4)	0.5592 (3)	1.274 (1)	0.059 (2)
O2	0.2486 (5)	0.3956 (3)	0.595 (1)	0.055 (3)
O3	0.0720 (4)	0.5013 (3)	1.286 (1)	0.057 (3)
C1	0.4521 (6)	0.6108 (4)	1.157 (2)	0.057 (3)
C2	0.5363 (6)	0.6263 (4)	1.066 (2)	0.040 (3)
C3	0.4449 (6)	0.5539 (4)	0.796 (2)	0.041 (3)
C4	0.3976 (5)	0.5700 (3)	1.003 (2)	0.039 (2)
C5	0.3066 (6)	0.5469 (4)	1.073 (2)	0.034 (2)
C6	0.2564 (5)	0.5072 (3)	0.890 (2)	0.040 (3)
C7	0.2176 (5)	0.4445 (3)	0.983 (2)	0.047 (3)

C8	0.1559 (5)	0.4971 (3)	0.906 (2)	0.045 (3)
C9	0.2298 (5)	0.3884 (4)	0.814 (2)	0.058 (3)
C10	0.2208 (6)	0.3225 (3)	0.921 (2)	0.069 (4)
C11	0.2499 (5)	0.2680 (4)	0.794 (2)	0.056 (3)
C12	0.2303 (7)	0.2126 (4)	0.914 (2)	0.044 (3)
C13	0.1780 (6)	0.3084 (4)	1.138 (2)	0.071 (6)
C14	0.0987 (5)	0.5301 (4)	1.102 (2)	0.039 (5)
C15	0.0727 (4)	0.5972 (2)	1.055 (1)	0.045 (2)
C16	0.1017 (5)	0.6292 (2)	0.850 (1)	0.044
C17	0.0041 (8)	0.6990 (3)	1.129 (2)	0.026 (4)
C18	0.0122 (8)	0.6370 (3)	1.236 (2)	0.071 (6)
C25	0.0727	0.5972	1.0552	0.045
C26	0.0160 (10)	0.6240 (3)	1.225 (2)	0.022 (6)
C27	0.0690 (10)	0.7045 (3)	0.905 (3)	0.040 (10)
C28	0.1090 (10)	0.6437 (4)	0.852 (3)	0.070 (10)

† S3 (C15–C18) and S23 (C25–C28) are two alternative conformations of the ring (see text).

Table 2. Selected geometric parameters (Å, °)

S1–C2	1.70 (1)	C10–C13	1.36 (1)
S1–C3	1.713 (9)	C11–C12	1.36 (1)
S2–C12	1.72 (1)	C14–C15	1.47 (0)
O1–C5	1.21 (1)	C14–C25	1.474 (8)
O2–C9	1.22 (1)	C15–C16	1.362 (8)
O3–C14	1.23 (1)	C15–C18	1.56 (1)
C1–C2	1.37 (1)	C15–C26	1.36 (1)
C1–C4	1.43 (1)	C15–C28	1.56 (1)
C3–C4	1.36 (1)	C16–C25	1.363 (5)
C4–C5	1.47 (1)	C16–C27	1.671 (9)
C5–C6	1.49 (1)	C17–C18	1.42 (1)
C6–C7	1.51 (1)	C17–C26	1.66 (1)
C6–C8	1.49 (1)	C17–C27	1.54 (1)
C7–C8	1.48 (1)	C18–C25	1.56 (1)
C7–C9	1.50 (1)	C25–C26	1.36 (1)
C8–C14	1.52 (1)	C25–C28	1.56 (1)
C9–C10	1.50 (1)	C27–C28	1.43 (1)
C10–C11	1.40 (1)		
C2–S1–C3	93.6 (4)	C16–C15–C26	122.6 (7)
C12–S2–C13	92.1 (4)	C16–C15–C18	115.2 (6)
C2–C1–C4	115.7 (8)	C15–C16–C28	123 (2)
S1–C2–C1	108.6 (7)	C15–C16–C27	103.2 (4)
S1–C3–C4	112.0 (6)	C15–C16–C25	0.0 (2)
C1–C4–C3	110.0 (8)	C27–C16–C28	37 (2)
C3–C4–C5	126.5 (8)	C25–C16–C28	123 (2)
C1–C4–C5	123.5 (7)	C25–C16–C27	103.2 (4)
O1–C5–C4	121.0 (8)	C26–C17–C27	104.5 (6)
C4–C5–C6	117.5 (7)	C18–C17–C27	109.6 (7)
O1–C5–C6	121.5 (9)	C18–C17–C26	5.8 (3)
C5–C6–C8	122.0 (7)	C15–C18–C17	106.1 (5)
C5–C6–C7	116.6 (6)	C17–C18–C26	143 (2)
C7–C6–C8	59.2 (5)	C17–C18–C25	106.1 (5)
C6–C7–C9	115.5 (6)	C15–C18–C26	41 (2)
C6–C7–C8	59.6 (4)	C15–C18–C25	0.0 (2)
C8–C7–C9	118.8 (6)	C25–C18–C26	41 (2)
C6–C8–C7	61.2 (5)	C16–C25–C18	115.2 (4)
C7–C8–C14	118.5 (6)	C15–C25–C18	129 (8)
C6–C8–C14	121.5 (7)	C14–C25–C18	123.1 (4)
O2–C9–C7	121.4 (9)	C14–C25–C16	121.7 (4)
C7–C9–C10	118.3 (6)	C14–C25–C15	99.9 (4)
O2–C9–C10	120.4 (8)	C18–C25–C28	107.6 (6)
C9–C10–C13	124.8 (8)	C18–C25–C26	7.9 (5)
C9–C10–C11	122.2 (6)	C16–C25–C28	10.0 (5)
C11–C10–C13	112.8 (9)	C16–C25–C26	122.6 (5)
C10–C11–C12	113.3 (7)	C15–C25–C28	45 (4)
S2–C12–C11	110.3 (8)	C15–C25–C26	133 (3)
S2–C13–C10	111.4 (6)	C14–C25–C28	128.7 (5)
O3–C14–C8	121.3 (8)	C14–C25–C26	115.7 (4)
C8–C14–C25	117.1 (6)	C26–C25–C28	115.3 (7)
C8–C14–C15	117.0 (6)	C18–C26–C25	131 (2)
O3–C14–C25	121.6 (7)	C17–C26–C25	104.0 (5)
O3–C14–C15	121.6 (8)	C17–C26–C18	31 (2)
C15–C14–C25	0.0 (2)	C15–C26–C25	0.0 (2)
C14–C15–C28	128.7 (6)	C15–C26–C18	131 (2)
C14–C15–C26	115.7 (5)	C15–C26–C17	104.0 (5)
C14–C15–C25	80.1 (4)	C16–C27–C17	104.2 (6)

C14—C15—C18	123.1 (5)	C17—C27—C28	110.1 (9)
C14—C15—C16	121.8 (5)	C16—C27—C28	7.8 (4)
C26—C15—C28	115.3 (8)	C25—C28—C27	105.8 (6)
C25—C15—C28	135 (4)	C16—C28—C27	135 (2)
C25—C15—C26	47 (3)	C16—C28—C25	47 (2)
C18—C15—C28	107.6 (7)	C15—C28—C27	105.9 (6)
C18—C15—C26	7.9 (5)	C15—C28—C25	0.0 (2)
C18—C15—C25	51 (7)	C15—C28—C16	47 (2)
C16—C15—C28	10.0 (5)		

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Saba, A. (1990). *J. Chem. Res. (S)*, pp. 288–289.

All reagents and solvents were of reagent grade and used without further purification. (2) was obtained as colourless needles, m.p. 469–470 K (crystallized from isobutyl methyl ketone) in 91% yield by treatment of 10 mmol of 3-(bromoacetyl)thiophene (McDowell & Greenwood, 1965) with 6 mmol of K₂CO₃ in DMF at room temperature for 6 h, water quenching, neutralization with 10% HCl and filtration. Analysis: calculated for C₁₈H₁₂O₃S₃ C 58.03, H 3.22, S 25.82%; found C 58.32, H 3.11, S 25.56%.

¹H (300 MHz) and ¹³C (75 MHz) NMR spectra of (2) were recorded on a Varian VXR-300 spectrometer, with CDCl₃ or Me₂SO as solvents and TMS (tetramethylsilane) as the internal standard. ¹H NMR (CDCl₃, 300 MHz) δ: 8.42 (1H, *dd*, *J* = 1.2, 3 Hz), 8.14 (2H, *dd*, *J* = 1.2, 3 Hz), 7.68 (1H, *dd*, *J* = 1.2, 5.1 Hz), 7.54 (2H, *dd*, *J* = 1.2, 5.1 Hz), 7.38 (1H, *dd*, *J* = 1.2, 5.1 Hz), 7.28 (2H, *dd*, *J* = 3, 5.1 Hz), 4.04 (1H, *t*, *J* = 5.7 Hz), 3.58 (2H, *d*, *J* = 5.7 Hz). ¹³C NMR (CDCl₃, 75 MHz): 189.7, 186.7, 141.8, 141.6, 134.2, 132.9, 126.9, 126.8, 126.7, 126.6, 36.6, 30.9.

Intensity data were corrected for Lorentz and polarization effects. H atoms were included in the structure-factor calculations in idealized positions (C—H = 0.95 Å), and were assigned isotropic displacement parameters 20% greater than the *U*_{eq} value of the atom to which they were bonded. Because the S3-thiophene ring in the molecule is disordered, the C₄S five-membered rigid-body rings were generated based on the average geometry of the other two thiophene rings, which were located from difference Fourier maps and refined by full-matrix least squares. The rigid-body rings were refined isotropically with populations of 0.65 (1) for the ring containing S3 and 0.35 (1) for the ring containing S23. H atoms were not included for the rigid-body rings owing to problems with disorder in the refinements. Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1985). Molecular graphics: *ORTEP* (Johnson, 1965). Anomalous-scattering coefficients were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71592 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: L11065]

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Structure of a Model for the Aranorosin Nucleus

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Abstract

The structure of a synthetic model system, 2-hydroxy-6,7,9,10-*cis,cis*-diepoxy-1-oxaspiro[4.5]decan-8-one, C₉H₁₀O₅, for the spirocyclic headgroup of the natural product aranorosin has been determined and shown to possess the natural product stereochemistry. Two crystallographically independent molecules cocrystallize in a centrosymmetric space group. The *syn* arrangement of the diepoxides and the lactol O atom about the cyclohexanone ring has been confirmed in both molecules. The cyclohexanone ring adopts a boat conformation with the carbonyl O atom *anti* to the lactol O atom.

Comment

The recently isolated natural product aranorosin (1) displays antibiotic, antifungal and antitumor properties (Roy, Mukhopadhyay, Reddy, Desikan, Rupp & Ganguli, 1988; Felhaber, Kogler, Mukhopadhyay, Vijayakumar, Roy, Rupp & Ganguli, 1988; Felhaber, Kogler, Mukhopadhyay, Vijayakumar & Ganguli, 1988). The novel spirocyclic cyclohexanone diepoxide moiety of this molecule provides a particularly challenging synthetic target. This challenge, as well as the biological properties of aranorosin (1), have stimulated interest in its synthesis (Rama Rao, Gurjar & Sharma, 1991) and a highly stereoselective route to the model aranorosin nucleus (2) has