

## Methyl 12-benzoyldehydroabietate

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## Key indicators

Single-crystal X-ray study  
 $T = 193\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.041  
 $wR$  factor = 0.088  
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>.

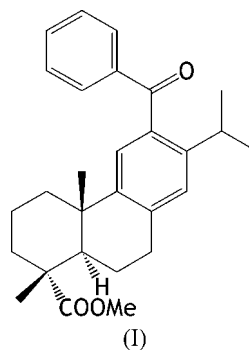
The title compound,  $\text{C}_{28}\text{H}_{34}\text{O}_3$ , is a new derivative of dehydroabietic acid, whose *A* and *B* rings exhibit a classical chair and half-chair conformation, respectively.

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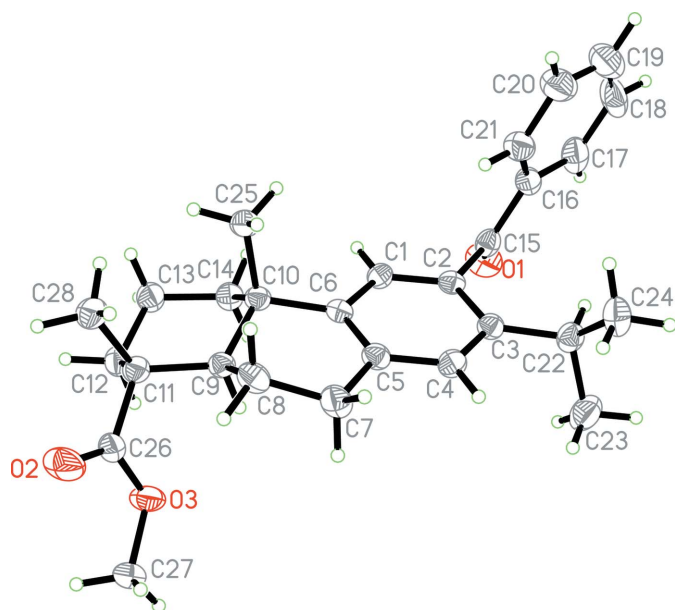
## Comment

Pine resin is a very abundant renewable source mainly composed of diterpenic resin acids of the general formula  $\text{C}_{19}\text{H}_{29}\text{COOH}$ . This raw material has a wide range of industrial applications and is also a source of fine chemicals (Zinkel & Russell, 1989). Dehydroabietic acid can be easily obtained by catalytic dehydrogenation of abietic acids. Dehydroabietic acid derivatives have shown a broad spectrum of biological activities, such as antifungal and tumor-inhibitory (Silvestre *et al.*, 1998; Fonseca *et al.*, 2004). Considerable interest has been devoted to this easily available compound as a starting material, either as the free acid or ester, for the synthesis of other important natural or bioactive compounds mainly through transformations that involve the benzylic or aromatic sites of the molecule (Roy *et al.*, 2003). Recently, we reported the structure of methyl 12-bromo-13,14-dinitrodeisopropyl dehydroabietate (Pan *et al.*, 2005), which is a derivative of dehydroabietic acid. The present structure determination was performed within the same project.

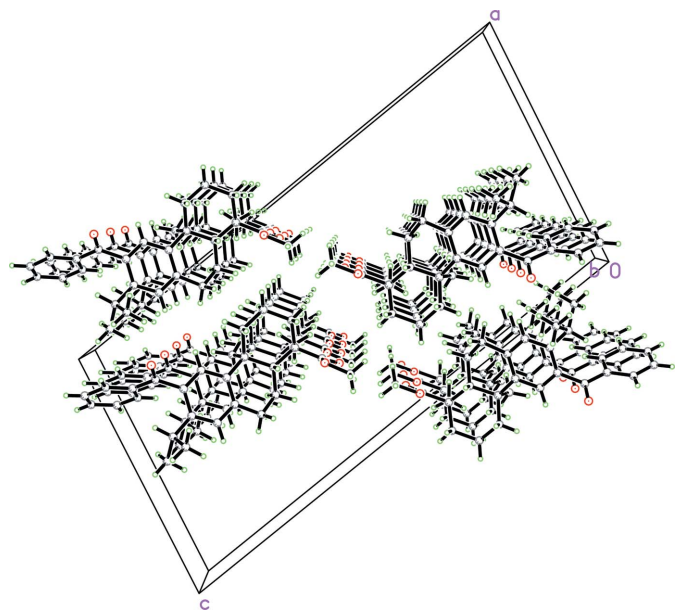


The molecular structure of the title compound, (I), is shown in Fig. 1. As expected for diterpenes (Pan *et al.*, 2005; Silvestre *et al.*, 1998), rings *A* (atoms C9–C14) and *B* (atoms C5–C10) show a *trans* ring junction, with two methyl groups in axial positions of the six-membered rings. The torsion angles are in accordance with a classical chair conformation and a half-chair conformation for rings *A* and *B*, respectively.

The overall geometry of (I) is comparable to that found for 12-acetyl dehydroabietate (Silvestre *et al.*, 1998), methyl dehydroabietate (Hamodrakas *et al.*, 1978) and methyl 12-bromo-13,14-dinitrodeisopropyl dehydroabietate (Pan *et al.*



**Figure 1**  
The structure of (I), showing 50% probability displacement ellipsoids and the atom labeling scheme. H atoms are represented by small spheres of arbitrary radii.



**Figure 2**  
The crystal structure of the title compound, viewed along the *b* axis.

2005), apart from the substituted benzoyl at the benzene ring. The ester group is planar.

In the crystal structure, the molecules are stacked in the direction of the crystallographic *b* axis, indicating weak  $\pi$ - $\pi$  stacking interactions (Fig. 2).

## Experimental

A mixture of methyl dehydroabietate (6.8 mmol), 1,1,2,2-tetrachloroethane (20 ml), benzoyl chloride (1.4 ml) and aluminium chloride (13.6 mmol) was stirred at room temperature for 24 h. The

resulting solution was treated with 0.5 mol l<sup>-1</sup> hydrochloric acid (28 ml). 1,1,2,2-Tetrachloroethane was then removed by steam distillation and the residue was filtered through a Buchner funnel to obtain a pale solid. Upon recrystallization from methanol, white crystals of (I) were obtained (yield 81%, m.p. 425.6–426.5 K). Single crystals were grown from a mixed solvent of absolute methanol and chloroform in the volume ratio 1:1.

## Crystal data

C<sub>28</sub>H<sub>34</sub>O<sub>3</sub>  
*M<sub>r</sub>* = 418.55  
 Monoclinic, *C*2  
*a* = 14.0617 (13) Å  
*b* = 6.0363 (5) Å  
*c* = 28.051 (4) Å  
 $\beta$  = 102.40 (2)°  
*V* = 2325.4 (4) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.196 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 $\mu$  = 0.08 mm<sup>-1</sup>  
*T* = 193 (2) K  
 Block, colorless  
 0.35 × 0.30 × 0.20 mm

## Data collection

Rigaku Mercury CCD  
 diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan  
 (REQABS; Jacobson, 1998)  
*T<sub>min</sub>* = 0.836, *T<sub>max</sub>* = 0.985

11429 measured reflections  
 4202 independent reflections  
 3794 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.027  
 $\theta_{\max}$  = 25.4°

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.041  
*wR* (*F*<sup>2</sup>) = 0.088  
*S* = 1.10  
 4202 reflections  
 285 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.4191P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.11 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.14 \text{ e \AA}^{-3}$

H atoms were positioned with idealized geometry and included in the refinement in the riding-model approximation [*C*–*H* = 0.95, 0.98, 0.99 and 1.00 Å for aromatic, methyl, CH<sub>2</sub> and CH groups, respectively, and *U<sub>iso</sub>*(H) = 1.5*U<sub>eq</sub>*(methyl C) and 1.2*U<sub>eq</sub>*(all other C)]. In the absence of significant anomalous scattering effects, Friedel pairs were averaged. The absolute configuration was assigned according to the known absolute configuration of the enantiomeric pure compound.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MS, 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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