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1-[(*E*)-4-Benzenesulfonyl-3-methyl-but-2-enyl]-2,3,4,5-tetramethoxy-6-methylbenzene

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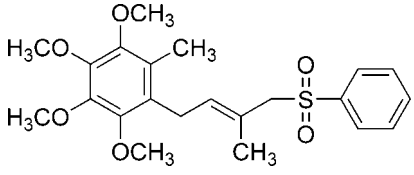
Key indicators: single-crystal X-ray study; T = 184 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.138; data-to-parameter ratio = 19.0.

The title compound, $C_{22}H_{28}O_6S$, was prepared as a key intermediate in the synthesis of coenzyme Q_{10} *via* a Friedel–Crafts reaction. The C=C double bond is *trans* configured. The crystal packing is stabilized by C—H···O contacts.

Related literature

For related literature, see: Min *et al.* (2003).



Experimental

Crystal data

$C_{22}H_{28}O_6S$
M_r = 420.50
Monoclinic, $P2_1/c$
a = 9.8351 (6) Å
b = 20.5513 (14) Å
c = 10.9955 (7) Å
β = 102.841 (1)°
V = 2166.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 184 (2) K
0.31 × 0.19 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.945, T_{max} = 0.969
17680 measured reflections
5102 independent reflections
3755 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.138
S = 1.03
5102 reflections
268 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12A···O6^{i}	0.99	2.58	3.554 (2)	168
C19—H19C···O6^{ii}	0.98	2.56	3.305 (3)	133
C22—H22B···O5^{iii}	0.98	2.26	3.231 (4)	171

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, y, z-1$; (iii) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2497).

References

Bruker (1998). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
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supplementary materials

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1-[(*E*)-4-Benzenesulfonyl-3-methyl-but-2-enyl]-2,3,4,5-tetramethoxy-6-methylbenzene

Q. Li, S.-J. Yan, X.-R. Lin, C.-H. Zhang and J. Lin

Comment

Coenzyme Q_{10} plays an essential role in the electron-transfer processes necessary for respiration, and is applied as a drug against many diseases. The title compound is the key intermediate to synthesize coenzyme Q_{10} *via* a coupling reactions with solanesyl bromide, and it was prepared by coupling tetramethoxytoluene (I) with 4-chioro-2-methyl-1-phenylsulfonyl- 2-butene (II) *via* Friedel-Crafts reaction.

The crystal packing of the title compound is stabilized by C—H···O contacts.

Experimental

2,3,4,5-Tetramethoxytoluene (I) (10 mmol, 1.0 equiv.), and 4-chioro-2- methyl-1-phenylsulfonyl-2-butene (II) (10 mmol, 1.0 equiv.) were stirred in nitromethane (25 ml), then anhydrous zinc chloride (20 mmol) was added and stirred at room temperature for 8 hrs. Water (50 ml) was added to the reaction mixture and stirred for 10 min. The organic layer was separated, dried with anhydrous Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography to give the title compound (III). The single-crystal of (III) was crystallized in the mixed solvent of petroleum ether and acetone (5:1). MS: 420, m. p. 350 K.

Refinement

All H atoms were placed in idealized positions and refined using a riding model, with C—H distances in the range 0.93–0.96 Å and with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

Figures

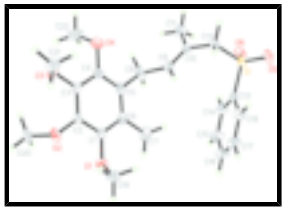


Fig. 1. View of (III) showing 30% displacement ellipsoids (arbitrary spheres for the H atoms).

Fig. 2. Reaction scheme illustrating the preparation of the title compound.

1-[(*E*)-4-Benzenesulfonyl-3-methyl-but-2-enyl]-2,3,4,5- tetramethoxy-6-methylbenzene

Crystal data

$C_{22}H_{28}O_6S$	$D_x = 1.289$ Mg m^{-3}
$M_r = 420.50$	Melting point: 350 K
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.8351$ (6) Å	Cell parameters from 5102 reflections
$b = 20.5513$ (14) Å	$\theta = 2.0$–28.3º
$c = 10.9955$ (7) Å	$\mu = 0.18$ mm^{-1}
$\beta = 102.8410$ (10)º	$T = 184$ (2) K
$V = 2166.9$ (2) Å^3	Block, colourless
$Z = 4$	0.31 × 0.19 × 0.17 mm
$F_{000} = 896$	

Data collection

Bruker SMART CCD area-detector diffractometer	5102 independent reflections
Radiation source: fine-focus sealed tube	3755 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.046$
$T = 184$(2) K	$\theta_{max} = 28.3$º
φ and ω scans	$\theta_{min} = 2.0$º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$h = -12 \rightarrow 12$
$T_{min} = 0.945$, $T_{max} = 0.969$	$k = -27 \rightarrow 27$
17680 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.138$	$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 1.2576P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
5102 reflections	$\Delta\rho_{max} = 0.57$ e Å^{-3}
268 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.29374 (6)	0.26316 (3)	0.94801 (4)	0.03766 (15)
O1	0.18082 (16)	0.46913 (7)	0.28067 (16)	0.0444 (4)
O2	−0.04892 (17)	0.42801 (8)	0.10933 (13)	0.0458 (4)
O3	−0.26927 (17)	0.36823 (9)	0.18912 (16)	0.0566 (5)
O4	−0.24997 (19)	0.34571 (9)	0.43847 (18)	0.0605 (5)
O5	0.3724 (2)	0.20406 (8)	0.97480 (15)	0.0566 (5)
O6	0.25134 (18)	0.29583 (8)	1.04922 (13)	0.0478 (4)
C1	0.0750 (2)	0.43765 (9)	0.32173 (19)	0.0333 (4)
C2	−0.0431 (2)	0.41838 (10)	0.23398 (18)	0.0332 (4)
C3	−0.1507 (2)	0.38721 (10)	0.27361 (19)	0.0373 (5)
C4	−0.1422 (2)	0.37809 (10)	0.4003 (2)	0.0390 (5)
C5	−0.0264 (2)	0.39874 (10)	0.48855 (19)	0.0385 (5)
C6	0.0848 (2)	0.42754 (10)	0.44898 (19)	0.0371 (5)
C7	0.2138 (3)	0.44833 (13)	0.5409 (2)	0.0565 (7)
H7A	0.1898	0.4818	0.5960	0.085*
H7B	0.2813	0.4660	0.4961	0.085*
H7C	0.2547	0.4108	0.5907	0.085*
C8	−0.0224 (3)	0.38985 (13)	0.6256 (2)	0.0549 (7)
H8A	0.0181	0.4293	0.6712	0.066*
H8B	−0.1191	0.3852	0.6369	0.066*
C9	0.0612 (2)	0.33153 (10)	0.68133 (18)	0.0371 (5)
H9	0.1258	0.3143	0.6372	0.045*
C10	0.0527 (2)	0.30221 (10)	0.78600 (17)	0.0323 (4)
C11	−0.0446 (3)	0.32196 (14)	0.8673 (2)	0.0526 (6)
H11A	−0.1069	0.3564	0.8259	0.079*
H11B	0.0097	0.3381	0.9474	0.079*
H11C	−0.1000	0.2843	0.8818	0.079*
C12	0.1445 (2)	0.24476 (9)	0.82806 (18)	0.0357 (4)
H12A	0.1759	0.2267	0.7555	0.043*
H12B	0.0891	0.2108	0.8587	0.043*
C13	0.3925 (2)	0.31897 (10)	0.88015 (18)	0.0346 (4)
C14	0.3929 (3)	0.38410 (11)	0.9102 (2)	0.0467 (5)
H14	0.3380	0.3995	0.9652	0.056*

C15	0.4738 (3)	0.42674 (13)	0.8599 (3)	0.0592 (7)
H15	0.4747	0.4717	0.8804	0.071*
C16	0.5532 (3)	0.40439 (14)	0.7800 (3)	0.0561 (7)
H16	0.6092	0.4339	0.7459	0.067*
C17	0.5515 (3)	0.33968 (13)	0.7496 (2)	0.0532 (6)
H17	0.6058	0.3246	0.6939	0.064*
C18	0.4713 (2)	0.29623 (12)	0.7996 (2)	0.0446 (5)
H18	0.4704	0.2514	0.7787	0.054*
C19	0.2678 (3)	0.42452 (12)	0.2322 (3)	0.0550 (7)
H19A	0.3112	0.3940	0.2979	0.082*
H19B	0.3405	0.4488	0.2034	0.082*
H19C	0.2110	0.4003	0.1622	0.082*
C20	−0.1450 (3)	0.47747 (13)	0.0544 (2)	0.0572 (7)
H20A	−0.2390	0.4657	0.0629	0.086*
H20B	−0.1441	0.4817	−0.0342	0.086*
H20C	−0.1178	0.5190	0.0968	0.086*
C21	−0.2566 (3)	0.30972 (14)	0.1250 (3)	0.0644 (7)
H21A	−0.1913	0.3161	0.0704	0.097*
H21B	−0.3480	0.2972	0.0747	0.097*
H21C	−0.2215	0.2753	0.1855	0.097*
C22	−0.3725 (3)	0.38408 (19)	0.4270 (4)	0.0894 (11)
H22A	−0.3517	0.4223	0.4811	0.134*
H22B	−0.4455	0.3582	0.4518	0.134*
H22C	−0.4046	0.3981	0.3402	0.134*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0501 (3)	0.0356 (3)	0.0260 (2)	0.0065 (2)	0.0059 (2)	0.00566 (19)
O1	0.0437 (9)	0.0280 (7)	0.0646 (10)	0.0002 (6)	0.0185 (8)	0.0025 (7)
O2	0.0567 (10)	0.0515 (9)	0.0292 (7)	0.0154 (8)	0.0092 (7)	0.0090 (6)
O3	0.0386 (9)	0.0654 (11)	0.0579 (11)	−0.0021 (8)	−0.0062 (8)	−0.0105 (9)
O4	0.0560 (11)	0.0553 (11)	0.0759 (13)	−0.0043 (9)	0.0269 (9)	0.0145 (9)
O5	0.0743 (12)	0.0468 (10)	0.0456 (9)	0.0223 (9)	0.0067 (8)	0.0153 (7)
O6	0.0669 (11)	0.0515 (9)	0.0271 (7)	0.0007 (8)	0.0147 (7)	0.0006 (6)
C1	0.0352 (10)	0.0243 (9)	0.0402 (11)	0.0029 (8)	0.0082 (8)	0.0006 (8)
C2	0.0383 (11)	0.0330 (10)	0.0273 (9)	0.0084 (8)	0.0051 (8)	0.0040 (7)
C3	0.0329 (10)	0.0378 (11)	0.0376 (11)	0.0027 (8)	0.0003 (8)	−0.0013 (8)
C4	0.0412 (11)	0.0351 (11)	0.0427 (12)	0.0047 (9)	0.0135 (9)	0.0070 (9)
C5	0.0477 (12)	0.0358 (11)	0.0321 (10)	0.0165 (9)	0.0088 (9)	0.0036 (8)
C6	0.0398 (11)	0.0320 (10)	0.0350 (11)	0.0088 (9)	−0.0018 (9)	−0.0056 (8)
C7	0.0520 (14)	0.0602 (15)	0.0492 (14)	0.0017 (12)	−0.0058 (11)	−0.0216 (12)
C8	0.0729 (17)	0.0610 (15)	0.0325 (11)	0.0353 (13)	0.0157 (11)	0.0078 (10)
C9	0.0432 (12)	0.0409 (11)	0.0279 (10)	0.0110 (9)	0.0096 (8)	−0.0009 (8)
C10	0.0332 (10)	0.0353 (10)	0.0281 (9)	−0.0033 (8)	0.0058 (8)	−0.0031 (8)
C11	0.0487 (14)	0.0724 (17)	0.0420 (13)	0.0064 (12)	0.0212 (11)	0.0019 (12)
C12	0.0477 (12)	0.0295 (10)	0.0313 (10)	−0.0061 (9)	0.0117 (9)	0.0004 (8)
C13	0.0336 (10)	0.0403 (11)	0.0270 (9)	0.0021 (8)	0.0006 (8)	−0.0009 (8)

C14	0.0501 (13)	0.0432 (12)	0.0501 (13)	−0.0005 (10)	0.0181 (11)	−0.0081 (10)
C15	0.0659 (17)	0.0475 (14)	0.0695 (17)	−0.0139 (12)	0.0264 (14)	−0.0151 (12)
C16	0.0503 (14)	0.0646 (16)	0.0561 (15)	−0.0209 (12)	0.0175 (12)	−0.0114 (12)
C17	0.0470 (14)	0.0681 (17)	0.0473 (13)	−0.0065 (12)	0.0167 (11)	−0.0144 (12)
C18	0.0494 (13)	0.0469 (13)	0.0375 (11)	−0.0007 (10)	0.0097 (10)	−0.0105 (9)
C19	0.0483 (14)	0.0399 (12)	0.0847 (19)	0.0056 (11)	0.0320 (13)	0.0089 (12)
C20	0.0610 (16)	0.0620 (16)	0.0449 (13)	0.0159 (13)	0.0037 (12)	0.0211 (12)
C21	0.0631 (17)	0.0601 (16)	0.0614 (17)	−0.0131 (14)	−0.0043 (13)	−0.0123 (13)
C22	0.0576 (19)	0.092 (2)	0.131 (3)	−0.0004 (18)	0.048 (2)	0.020 (2)

Geometric parameters (Å, °)

S1—O5	1.4348 (16)	C10—C11	1.503 (3)
S1—O6	1.4385 (16)	C11—H11A	0.9800
S1—C13	1.772 (2)	C11—H11B	0.9800
S1—C12	1.783 (2)	C11—H11C	0.9800
O1—C1	1.384 (2)	C12—H12A	0.9900
O1—C19	1.435 (3)	C12—H12B	0.9900
O2—C2	1.373 (2)	C13—C14	1.378 (3)
O2—C20	1.427 (3)	C13—C18	1.382 (3)
O3—C3	1.376 (2)	C14—C15	1.379 (3)
O3—C21	1.413 (3)	C14—H14	0.9500
O4—C4	1.393 (3)	C15—C16	1.378 (4)
O4—C22	1.422 (4)	C15—H15	0.9500
C1—C2	1.393 (3)	C16—C17	1.370 (4)
C1—C6	1.396 (3)	C16—H16	0.9500
C2—C3	1.387 (3)	C17—C18	1.383 (3)
C3—C4	1.390 (3)	C17—H17	0.9500
C4—C5	1.388 (3)	C18—H18	0.9500
C5—C6	1.396 (3)	C19—H19A	0.9800
C5—C8	1.510 (3)	C19—H19B	0.9800
C6—C7	1.497 (3)	C19—H19C	0.9800
C7—H7A	0.9800	C20—H20A	0.9800
C7—H7B	0.9800	C20—H20B	0.9800
C7—H7C	0.9800	C20—H20C	0.9800
C8—C9	1.505 (3)	C21—H21A	0.9800
C8—H8A	0.9900	C21—H21B	0.9800
C8—H8B	0.9900	C21—H21C	0.9800
C9—C10	1.318 (3)	C22—H22A	0.9800
C9—H9	0.9500	C22—H22B	0.9800
C10—C12	1.496 (3)	C22—H22C	0.9800
O5—S1—O6	118.16 (10)	H11A—C11—H11C	109.5
O5—S1—C13	108.27 (11)	H11B—C11—H11C	109.5
O6—S1—C13	107.70 (10)	C10—C12—S1	113.74 (14)
O5—S1—C12	106.86 (11)	C10—C12—H12A	108.8
O6—S1—C12	109.80 (10)	S1—C12—H12A	108.8
C13—S1—C12	105.33 (9)	C10—C12—H12B	108.8
C1—O1—C19	112.09 (15)	S1—C12—H12B	108.8
C2—O2—C20	113.69 (17)	H12A—C12—H12B	107.7

C3—O3—C21	114.98 (19)	C14—C13—C18	120.7 (2)
C4—O4—C22	113.2 (2)	C14—C13—S1	120.01 (17)
O1—C1—C2	118.63 (18)	C18—C13—S1	119.30 (17)
O1—C1—C6	120.44 (18)	C13—C14—C15	119.5 (2)
C2—C1—C6	120.89 (19)	C13—C14—H14	120.3
O2—C2—C3	120.99 (18)	C15—C14—H14	120.3
O2—C2—C1	119.57 (19)	C16—C15—C14	120.2 (2)
C3—C2—C1	119.38 (18)	C16—C15—H15	119.9
O3—C3—C2	120.66 (19)	C14—C15—H15	119.9
O3—C3—C4	119.4 (2)	C17—C16—C15	120.0 (2)
C2—C3—C4	119.84 (19)	C17—C16—H16	120.0
C5—C4—C3	121.1 (2)	C15—C16—H16	120.0
C5—C4—O4	119.79 (19)	C16—C17—C18	120.5 (2)
C3—C4—O4	119.1 (2)	C16—C17—H17	119.8
C4—C5—C6	119.35 (19)	C18—C17—H17	119.8
C4—C5—C8	119.7 (2)	C13—C18—C17	119.1 (2)
C6—C5—C8	121.0 (2)	C13—C18—H18	120.4
C5—C6—C1	119.39 (19)	C17—C18—H18	120.4
C5—C6—C7	121.0 (2)	O1—C19—H19A	109.5
C1—C6—C7	119.6 (2)	O1—C19—H19B	109.5
C6—C7—H7A	109.5	H19A—C19—H19B	109.5
C6—C7—H7B	109.5	O1—C19—H19C	109.5
H7A—C7—H7B	109.5	H19A—C19—H19C	109.5
C6—C7—H7C	109.5	H19B—C19—H19C	109.5
H7A—C7—H7C	109.5	O2—C20—H20A	109.5
H7B—C7—H7C	109.5	O2—C20—H20B	109.5
C9—C8—C5	113.14 (18)	H20A—C20—H20B	109.5
C9—C8—H8A	109.0	O2—C20—H20C	109.5
C5—C8—H8A	109.0	H20A—C20—H20C	109.5
C9—C8—H8B	109.0	H20B—C20—H20C	109.5
C5—C8—H8B	109.0	O3—C21—H21A	109.5
H8A—C8—H8B	107.8	O3—C21—H21B	109.5
C10—C9—C8	125.77 (19)	H21A—C21—H21B	109.5
C10—C9—H9	117.1	O3—C21—H21C	109.5
C8—C9—H9	117.1	H21A—C21—H21C	109.5
C9—C10—C12	118.66 (18)	H21B—C21—H21C	109.5
C9—C10—C11	124.6 (2)	O4—C22—H22A	109.5
C12—C10—C11	116.71 (18)	O4—C22—H22B	109.5
C10—C11—H11A	109.5	H22A—C22—H22B	109.5
C10—C11—H11B	109.5	O4—C22—H22C	109.5
H11A—C11—H11B	109.5	H22A—C22—H22C	109.5
C10—C11—H11C	109.5	H22B—C22—H22C	109.5
C19—O1—C1—C2	−79.7 (2)	O1—C1—C6—C5	176.67 (17)
C19—O1—C1—C6	102.4 (2)	C2—C1—C6—C5	−1.1 (3)
C20—O2—C2—C3	73.4 (3)	O1—C1—C6—C7	−2.7 (3)
C20—O2—C2—C1	−109.4 (2)	C2—C1—C6—C7	179.49 (19)
O1—C1—C2—O2	3.2 (3)	C4—C5—C8—C9	99.8 (3)
C6—C1—C2—O2	−178.95 (17)	C6—C5—C8—C9	−80.3 (3)
O1—C1—C2—C3	−179.51 (17)	C5—C8—C9—C10	−160.8 (2)

C6—C1—C2—C3	−1.7 (3)	C8—C9—C10—C12	−179.7 (2)
C21—O3—C3—C2	79.9 (3)	C8—C9—C10—C11	0.8 (4)
C21—O3—C3—C4	−103.6 (3)	C9—C10—C12—S1	101.1 (2)
O2—C2—C3—O3	−3.5 (3)	C11—C10—C12—S1	−79.4 (2)
C1—C2—C3—O3	179.31 (18)	O5—S1—C12—C10	−178.22 (15)
O2—C2—C3—C4	−179.99 (18)	O6—S1—C12—C10	52.48 (17)
C1—C2—C3—C4	2.8 (3)	C13—S1—C12—C10	−63.22 (17)
O3—C3—C4—C5	−177.67 (19)	O5—S1—C13—C14	−141.46 (18)
C2—C3—C4—C5	−1.1 (3)	O6—S1—C13—C14	−12.6 (2)
O3—C3—C4—O4	4.5 (3)	C12—S1—C13—C14	104.51 (19)
C2—C3—C4—O4	−178.90 (19)	O5—S1—C13—C18	37.00 (19)
C22—O4—C4—C5	107.8 (3)	O6—S1—C13—C18	165.83 (17)
C22—O4—C4—C3	−74.4 (3)	C12—S1—C13—C18	−77.03 (18)
C3—C4—C5—C6	−1.7 (3)	C18—C13—C14—C15	−0.5 (4)
O4—C4—C5—C6	176.06 (18)	S1—C13—C14—C15	177.9 (2)
C3—C4—C5—C8	178.20 (19)	C13—C14—C15—C16	0.1 (4)
O4—C4—C5—C8	−4.0 (3)	C14—C15—C16—C17	0.4 (4)
C4—C5—C6—C1	2.8 (3)	C15—C16—C17—C18	−0.6 (4)
C8—C5—C6—C1	−177.12 (18)	C14—C13—C18—C17	0.3 (3)
C4—C5—C6—C7	−177.8 (2)	S1—C13—C18—C17	−178.12 (18)
C8—C5—C6—C7	2.3 (3)	C16—C17—C18—C13	0.2 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12A···O6^{i}	0.99	2.58	3.554 (2)	168
C19—H19C···O6^{ii}	0.98	2.56	3.305 (3)	133
C22—H22B···O5^{iii}	0.98	2.26	3.231 (4)	171

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*, *y*, *z*−1; (iii) *x*−1, −*y*+1/2, *z*−1/2.

Fig. 1

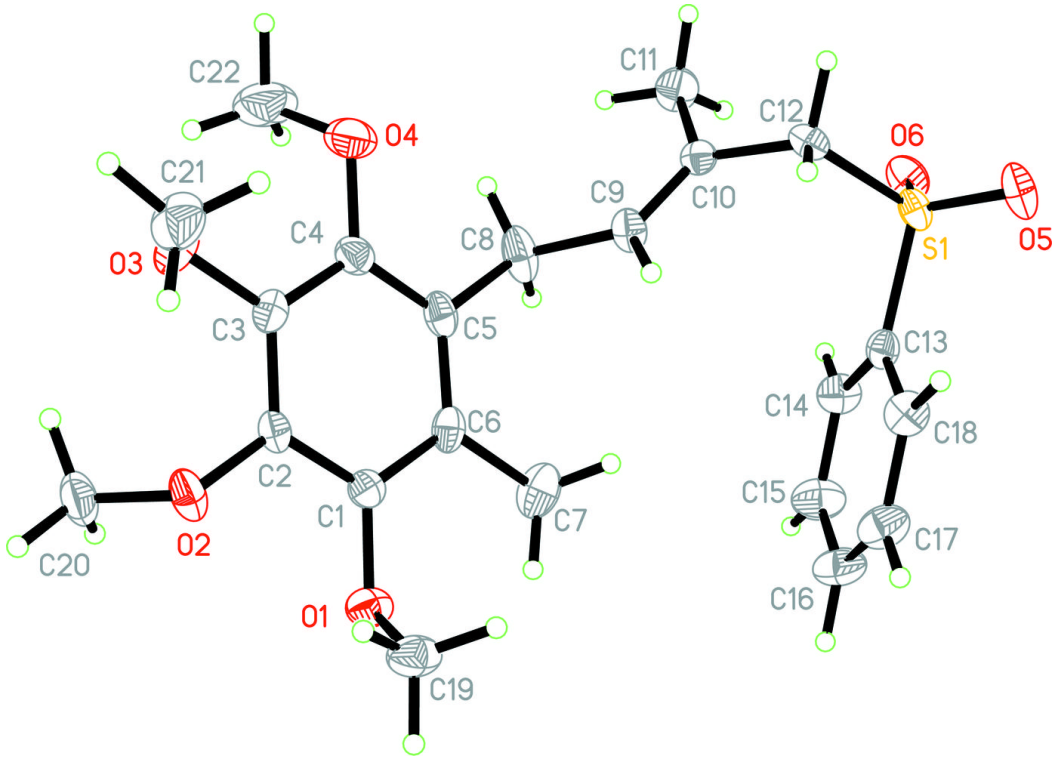
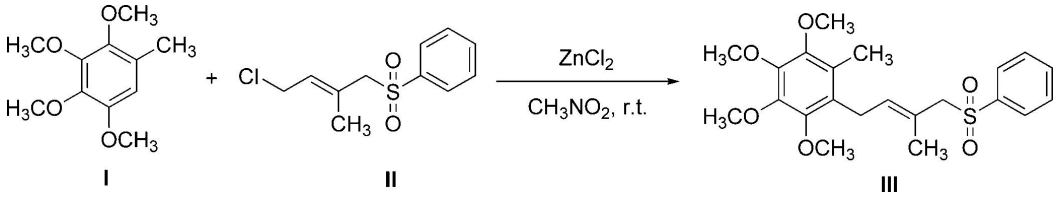

C22
O4
C21
O3
C4
C3
C5
C8
C9
C10
C11
C12
O6
S1
O5
C13
C14
C18
C15
C17
C16
C6
C2
C1
C7
O2
C20
O1
C19

Fig. 2


OCH3
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CH3
H3CO
OCH3
I
+
Cl
CH3
O
S
O
II
ZnCl2
CH3NO2, r.t.
OCH3
H3CO
CH3
H3CO
OCH3
CH3
O
S
O
III