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Structure of Isofutoquinol A

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Abstract. 7-(1,3-Benzodioxol-5-yl)-2,3-dimethoxy-8-methyl-6-(2-propenyl)tricyclo[4.2.0.0^{2,8}]oct-3-en-5-one, $C_{21}H_{22}O_5$, $M_r = 354{\cdot}4$, triclinic, $P\bar{1}$, $a = 9{\cdot}653$ (2), $b = 12{\cdot}474$ (2), $c = 8{\cdot}182$ (1) Å, $\alpha = 91{\cdot}30$ (2), $\beta = 97{\cdot}79$ (1), $\gamma = 109{\cdot}15$ (1)°, $V = 919{\cdot}8$ (3) Å^3, $Z = 2$, $D_m = 1{\cdot}26$ (1), $D_x = 1{\cdot}28$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}085$ mm^{-1}, $F(000) = 376$, $T = 294$ (1) K, $R = 0{\cdot}040$ for 2336 observed unique reflections. The title compound, synthesized by means of electrochemical and photochemical methods, has been confirmed as identical with a racemic authentic sample of natural isofutoquinol A, whose relative configurations are now established. The 3–4–6 fused ring structure is strained, resulting in a C—C bond in the cyclobutane ring as long as 1·606 (3) Å.

Experimental. Colorless plate-like crystals grown from *n*-hexane. D_m by flotation in KI aqueous solution. Crystal size 0·6 × 0·5 × 0·35 mm, Rigaku AFC-5 four-circle diffractometer, graphite-monochromatized Mo $K\alpha$, Laue group $\bar{1}$, cell parameters from least-squares refinement of 20 reflections ($20 < 2\theta < 30°$). Intensity measurement performed to $2\theta = 55°$ (h −12→12, k 0→16, l −10→10), θ–2θ scan with speed 4° min^{-1} in θ. Five standard reflections showed no significant decay, $0{\cdot}979 \le |F_o|/|F_o|_{\text{initial}} \le 1{\cdot}009$. 4432 unique reflections measured with 2451 observed $|F_o| > 3\sigma(|F_o|)$, absorption correction ignored. Structure solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). 25 among 26 non-H atoms determined from *E*

Table 1. *Positional parameters* (× 10^4) *and equivalent isotropic temperature factors* (*Hamilton*, 1959)

	x	y	z	B_{eq}(Å^2)
O(1)	−5116 (1)	305 (1)	2331 (2)	4·3
O(2)	−1819 (1)	2849 (1)	6623 (1)	3·5
O(3)	−2552 (1)	4561 (1)	5284 (2)	3·9
O(4)	2262 (2)	2508 (1)	5054 (2)	5·5
O(5)	1850 (2)	680 (1)	4001 (2)	5·2
C(1)	−4012 (2)	2265 (1)	1817 (2)	2·9
C(2)	−4318 (2)	1262 (1)	2888 (2)	3·1
C(3)	−3528 (2)	1488 (1)	4563 (2)	3·0
C(4)	−2733 (2)	2555 (1)	5165 (2)	2·7
C(5)	−2788 (2)	3577 (1)	4277 (2)	2·9
C(6)	−2198 (2)	3872 (1)	2643 (2)	3·1
C(7)	−2282 (2)	2798 (1)	1661 (2)	3·0
C(8)	−3859 (2)	3400 (1)	2714 (2)	3·1
C(9)	−5044 (2)	2015 (2)	151 (2)	3·8
C(10)	−6589 (2)	1918 (2)	358 (3)	4·6
C(11)	−7259 (3)	2627 (3)	−167 (3)	7·0
C(12)	−1237 (2)	5050 (2)	2361 (3)	4·5
C(13)	−1188 (2)	2197 (1)	2207 (2)	3·0
C(14)	58 (2)	2745 (2)	3402 (2)	3·4
C(15)	989 (2)	2155 (2)	3903 (2)	3·5
C(16)	742 (2)	1065 (2)	3276 (2)	3·7
C(17)	−438 (2)	517 (2)	2091 (2)	3·9
C(18)	−1403 (2)	1101 (2)	1574 (2)	3·4
C(19)	−1501 (2)	1946 (2)	7475 (2)	4·4
C(20)	−3632 (3)	4457 (2)	6348 (3)	5·4
C(21)	2738 (2)	1546 (2)	5225 (3)	5·4

Table 2. *Bond lengths* (Å) *and bond angles* (°)

O(1)–C(2)	1·226 (2)	C(4)–C(5)	1·494 (2)
O(2)–C(4)	1·349 (2)	C(5)–C(6)	1·529 (2)
O(2)–C(19)	1·433 (3)	C(5)–C(8)	1·494 (2)
O(3)–C(5)	1·399 (2)	C(6)–C(7)	1·520 (2)
O(3)–C(20)	1·423 (3)	C(6)–C(8)	1·526 (3)
O(4)–C(15)	1·380 (2)	C(6)–C(12)	1·501 (2)
O(4)–C(21)	1·422 (3)	C(7)–C(13)	1·511 (3)
O(5)–C(16)	1·380 (3)	C(9)–C(10)	1·490 (3)
O(5)–C(21)	1·420 (2)	C(10)–C(11)	1·305 (5)
C(1)–C(2)	1·516 (2)	C(13)–C(14)	1·408 (2)
C(1)–C(7)	1·606 (3)	C(13)–C(18)	1·390 (3)
C(1)–C(8)	1·533 (2)	C(14)–C(15)	1·366 (4)
C(1)–C(9)	1·535 (2)	C(15)–C(16)	1·375 (3)
C(2)–C(3)	1·448 (2)	C(16)–C(17)	1·366 (2)
C(3)–C(4)	1·342 (2)	C(17)–C(18)	1·389 (3)
C(4)–O(2)–C(19)	116·8 (1)	C(5)–C(6)–C(12)	122·2 (1)
C(5)–O(3)–C(20)	114·3 (1)	C(7)–C(6)–C(8)	90·8 (1)
C(15)–O(4)–C(21)	105·6 (1)	C(7)–C(6)–C(12)	124·6 (2)
C(16)–O(5)–C(21)	105·7 (2)	C(8)–C(6)–C(12)	130·1 (2)
C(2)–C(1)–C(7)	111·9 (1)	C(1)–C(7)–C(6)	89·6 (1)
C(2)–C(1)–C(8)	114·7 (1)	C(1)–C(7)–C(13)	122·3 (1)
C(2)–C(1)–C(9)	113·2 (1)	C(6)–C(7)–C(13)	119·3 (1)
C(7)–C(1)–C(8)	87·3 (1)	C(1)–C(8)–C(5)	108·8 (1)
C(7)–C(1)–C(9)	113·9 (1)	C(1)–C(8)–C(6)	92·2 (1)
C(8)–C(1)–C(9)	113·4 (1)	C(5)–C(8)–C(6)	60·8 (1)
O(1)–C(2)–C(1)	121·6 (1)	C(1)–C(9)–C(10)	111·7 (2)
O(1)–C(2)–C(3)	122·3 (1)	C(9)–C(10)–C(11)	125·0 (2)
C(1)–C(2)–C(3)	116·0 (1)	C(7)–C(13)–C(14)	119·6 (1)
C(2)–C(3)–C(4)	120·8 (1)	C(7)–C(13)–C(18)	121·2 (2)
O(2)–C(4)–C(3)	125·6 (1)	C(14)–C(13)–C(18)	119·2 (2)
O(2)–C(4)–C(5)	111·6 (1)	C(13)–C(14)–C(15)	117·6 (2)
C(3)–C(4)–C(5)	122·8 (1)	O(4)–C(15)–C(14)	128·1 (2)
O(3)–C(5)–C(4)	115·7 (1)	O(4)–C(15)–C(16)	109·8 (2)
O(3)–C(5)–C(6)	111·1 (1)	C(14)–C(15)–C(16)	122·1 (2)
O(3)–C(5)–C(8)	116·5 (1)	O(5)–C(16)–C(15)	109·7 (2)
C(4)–C(5)–C(6)	123·8 (1)	O(5)–C(16)–C(17)	128·5 (2)
C(4)–C(5)–C(8)	118·2 (1)	C(15)–C(16)–C(17)	121·8 (2)
C(6)–C(5)–C(8)	60·6 (1)	C(16)–C(17)–C(18)	116·9 (2)
C(5)–C(6)–C(7)	110·4 (1)	C(13)–C(18)–C(17)	122·4 (2)
C(5)–C(6)–C(8)	58·6 (1)	O(4)–C(21)–O(5)	108·6 (2)

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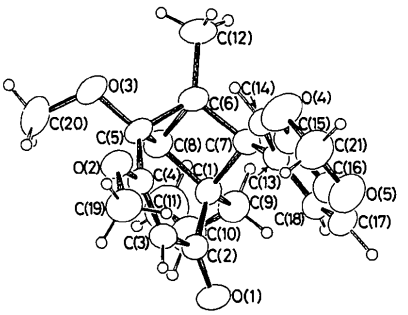
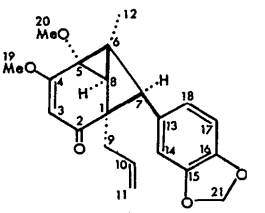


Fig. 1. An *ORTEP* drawing (Johnson, 1965) of the molecule with thermal ellipsoids scaled at the 50% probability level. H atoms are represented by circles of radius 0·08 Å.

map and the remaining from Fourier synthesis; block-diagonal least-squares refinement with anisotropic thermal parameters ($R = 0{\cdot}080$) using *UNICS*-III computation program system (Sakurai & Kobayashi, 1979). All H atoms found from difference synthesis and refined with isotropic thermal parameters. $\sum w||F_o| - |F_c||^2$ minimized, $w^{-1} = \sigma^2(|F_o|) + (0{\cdot}015\,|F_o|)^2$. Final $R = 0{\cdot}040$, $wR = 0{\cdot}048$, $S = 2{\cdot}2$.* Reflection/parameter (refined) ratio 7·2, $\Delta/\sigma < 0{\cdot}5$, $-0{\cdot}23 < \Delta\rho < 0{\cdot}14$ e Å^{-3}. Complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters in Table 1. Bond lengths and bond angles in Table 2. Molecular structure in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters, atomic parameters for H atoms, bond lengths and bond angles involving H atoms, torsion angles, and a projection of the crystal structure have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43321 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Title compound (I) having anti-feedant activity against insects was totally synthesized (Shizuri, Nakamura, Yamamura, Ohba, Yamashita & Saito, 1986). The elongation of the C(6)–C(7) bond length results from the strain in the 3–4–6 fused ring structure, which seems larger than that in 5–5–5 fused ring structures (Luyten, Luef, Beck & Buergi, 1986; Iball, Motherwell, Barnes & Golnazarians, 1986).



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Structure of barium germanium hydroxide oxide: erratum.

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Abstract

In the paper by Ozima [*Acta Cryst.* (1986). C**42**, 513–515], the space group of $BaGe_3O_6(OH)_2$ was reported to be *Cc*. Further calculation revealed that the correct space group is *C*2/*c*. Refinement based on the correct space group gave an *R* value of 0·042 for 843 independent reflections.

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