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1-(Cholest-4-en-3β-yl)-2,2,2-trichloroethanimidate *tert*-butyl methyl ether hemisolvate

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Key indicators

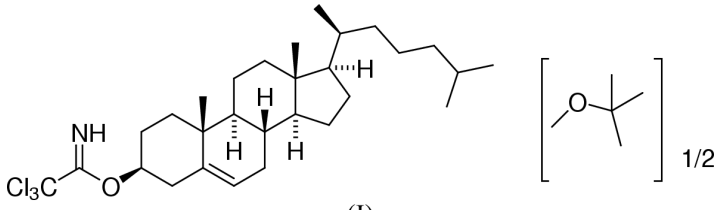
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.010 Å
Disorder in solvent or counterion
R factor = 0.078
wR factor = 0.221
Data-to-parameter ratio = 8.2

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

The title compound, $C_{29}H_{45}Cl_3NO{\cdot}0.5C_5H_{12}O$, is a rare example of a crystalline trichloroacetimidate and has a structure similar to that reported for analogous species.

Comment

As part of our studies on the synthesis and biological properties of certain terpene peroxides (Fielder *et al.*, 1996, 1998), we investigated the preparation of hydroperoxides from trichloroacetimidates by a modification of the reported procedure for dialkyl peroxides (Bourgeois *et al.*, 1993). The X-ray structure determination of the title compound, (I), was performed as a means of structural identification, since (i) the compound could not be transformed into peroxide analogues, and (ii) the compound was crystalline, evidently a very unusual property for trichloroacetimidates (Clegg *et al.*, 1995).



(I)

The structure was solved in C2 with $Z = 2$ and so (I) is chiral: the correct enantiomer was determined unambiguously. While there are very few structurally characterized trichloroacetimidates (Nakano *et al.*, 1988; Clegg *et al.*, 1995; Ernst *et al.*, 1998), they all show generally similar features to the title compound. The C_3NO skeleton has essentially a planar configuration with a r.m.s. deviation from planarity of 0.024 Å.

Experimental

The title compound was prepared in 67% yield, using a modification of the standard procedure (Overman, 1976). IR max (KBr disc): 3342 (N—H), 1654 (C═NH) cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$): δ 8.24 (1H, *s*, ═NH), 5.42 (1H, *m*, ═CH–), 4.77 [1H, *m*, –CH(O*R*)–], 2.47 (2H, *m*, –CH_2–C═), 2.05–0.92 (26H, *m*), 1.07 (3H, *s*, –CH_3), 0.93 [3H, *d*, *J* = 6.4 Hz, –CH(CH_3)–], 0.88 [3H, *d*, *J* = 6.6 Hz, –CH(CH_3)CH_3], 0.87 [3H, *d*, *J* = 6.6 Hz, –CH(CH_3)CH_3], 0.69 (3H, *s*, –CH_3) p.p.m.

Crystal data

$C_{29}H_{45}Cl_3NO{\cdot}0.5C_5H_{12}O$
M_r = 574.08
Monoclinic, *C*2
a = 29.037 (13) Å
b = 9.072 (6) Å
c = 12.628 (6) Å
β = 109.14 (7)°
V = 3143 (3) Å^3
Z = 4
D_x = 1.213 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9–16°
μ = 0.32 mm^{-1}
T = 173 (2) K
Plate, colorless
0.50 × 0.50 × 0.30 mm

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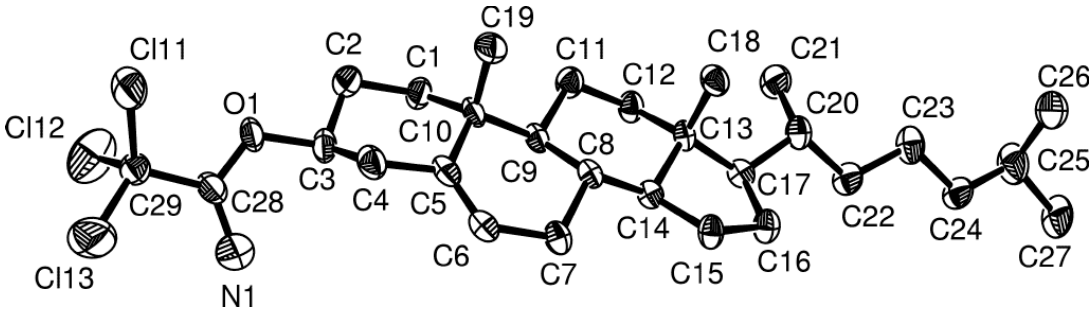

Figure 1
The molecular structure of the title compound showing 50% probability displacement ellipsoids (Farrugia, 1997). The *tert*-butyl methyl ether solvate and all H atoms have been omitted for clarity.

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: empirical (North *et al.*, 1968)
T_{min} = 0.857, T_{max} = 0.911
3008 measured reflections
2926 independent reflections
2640 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -33 \rightarrow 0$
$k = 0 \rightarrow 10$
$l = -12 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.221
S = 1.03
2926 reflections
358 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1249P)^2 + 13.5363P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.048
$\Delta\rho_{max}$ = 0.97 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.00 (18)

A disordered half molecule of the crystallization solvent, *tert*-butyl methyl ether, is present in the lattice and therefore the C—C distances and the C—O distances were fixed at 1.55 (1) and 1.45 (1) Å, respectively, using the command *DFIX* (Sheldrick, 1997). All non-H atoms are refined anisotropically. All H atoms were placed in calculated positions, refined using a riding model, and given an isotropic displacement parameter equal to 1.2 times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: *CAD*-4/*PC* (Enraf–Nonius, 1993); cell refinement: *CAD*-4/*PC*; data reduction: *XCAD*4*B* (Harms, 1994); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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