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2-Butanoyloxy-5-nitrotropone

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.006 Å
R factor = 0.055
wR factor = 0.168
Data-to-parameter ratio = 15.8

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

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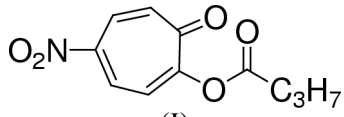
In the title compound, 4-nitro-7-oxocyclohepta-1,3,5-trienyl butanoate, $C_{11}H_{11}NO_5$, the tropolone ring is approximately perpendicular to the ester plane [dihedral angle 71.8 (1)°] and the paraffin chain has *trans* and *gauche* conformations. Intermolecular π–π interactions between the tropolone planes are observed in the crystalline state.

Comment

Troponoids have been an important building block for constructing liquid crystals (Mori & Takeshita, 1995). Recently, we prepared liquid crystals with a troponoid core which has enhanced the formation of smectic phases when compared with the corresponding benzenoids (Mori & Takeshita, 1995; Hashimoto *et al.*, 2000). The crystal structures of cores such as tropolone, 5-nitrotropolone and 5-cyanotropolone rings have been elucidated by X-ray analyses (Shimanouchi & Sasada, 1973; Kubo *et al.*, 2001). In order to reveal the effect upon crystal packing of substitution at O2 of 5-nitrotropolone, we now report the structure of the title compound, (I), as shown in Fig. 1.



(I)

The seven-membered ring in (I) is nearly planar; the respective deviations of each atom from the least-squares plane *A*, defined by atoms C1–C7/O1/O2, are 0.033 (4), 0.064 (4), 0.048 (4), −0.031 (5), −0.064 (4), 0.022 (4), 0.083 (5), −0.040 (3) and −0.015 (3) Å. The dihedral angle between the least-squares planes through *A* and *B* [defined by atoms O2, O5 and C8] is 71.8 (1)°, which is similar to that in tropolonyl *p*-chlorobenzoate, 71.5° (Shaefer & Reed, 1971). The C—C bond lengths of the seven-membered ring of (I) are similar to those of tropone (Barrow *et al.*, 1973), but are distinct from 5-nitrotropolone (Kubo *et al.*, 2001). The paraffin chain has *trans* and *gauche* conformations.

Intermolecular π–π interactions are observed between the tropolone dimer planes (head-to-tail) of (I) (Fig. 2). The distance between intermolecular tropolone planes is 3.461 (5) Å for C1—C4^{i} [symmetry code: (i) $1-x, -y, 1-z$], which is similar to the distance of 3.40 Å found in 5-nitrotropolone (Kubo *et al.*, 2001). However, the packing in (I) is distinct from that of 5-nitrotropolone (Kubo *et al.*, 2001), which features intermolecular $NO_2\cdots\pi$–π interactions. Thus, the substitution at O2 results in a different crystal-packing arrangement.

Experimental

Compound (I) was prepared by esterification of 5-nitrotropolone with butanoyl chloride. The single crystals of (I) were obtained by recrystallization from a chloroform solution of the compound.

Crystal data

$C_{11}H_{11}NO_5$	D_x = 1.404 Mg m^{-3}
M_r = 237.21	Mo $K\alpha$ radiation
Monoclinic, $P2_1/a$	Cell parameters from 21 reflections
a = 12.8585 (14) Å	θ = 9.1–18.1°
b = 10.7092 (11) Å	μ = 0.11 mm^{-1}
c = 8.3657 (14) Å	T = 296 (2) K
β = 103.052 (11)°	Prism, yellow
V = 1122.2 (3) Å^3	0.33 × 0.23 × 0.23 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.073
ω–2θ scans	θ_{max} = 27.0°
ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 16$
T_{min} = 0.981, T_{max} = 1.000	$k = -13 \rightarrow 0$
2559 measured reflections	$l = -10 \rightarrow 10$
2450 independent reflections	3 standard reflections
953 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: 0.1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.168	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
S = 0.95	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
2450 reflections	Extinction correction: *SHELXL*97
155 parameters	Extinction coefficient: 0.018 (3)
H-atom parameters constrained	

All H atoms were fixed at ideal positions and restrained with U_{iso} held fixed to 1.2U_{eq} of the parent atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Xtal_GX* (Hall & du Boulay, 1995); software used to prepare material for publication: *SHELXL*97.

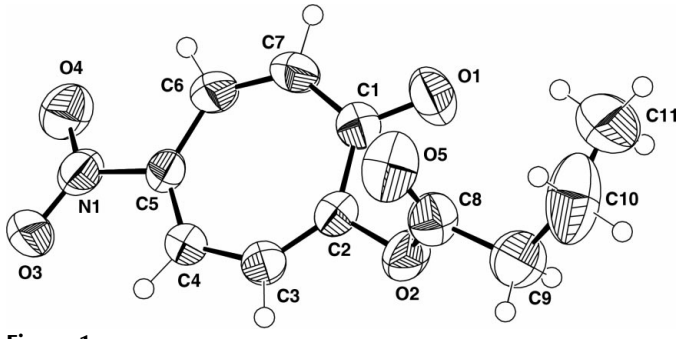


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids (Johnson, 1976).

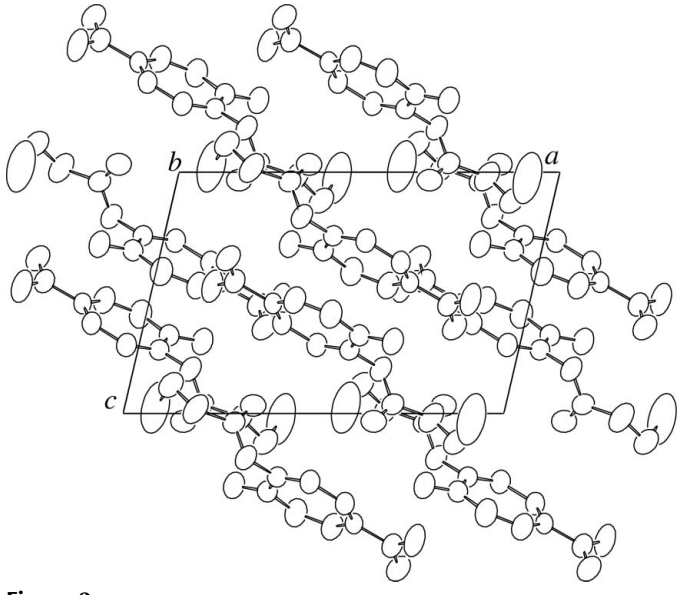


Figure 2
Packing diagram of (I) viewed down the *b* axis. H atoms have been omitted for clarity.

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