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rac-Methyl 2-(2-formyl-4-nitrophenoxy)-hexanoate

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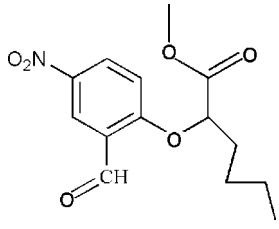
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.076; wR factor = 0.172; data-to-parameter ratio = 15.8.

In the racemic title compound, $C_{14}H_{17}NO_6$, the plane of the ester group of the methyl hexanoate side chain makes a dihedral angle of 80.0 (2)° with the benzene ring, while the nitro group is approximately coplanar with the benzene ring [dihedral angle = 10.3 (2)°]. In the crystal, molecules form weak aromatic C—H···O_{nitro} hydrogen-bonding interactions, giving inversion dimers [graph set $R_2^2(8)$].

Related literature

For applications of the title compound, see: Dale & White (2007). For graph-set analysis, see: Etter *et al.* (1990)



Experimental

Crystal data

$C_{14}H_{17}NO_6$ M_r = 295.29
Monoclinic, $P2_1/n$
a = 14.918 (3) Å
b = 4.922 (1) Å
c = 20.928 (4) Å
β = 103.26 (3)°
V = 1495.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.980, T_{max} = 0.990
2722 measured reflections
2722 independent reflections
1228 reflections with $I > 2\sigma(I)$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.172
S = 1.00
2722 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O1^{i}	0.93	2.52	3.442 (6)	169

Symmetry code: (i) $-x, -y+2, -z+1$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2196).

References

Dale, K. M. & White, C. M. (2007). *Ann. Pharmacother.* **41**, 599–605.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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rac-Methyl 2-(2-formyl-4-nitrophenoxy)hexanoate

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S1. Comment

The title compound, $C_{14}H_{17}NO_6$ is a good organic intermediate for the synthesis of the drug dronedarone, an important drug used to treat cardiac arrhythmia (Dale & White, 2007), and its crystal structure is reported herein.

In the title compound (Fig. 1), both the nitro group and the aldehyde group are approximately coplanar with the benzene ring, as shown by the torsion angles O1—N—C3—C4 [170.9 (4)°] and C6—C5—C7—O3 [177.6 (4)°]. The plane of the ester group of the methyl hexanoate side chain makes a dihedral angle of 80.0 (2)° with the benzene ring. In the crystal, the molecules are linked by weak intermolecular aromatic C2—H···$O1_{nitro}$ hydrogen-bonding interactions (Table 1), giving centrosymmetric cyclic dimers [graph set $R^2_2(8)$ (Etter *et al.*, 1990)]. Also present are intramolecular interactions between the aldehyde and methylene C—H groups and the ether O-atom.

S2. Experimental

A mixture of 5-nitrosalicylaldehyde (0.2 mol, 33.4 g), methyl 2-bromohexanoate (2-bromhexine acid methyl ester) (0.2 mol, 41.8g) and anhydrous potassium carbonate (0.2 mol, 27.6g) in DMF (400 ml) was reacted for 3.5h at 365-367 K. After the completion of the reaction, the precipitate was filtered and washed and the product (0.1 g) was crystallized from 15 ml of CH_3OH at room temperature to give colorless crystals from which a specimen was selected for X-ray data collection.

S3. Refinement

All H atoms were placed in calculated positions and treated as riding, with C—H = 0.93, 0.98, 0.97 and 0.96 Å for CH(aromatic), C—H(aliphatic), CH, CH_2 and CH_3 H atoms, respectively and with U_{iso}(H) = k × U_{eq}(C), where k = 1.5 for CH_3 H-atoms and k = 1.2 for all other H-atoms.

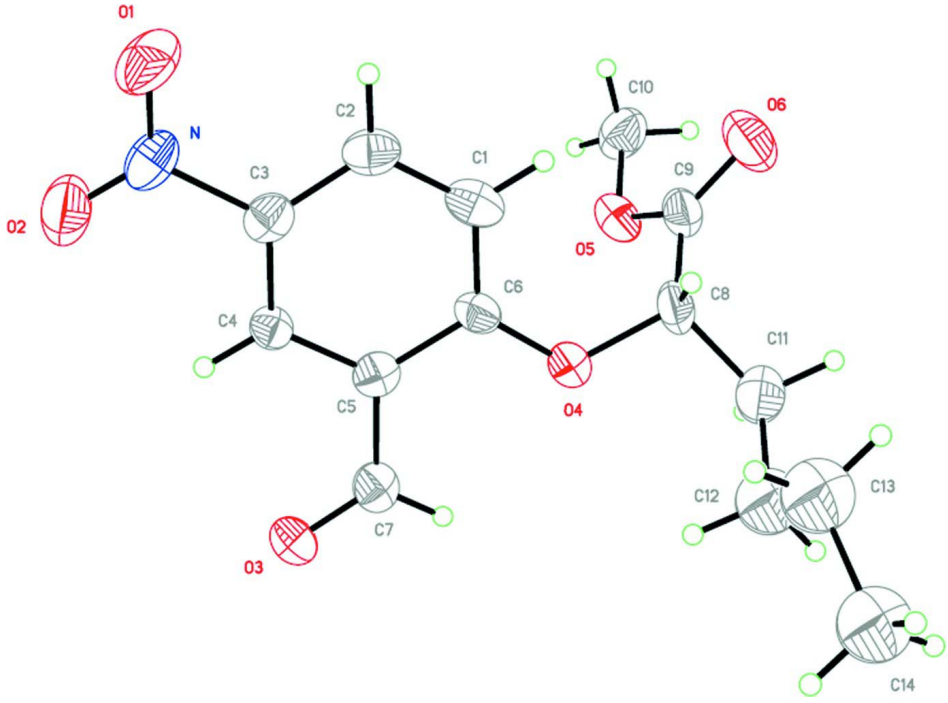

Figure 1

The structure of the title compound, showing the atom numbering scheme. Non-H atoms are shown as 30% probability displacement ellipsoids.

rac-Methyl 2-(2-formyl-4-nitrophenoxy)hexanoate

Crystal data

$C_{14}H_{17}NO_6$
$M_r = 295.29$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 14.918$ (3) Å
$b = 4.922$ (1) Å
$c = 20.928$ (4) Å
$\beta = 103.26$ (3)°
$V = 1495.7$ (5) Å^3
$Z = 4$

$F(000) = 624$
$D_x = 1.311$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω–2θ scans
Absorption correction: ψ scan (North et al., 1968)
$T_{min} = 0.980$, $T_{max} = 0.990$
2722 measured reflections

2722 independent reflections
1228 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.5°$
$h = -17\rightarrow17$
$k = 0\rightarrow5$
$l = 0\rightarrow25$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.172$
$S = 1.00$
2722 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.050P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N	−0.0326 (2)	0.6463 (8)	0.59117 (19)	0.0672 (11)
C1	0.2020 (3)	0.5437 (8)	0.56249 (18)	0.0592 (11)
H1A	0.2389	0.6148	0.5363	0.071*
O1	−0.0535 (2)	0.8534 (7)	0.55953 (18)	0.0926 (11)
C2	0.1162 (3)	0.6501 (9)	0.55803 (19)	0.0604 (11)
H2A	0.0946	0.7942	0.5300	0.072*
O2	−0.0834 (2)	0.5218 (8)	0.61742 (19)	0.1013 (13)
C3	0.0617 (3)	0.5338 (9)	0.59740 (18)	0.0544 (10)
O3	0.16665 (19)	−0.0836 (7)	0.72916 (13)	0.0747 (10)
O4	0.31794 (16)	0.2096 (6)	0.61157 (12)	0.0598 (8)
C4	0.0913 (2)	0.3232 (7)	0.64028 (16)	0.0437 (9)
H4A	0.0533	0.2496	0.6654	0.052*
C5	0.1794 (2)	0.2243 (8)	0.64485 (17)	0.0496 (9)
O5	0.2966 (2)	0.0517 (6)	0.48813 (13)	0.0675 (9)
C6	0.2353 (3)	0.3330 (8)	0.60504 (17)	0.0529 (10)
O6	0.3773 (2)	0.3910 (8)	0.46304 (16)	0.0990 (12)
C7	0.2119 (3)	0.0063 (8)	0.69327 (17)	0.0549 (10)
H7A	0.2704	−0.0651	0.6961	0.066*
C8	0.3791 (3)	0.3212 (9)	0.5738 (2)	0.0645 (12)
H8A	0.3839	0.5183	0.5803	0.077*
C9	0.3496 (3)	0.2609 (11)	0.5030 (2)	0.0663 (12)
C10	0.2656 (3)	−0.0163 (11)	0.41826 (19)	0.0806 (15)
H10A	0.2268	−0.1741	0.4135	0.121*
H10B	0.3180	−0.0529	0.4003	0.121*
H10C	0.2315	0.1337	0.3953	0.121*

C11	0.4715 (3)	0.1900 (12)	0.6028 (2)	0.0907 (17)
H11A	0.4652	−0.0034	0.5941	0.109*
H11B	0.5153	0.2588	0.5790	0.109*
C12	0.5116 (4)	0.2243 (12)	0.6721 (3)	0.107
H12A	0.4667	0.1703	0.6967	0.128*
H12B	0.5642	0.1040	0.6846	0.128*
C13	0.5406 (4)	0.4958 (13)	0.6900 (3)	0.122
H13A	0.4881	0.6034	0.6949	0.147*
H13B	0.5656	0.5768	0.6555	0.147*
C14	0.6167 (4)	0.4976 (13)	0.7569 (3)	0.123
H14A	0.6367	0.6807	0.7676	0.184*
H14B	0.6683	0.3891	0.7521	0.184*
H14C	0.5912	0.4241	0.7913	0.184*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.055 (2)	0.056 (3)	0.086 (3)	0.014 (2)	0.008 (2)	−0.010 (2)
C1	0.088 (3)	0.040 (2)	0.050 (2)	−0.006 (2)	0.018 (2)	−0.0012 (19)
O1	0.079 (2)	0.073 (2)	0.122 (3)	0.031 (2)	0.016 (2)	0.010 (2)
C2	0.073 (3)	0.048 (3)	0.057 (2)	0.008 (2)	0.008 (2)	0.004 (2)
O2	0.063 (2)	0.111 (3)	0.138 (3)	0.010 (2)	0.040 (2)	0.022 (3)
C3	0.053 (2)	0.057 (3)	0.050 (2)	0.003 (2)	0.0052 (17)	−0.010 (2)
O3	0.0661 (19)	0.094 (3)	0.0691 (18)	−0.0031 (18)	0.0249 (15)	0.0283 (18)
O4	0.0533 (16)	0.074 (2)	0.0563 (16)	0.0033 (15)	0.0203 (13)	0.0139 (15)
C4	0.046 (2)	0.036 (2)	0.049 (2)	−0.0041 (17)	0.0126 (16)	0.0033 (18)
C5	0.051 (2)	0.044 (2)	0.051 (2)	−0.0027 (19)	0.0062 (18)	−0.0037 (18)
O5	0.075 (2)	0.070 (2)	0.0647 (19)	−0.0062 (17)	0.0305 (15)	0.0095 (17)
C6	0.057 (2)	0.065 (3)	0.0377 (18)	−0.003 (2)	0.0119 (18)	0.0054 (19)
O6	0.102 (3)	0.116 (3)	0.088 (2)	−0.025 (2)	0.042 (2)	0.016 (2)
C7	0.053 (2)	0.052 (3)	0.058 (2)	−0.019 (2)	0.010 (2)	−0.010 (2)
C8	0.048 (2)	0.071 (3)	0.083 (3)	−0.005 (2)	0.031 (2)	0.018 (2)
C9	0.053 (2)	0.077 (3)	0.078 (3)	0.010 (3)	0.032 (2)	0.021 (3)
C10	0.061 (3)	0.115 (4)	0.063 (3)	0.008 (3)	0.009 (2)	−0.011 (3)
C11	0.063 (3)	0.131 (5)	0.078 (3)	−0.010 (3)	0.018 (2)	0.002 (3)
C12	0.107	0.107	0.107	0.000	0.024	0.000
C13	0.122	0.122	0.122	0.000	0.028	0.000
C14	0.123	0.123	0.123	0.000	0.028	0.000

Geometric parameters (Å, º)

N—O2	1.200 (4)	C7—H7A	0.9300
N—O1	1.216 (4)	C8—C9	1.476 (6)
N—C3	1.490 (5)	C8—C11	1.517 (6)
C1—C2	1.366 (5)	C8—H8A	0.9800
C1—C6	1.383 (5)	C10—H10A	0.9600
C1—H1A	0.9300	C10—H10B	0.9600
C2—C3	1.405 (5)	C10—H10C	0.9600

C2—H2A	0.9300	C11—C12	1.447 (6)
C3—C4	1.376 (5)	C11—H11A	0.9700
O3—C7	1.204 (4)	C11—H11B	0.9700
O4—C6	1.353 (4)	C12—C13	1.428 (7)
O4—C8	1.446 (4)	C12—H12A	0.9700
C4—C5	1.385 (5)	C12—H12B	0.9700
C4—H4A	0.9300	C13—C14	1.587 (7)
C5—C6	1.412 (5)	C13—H13A	0.9700
C5—C7	1.479 (5)	C13—H13B	0.9700
O5—C9	1.291 (5)	C14—H14A	0.9600
O5—C10	1.468 (4)	C14—H14B	0.9600
O6—C9	1.200 (5)	C14—H14C	0.9600
O2—N—O1	124.6 (4)	O6—C9—C8	121.4 (5)
O2—N—C3	116.9 (4)	O5—C9—C8	115.3 (4)
O1—N—C3	118.5 (4)	O5—C10—H10A	109.5
C2—C1—C6	121.6 (4)	O5—C10—H10B	109.5
C2—C1—H1A	119.2	H10A—C10—H10B	109.5
C6—C1—H1A	119.2	O5—C10—H10C	109.5
C1—C2—C3	117.6 (4)	H10A—C10—H10C	109.5
C1—C2—H2A	121.2	H10B—C10—H10C	109.5
C3—C2—H2A	121.2	C12—C11—C8	118.7 (5)
C4—C3—C2	123.2 (4)	C12—C11—H11A	107.6
C4—C3—N	119.5 (4)	C8—C11—H11A	107.6
C2—C3—N	117.3 (4)	C12—C11—H11B	107.6
C6—O4—C8	116.7 (3)	C8—C11—H11B	107.6
C3—C4—C5	117.8 (3)	H11A—C11—H11B	107.1
C3—C4—H4A	121.1	C13—C12—C11	113.7 (6)
C5—C4—H4A	121.1	C13—C12—H12A	108.8
C4—C5—C6	120.4 (4)	C11—C12—H12A	108.8
C4—C5—C7	117.3 (3)	C13—C12—H12B	108.8
C6—C5—C7	122.3 (4)	C11—C12—H12B	108.8
C9—O5—C10	117.2 (4)	H12A—C12—H12B	107.7
O4—C6—C1	125.9 (4)	C12—C13—C14	110.4 (6)
O4—C6—C5	114.7 (3)	C12—C13—H13A	109.6
C1—C6—C5	119.4 (4)	C14—C13—H13A	109.6
O3—C7—C5	123.5 (4)	C12—C13—H13B	109.6
O3—C7—H7A	118.3	C14—C13—H13B	109.6
C5—C7—H7A	118.3	H13A—C13—H13B	108.1
O4—C8—C9	113.1 (3)	C13—C14—H14A	109.5
O4—C8—C11	104.4 (3)	C13—C14—H14B	109.5
C9—C8—C11	110.5 (4)	H14A—C14—H14B	109.5
O4—C8—H8A	109.6	C13—C14—H14C	109.5
C9—C8—H8A	109.6	H14A—C14—H14C	109.5
C11—C8—H8A	109.6	H14B—C14—H14C	109.5
O6—C9—O5	123.2 (5)		
C6—C1—C2—C3	−1.3 (6)	C4—C5—C6—C1	1.5 (5)

C1—C2—C3—C4	1.0 (6)	C7—C5—C6—C1	−177.6 (3)
C1—C2—C3—N	−178.6 (3)	C4—C5—C7—O3	−1.5 (5)
O2—N—C3—C4	−10.1 (6)	C6—C5—C7—O3	177.6 (4)
O1—N—C3—C4	170.9 (4)	C6—O4—C8—C9	−72.8 (5)
O2—N—C3—C2	169.5 (4)	C6—O4—C8—C11	166.9 (4)
O1—N—C3—C2	−9.6 (5)	C10—O5—C9—O6	−3.7 (6)
C2—C3—C4—C5	0.6 (5)	C10—O5—C9—C8	−180.0 (3)
N—C3—C4—C5	−179.9 (3)	O4—C8—C9—O6	160.8 (4)
C3—C4—C5—C6	−1.8 (5)	C11—C8—C9—O6	−82.5 (6)
C3—C4—C5—C7	177.3 (3)	O4—C8—C9—O5	−22.8 (5)
C8—O4—C6—C1	4.5 (5)	C11—C8—C9—O5	93.8 (4)
C8—O4—C6—C5	−177.5 (3)	O4—C8—C11—C12	−57.5 (6)
C2—C1—C6—O4	178.0 (4)	C9—C8—C11—C12	−179.5 (5)
C2—C1—C6—C5	0.1 (6)	C8—C11—C12—C13	−68.5 (7)
C4—C5—C6—O4	−176.6 (3)	C11—C12—C13—C14	−157.4 (5)
C7—C5—C6—O4	4.3 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*A*···O4	0.97	2.51	2.877 (6)	102
C7—H7*A*···O4	0.93	2.46	2.769 (5)	100
C2—H2*A*···O1^{i}	0.93	2.52	3.442 (6)	169

Symmetry code: (i) −*x*, −*y*+2, −*z*+1.