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Methyl 12-chlorodehydroabietate

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Key indicators

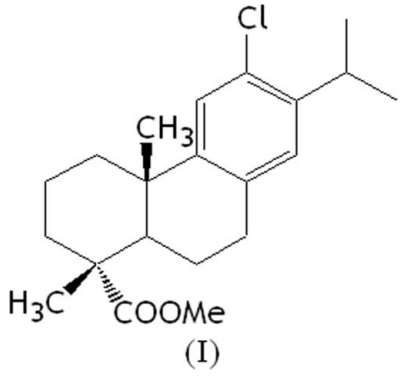
Single-crystal X-ray study
T = 163 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.081
Data-to-parameter ratio = 14.8

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

The structure of the title compound (systematic name: methyl 6-chloro-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthrene-1-carboxylate), $C_{21}H_{29}ClO_2$, has been determined by X-ray diffraction and is compared with the structures of methyl 12-acetyldehydroabietate and methyl dehydroabietate.

Comment

The structure of methyl dehydroabietate, its use as a source of fine chemicals (Zinkel & Russell, 1989), and the use of its chloro derivatives as important intermediates led us to the synthesis of methyl 12-chlorodehydroabietate, (I).



(I)

The bond dimensions of (I) compare well with those of methyl 12-acetyldehydroabietate (Silvestre *et al.*, 1998) and methyl dehydroabietate (Hamodrakas *et al.*, 1978).

The absolute configuration of the title compound, determined from anomalous scattering effects, is consistent with those of methyl 12-acetyldehydroabietate (Silvestre *et al.*, 1998) and methyl dehydroabietate (Hamodrakas *et al.*, 1978). In addition, NMR studies of analgous compounds suggest that the configuration is retained through the course of the reaction.

Experimental

To a mixture of methyl 12-aminedehydroabietate (1.0 g, 3.04 mmol) and hydrochloric acid (20 ml), sodium nitrite (0.21 g, 3.10 mmol) dissolved in water (5 ml) was added dropwise with stirring in an ice bath. The resultant solution was stirred for another 30 min and enough sodium acetate was added to bring the solution to pH 4. The mixture was then treated with a solution of sodium ethoxide (0.25 g, 3.10 mmol) in ethanol (15 ml) for 1.5 h and filtered to obtain a yellow solid. Upon recrystallization from hot ethanol, colourless crystals (0.3 g, 0.86 mmol) were obtained in 28% yield (m.p. 409–411 K).

Crystal data

$C_{21}H_{29}ClO_2$
M_r = 348.89
Monoclinic, $C2$
a = 13.6507 (17) Å
b = 6.0856 (6) Å
c = 23.120 (3) Å
β = 102.760 (16)°
V = 1873.2 (4) Å^3
Z = 4
D_x = 1.237 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 163 (2) K
Platelet, colourless
0.56 × 0.41 × 0.06 mm

Data collection

Rigaku Mercury diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.856, T_{max} = 0.987
9174 measured reflections
3282 independent reflections
3079 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.081
S = 1.09
3282 reflections
222 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 0.9195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), with 1402 Friedel pairs
Flack parameter: 0.02 (6)

H atoms bound to C atoms were positioned geometrically and included in the refinement in the riding-model approximation, with C—H = 0.95, 0.98, 0.99 and 1.00 Å for aromatic, methyl, CH_2 and CH groups, respectively, and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H and $1.2U_{eq}(C)$ for all others.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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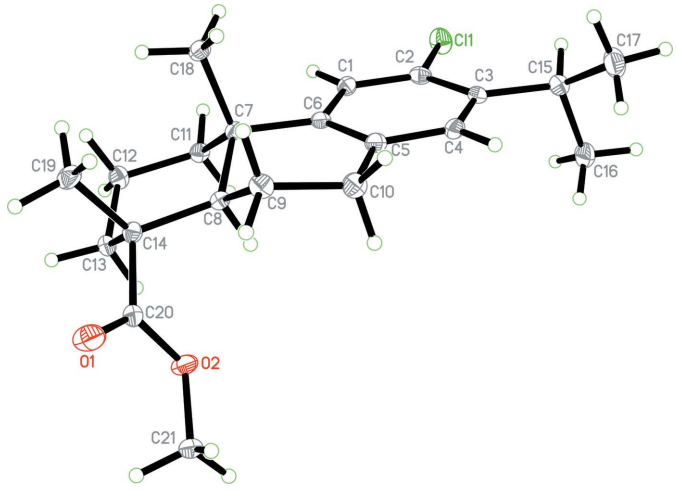

Figure 1
The absolute configuration of (I), showing 30% probability displacement ellipsoids and the atom-labelling scheme. H atoms are represented by small spheres of arbitrary radii.

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