H atoms were placed geometrically and thereafter allowed to ride on their parent atoms with common isotropic displacement parameters ($U_{\rm iso}$ = 0.08 Å^2). For the H atoms of the C6 methyl group, the torsion angle was also refined ($U_{\rm iso}$ = 0.13 Å^2). The H atom on N1 was located from a difference map and refined isotropically. Programs used include *PARST* (Nardelli, 1983).

Data collection: *TEXSAN* (Molecular Structure Corporation, 1985). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,4,6-Tri-*O*-benzyl-*myo*-inositol 1,3,5-Tris(dibenzylphosphate)

Valerie Graingeot, Cedric Brigando, Bruno Faure and David Benlian

Laboratoire de Chimie de Coordination, Faculté des Sciences et Techniques de Saint-Jérôme, Case D22, 13397 Marseille CEDEX 20, France. E-mail: david.benlian@lcc.u-3mrs.fr

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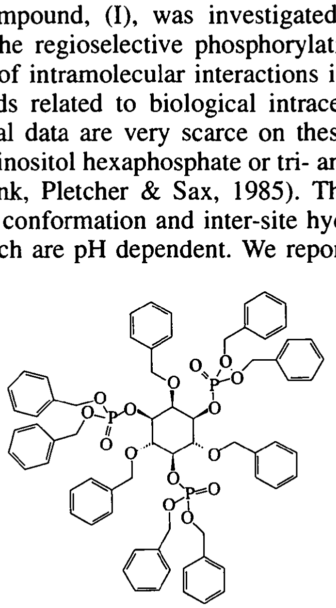
Abstract

This paper reports the crystal structure of 2,4,6-tri-*O*-benzyl-*myo*-inositol 1,3,5-tris(dibenzylphosphate), $C_{69}H_{69}O_{15}P_3$. The X-ray analysis reveals a cyclohexyl ring in a chair conformation with five substituents in equatorial orientations and one in an axial orientation.

Comment

The title compound, (I), was investigated as part of a study on the regioselective phosphorylation of *myo*-inositol and of intramolecular interactions in this series of compounds related to biological intracellular carriers. Structural data are very scarce on these analogues (*i.e.* on *myo*-inositol hexaphosphate or tri- and tetraphosphates) (Blank, Pletcher & Sax, 1985). These interactions induce conformation and inter-site hydrogen-bond changes which are pH dependent. We reported recently on NMR investigations of conformational variations with pH (Brigando, Mossoyan, Favier & Benlian, 1995) and on the influence of intramolecular labile hydrogen bonds (Brigando & Mossoyan, 1996). It appears that certain chemical shifts occur when the pH is raised, by the H1–H6 protons of the *myo*-inositol ring C atoms, which could be assigned to the direct through-space interaction with the vicinal phosphate-O atom. This is observed in aqueous and mixed-solvent solution. It appears that, even in the case of esters, the phosphorylation on the 1, 3 and 5 positions has a noticeable but non-uniform influence on the vicinal C2, C4 and C6 centres. This is sustained by comparison with the structure of the non-phosphorylated molecule (Graingeot, Brigando & Benlian, 1996). The phosphorylation is the crucial step of the synthesis of *myo*-inositol trisphosphates. The structure of (I) was determined by X-ray diffraction. An *ORTEP*II (Johnson, 1976) view is shown in Fig. 1.



(I)

The molecule assumes a chair conformation with three dibenzyl phosphate groups at the C1, C3 and C5 positions. The three other substituents on the central ring are benzyloxy groups (at the C2, C4 and C6 positions). The only substituent in an axial position is the benzyloxy group on C2, the others are in equatorial positions. The stereochemistry of the central ring is shown in Fig. 2.

The adoption of these orientations by the substituents appears to have been determined by the requirement of minimum steric interactions. Two remarkable features of this structure are worth a closer look. One is the relative mobility of the outer rims of the benzyl groups in the structure as shown by the $U_{\rm eq}$ values (>0.12)

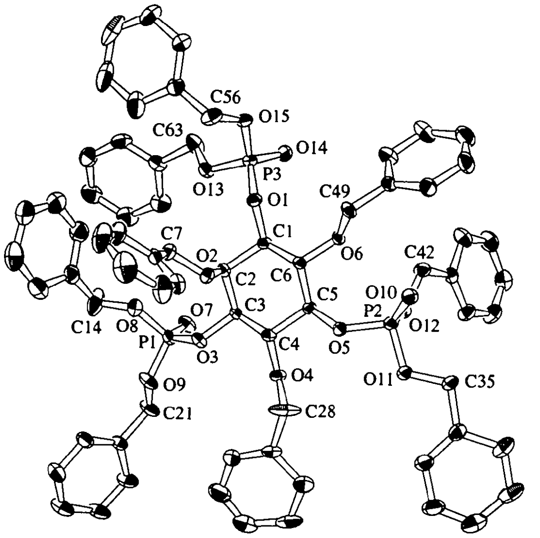


Fig. 1. A perspective *ORTEP*II (Johnson, 1976) view showing 20% probability displacement ellipsoids.

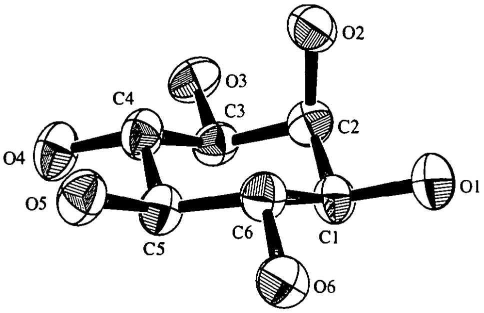


Fig. 2. Stereochemistry of the central inositol ring.

on C11, C12, C17, C18, C19, C28, C31, C38 and C58, C59, C60, C61, C62. The packing of this crystal appears much less tight than in the non-phosphorylated compound (U_{eq} in the region of 0.06). This is not unexpected as the protective phenyl groups are mutually disaligned with no stabilizing interaction possible. The central part of the molecule seems quite static. The model could not be improved by including more atoms with partial occupancy factors in the refinements. The other main difference appears to be the alteration of the C—O bond lengths after phosphorylation. At the axial *O*-benzyl group on C2, both the C2—O2 [1.425 (6) *cf.* 1.435 (3) Å] and the C7—O2 [1.412 (5) *cf.* 1.440 (2) Å] distances are significantly shorter than in 2,4,6-tri-*O*-benzyl inositol (Graingeot *et al.*, 1996). This is observed to a lesser degree at C6 [1.423 (5) *cf.* 1.436 (2) Å]. On the other hand, PO_4-bearing C—O bonds are lengthened at C1 [1.458 (5) *cf.* 1.423 (3) Å], C3 [1.453 (5) *cf.* 1.441 (2) Å] and C5 [1.443 (6) *cf.* 1.418 (3) Å]. This will be correlated with the spectral consequences of charge-density transfers observed in solutions of similar molecules in this family.

Experimental

The title compound was prepared by a method inspired by Vacca, de Solms & Huff (1987). Single crystals were obtained by vapour diffusion of pentane into a solution of the title compound in diethyl ether at room temperature.

Crystal data

$C_{69}H_{69}O_{15}P_3$
$M_r = 1231.23$
Triclinic
$P\overline{1}$
$a = 14.438\ (2)$ Å
$b = 14.508\ (5)$ Å
$c = 16.827\ (1)$ Å
$\alpha = 73.89\ (2)°$
$\beta = 68.23\ (1)°$
$\gamma = 87.44\ (2)°$
$V = 3138\ (1)$ Å^3
$Z = 2$
$D_x = 1.30$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11–15°$
$\mu = 0.156$ mm^{-1}
$T = 294$ K
Prism
0.60 × 0.40 × 0.40 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
10198 measured reflections
7095 independent reflections
6535 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.028$
$\theta_{max} = 24°$
$h = -15 \rightarrow 16$
$k = -15 \rightarrow 16$
$l = 0 \rightarrow 19$
2 standard reflections
frequency: 120 min
intensity decay: 1.1%

Refinement

Refinement on F
$R = 0.050$
$wR = 0.066$
$S = 2.18$
6535 reflections
784 parameters
H-atoms introduced at idealized positions before the last refinement cycle and not refined

$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0016F_o^4]$
$(\Delta/\sigma)_{max} = 0.201$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	U_{eq}
P1	0.78090 (6)	0.49627 (6)	0.05389 (5)	0.0513 (3)
P2	1.13073 (6)	0.13503 (6)	−0.13455 (5)	0.0476 (2)
P3	1.00318 (6)	0.21279 (6)	0.27500 (5)	0.0481 (2)
O2	0.7969 (1)	0.2189 (1)	0.1538 (1)	0.0496 (6)
O3	0.8057 (1)	0.4003 (1)	0.0285 (1)	0.0513 (6)
O4	0.9307 (2)	0.3489 (1)	−0.1166 (1)	0.0544 (7)
O5	1.0295 (1)	0.1765 (1)	−0.0876 (1)	0.0478 (6)
O6	1.0952 (1)	0.1403 (1)	0.0582 (1)	0.0436 (6)
O1	0.9580 (1)	0.1799 (1)	0.2149 (1)	0.0467 (6)

O7	0.8656 (2)	0.5576 (1)	0.0387 (2)	0.0695 (8)
O8	0.7088 (2)	0.4535 (2)	0.1541 (2)	0.101 (1)
O9	0.7097 (2)	0.5386 (2)	0.0049 (2)	0.0747 (8)
O10	1.1436 (2)	0.0355 (1)	−0.0947 (1)	0.0574 (7)
O11	1.1251 (2)	0.1550 (2)	−0.2293 (1)	0.0614 (7)
O12	1.2157 (1)	0.2095 (1)	−0.1514 (1)	0.0539 (7)
O13	0.9318 (1)	0.2922 (1)	0.3034 (1)	0.0556 (6)
O14	1.1085 (2)	0.2426 (2)	0.2368 (1)	0.0720 (8)
O15	0.9803 (2)	0.1237 (2)	0.3570 (1)	0.0640 (8)
C2	0.8759 (2)	0.2901 (2)	0.1269 (2)	0.0428 (9)
C3	0.8940 (2)	0.3517 (2)	0.0324 (2)	0.0423 (9)
C4	0.9178 (2)	0.2890 (2)	−0.0306 (2)	0.0436 (9)
C5	1.0121 (2)	0.2380 (2)	−0.0296 (2)	0.0408 (8)
C6	1.0013 (2)	0.1774 (2)	0.0636 (2)	0.0397 (8)
C1	0.9724 (2)	0.2413 (2)	0.1256 (2)	0.0408 (9)
C7	0.7246 (2)	0.2064 (3)	0.2405 (2)	0.063 (1)
C8	0.6511 (2)	0.1246 (2)	0.2643 (2)	0.055 (1)
C9	0.6520 (3)	0.0749 (2)	0.2062 (2)	0.069 (1)
C10	0.5795 (3)	0.0019 (3)	0.2306 (3)	0.108 (2)
C11	0.5072 (4)	−0.0210 (4)	0.3131 (4)	0.135 (3)
C12	0.5066 (4)	0.0273 (4)	0.3728 (4)	0.131 (2)
C13	0.5766 (3)	0.1007 (3)	0.3484 (3)	0.095 (2)
C14	0.6434 (5)	0.4980 (4)	0.2069 (4)	0.152 (2)
C15	0.5818 (3)	0.4310 (3)	0.2934 (2)	0.078 (1)
C16	0.6249 (3)	0.3996 (3)	0.3554 (3)	0.099 (2)
C17	0.5708 (4)	0.3400 (5)	0.4391 (3)	0.133 (2)
C18	0.4766 (4)	0.3155 (4)	0.4615 (4)	0.140 (3)
C19	0.4309 (3)	0.3435 (3)	0.4058 (4)	0.120 (2)
C20	0.4829 (3)	0.4002 (3)	0.3181 (3)	0.108 (2)
C21	0.7271 (3)	0.6292 (3)	−0.0592 (2)	0.081 (1)
C22	0.6341 (2)	0.6560 (2)	−0.0780 (2)	0.057 (1)
C23	0.5411 (2)	0.6203 (3)	−0.0187 (2)	0.077 (1)
C24	0.4569 (3)	0.6496 (3)	−0.0375 (3)	0.093 (2)
C25	0.4667 (3)	0.7148 (3)	−0.1163 (3)	0.098 (2)
C26	0.5583 (3)	0.7505 (3)	−0.1760 (3)	0.108 (2)
C27	0.6443 (3)	0.7211 (3)	−0.1580 (3)	0.081 (1)
C28	0.8959 (4)	0.3092 (3)	−0.1681 (2)	0.125 (2)
C29	0.8647 (2)	0.3868 (2)	−0.2313 (2)	0.063 (1)
C30	0.7941 (3)	0.4454 (3)	−0.2022 (2)	0.091 (2)
C31	0.7649 (3)	0.5166 (4)	−0.2587 (3)	0.129 (2)
C32	0.8081 (3)	0.5294 (3)	−0.3456 (3)	0.115 (2)
C33	0.8796 (3)	0.4747 (4)	−0.3804 (2)	0.107 (2)
C34	0.9100 (3)	0.3999 (3)	−0.3217 (2)	0.095 (2)
C35	1.1937 (3)	0.1082 (3)	−0.2904 (2)	0.080 (1)
C36	1.1845 (3)	0.1424 (2)	−0.3795 (2)	0.063 (1)
C37	1.1022 (3)	0.1860 (3)	−0.3914 (2)	0.100 (2)
C38	1.0949 (4)	0.2119 (4)	−0.4734 (3)	0.123 (2)
C39	1.1663 (4)	0.1953 (3)	−0.5445 (3)	0.109 (2)
C40	1.2503 (4)	0.1514 (3)	−0.5354 (2)	0.105 (2)
C41	1.2594 (3)	0.1252 (3)	−0.4522 (2)	0.085 (1)
C42	1.2881 (2)	0.1866 (3)	−0.1088 (2)	0.067 (1)
C43	1.3867 (2)	0.2409 (2)	−0.1693 (2)	0.054 (1)
C44	1.4113 (3)	0.2857 (3)	−0.2583 (2)	0.076 (1)
C45	1.5040 (3)	0.3359 (3)	−0.3094 (3)	0.091 (2)
C46	1.5716 (3)	0.3393 (3)	−0.2708 (3)	0.089 (2)
C47	1.5483 (3)	0.2944 (3)	−0.1834 (3)	0.091 (2)
C48	1.4557 (3)	0.2455 (3)	−0.1317 (2)	0.075 (1)
C49	1.0898 (2)	0.0404 (2)	0.1070 (2)	0.054 (1)
C50	1.1937 (2)	0.0142 (2)	0.0993 (2)	0.053 (1)
C51	1.2370 (3)	−0.0572 (2)	0.0604 (2)	0.071 (1)
C52	1.3340 (3)	−0.0827 (3)	0.0552 (3)	0.093 (2)
C53	1.3845 (3)	−0.0366 (3)	0.0877 (3)	0.102 (2)
C54	1.3436 (3)	0.0351 (3)	0.1247 (3)	0.102 (2)
C55	1.2479 (3)	0.0609 (3)	0.1304 (2)	0.076 (1)
C56	0.8818 (3)	0.0787 (3)	0.4105 (2)	0.078 (1)
C57	0.8398 (3)	0.1028 (3)	0.4982 (2)	0.069 (1)
C58	0.8735 (4)	0.0579 (4)	0.5639 (3)	0.121 (2)
C59	0.8368 (5)	0.0814 (6)	0.6438 (3)	0.182 (3)
C60	0.7673 (5)	0.1472 (5)	0.6587 (3)	0.167 (3)
C61	0.7312 (5)	0.1862 (4)	0.5931 (4)	0.157 (3)
C62	0.7693 (4)	0.1647 (3)	0.5127 (3)	0.115 (2)
C63	0.9597 (3)	0.3520 (3)	0.3485 (3)	0.093 (1)
C64	0.8910 (2)	0.4330 (2)	0.3564 (2)	0.064 (1)
C65	0.8268 (3)	0.4375 (3)	0.4394 (2)	0.082 (1)
C66	0.7694 (3)	0.5142 (3)	0.4479 (3)	0.100 (2)
C67	0.7762 (3)	0.5861 (3)	0.3735 (3)	0.105 (2)
C68	0.8380 (4)	0.5807 (3)	0.2911 (3)	0.103 (2)
C69	0.8950 (3)	0.5052 (3)	0.2828 (3)	0.079 (1)

Table 2. *Geometric parameters (Å, °)*

P1—O3	1.556 (2)	C22—C23	1.359 (4)
P1—O7	1.446 (3)	C22—C27	1.374 (5)
P1—O8	1.577 (2)	C23—C24	1.387 (6)
P1—O9	1.551 (3)	C24—C25	1.360 (6)
P2—O5	1.565 (2)	C25—C26	1.347 (5)
P2—O10	1.450 (2)	C26—C27	1.404 (6)
P2—O11	1.571 (2)	C28—C29	1.500 (5)
P2—O12	1.564 (2)	C29—C30	1.335 (5)
P3—O1	1.568 (3)	C29—C34	1.375 (5)
P3—O13	1.566 (2)	C30—C31	1.365 (6)
P3—O14	1.447 (2)	C31—C32	1.321 (6)
P3—O15	1.548 (2)	C32—C33	1.333 (7)
O2—C2	1.427 (4)	C33—C34	1.425 (6)
O2—C7	1.410 (3)	C35—C36	1.497 (5)
O3—C3	1.444 (3)	C36—C37	1.375 (6)
O4—C4	1.416 (3)	C36—C41	1.374 (5)
O4—C28	1.399 (6)	C37—C38	1.369 (7)
O5—C5	1.447 (4)	C38—C39	1.332 (6)
O6—C6	1.418 (3)	C39—C40	1.383 (7)
O6—C49	1.443 (3)	C40—C41	1.399 (6)
O1—C1	1.461 (3)	C42—C43	1.505 (4)
O8—C14	1.320 (6)	C43—C44	1.370 (5)
O9—C21	1.412 (4)	C43—C48	1.376 (6)
O11—C35	1.440 (4)	C44—C45	1.396 (5)
O12—C42	1.452 (5)	C45—C46	1.367 (7)
O13—C63	1.453 (6)	C46—C47	1.349 (6)
O15—C56	1.438 (4)	C47—C48	1.387 (5)
C2—C3	1.523 (4)	C49—C50	1.499 (5)
C2—C1	1.530 (4)	C50—C51	1.379 (5)
C3—C4	1.518 (4)	C50—C55	1.376 (6)
C4—C5	1.523 (4)	C51—C52	1.409 (6)
C5—C6	1.524 (4)	C52—C53	1.350 (8)
C6—C1	1.517 (4)	C53—C54	1.360 (7)
C7—C8	1.492 (5)	C54—C55	1.391 (6)
C8—C9	1.364 (6)	C56—C57	1.507 (6)
C8—C13	1.382 (4)	C57—C58	1.363 (7)
C9—C10	1.387 (6)	C57—C62	1.326 (6)
C10—C11	1.352 (7)	C58—C59	1.383 (8)
C11—C12	1.37 (1)	C59—C60	1.36 (1)
C12—C13	1.368 (7)	C60—C61	1.37 (1)
C14—C15	1.478 (6)	C61—C62	1.378 (8)
C15—C16	1.370 (7)	C63—C64	1.505 (5)
C15—C20	1.387 (6)	C64—C65	1.377 (5)
C16—C17	1.383 (6)	C64—C69	1.367 (5)
C17—C18	1.306 (8)	C65—C66	1.365 (6)
C18—C19	1.305 (9)	C66—C67	1.363 (6)
C19—C20	1.405 (6)	C67—C68	1.364 (6)
C21—C22	1.502 (5)	C68—C69	1.347 (6)
O3—P1—O7	116.0 (1)	C15—C20—C19	119.1 (5)
O3—P1—O8	98.5 (1)	O9—C21—C22	109.7 (3)
O3—P1—O9	102.0 (1)	C21—C22—C23	122.9 (3)
O7—P1—O8	116.2 (2)	C21—C22—C27	117.9 (3)
O7—P1—O9	117.4 (1)	C23—C22—C27	119.2 (3)
O8—P1—O9	104.1 (1)	C22—C23—C24	121.0 (3)
O5—P2—O10	116.9 (1)	C23—C24—C25	120.0 (3)
O5—P2—O11	98.2 (1)	C24—C25—C26	119.8 (4)
O5—P2—O12	106.7 (1)	C25—C26—C27	120.8 (4)
O10—P2—O11	114.2 (1)	C22—C27—C26	119.3 (3)
O10—P2—O12	116.0 (1)	O4—C28—C29	110.3 (3)
O11—P2—O12	102.7 (1)	C28—C29—C30	121.6 (3)
O1—P3—O13	101.5 (1)	C28—C29—C34	119.9 (3)
O1—P3—O14	117.4 (1)	C30—C29—C34	118.4 (3)
O1—P3—O15	103.1 (1)	C29—C30—C31	122.5 (4)
O13—P3—O14	114.8 (1)	C30—C31—C32	119.4 (4)
O13—P3—O15	108.3 (1)	C31—C32—C33	122.1 (4)
O14—P3—O15	110.7 (1)	C32—C33—C34	118.7 (4)
C2—O2—C7	115.4 (3)	C29—C34—C33	118.9 (4)
P1—O3—C3	121.8 (2)	O11—C35—C36	110.1 (3)
C4—O4—C28	116.0 (2)	C35—C36—C37	123.0 (3)
P2—O5—C5	127.1 (2)	C35—C36—C41	118.6 (3)
C6—O6—C49	114.3 (2)	C37—C36—C41	118.3 (4)
P3—O1—C1	120.5 (2)	C36—C37—C38	121.0 (4)
P1—O8—C14	128.4 (3)	C37—C38—C39	121.7 (5)
P1—O9—C21	124.3 (2)	C38—C39—C40	119.2 (5)
P2—O11—C35	117.4 (2)	C39—C40—C41	119.9 (4)

P2—O12—C42	122.2 (2)	C36—C41—C40	120.0 (4)
P3—O13—C63	118.0 (2)	O12—C42—C43	110.0 (2)
P3—O15—C56	123.7 (2)	C42—C43—C44	124.3 (4)
O2—C2—C3	109.2 (3)	C42—C43—C48	117.1 (3)
O2—C2—C1	109.7 (2)	C44—C43—C48	118.6 (3)
C3—C2—C1	107.6 (2)	C43—C44—C45	120.6 (4)
O3—C3—C2	110.7 (2)	C44—C45—C46	119.8 (4)
O3—C3—C4	107.1 (3)	C45—C46—C47	119.9 (4)
C2—C3—C4	110.2 (2)	C46—C47—C48	120.7 (5)
O4—C4—C3	108.1 (2)	C43—C48—C47	120.3 (4)
O4—C4—C5	111.6 (2)	O6—C49—C50	107.7 (2)
C3—C4—C5	108.6 (3)	C49—C50—C51	120.5 (4)
O5—C5—C4	106.6 (3)	C49—C50—C55	120.7 (3)
O5—C5—C6	109.5 (2)	C51—C50—C55	118.8 (3)
C4—C5—C6	112.2 (2)	C50—C51—C52	120.0 (4)
O6—C6—C5	107.6 (2)	C51—C52—C53	119.7 (4)
O6—C6—C1	109.9 (3)	C52—C53—C54	121.0 (4)
C5—C6—C1	108.8 (2)	C53—C54—C55	119.8 (5)
O1—C1—C2	109.5 (2)	C50—C55—C54	120.6 (4)
O1—C1—C6	107.2 (2)	O15—C56—C57	111.5 (4)
C2—C1—C6	111.6 (3)	C56—C57—C58	119.0 (4)
O2—C7—C8	110.7 (3)	C56—C57—C62	121.0 (4)
C7—C8—C9	123.1 (3)	C58—C57—C62	120.0 (4)
C7—C8—C13	118.2 (4)	C57—C58—C59	119.1 (5)
C9—C8—C13	118.7 (3)	C58—C59—C60	121.8 (6)
C8—C9—C10	120.8 (3)	C59—C60—C61	117.2 (6)
C9—C10—C11	119.9 (5)	C60—C61—C62	121.0 (6)
C10—C11—C12	119.8 (5)	C57—C62—C61	120.7 (5)
C11—C12—C13	120.4 (4)	O13—C63—C64	109.3 (3)
C8—C13—C12	120.3 (5)	C63—C64—C65	119.7 (3)
O8—C14—C15	112.2 (4)	C63—C64—C69	121.0 (3)
C14—C15—C16	117.2 (4)	C65—C64—C69	119.2 (3)
C14—C15—C20	125.5 (5)	C64—C65—C66	120.3 (3)
C16—C15—C20	117.3 (3)	C65—C66—C67	119.3 (4)
C15—C16—C17	120.5 (4)	C66—C67—C68	120.5 (4)
C16—C17—C18	120.6 (6)	C67—C68—C69	120.1 (4)
C17—C18—C19	121.9 (5)	C64—C69—C68	120.6 (4)
C18—C19—C20	120.5 (5)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (Frenz *et al.*, 1985). Program(s) used to solve structure: *MULTAN* (Main *et al.*, 1982) (direct methods). Program(s) used to refine structure: *SDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF VAX MolEN* (Fair, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1235). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Conformation of Crystalline 3-(4-Methoxyphenyl)-2,2,4,4-tetramethylpentane

David J. Collins, Gary D. Fallon and Howard A. Jacobs

Chemistry Department, Monash University, Clayton, Victoria, Australia 3168. E-mail: gary.d.fallon@sci.monash.edu.au

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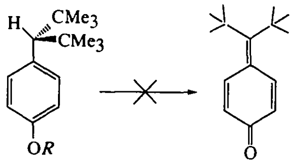
Abstract

The structure of the title compound, $C_{16}H_{26}O$, has shown that the plane of the benzene ring lies almost perpendicular [87.9 (2)°] to the plane C(2),C(3),C(4).

Comment

3-(4-Methoxyphenyl)-2,2,4,4-tetramethylpentane, (1*a*), and the corresponding phenol, (1*b*), were synthesized with the aim of converting the latter into the non-tautomerizable quinone methide, (2) (Collins & Jacobs, 1986). All attempts to oxidize (1*b*) to (2) with a variety of reagents failed, presumably because of severe non-bonded interactions between the *tert*-butyl groups and the *ortho* aromatic protons in (1*a*) (Collins & Jacobs, 1986). The quinone methide (2) remains an elusive target.



(1*a*) $R = \mathrm{Me}$
(1*b*) $R = \mathrm{H}$

(2)

The ^{1}H NMR spectra of (1*a*) and (1*b*) (Collins & Jacobs, 1986) showed asymmetric *ABCD*-type patterns for the four respective aromatic protons, indicative of