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1-(5-Chloro-6-methoxynaphthalen-2-yl)-propan-1-one

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.082
Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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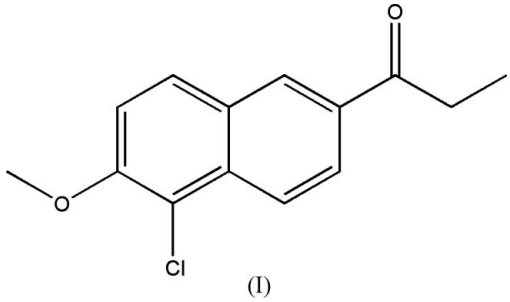
The title compound, $C_{14}H_{13}ClO_2$, has been synthesized from 2-methoxynaphthalene *via* chlorination by cupric chloride and reaction with propionyl chloride. The 6-methoxy and 2-propionyl groups are coplanar with the naphthalene ring system. The molecules are packed in a head-to-tail arrangement showing π–π stacking interactions.

Comment

1-(5-Chloro-6-methoxynaphthalen-2-yl)propan-1-one, (I), is an important intermediate in the synthesis of (*S*)-(+)-2-(6-methoxynaphthalen-2-yl)propanoic acid, known as naproxen, which is a medicament possessing anti-inflammatory and analgesic activity. It is used to reduce pain, inflammation and stiffness caused by many conditions, such as osteoarthritis, rheumatoid arthritis, gout, ankylosing spondylitis, injury, abdominal cramps associated with menstruation, tendinitis and bursitis. Compound (I) was synthesized by a Friedel–Crafts reaction between propionyl chloride and 1-chloro-2-methoxynaphthalene, conducted in dichloromethane in the presence of aluminium trichloride (Claudio, 1989). 1-Chloro-2-methoxynaphthalene was synthesized from 2-methoxynaphthalene, using cupric chloride as chlorinating agent.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The 6-methoxy and 2-propionyl groups are coplanar with the naphthalene ring system. Molecules exhibit a head-to-tail arrangement in the crystal structure, which is stabilized by face-to-face π–π stacking interactions. Adjacent naphthalene units are exactly parallel and the centroid–centroid separations between C1–C4/C9/C10 rings are 3.690 and 3.776 Å (Fig.2).

Experimental

To a 250 ml three-necked flask were added 2-methoxynaphthalene (3.2 g), $CuCl_2$ (5.4 g) and chlorobenzene (100 ml). The mixture was stirred and heated to reflux for 6 h. After the reaction was complete (monitored by thin-layer chromatography), the CuCl was removed by

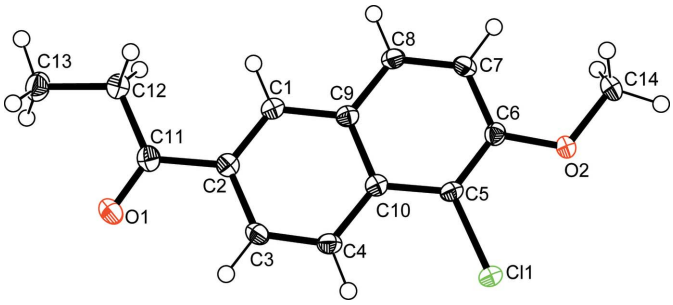


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

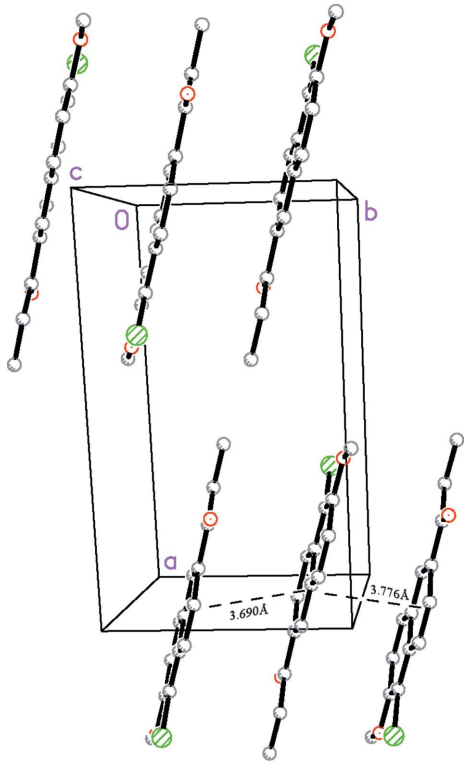


Figure 2
The crystal packing for (I), showing π–π stacking interactions as dashed lines.

filtration. The filtrate was cooled in an ice bath, giving a white precipitate, which was filtered off and dried to give 3.75 g 1-chloro-2-methoxynaphthalene in 97.5% yield (m.p. 339–339 K). A mixture of propionyl chloride (1.11 g), $AlCl_3$ (1.8 g) and dichloromethane (30 ml) was cooled to about 273 K, and a solution of 1-chloro-2-methoxynaphthalene (1.93 g) in dichloromethane (30 ml) was added dropwise to the mixture with stirring, maintaining the temperature below 278 K. The reaction mixture was stirred for 15 min at this temperature and then poured into ice-cold hydrochloric acid (50 ml, 2 mol l^{-1}). The organic layer was washed with hydrochloric acid (10 ml) once and three times with water (20 ml). It was then dried over anhydrous Na_2SO_4, and dichloromethane was removed by distillation to yield the crude product. This was dissolved in hot ethanol (25 ml), cooled to crystallize, filtered off and dried to give 2.42 g of (I) in 97.2% yield (m.p. 402–404 K). Single crystals of (I) were obtained by slow evaporation of an ethanol solution at room temperature.

Crystal data

$C_{14}H_{13}ClO_2$
M_r = 248.69
Monoclinic, $P2_1/n$
a = 12.9248 (6) Å
b = 7.1566 (3) Å
c = 13.2007 (6) Å
β = 105.0500 (10)°
V = 1179.15 (9) Å^3
Z = 4
D_x = 1.401 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 173 (2) K
Block, colourless
0.46 × 0.34 × 0.18 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.871, T_{max} = 0.946
8862 measured reflections
2309 independent reflections
2078 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.082
S = 1.00
2309 reflections
157 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.5817P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.019 (2)

H atoms were placed in calculated positions, with C—H distances of 0.99 (methylene), 0.98 (methyl) and 0.95 Å (aromatic), and with U_{iso}(H)= 1.5U_{eq}(methyl C) and 1.2U_{eq}(C). The methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

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