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2-Isopropyl-4-methoxy-5-methylphenyl acetate

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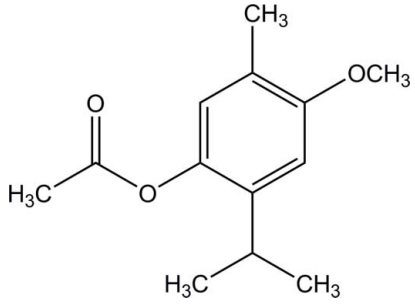
Key indicators: single-crystal X-ray study; T = 300 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.132; data-to-parameter ratio = 18.4.

In the title compound, $C_{13}H_{18}O_3$, the benzene ring is almost perpendicular to the acetoxy plane, making a dihedral angle of 89.33 (11)°. In the crystal, molecules are linked by weak C—H···O hydrogen bonds, forming a zigzag chain along the c-axis direction.

Related literature

For background to natural monoterpenic phenols and their derivatives, see: Yuan-Lang & Erdtman (1962); Ündeğer *et al.* (2009); Osorio *et al.* (2006). For a related structure, see: Rajouani *et al.* (2008).



Experimental

Crystal data

$C_{13}H_{18}O_3$
M_r = 222.27
Monoclinic, $P2_1/c$
a = 10.829 (2) Å
b = 9.600 (2) Å
c = 12.530 (3) Å
β = 100.34 (2)°
V = 1281.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 300 K
0.3 × 0.15 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.521, T_{max} = 0.992
3366 measured reflections
2780 independent reflections
1594 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
2 standard reflections every 60 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.132
S = 1.02
2780 reflections
151 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H1···O2^{i}	0.93	2.60	3.523 (3)	171

Symmetry code: (i) $x, -y-\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD-4 EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5314).

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supporting information

Acta Cryst. (2013). E**69**, o1715 [doi:10.1107/S160053681302922X]

2-Isopropyl-4-methoxy-5-methylphenyl acetate

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S1. Comment

The *p*-methoxythymol is a natural phenol extracted from heartwood of tetraclinis articulate (Yuan-Lang *et al.*, 1962). In several recent studies, natural monoterpenic phenols and their derivatives have revealed extensive biological activities, ranging from antimicrobial (Ündeğer *et al.*, 2009) to Antileishmanial and Cytotoxic activities (Osorio *et al.*, 2006). In the aim of preparing monoterpenic phenol derivatives, we report here, the hemisynthesis of 2-isopropyl-4-methoxy-5-methylphenyl acetate from naturally occurred *p*-methoxythymol. Thus, treatment of *p*-methoxythymol with acetic anhydride in pyridine, and provides the title compound as colorless crystals in 72.4% yield. Its structure was fully characterized by its mass and NMR spectroscopic data. Furthermore, the crystallographic study made it possible to determine the stereochemistry. The main geometric features of this group are in good agreement with those observed in a similar compound (Rajouani *et al.*, 2008).

S2. Experimental

A solution of *p*-methoxythymol (111 mg, 0.617 mmol) in acetic anhydride (20 ml) and pyridine (20 ml) was heated under reflux for 24 h. After cooling, the mixture was acidified with 1 N HCl solution then extracted with ether (3 × 20 ml). The organic layer was washed with water, dried on anhydrous Na_2SO_4 and then evaporated under reduced pressure. The obtained residue was chromatographied on silica gel column using hexane and ethyl acetate (97/3) as eluent, to give 2-isopropyl-4-methoxy-5-methylphenyl acetate (100 mg) in 72.4% yield.

S3. Refinement

All H-atoms were located in a difference map and refined using a riding model with C—H = 0.93–0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

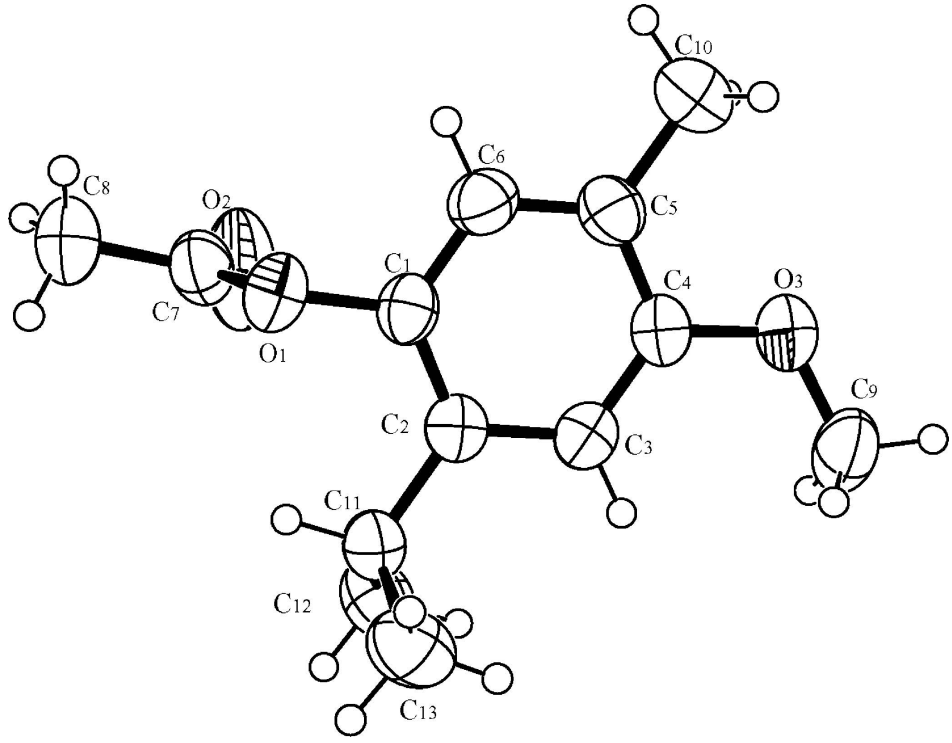

Figure 1

The molecule structure of the title compound with 50% probability ellipsoids.

2-Isopropyl-4-methoxy-5-methylphenyl acetate

Crystal data

$C_{13}H_{18}O_3$
$M_r = 222.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.829$ (2) Å
$b = 9.600$ (2) Å
$c = 12.530$ (3) Å
$\beta = 100.34$ (2)°
$V = 1281.4$ (5) Å^3
$Z = 4$
$F(000) = 480$
$D_x = 1.152$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 0.08$ mm^{-1}
$T = 300$ K
Prism, colourless
0.3 × 0.15 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.521$, $T_{max} = 0.992$
3366 measured reflections
2780 independent reflections
1594 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.7°$
$h = -13 \rightarrow 1$
$k = -1 \rightarrow 12$
$l = -15 \rightarrow 15$
2 standard reflections every 60 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.132$
$S = 1.02$
2780 reflections
151 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.2201P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.016 (3)

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 $F^2 > \sigma(F^2)$ 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}
O1	0.76460 (12)	0.08305 (13)	0.33870 (9)	0.0587 (4)
O2	0.76646 (18)	−0.07552 (19)	0.46746 (13)	0.1062 (7)
O3	0.43208 (12)	−0.20942 (15)	0.03401 (10)	0.0673 (4)
C1	0.68418 (16)	−0.00154 (18)	0.26312 (13)	0.0496 (4)
C2	0.73387 (16)	−0.09160 (17)	0.19660 (13)	0.0476 (4)
C3	0.64806 (16)	−0.16266 (18)	0.11839 (14)	0.0522 (4)
H1	0.6777	−0.2242	0.0715	0.063*
C4	0.52031 (16)	−0.14316 (18)	0.10945 (13)	0.0514 (4)
C5	0.47183 (17)	−0.0533 (2)	0.17877 (14)	0.0562 (5)
C6	0.55666 (17)	0.0175 (2)	0.25510 (14)	0.0570 (5)
H2	0.5272	0.0792	0.3020	0.068*
C7	0.79534 (17)	0.0364 (2)	0.44091 (15)	0.0612 (5)
C8	0.8695 (2)	0.1412 (3)	0.51249 (17)	0.0803 (7)
H3	0.8841	0.1081	0.5860	0.096*
H4	0.8239	0.2274	0.5082	0.096*
H5	0.9485	0.1559	0.4896	0.096*
C9	0.4748 (2)	−0.2910 (2)	−0.04666 (16)	0.0742 (6)
H6	0.5283	−0.2355	−0.0830	0.089*
H7	0.4041	−0.3229	−0.0983	0.089*
H8	0.5210	−0.3697	−0.0132	0.089*
C10	0.33271 (18)	−0.0334 (3)	0.16960 (19)	0.0812 (7)
H9	0.3168	0.0323	0.2233	0.097*
H10	0.2943	−0.1210	0.1810	0.097*

H11	0.2982	0.0011	0.0986	0.097*
C11	0.87402 (16)	−0.11360 (19)	0.20594 (15)	0.0546 (5)
H12	0.9162	−0.0529	0.2641	0.066*
C12	0.9114 (2)	−0.2629 (2)	0.23722 (18)	0.0747 (6)
H13	0.8718	−0.3250	0.1813	0.090*
H14	0.8850	−0.2857	0.3043	0.090*
H15	1.0009	−0.2723	0.2458	0.090*
C13	0.9195 (2)	−0.0725 (3)	0.10231 (18)	0.0785 (6)
H16	1.0093	−0.0797	0.1133	0.094*
H17	0.8949	0.0218	0.0837	0.094*
H19	0.8830	−0.1335	0.0445	0.094*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0685 (8)	0.0536 (7)	0.0512 (7)	−0.0086 (6)	0.0036 (6)	−0.0056 (6)
O2	0.1266 (15)	0.1007 (13)	0.0758 (10)	−0.0443 (11)	−0.0236 (10)	0.0304 (10)
O3	0.0564 (7)	0.0792 (9)	0.0621 (8)	−0.0073 (7)	−0.0007 (6)	−0.0123 (7)
C1	0.0553 (10)	0.0460 (10)	0.0448 (9)	−0.0017 (8)	0.0022 (8)	0.0014 (8)
C2	0.0509 (9)	0.0444 (9)	0.0464 (9)	−0.0011 (8)	0.0054 (7)	0.0033 (8)
C3	0.0574 (11)	0.0510 (10)	0.0475 (9)	−0.0004 (8)	0.0079 (8)	−0.0034 (8)
C4	0.0508 (10)	0.0521 (10)	0.0481 (9)	−0.0037 (8)	0.0006 (8)	0.0019 (8)
C5	0.0516 (10)	0.0583 (11)	0.0562 (10)	0.0076 (9)	0.0030 (8)	0.0052 (9)
C6	0.0624 (11)	0.0543 (11)	0.0543 (11)	0.0077 (9)	0.0108 (9)	−0.0040 (9)
C7	0.0533 (11)	0.0727 (13)	0.0545 (11)	−0.0043 (10)	0.0013 (9)	0.0009 (10)
C8	0.0706 (13)	0.0957 (17)	0.0675 (13)	−0.0095 (12)	−0.0071 (11)	−0.0155 (12)
C9	0.0796 (14)	0.0819 (15)	0.0586 (11)	−0.0200 (12)	0.0054 (10)	−0.0148 (11)
C10	0.0567 (12)	0.0940 (17)	0.0900 (16)	0.0120 (12)	0.0052 (11)	−0.0100 (13)
C11	0.0504 (10)	0.0560 (11)	0.0559 (10)	−0.0048 (9)	0.0053 (8)	−0.0048 (9)
C12	0.0615 (12)	0.0719 (14)	0.0860 (15)	0.0071 (11)	0.0006 (11)	0.0076 (12)
C13	0.0652 (13)	0.0903 (16)	0.0839 (15)	−0.0030 (12)	0.0236 (11)	0.0106 (13)

Geometric parameters (Å, º)

O1—C7	1.341 (2)	C11—C12	1.522 (3)
O1—C1	1.421 (2)	C8—H3	0.9600
O2—C7	1.183 (2)	C8—H4	0.9600
O3—C4	1.374 (2)	C8—H5	0.9600
O3—C9	1.420 (2)	C9—H6	0.9600
C1—C2	1.376 (2)	C9—H7	0.9600
C1—C6	1.379 (2)	C9—H8	0.9600
C2—C3	1.401 (2)	C10—H9	0.9600
C2—C11	1.516 (2)	C10—H10	0.9600
C3—C4	1.380 (2)	C10—H11	0.9600
C3—H1	0.9300	C11—H12	0.9800
C4—C5	1.392 (3)	C12—H13	0.9600
C5—C6	1.380 (2)	C12—H14	0.9600
C5—C10	1.502 (3)	C12—H15	0.9600

C6—H2	0.9300	C13—H16	0.9600
C7—C8	1.484 (3)	C13—H17	0.9600
C11—C13	1.521 (3)	C13—H19	0.9600
C7—O1—C1	117.57 (14)	O3—C9—H7	109.5
C4—O3—C9	118.02 (15)	H6—C9—H7	109.5
C2—C1—C6	122.33 (16)	O3—C9—H8	109.5
C2—C1—O1	120.23 (15)	H6—C9—H8	109.5
C6—C1—O1	117.31 (16)	H7—C9—H8	109.5
C1—C2—C3	116.58 (16)	C5—C10—H9	109.5
C1—C2—C11	122.40 (15)	C5—C10—H10	109.5
C3—C2—C11	121.02 (16)	H9—C10—H10	109.5
C4—C3—C2	121.33 (17)	C5—C10—H11	109.5
C4—C3—H1	119.3	H9—C10—H11	109.5
C2—C3—H1	119.3	H10—C10—H11	109.5
O3—C4—C3	123.79 (16)	C2—C11—C13	111.77 (15)
O3—C4—C5	115.01 (16)	C2—C11—C12	111.54 (15)
C3—C4—C5	121.20 (16)	C13—C11—C12	110.65 (17)
C6—C5—C4	117.32 (16)	C2—C11—H12	107.5
C6—C5—C10	121.61 (18)	C13—C11—H12	107.5
C4—C5—C10	121.07 (17)	C12—C11—H12	107.5
C1—C6—C5	121.23 (17)	C11—C12—H13	109.5
C1—C6—H2	119.4	C11—C12—H14	109.5
C5—C6—H2	119.4	H13—C12—H14	109.5
O2—C7—O1	122.63 (18)	C11—C12—H15	109.5
O2—C7—C8	125.96 (19)	H13—C12—H15	109.5
O1—C7—C8	111.41 (18)	H14—C12—H15	109.5
C7—C8—H3	109.5	C11—C13—H16	109.5
C7—C8—H4	109.5	C11—C13—H17	109.5
H3—C8—H4	109.5	H16—C13—H17	109.5
C7—C8—H5	109.5	C11—C13—H19	109.5
H3—C8—H5	109.5	H16—C13—H19	109.5
H4—C8—H5	109.5	H17—C13—H19	109.5
O3—C9—H6	109.5		
C7—O1—C1—C2	−95.9 (2)	C3—C4—C5—C6	1.3 (3)
C7—O1—C1—C6	88.2 (2)	O3—C4—C5—C10	0.3 (3)
C6—C1—C2—C3	0.8 (3)	C3—C4—C5—C10	−179.34 (18)
O1—C1—C2—C3	−174.89 (14)	C2—C1—C6—C5	−0.3 (3)
C6—C1—C2—C11	−179.47 (17)	O1—C1—C6—C5	175.57 (16)
O1—C1—C2—C11	4.8 (2)	C4—C5—C6—C1	−0.8 (3)
C1—C2—C3—C4	−0.3 (2)	C10—C5—C6—C1	179.85 (19)
C11—C2—C3—C4	179.98 (16)	C1—O1—C7—O2	6.1 (3)
C9—O3—C4—C3	−7.0 (3)	C1—O1—C7—C8	−174.65 (16)
C9—O3—C4—C5	173.38 (16)	C1—C2—C11—C13	−118.73 (19)
C2—C3—C4—O3	179.64 (15)	C3—C2—C11—C13	61.0 (2)
C2—C3—C4—C5	−0.8 (3)	C1—C2—C11—C12	116.80 (19)
O3—C4—C5—C6	−179.06 (15)	C3—C2—C11—C12	−63.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H1···O2^{i}	0.93	2.60	3.523 (3)	171

Symmetry code: (i) *x*, −*y*−1/2, *z*−1/2.