C2	0.1203 (2)	0.1948 (2)	0.81824 (9)	0.0329 (4)
C3	0.2730 (2)	0.2522 (3)	0.81227 (11)	0.0438 (5)
C4	0.3746 (3)	0.2436 (3)	0.87460 (14)	0.0559 (6)
C5	0.3206 (3)	0.1802 (3)	0.94295 (12)	0.0542 (6)
C6	0.1690 (3)	0.1259 (2)	0.94869 (10)	0.0497 (5)
C7	0.0633 (3)	0.1303 (2)	0.88655 (9)	0.0383 (4)
C8	−0.1016 (3)	0.0665 (3)	0.89237 (13)	0.0505 (5)
C9	−0.1112 (2)	0.0771 (2)	0.56451 (10)	0.0344 (4)
C10	−0.0462 (3)	0.1711 (3)	0.49793 (11)	0.0436 (5)
C11	−0.2299 (2)	0.2700 (2)	0.65445 (10)	0.0352 (4)
C12	−0.3668 (3)	0.1924 (4)	0.6958 (2)	0.0578 (6)

Table 4. *Selected geometric parameters (Å, °) for DETTU*

S—C1	1.702 (2)	C3—C4	1.385 (3)
N1—C1	1.358 (2)	C4—C5	1.384 (4)
N1—C2	1.432 (2)	C5—C6	1.363 (4)
N2—C1	1.344 (2)	C6—C7	1.403 (3)
N2—C11	1.467 (2)	C7—C8	1.496 (3)
N2—C9	1.470 (2)	C9—C10	1.511 (3)
C2—C3	1.381 (3)	C11—C12	1.512 (3)
C2—C7	1.395 (2)		
C1—N1—C2	126.24 (14)	C2—C3—C4	120.0 (2)
C1—N2—C11	123.43 (13)	C5—C4—C3	119.4 (2)
C1—N2—C9	121.51 (14)	C6—C5—C4	120.2 (2)
C11—N2—C9	115.02 (13)	C5—C6—C7	122.1 (2)
N2—C1—N1	115.63 (14)	C2—C7—C6	116.7 (2)
N2—C1—S	123.07 (12)	C2—C7—C8	121.3 (2)
N1—C1—S	121.30 (12)	C6—C7—C8	121.9 (2)
C3—C2—C7	121.6 (2)	N2—C9—C10	112.69 (15)
C3—C2—N1	120.4 (2)	N2—C11—C12	113.3 (2)
C7—C2—N1	117.9 (2)		

The two title structures were solved by direct methods and refined by a full-matrix least-squares technique. All H atoms in both structures were located from difference maps and refined isotropically.

For both compounds, data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990*a*); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*); software used to prepare material for publication: *SHELXL*93. Geometric calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1206). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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αββα-3,4-Dimethyl-2,5-bis(3,4,5-trimethoxyphenyl)tetrahydrofuran

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Abstract

The crystal structure determination of the title compound, $C_{24}H_{32}O_7$, a second isomer of the natural 2,5-bisaryltetrahydrofuran lignans, reveals that the molecule adopts an αββα configuration. The tetrahydrofuran ring is in an intermediate conformation between half-chair and envelope.

Comment

There are ten possible stereoisomers for natural 2,5-bisaryltetrahydrofuran lignans, (I), consisting of four diastereomeric pairs and two *meso* forms (Biftu & Stevenson, 1987). Although these isomers are found in nature, only the crystal structure of (−)-grandisin, an αβαβ isomer, has been reported (Saad, Soepadmo, Fang, McLaughlin & Fanwick, 1991). We have now determined the crystal structure of one of the *meso* isomers, (II), a compound isolated from the roots of *Polyalthia bullata* (*Annonaceae*), a medicinal plant of Malaysia used for various ailments (Burkhill, 1966). The chemical structure was verified by its melting point, NMR and mass spectral data, which are similar to those of a compound isolated earlier from *Aristolochia birotris* (Conserva, Da Silva & Filho, 1990) and also to those of the synthetic compound (Biftu, Hazra & Stevenson, 1979).

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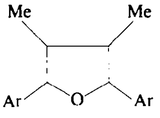
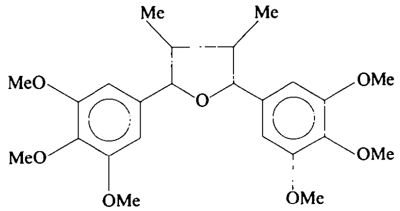


(I)



(II)

The title compound, (II), has been found to have the $\alpha\beta\beta\alpha$ configuration (Fig. 1). Bond lengths for the trimethoxyphenyl groups are comparable with those observed in (−)-grandisin. The C—O bond lengths [C7—O4 1.426 (2), C10—O4 1.431 (2) Å] of the furan ring are also in good agreement with those of (−)-grandisin, whereas the ring C—C bond lengths are slightly longer. Two of the three methoxy groups attached to each phenyl ring (attached to the *meta* positions) lie close to the ring plane whereas the methyl C atom of the *para* substituent is well away from that plane. The dihedral angle between the planes of the two phenyl rings is 139.25 (6)°.

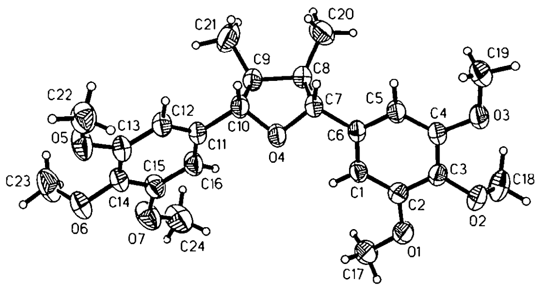


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title molecule with the numbering scheme, showing 50% probability displacement ellipsoids.

In (−)-grandisin the tetrahydrofuran five-membered ring adopts a half-chair conformation with a twofold axis passing through the O atom, whereas in the title compound an intermediate conformation between a half-chair and envelope is observed [asymmetry parameters $\Delta S(\mathrm{O4})$ = 0.039 (1), $\Delta 2(\mathrm{C8})$ = 0.052 (1) (Nardelli, 1983*a*)].

There are several possible C—H···O hydrogen bonds in this structure, of which the most significant are: C18···O6$^{\mathrm{i}}$ 3.355 (3) Å, C18—H18*A*···O6$^{\mathrm{i}}$ 143 (2)°; C19···O5$^{\mathrm{ii}}$ 3.446 (3) Å, C19—H19*A*···O5$^{\mathrm{ii}}$ 145 (2)°; C19···O3$^{\mathrm{iii}}$ 3.258 (4) Å, C19—H19*B*···O3$^{\mathrm{iii}}$ 128 (2)°; symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $\frac{3}{2} - x, -y, z - \frac{1}{2}$; (iii) $\frac{3}{2} - x, y - \frac{1}{2}, z$.

Experimental

The title compound [CA Reg. No. 50394-05-3] was obtained by column chromatography using silica gel (230–400 mesh) and eluted with hexane–ethyl acetate (4:1), forming colourless crystals on standing.

Crystal data

$C_{24}H_{32}O_7$
M_r = 432.50
Orthorhombic
Pbca
a = 20.660 (2) Å
b = 7.706 (1) Å
c = 29.217 (4) Å
V = 4651.5 (10) Å^3
Z = 8
D_x = 1.235 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 29 reflections
θ = 7–12.5°
μ = 0.090 mm^{-1}
T = 293 (2) K
Transparent block
0.60 × 0.56 × 0.38 mm
Colourless

Data collection

Siemens *P*4 four-circle diffractometer
$\theta/2\theta$ scans
Absorption correction: none
6550 measured reflections
5260 independent reflections
3238 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0303
θ_{max} = 27.49°
$h = -1 \rightarrow 26$
$k = -1 \rightarrow 10$
$l = -1 \rightarrow 37$
3 standard reflections monitored every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0470
$wR(F^2)$ = 0.2237
S = 0.935
5244 reflections
408 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0998P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\mathrm{max}}$ = 0.001
$\Delta\rho_{\mathrm{max}}$ = 0.18 e Å^{-3}
$\Delta\rho_{\mathrm{min}}$ = −0.22 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.97104 (7)	0.2728 (2)	0.49732 (5)	0.0554 (4)
O2	0.90239 (7)	0.2480 (2)	0.42064 (5)	0.0513 (4)
O3	0.80746 (7)	0.0150 (2)	0.41128 (4)	0.0510 (4)
O4	0.84441 (7)	−0.0991 (2)	0.61338 (4)	0.0475 (3)
O5	0.77155 (7)	−0.1271 (2)	0.80590 (5)	0.0665 (5)
O6	0.88882 (8)	−0.0079 (2)	0.82564 (4)	0.0584 (4)
O7	0.98384 (7)	−0.0072 (3)	0.76424 (5)	0.0731 (5)
C1	0.90635 (9)	0.0476 (2)	0.53462 (6)	0.0428 (4)
C2	0.92302 (9)	0.1509 (2)	0.49740 (6)	0.0422 (4)
C3	0.88859 (9)	0.1378 (2)	0.45646 (6)	0.0413 (4)
C4	0.83773 (9)	0.0197 (2)	0.45280 (6)	0.0406 (4)
C5	0.82086 (10)	−0.0821 (3)	0.49020 (6)	0.0421 (4)
C6	0.85505 (9)	−0.0682 (2)	0.53106 (6)	0.0396 (4)
C7	0.83544 (9)	−0.1827 (3)	0.57030 (6)	0.0411 (4)
C8	0.87193 (10)	−0.3578 (2)	0.57405 (6)	0.0431 (4)
C9	0.87468 (11)	−0.3883 (3)	0.62669 (6)	0.0472 (5)
C10	0.83456 (10)	−0.2367 (3)	0.64553 (6)	0.0441 (4)
C11	0.84990 (9)	−0.1757 (3)	0.69346 (6)	0.0433 (4)
C12	0.80191 (10)	−0.1818 (3)	0.72652 (6)	0.0457 (5)

C13	0.81515 (10)	−0.1281 (3)	0.77101 (6)	0.0463 (5)
C14	0.87689 (10)	−0.0704 (3)	0.78221 (6)	0.0458 (4)
C15	0.92491 (10)	−0.0651 (3)	0.74900 (7)	0.0500 (5)
C16	0.91160 (11)	−0.1171 (3)	0.70461 (7)	0.0517 (5)
C17	1.00865 (12)	0.2936 (3)	0.53767 (9)	0.0576 (6)
C18	0.94625 (14)	0.1770 (4)	0.38825 (9)	0.0663 (7)
C19	0.75146 (15)	−0.0931 (4)	0.40725 (9)	0.0682 (7)
C20	0.8398 (2)	−0.4960 (3)	0.54555 (10)	0.0679 (7)
C21	0.8513 (3)	−0.5626 (4)	0.64410 (11)	0.0915 (11)
C22	0.70538 (12)	−0.1482 (5)	0.79541 (10)	0.0672 (7)
C23	0.9065 (2)	−0.1339 (5)	0.85823 (9)	0.0846 (9)
C24	1.03674 (13)	−0.0203 (5)	0.73362 (12)	0.0824 (9)

Table 2. *Selected geometric parameters (Å, °)*

O1—C2	1.367 (2)	C3—C4	1.394 (3)
O1—C17	1.421 (3)	C4—C5	1.390 (3)
O2—C3	1.377 (2)	C5—C6	1.391 (3)
O2—C18	1.420 (3)	C6—C7	1.503 (2)
O3—C4	1.365 (2)	C7—C8	1.549 (3)
O3—C19	1.431 (3)	C8—C20	1.506 (3)
O4—C7	1.426 (2)	C8—C9	1.557 (3)
O4—C10	1.431 (2)	C9—C21	1.515 (3)
O5—C13	1.360 (2)	C9—C10	1.534 (3)
O5—C22	1.410 (3)	C10—C11	1.511 (2)
O6—C14	1.380 (2)	C11—C12	1.385 (3)
O6—C23	1.408 (3)	C11—C16	1.391 (3)
O7—C15	1.371 (2)	C12—C13	1.391 (3)
O7—C24	1.416 (3)	C13—C14	1.390 (3)
C1—C6	1.390 (3)	C14—C15	1.388 (3)
C1—C2	1.391 (3)	C15—C16	1.385 (3)
C2—C3	1.395 (3)		
C2—O1—C17	118.2 (2)	C20—C8—C7	111.2 (2)
C3—O2—C18	113.7 (2)	C20—C8—C9	117.1 (2)
C4—O3—C19	117.3 (2)	C7—C8—C9	102.65 (15)
C7—O4—C10	103.06 (14)	C21—C9—C10	112.5 (2)
C13—O5—C22	118.6 (2)	C21—C9—C8	117.0 (2)
C14—O6—C23	115.3 (2)	C10—C9—C8	102.70 (15)
C15—O7—C24	117.2 (2)	O4—C10—C11	110.3 (2)
C6—C1—C2	119.9 (2)	O4—C10—C9	104.59 (14)
O1—C2—C1	125.1 (2)	C11—C10—C9	117.1 (2)
O1—C2—C3	114.7 (2)	C12—C11—C16	120.2 (2)
C1—C2—C3	120.2 (2)	C12—C11—C10	119.1 (2)
O2—C3—C4	120.0 (2)	C16—C11—C10	120.7 (2)
O2—C3—C2	120.1 (2)	C11—C12—C13	120.0 (2)
C4—C3—C2	119.8 (2)	O5—C13—C14	115.4 (2)
O3—C4—C5	124.6 (2)	O5—C13—C12	124.9 (2)
O3—C4—C3	115.5 (2)	C14—C13—C12	119.7 (2)
C5—C4—C3	119.8 (2)	O6—C14—C15	120.3 (2)
C4—C5—C6	120.3 (2)	O6—C14—C13	119.5 (2)
C1—C6—C5	120.1 (2)	C15—C14—C13	120.0 (2)
C1—C6—C7	121.7 (2)	O7—C15—C16	125.1 (2)
C5—C6—C7	118.2 (2)	O7—C15—C14	114.6 (2)
O4—C7—C6	111.9 (2)	C16—C15—C14	120.3 (2)
O4—C7—C8	105.54 (14)	C15—C16—C11	119.7 (2)
C6—C7—C8	115.8 (2)		

The structure was solved by direct methods and refined by the full-matrix least-squares method. All H atoms were located from difference Fourier maps and were refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93. Geometrical calculations: *PARST* (Nardelli, 1983*b*).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 123-3417-2201. One of the authors (KS) thanks Universiti Sains Malaysia for a Visiting Postdoctoral Research Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1215). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl 3-*O*-Benzoyl-4,6-*O*-benzylidene-2-*O*-trifluoromethylsulfonyl-α-D-mannopyranoside

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(*Received 30 June 1995; accepted 4 September 1995*)

Abstract

The title compound, $C_{22}H_{21}F_3O_9S$, is of interest as a potential precursor of 2-azido-2-deoxy-D-glucopyranose derivatives. The conformation of the molecule is as expected.

Comment

In connection with the synthesis of glycosyl-phosphatidylinositols (Cottaz, Brimacombe & Ferguson, 1993),

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