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Key indicators

Single-crystal X-ray study
 $T = 161$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.043
 wR factor = 0.088
Data-to-parameter ratio = 9.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.5-[2-(Hydroxymethyl)phenyl]-*N,N*-dimethylpent-4-ynamide

The title compound, $\text{C}_{14}\text{H}_{17}\text{NO}_2$, crystallizes with four independent molecules (*A*, *B*, *C* and *D*) in the asymmetric unit. In the crystal structure, molecules *A* and *C* are hydrogen bonded to their symmetry-related molecules to form parallel chains extending in the *a* direction. Molecules *B* and *D* are hydrogen bonded to one another and form alternating zigzag chains running in the *c* direction. These hydrogen-bonded chains are connected *via* intermolecular $\text{C}-\text{H}\cdots\pi$ and $\text{C}-\text{H}\cdots\text{O}$ contacts. The four independent molecules are related by non-crystallographic pseudo-symmetry.

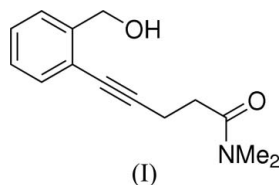
Received 1 December 2005

Accepted 6 December 2005

Online 14 December 2005

Comment

The title compound, (I), was prepared for an investigation of the addition of nucleophiles to $\text{C}\equiv\text{C}$ triple bonds and $\text{C}-\text{H}$ activation in the benzylic position by homogeneous gold catalysts (Hashmi, 2004).



Compound (I) crystallizes with four independent molecules (*A*, *B*, *C* and *D*) per asymmetric unit. The molecular structure of molecule *A* is illustrated in Fig. 1. The dimensions of all four molecules are very similar. The amide groups are planar; the $\text{O}2-\text{C}12-\text{N}1-\text{C}14$ torsion angles are 175.9 (2), -179.4 (2), 179.2 (2) and -179.7 (2) $^\circ$ for molecules *A*, *B*, *C* and *D*, respectively. The N atoms show no significant deviations from planarity; the sum of the three valence angles about the N atoms are 359.9 , 360.0 , 360.0 and 359.9 $^\circ$ for *N1A*, *N1B*, *N1C* and *N1D*, respectively. The angles between the plane of the

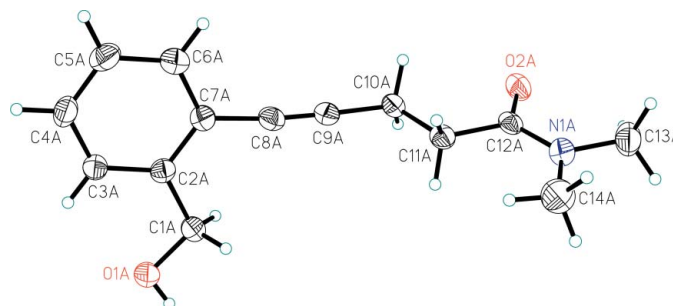


Figure 1

The molecular structure of molecule *A* of compound (I), showing the numbering scheme and displacement ellipsoids drawn at the 50% probability level. Molecules *B*, *C* and *D* are very similar.

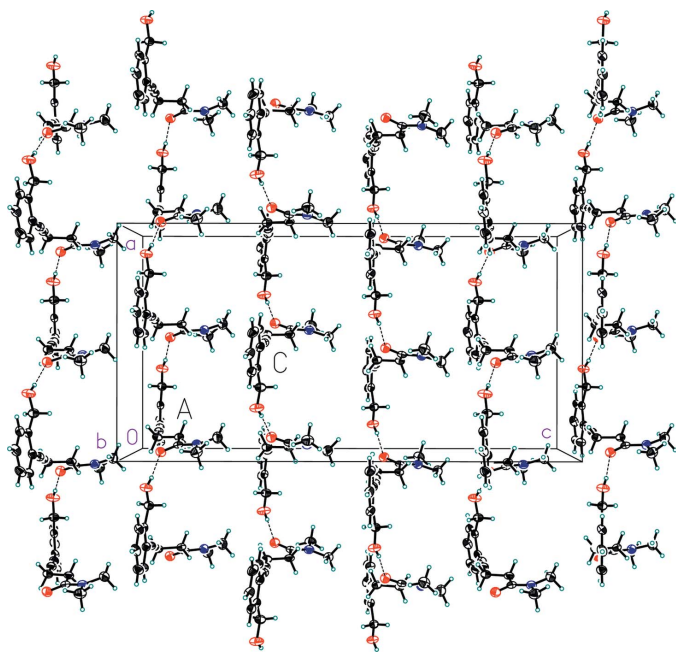


Figure 2
A perspective view down the *b* axis of the crystal packing of compound (I), showing only the hydrogen-bonded chains of molecules A and C. Hydrogen bonds are shown as dashed lines and displacement ellipsoids are drawn at the 50% probability level.

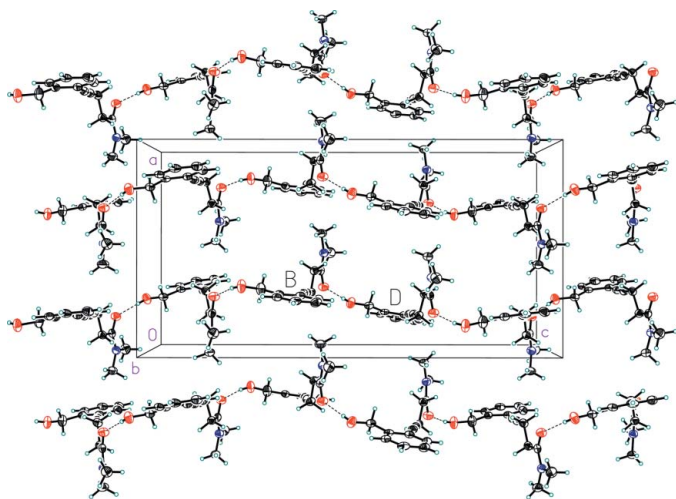


Figure 3
A perspective view down the *b* axis of the crystal packing of compound (I), showing only the hydrogen-bonded chains of molecules B and D. Hydrogen bonds are shown as dashed lines and displacement ellipsoids are drawn at the 50% probability level.

benzene ring and the plane of the *N,N*-dimethylamide group are 77.2 (1), 82.2 (1), 80.2 (1) and 74.6 (1)° for molecules A, B, C and D, respectively. This confirms the similarity of the four molecules.

The C1—O1 bond is coplanar with the benzene ring; the torsion angles O1—C1—C2—C3 are 2.6 (3), 3.9 (4), 1.9 (4) and 0.4 (3)° for molecules A, B, C and D, respectively. This conformation results in contact distances between atoms O1 and H3 of 2.40, 2.43, 2.41 and 2.38 Å, for molecules A, B, C and D, respectively.

In the crystal structure the molecules are connected by intermolecular hydrogen bonds between the hydroxyl groups and the keto groups to form chains. Details of the hydrogen bonding are given in Table 1, and the crystal packing is illustrated in Figs. 2 and 3. Molecules A and C are hydrogen bonded to their symmetry-related molecules to form chains extending in the *a* direction (Fig. 2). Molecules in these chains are related by an *a*-glide plane. Molecules B and D are hydrogen bonded to one another and form alternating zigzag chains running in the *c* direction (Fig. 3). The hydrogen-bonded chains are connected by intermolecular C—H··· π and C—H···O contacts. The crystal packing shows nine different intermolecular C—H···O interactions with H···O distances between 2.38 and 2.59 Å and five weak intermolecular C_{methylene}—H··· π _{phenyl} interactions.

Each of the hydrogen-bonded motifs shows pseudo-symmetry. Molecules A and C are related by the pseudo-relationship $x_C \simeq 0.60 - x_A$, $y_C \simeq 1.02 - y_A$ and $z_C \simeq \frac{1}{4} + z_A$. This is a pseudo-twofold screw axis about (0.30, 0.51, *z*) with a translation of *c*/4. Thus molecule C and molecule A at ($-x$, $-y$, $\frac{1}{2} + z$) are related by a pseudo-translation vector (0.60, 1.02, -0.25). The coordinates of molecules B and D also show a pseudo-relationship: $x_D \simeq x_B - 0.11$, $y_D \simeq 2.02 - y_B$ and $z_D \simeq \frac{1}{4} + z_B$. This is a pseudo-glide plane perpendicular to the *b* axis through $y = 1.01$ with an unusual translation vector of (-0.11 , 0, 0.25). Thus molecule D and molecule B, at ($\frac{1}{2} + x$, $-y$, *z*), are related by a pseudo-translation vector (-0.61 , 2.02, 0.25). This shows the pseudo-symmetry between the AC and BD structures to be very similar.

Experimental

The title compound was prepared using the Sonogashira coupling of 2-iodobenzyl alcohol and *N,N*-dimethylpent-4-ynamide (Thorand & Krause, 1998). Single crystals were obtained by evaporation of a solution of (I) in diethyl ether/hexane (1:1 *v/v*).

Crystal data

C₁₄H₁₇NO₂
M_r = 231.29
 Orthorhombic, *Pca*2₁
a = 14.166 (2) Å
b = 12.9093 (14) Å
c = 27.669 (5) Å
V = 5060.1 (14) Å³
Z = 16
D_x = 1.214 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 234 reflections
 $\theta = 3\text{--}23^\circ$
 $\mu = 0.08 \text{ mm}^{-1}$
T = 161 (2) K
 Block, colorless
 0.50 × 0.36 × 0.30 mm

Data collection

Siemens SMART 1K CCD diffractometer
 ω scans
 Absorption correction: none
 57442 measured reflections
 6075 independent reflections

4295 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.088$
 $\theta_{\text{max}} = 28.2^\circ$
 $h = -18 \rightarrow 18$
 $k = -16 \rightarrow 16$
 $l = -35 \rightarrow 36$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.088$
 $S = 1.06$
 6075 reflections
 637 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1A-H1C\cdots O2A^i$	0.80 (3)	1.96 (3)	2.757 (3)	173 (3)
$O1B-H1F\cdots O2D^{ii}$	0.84 (4)	1.92 (4)	2.747 (3)	170 (3)
$O1C-H1I\cdots O2C^{iii}$	0.80 (3)	1.96 (3)	2.759 (3)	173 (3)
$O1D-H1L\cdots O2B$	0.80 (3)	1.92 (3)	2.718 (3)	173 (3)

Symmetry codes: (i) $x + \frac{1}{2}, -y + 1, z$; (ii) $-x + \frac{1}{2}, y, z - \frac{1}{2}$; (iii) $x - \frac{1}{2}, -y + 1, z$.

Equivalent reflections, including Friedel pairs, were merged. H atoms attached to C atoms were positioned geometrically and refined as riding atoms [$C_{sp^2}-H = 0.95 \text{ \AA}$, $C_{secondary}-H = 0.99 \text{ \AA}$, with $U_{iso}(H) = 1.2U_{eq}(\text{parent C atom})$, and $C_{methyl}-H = 0.98 \text{ \AA}$, with $U_{iso}(H) = 1.5U_{eq}(\text{parent } C_{methyl} \text{ atom})$. The hydroxyl H atoms were located in difference Fourier maps and refined isotropically [$O-H = 0.80(3)-0.84(4) \text{ \AA}$].

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *SHELXL97*.

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