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Key indicators

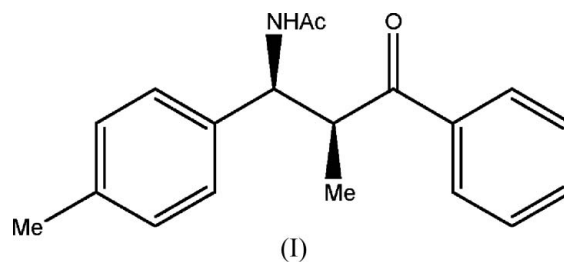
Single-crystal X-ray study
 $T = 153\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.058
 wR factor = 0.137
Data-to-parameter ratio = 14.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.***syn*-N-[2-Benzoyl-1-(4-methylphenyl)propyl]acetamide: supramolecular structure formed by N—H···O, C—H···O and C—H··· π (arene) hydrogen bonds**

In the title compound, $\text{C}_{19}\text{H}_{21}\text{NO}_2$, the asymmetric unit contains two independent molecules (*A* and *B*) linked by a strong N—H···O hydrogen bond. The molecular assembly in the structure is established by N—H···O, C—H···O and C—H··· π (arene) interactions. Intermolecular N—H···O hydrogen bonds involving different types of molecules generate an infinite *ABAB*... chain running parallel to the [001] direction. The combination of N—H···O and C—H···O hydrogen bonds results into a two-dimensional supramolecular framework of $R_2^2(32)$ rings in the (011) plane.

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Comment

β -Acetamido ketones serve as potential intermediates in the synthesis of natural products and antibiotics (Wabnitz & Spencer, 2002). Depending on the types of substitution at the C_α , C_β and keto-C atoms, and the conformational flexibility of the substituent groups, a variety of β -acetamido ketones offering the possibility of intermolecular interactions can be obtained (Ghosh *et al.*, 2005). In a continuation of our studies (Ghosh *et al.*, 2006) of one-pot stereoselective synthesis of β -acetamido ketones, the title compound, (I), has been prepared and the crystal structure is reported here.



The asymmetric unit of (I) consists of two molecules, *A* (C1–C19, N1, O1, O2) and *B* (C20–C38, N2, O3, O4), with similar bond lengths and angles. The normal probability plot analyses (International Tables for X-ray Crystallography, 1974, Vol. IV, pp. 293–309) for both bond lengths and angles show that the differences between the two symmetry independent molecules (Fig. 1) are of a statistical nature; the squares of the correlation factors for bond lengths and angles are 0.97 and 0.95, respectively. The difference in the conformation of the molecules in (I) can be described by the torsion angles (Table 1) involving the C atoms of the propyl chain, which links the phenyl and benzene rings. In both symmetry-independent molecules, the orientations of the carbonyl and the acetamido groups with respect to the $\text{C}_\alpha\text{—C}_\beta$ bond is antiperiplanar; the values of the C(carbonyl)— $\text{C}_\alpha\text{—C}_\beta\text{—N}$ (acetamide) torsion angles are close to 180° (Table 1). The

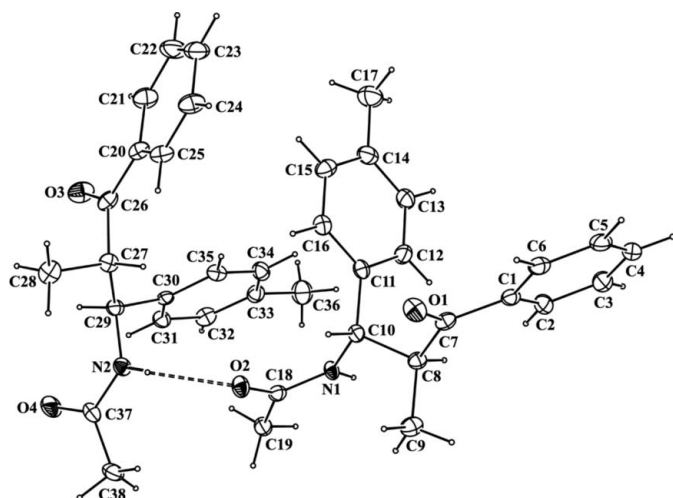


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

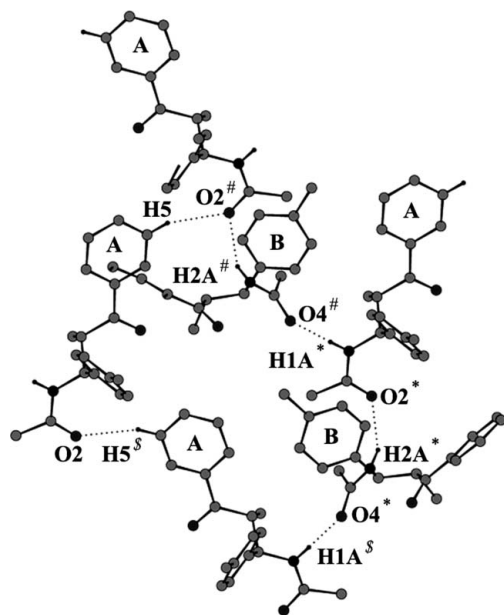


Figure 2
Part of the crystal structure, showing the formation of a $R_5^6(32)$ ring involving four *A* type and two *B* type molecules. H atoms not involved in hydrogen bonding have been omitted for clarity. Atoms marked with the symbols (#), (\$) and (*) are at the symmetry positions $(x, \frac{1}{2} - y, \frac{1}{2} + z)$, $(x, \frac{1}{2} - y, -\frac{1}{2} + z)$ and $(x, y - 1, z)$, respectively. Hydrogen bonds are shown as dotted lines.

bond lengths and angles in (I) are comparable with those reported for the *anti* isomer of the title β -acetamido ketone (Ghosh *et al.*, 2006).

The intermolecular aggregation in (I) is determined by a combination of N—H \cdots O, C—H \cdots O and C—H $\cdots\pi$ (arene) hydrogen bonds (Table 2). Considering only *A*-type molecules, the phenyl atom C5 acts as a donor to the acetamido atom O2, forming a polymeric C(10) chain (Bernstein *et al.*, 1995) running parallel to the [001] direction. The interlinking of *A* and *B* molecules *via* strong N—H \cdots O hydrogen bonds

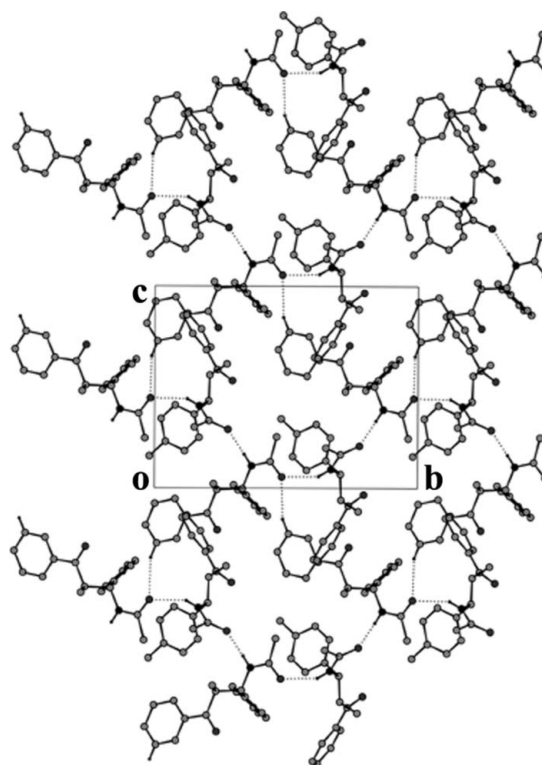


Figure 3
Part of the crystal structure of (I), showing the formation of a (011) sheet of $R_5^6(32)$ rings. H atoms not involved in hydrogen bonding have been omitted for clarity. Hydrogen bonds are shown as dotted lines.

generates an infinite *ABAB*... chain running parallel to the [001] direction. Additional reinforcement between the molecules of the *ABAB*... chain is provided by C—H $\cdots\pi$ (arene) hydrogen bonds. Intermolecular N—H \cdots O and C—H \cdots O hydrogen bonds connect four *A* and two *B* molecules into an $R_5^6(32)$ ring (Fig. 2); these rings are edge-fused, forming a two-dimensional supramolecular grid parallel to the (011) plane (Fig. 3).

Experimental

To a solution of *p*-tolualdehyde (1 mmol) and propiophenone (1 mmol) in dry acetonitrile (4 ml), $ZrOCl_2 \cdot 8H_2O$ (20 mol%) was added. To the resulting suspension, acetyl chloride (2 mmol) was added and the reaction mixture was stirred at ambient temperature. After completion of the reaction [checked by thin-layer chromatography (TLC) with ethyl acetate–petroleum ether (333–353 K)], the mixture was diluted with CH_2Cl_2 , washed with brine (1 \times 20 ml) and the aqueous layer was extracted with CH_2Cl_2 (3 \times 15 ml). The combined organic layer was washed with $NaHCO_3$ solution (1 \times 15 ml) followed by water (1 \times 20 ml). The organic layer was dried over Na_2SO_4 and the crude residue was purified on a silica gel (60–120 mesh) column after elution with 3:2 EtOAc–petroleum ether (333–353 K) (yield 94%). Diastereomers were separated by preparative TLC on silica plates using 1:2 EtOAc–petroleum ether (333–353 K). *syn*-Isomer (I): m.p. 392 (1) K; analysis found: C 77.23, H 6.94, N 4.35%; calculated for $C_{19}H_{21}O_2N$: C 77.25, H 7.16, N 4.74%. Single crystals of the *syn*-isomer of (I) were grown by slow evaporation of an EtOAc–hexane (1:10) solution.

Crystal data

C₁₉H₂₁NO₂
M_r = 295.37
 Monoclinic, *P*₂₁/*c*
a = 18.3820 (18) Å
b = 11.7845 (12) Å
c = 16.4961 (16) Å
 β = 111.444 (2)°
V = 3326.1 (6) Å³
Z = 8
D_x = 1.180 Mg m⁻³
 Mo *K*α radiation
 μ = 0.08 mm⁻¹
T = 153 (2) K
 Block, colourless
 0.35 × 0.12 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.984, *T_{max}* = 0.992
 17160 measured reflections
 5844 independent reflections
 4060 reflections with *I* > 2σ(*I*)
R_{int} = 0.063
 θ_{max} = 25.0°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.058
wR(*F*²) = 0.137
S = 1.04
 5844 reflections
 398 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.971P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.33 e Å⁻³
 Δρ_{min} = -0.26 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1—C7	1.224 (3)	C8—C9	1.525 (3)
O2—C18	1.242 (3)	C8—C10	1.538 (3)
O3—C26	1.218 (3)	C10—C11	1.521 (3)
O4—C37	1.237 (3)	C18—C19	1.497 (3)
N1—C18	1.337 (3)	C20—C26	1.498 (3)
N1—C10	1.458 (3)	C26—C27	1.526 (3)
N2—C37	1.338 (3)	C27—C29	1.528 (3)
N2—C29	1.468 (3)	C27—C28	1.530 (3)
C1—C7	1.498 (3)	C29—C30	1.524 (3)
C7—C8	1.517 (3)	C37—C38	1.498 (3)
C1—C7—C8—C10	-128.1 (2)	C20—C26—C27—C29	-141.3 (2)
C18—N1—C10—C11	-108.4 (2)	C37—N2—C29—C30	-101.4 (2)
C7—C8—C10—N1	-179.21 (18)	C26—C27—C29—N2	174.28 (18)
C7—C8—C10—C11	57.0 (3)	C26—C27—C29—C30	53.6 (3)
N1—C10—C11—C16	105.7 (2)	C27—C29—C30—C35	30.1 (3)
C8—C10—C11—C12	50.7 (3)	N2—C29—C30—C31	82.3 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N2—H2A···O2	0.86	2.15	2.902 (3)	145
N1—H1A···O4 ⁱ	0.86	1.96	2.818 (3)	175
C5—H5···O2 ⁱⁱ	0.93	2.47	3.354 (3)	160
C34—H34···Cg1 ⁱ	0.93	2.75	3.564 (3)	146

Symmetry codes: (i) *x*, -*y* + $\frac{3}{2}$, *z* + $\frac{1}{2}$; (ii) *x*, -*y* + $\frac{1}{2}$, *z* + $\frac{1}{2}$. Cg1 is the centroid of ring C11—C16

H atoms were placed in calculated positions and treated as riding, with N—H = 0.86 Å, C—H = 0.93 (aromatic), 0.96 (methyl H) and 0.98 Å (CH group), and *U*_{iso}(H) = 1.2*U*_{eq}(C,N).

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); 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 for Windows (Farrugia, 1997) and CAMERON (Watkin *et al.*, 1993); software used to prepare material for publication: SHELXL97, PARST95 (Nardelli, 1995) and PLATON (Spek, 2003).

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