indicating that both rings have approximately the $_3E$ (C3′-*exo*) conformation ($P = 198°$) with (1) distorted towards ^3_2T and (2) towards ^3_4T. The sugar conformations of the present compounds, which lack oxygen substituents at both C2′ and C3′, are in the range which Sundaralingam (1975) has indicated as usual for 2′-monodeoxyribofuranoses. The glycosidic bonds, C1′—N9 in (1) and C1′—N1 in (2), have χ torsion angles of 264·1 (2) and 203·2 (2)°, respectively, and both molecular conformations are *anti* although that of (1) is on the borderline with the *syn* conformation ($\chi = 270°$). The C1′—O4′ bond is significantly shorter than the C4′—O4′ bond in conformity with the observations of Bugg, Thomas, Sundaralingam & Rao (1971). In general, other molecular dimensions in both molecules are comparable with similar compounds.

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Structure of 1,5-Bis(α-isopropyl-3,4-dimethoxybenzyl)-2,4-dimethoxybenzene

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Abstract. $C_{32}H_{42}O_6$, $M_r = 522·7$, monoclinic, $P2_1/c$, $a = 13·196$ (1), $b = 13·692$ (2), $c = 16·770$ (2) Å, $\beta = 104·45$ (1)°, $V = 2934·0$ (6) Å^3, $Z = 4$, $D_x = 1·18$ g cm^{-3}, $F(000) = 1128$, $T = 295$ K, $R = 0·052$ and $wR = 0·059$ for 2107 unique observed reflections (Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å). This isomer is defined as *anti*, having the terminal benzyl rings on opposite sides with respect to the central benzene ring.

Introduction. The synthesis and physicochemical characterization of the title compound is a continuation of our investigations on molecules containing dimethoxy-substituted aromatic rings (Bruno, Bombieri, Rizzardi, Polizzotti & Natoli, 1983; Natoli, Agozzino, Ceraulo & Lamartina, 1983). These compounds are of particular interest because dimethoxy aromatic moieties are frequently present in synthetic and natural drugs.

By reacting 1,3-dimethoxybenzene (1) with 1-(3,4-dimethoxyphenyl)-2-methyl-1-propanol (2) in acid medium, different conformers due to the allowed rotation about C(sp^3)—C(sp^2) single bonds could be obtained as with the 1,5-bis(α-isopropyl-2,4-dimethoxybenzyl)-2,4-dimethoxybenzene derivative (Natoli, Agozzino, Ceraulo & Lamartina, 1982). In the present synthesis carried out in identical conditions only

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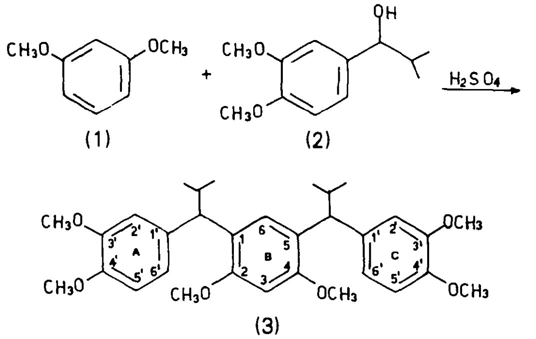
one conformer (3) was obtained, as white crystals from EtOH, m.p. 413 K (see scheme).



The mass spectrum showed the molecular ion peak at *m*/*z* 522 (12) and evidenced, as main fragmentation route, the loss of isopropyl radical (ion *a*), followed by the formation of the dimethoxybenzylic cation *b* through hydrogen transferring from the '*ortho*' methoxy group of the central ring. The subsequent losses of two isopropyl radicals from the doubly-charged molecular ion resulting in the ion at *m*/*z* 218 (35) were also observed (Natoli *et al.*, 1982).

The ^{1}H NMR spectrum in $CDCl_3$ solution, using TMS as internal standard, showed signals for isopropyl and methoxy groups at high fields. The analysis of the aromatic region revealed a multiplet centered at $\delta 6{\cdot}80$ integrated for six protons, together with two sharp singlets (each corresponding to one proton) at $\delta 6{\cdot}35$ for the strongly shielded proton at C(3) and $\delta 7{\cdot}37$ for the proton at C(6). The signal of benzylic protons, overlapped by the methoxy ones in $CDCl_3$ solution, appeared as doubled at $\delta 4{\cdot}17$ (J 11·0 Hz) in a spectrum performed in C_6D_6 solution.

In this paper we report on the crystal and molecular structure of (3) derived from a single-crystal X-ray analysis.

Experimental. White prismatic crystal, 0·25 × 0·30 × 0·20 mm, Siemens–Stoe diffractometer, graphite-monochromated Mo $K\alpha$ radiation, lattice parameters refined by least squares from 25 reflections with $6 < \theta < 10°$. Standard reflections 133, 425 and 145 were measured at 120 min intervals. No significant fluctuations on intensities other than those expected from Poisson statistics, intensities measured up to $2\theta = 50°$, ω/θ scan technique, scan width 1·2°, scan speed 1·8° min^{-1}. Intensities corrected for Lorentz and polarization effects. 4600 reflections measured, index range $0 < h < 15$, $0 < k < 16$, $-19 < l < 19$, 2107 independent with $I > 3\sigma(I)$. Structure solved by direct methods (*MULTAN*; Germain, Main & Woolfson, 1971), refined by full-matrix least-squares procedures; function minimized $\sum w(|F_o| - F_c|)^2$ with $w = 1{\cdot}7667[\sigma^2(F_o) + 0{\cdot}001541(F_o)^2]^{-1}$; anisotropic thermal parameters applied to the non-phenyl, non-H atoms, H atoms introduced at calculated positions (C—H 0·95 Å) riding on their bonded atoms with fixed isotropic temperature factors, $U = 0{\cdot}06$ Å^2 (256 parameters); $(\Delta/\sigma)_{\max}$ in final least-squares cycle 0·06. Final $R = 0{\cdot}052$, $wR = 0{\cdot}059$; $\Delta\rho$ in final difference map 0·2 e Å^{-3} and $S = 1{\cdot}1$. Scattering factors from *International Tables for X-ray Crystallography* (1974). Computations carried out on IBM 4331 computer of the University of Messina. Computer programs used: *SHELX*76 (Sheldrick, 1976), *PARST* (Nardelli, 1983) and *PLUTO* (Motherwell & Clegg, 1978).

Table 1. *Atomic fractional coordinates* (× 10^4) *and equivalent isotropic or isotropic thermal parameters* (× 10^3) *for non-*H *atoms with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* (\mathbf{a}_i . \mathbf{a}_j).$$

	x	y	z	U_{eq}/U_{iso}(Å^2)
C(1)	3494 (3)	−1589 (3)	2071 (3)	50 (2)
C(2)	2468 (4)	−1542 (3)	2329 (3)	56 (2)
C(3)	2411 (4)	−645 (4)	2843 (4)	82 (2)
C(4)	2298 (4)	−2448 (4)	2793 (4)	73 (2)
C(6)	2174 (3)	1101 (3)	−9 (2)	48 (2)
C(7)	1235 (3)	456 (3)	−414 (3)	52 (2)
C(8)	1537 (4)	−374 (4)	−900 (3)	78 (2)
C(9)	364 (4)	1041 (4)	−965 (3)	75 (2)
O(17)	986 (2)	4280 (2)	1689 (2)	69 (2)
C(17)	992 (5)	5240 (4)	1395 (4)	97 (3)
O(18)	904 (3)	2572 (3)	2247 (2)	88 (2)
C(18)	1040 (5)	1721 (4)	2679 (3)	99 (3)
O(27)	4234 (2)	1555 (2)	113 (2)	58 (1)
C(27)	5271 (3)	1838 (3)	131 (3)	63 (2)
O(28)	5468 (2)	−944 (2)	2138 (2)	63 (1)
C(28)	6509 (4)	−775 (5)	2117 (3)	87 (3)
O(37)	3680 (3)	−5058 (2)	313 (2)	70 (1)
C(37)	4403 (4)	−5815 (4)	645 (4)	86 (2)
O(38)	2162 (2)	−3874 (2)	−203 (2)	64 (1)
C(38)	1280 (4)	−3274 (4)	−471 (3)	68 (2)
C(12)	1249 (4)	2625 (3)	1548 (3)	57 (1)*
C(13)	1533 (3)	1847 (3)	1145 (3)	54 (1)*
C(14)	1879 (3)	1962 (3)	434 (2)	45 (1)*
C(15)	1927 (3)	2888 (3)	151 (3)	54 (1)*
C(16)	1633 (3)	3683 (3)	550 (3)	59 (1)*
C(11)	1294 (4)	3562 (3)	1241 (3)	54 (1)*
C(22)	2902 (3)	−217 (3)	1040 (2)	44 (1)
C(23)	3066 (3)	541 (3)	545 (2)	42 (1)*
C(24)	4093 (3)	808 (3)	608 (2)	45 (1)*
C(25)	4907 (3)	330 (3)	1134 (2)	48 (1)*
C(26)	4709 (3)	−420 (3)	1611 (3)	47 (1)*
C(21)	3694 (3)	−713 (3)	1579 (2)	42 (1)*
C(32)	2888 (3)	−3588 (3)	482 (3)	49 (1)*
C(33)	2848 (3)	−2742 (3)	896 (2)	48 (1)*
C(34)	3588 (3)	−2516 (3)	1613 (3)	47 (1)*
C(35)	4399 (4)	−3163 (3)	1884 (3)	58 (1)*
C(36)	4455 (4)	−4020 (3)	1465 (3)	59 (1)*
C(31)	3709 (4)	−4237 (3)	769 (3)	54 (1)*

* Isotropic

Discussion. Atomic parameters are given in Table 1,* bond lengths and bond angles in Table 2.

The stereochemical arrangement of a molecule showing its solid-state conformation and the atomic numbering scheme used is given in Fig. 1 which is a view projected on the central benzene-ring (*B*) plane.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44386 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The molecule adopts a conformation which we describe by the mutual position of the external benzyl rings (*A* and *C*) which respect to the plane through the central benzene ring *B*. In this isomer the two dimethoxybenzyl groups (*A* and *C*) and the two isopropyl groups are pointing in *anti* directions with respect to the central ring *B*. In fact the molecule has approximate C_2 symmetry about a binary axis passing through C(25) and C(22). [The two hydrogens at C(2) and C(7) of the two isopropyl groups are pointing in opposite directions.]

The two dihedral angles $A \wedge B$ 74·5 (1) and $B \wedge C$ 75·4 (1)° are very close to each other while $A \wedge C$ is 108·4 (1)°. The relevant torsion angles about C(sp^2)–C(sp^3) bonds are C(21)–C(1)–C(34)–C(33) −69·3 (5), C(34)–C(1)–C(21)–C(22) +93·0 (5), C(14)–C(6)–C(23)–C(22) +90·4 (4) and C(23)–C(6)–C(14)–C(13) −60·3 (9)°.

Bond distances and angles are comparable with those of 1,5-bis(α-isopropyl-2,4-dimethoxybenzyl)-2,4-dimethoxybenzene (Bruno *et al.*, 1983).

The crystal packing shown in Fig. 2 is determined by van der Waals forces and no significant intermolecular contacts are present in the structure.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·522 (7)	O(38)–C(38)	1·404 (6)
C(1)–C(21)	1·515 (6)	O(38)–C(32)	1·357 (5)
C(1)–C(34)	1·505 (6)	C(12)–C(13)	1·363 (7)
C(2)–C(3)	1·512 (8)	C(12)–C(11)	1·389 (6)
C(2)–C(4)	1·511 (8)	C(13)–C(14)	1·388 (6)
C(6)–C(7)	1·535 (5)	C(14)–C(15)	1·361 (6)
C(6)–C(14)	1·496 (6)	C(15)–C(16)	1·383 (6)
C(6)–C(23)	1·515 (5)	C(16)–C(11)	1·354 (8)
C(7)–C(8)	1·510 (7)	C(22)–C(23)	1·379 (6)
C(7)–C(9)	1·512 (6)	C(22)–C(21)	1·378 (5)
O(17)–C(17)	1·405 (6)	C(23)–C(24)	1·382 (6)
O(17)–C(11)	1·359 (6)	C(24)–C(25)	1·373 (5)
O(18)–C(18)	1·361 (7)	C(25)–C(26)	1·367 (6)
O(18)–C(12)	1·361 (7)	C(26)–C(21)	1·386 (6)
O(27)–C(27)	1·415 (5)	C(32)–C(33)	1·357 (6)
O(27)–C(24)	1·359 (5)	C(32)–C(31)	1·391 (6)
O(28)–C(28)	1·402 (6)	C(33)–C(34)	1·383 (5)
O(28)–C(26)	1·363 (5)	C(34)–C(35)	1·376 (6)
O(37)–C(37)	1·424 (6)	C(35)–C(36)	1·379 (6)
O(37)–C(31)	1·355 (5)	C(36)–C(31)	1·359 (6)

C(21)–C(1)–C(34)	110·1 (4)	O(17)–C(11)–C(16)	126·4 (4)
C(2)–C(1)–C(34)	111·9 (4)	O(17)–C(11)–C(12)	114·7 (4)
C(2)–C(1)–C(21)	114·2 (4)	C(23)–C(22)–C(21)	123·9 (4)
C(1)–C(2)–C(4)	111·6 (4)	C(6)–C(23)–C(22)	122·4 (4)
C(1)–C(2)–C(3)	111·8 (4)	C(22)–C(23)–C(24)	116·9 (3)
C(3)–C(2)–C(4)	109·9 (4)	C(6)–C(23)–C(24)	120·7 (3)
C(14)–C(6)–C(23)	110·6 (3)	O(27)–C(24)–C(23)	115·7 (3)
C(7)–C(6)–C(23)	113·5 (3)	C(23)–C(24)–C(25)	121·1 (4)
C(7)–C(6)–C(14)	113·0 (3)	O(27)–C(24)–C(25)	123·1 (4)
C(6)–C(7)–C(9)	111·8 (4)	C(24)–C(25)–C(26)	120·1 (4)
C(6)–C(7)–C(8)	112·3 (4)	O(28)–C(26)–C(25)	124·0 (4)
C(8)–C(7)–C(9)	109·4 (4)	C(25)–C(26)–C(21)	121·2 (4)
C(17)–O(17)–C(11)	117·0 (4)	O(28)–C(26)–C(21)	114·8 (4)
C(18)–O(18)–C(12)	118·4 (4)	C(22)–C(21)–C(26)	116·8 (4)
C(27)–O(27)–C(24)	118·2 (3)	C(1)–C(21)–C(26)	120·4 (4)
C(28)–O(28)–C(26)	117·6 (4)	C(1)–C(21)–C(22)	122·7 (4)
C(37)–O(37)–C(31)	117·5 (4)	O(38)–C(32)–C(31)	115·7 (4)
C(38)–O(38)–C(32)	117·2 (3)	O(38)–C(32)–C(33)	124·7 (4)
O(18)–C(12)–C(11)	115·0 (4)	C(33)–C(32)–C(31)	119·6 (4)
O(18)–C(12)–C(13)	125·3 (4)	C(32)–C(33)–C(34)	121·8 (4)
C(13)–C(12)–C(11)	119·8 (4)	C(1)–C(34)–C(33)	120·2 (4)
C(12)–C(13)–C(14)	121·8 (4)	C(33)–C(34)–C(35)	117·7 (4)
C(6)–C(14)–C(13)	121·3 (4)	C(1)–C(34)–C(35)	122·1 (4)
C(13)–C(14)–C(15)	117·4 (4)	C(34)–C(35)–C(36)	121·1 (4)
C(6)–C(14)–C(15)	121·3 (3)	C(35)–C(36)–C(31)	120·2 (4)
C(14)–C(15)–C(16)	121·4 (4)	C(32)–C(31)–C(36)	119·6 (4)
C(15)–C(16)–C(11)	120·7 (4)	O(37)–C(31)–C(36)	125·3 (4)
C(12)–C(11)–C(16)	118·9 (4)	O(37)–C(31)–C(32)	115·1 (4)

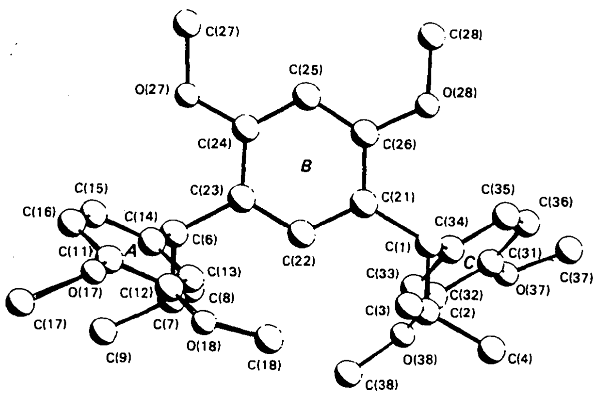

Fig. 1. View of a molecule down the central phenyl ring (*B*).

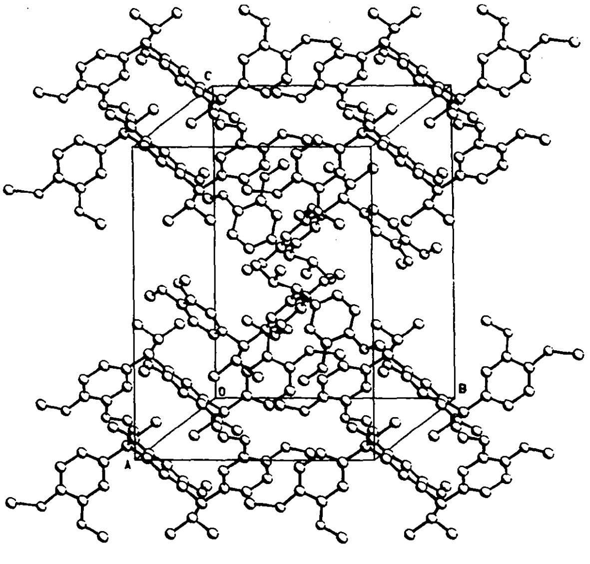

Fig. 2. Packing diagram.

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