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Benzyl *N*-(2-formylphenyl)carbamate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.055
wR factor = 0.166
Data-to-parameter ratio = 13.1

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

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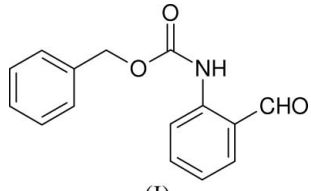
In the title compound, $C_{15}H_{13}NO_3$, an intramolecular N—H···O bond helps to establish the molecular conformation. In the crystal structure, adjacent molecules interact by way of weak C—H···π interactions.

Comment

As part of our studies of McMurry coupling reactions (Ephritikhine, 1998), the title compound, (I), $C_{15}H_{13}NO_3$, has been synthesised and structurally characterised (Fig. 1).



(I)

Compound (I) possesses normal geometrical parameters (Allen *et al.*, 1987). The dihedral angle between the C2–C7 and C10–C15 ring mean planes is 61.90 (7)°. The C8/N1/O2/O3 fragment is close to coplanar with the C2–C7 ring [dihedral angle = 4.72 (2)°], perhaps due to the stabilizing influence of an intramolecular N—H···O bond (Table 1). An intramolecular C—H···O interaction with a short H···O contact distance of 2.31 Å also arises. The bond-angle sum at N1 of 360° implies sp^2 hybridization for this atom.

The crystal packing for (I) may be consolidated by a weak intermolecular C—H···π interaction (Fig. 2), leading to [010] chains of molecules, generated by the 2_1 screw axis. Conversely, there are no π–π stacking interactions in (I), the shortest separation of aromatic ring centroids being greater than 4.5 Å.

Experimental

2-Aminobenzaldehyde (5.90 mmol, 0.714 g) in dry dichloromethane (50 ml) was stirred under argon in an ice bath for 20 minutes before adding *N*,*N*-ethyldiisopropylamine (5.90 mmol, 1 ml). This was followed by the addition of benzyl chloroformate (5.90 mmol, 0.84 ml) and the solution was allowed to warm to room temperature. It was stirred for a further 3 days, monitoring the reaction by TLC before working-up. The crude solution was concentrated and extracted using diethyl ether (3 × 15 ml) and distilled water (40 ml). The organics were combined, dried over $MgSO_4$, filtered and concentrated *in vacuo*. The crude mixture was purified using flash column chromatography (5:4 *v*:*v* dichloromethane:hexane, R_f = 0.36), yielding the title compound, which was recrystallized from dichloromethane, yielding colourless blocks of (I) [m.p. 337–338 K, 0.860 g (57%)]. ν_{max} (KBr) 3278 (NH), 3032, 2956 (CH), 2771 (CO—

H), 1726 (COO*R*), 1670 (CHO), 1609 (Ar), 1533 (Ar), 1453 (Ar), 1239 (C—O).

Crystal data

$C_{15}H_{13}NO_3$
M_r = 255.26
Monoclinic, $P2_1/n$
a = 12.5862 (8) Å
b = 6.7950 (4) Å
c = 15.8427 (10) Å
β = 108.386 (1)°
V = 1285.75 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.45 × 0.27 × 0.12 mm

Data collection

Bruker SMART1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.851, T_{max} = 0.929
(expected range = 0.906–0.989)
7557 measured reflections
2260 independent reflections
1533 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.166
S = 1.04
2260 reflections
172 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

π is the centroid of the C10–C15 ring

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	2.01	2.719 (3)	139
C6—H6···O2	0.93	2.31	2.909 (4)	122
C13—H13···π^{i}	0.93	2.91	3.696 (3)	143

Symmetry code: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

H atoms were placed in idealized locations (C—H = 0.93-0.97 Å, N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

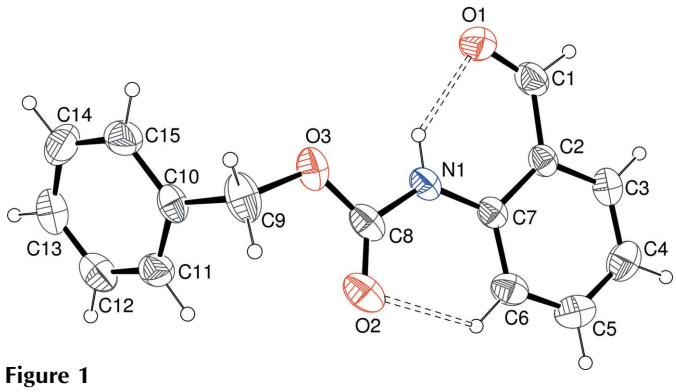


Figure 1
View of the molecular structure of (I) showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). The N—H···O and C—H···O interactions are drawn as double dashed lines.

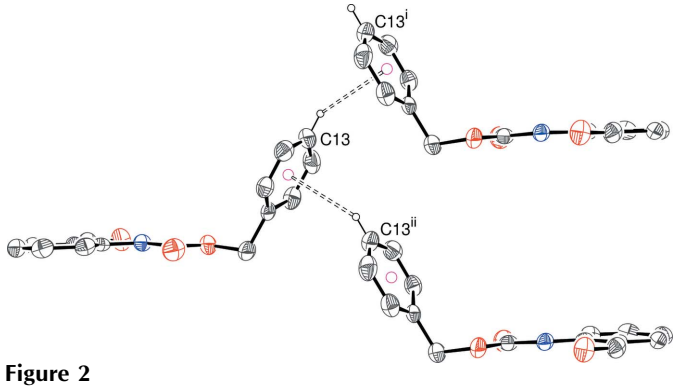


Figure 2
Fragment of a chain of molecules of (I) linked by C—H···π interactions (dashed lines). All H atoms except H13 are omitted for clarity. The pink circles represent the centroids of the C10–C15 ring. Symmetry code as in Table 1, additionally (ii) $\frac{1}{2}-x, y+\frac{1}{2}, \frac{3}{2}-z$.

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