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N-(2-Bromo-3-pyridyl)-3-methylbut-2-enamide

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.006 Å
R factor = 0.045
wR factor = 0.125
Data-to-parameter ratio = 18.0

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

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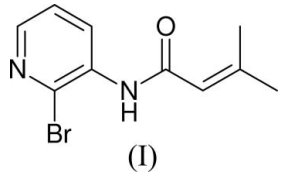
In the title compound, $C_8H_8BrN_3O$, the crystal packing is influenced by weak intermolecular C—H···O and C—H···N bonds rather than the more typical N—H···O interactions.

Comment

As part of our ongoing investigations of radical-mediated cyclization reactions (Kirsop *et al.*, 2007), the title compound, (I), has been synthesized and structurally characterized (Fig. 1).



(I)

The dihedral angle between the mean planes of the aromatic ring and the atoms C6/C7/N2/O1 is 4.0 (3)°. The angle between the C6/C7/N2/O1 and C7/C8/C9/C10 mean planes is 16.2 (3)°. Atom Br1 is displaced from the aromatic ring plane by 0.059 (6) Å. The bond-angle sum at N2 is 360.0°, indicative of sp^2 hybridization for this atom. Thus, the *p* orbital of N2 is well aligned to interact with the aromatic ring. Even so, the C5—N2 bond length of 1.392 (6) Å in (I) is significantly longer than the reference value (Allen *et al.*, 1987) of 1.353±0.007Å for this bond type. Otherwise, the geometrical parameters for (I) may be regarded as normal (Allen *et al.*, 1987).

The crystal packing for (I) is influenced by weak intermolecular C—H···O and C—H···N interactions (Table 1). The N2—H1 group in (I) participates in an intramolecular N—H···Br bond, rather than the more typical intermolecular N—H···O bond to the amide O atom in an adjacent molecule, which would result in a *C*(4) chain (Etter, 1990). The C—H···O bond in (I) leads to centrosymmetric dimers of molecules, and the C—H···N bond leads to a zigzag chain of molecules propagating along [010], generated by the 2_1 screw axis. Overall, (001) sheets of molecules result from this connectivity. There are no π–π stacking interactions in (I), the shortest separation of the centroids of nearby aromatic rings being greater than 5.7 Å.

Experimental

3-Amino-2-bromopyridine (0.20 g, 1.16 mmol) and 25 ml dichloromethane were placed in a dry twin-necked flask. The mixture was stirred for 10 min in an ice bath before adding *N*,*N*-ethyldiisopropylamine (0.24 ml, 1.36 mmol). The crude mixture was then

stirred for 20 min before dropwise addition of acryloyl chloride (0.11 ml, 1.36 mmol). The ice bath was removed and the crude mixture was warmed to room temperature and allowed to stand for 20–25 min before work-up. Saturated brine (20 ml) followed by distilled water (10 ml) were added and the organics extracted with chloroform (3 × 10 ml). The fractions were combined, dried using $MgSO_4$, filtered and concentrated under reduced pressure. The oily residue was then purified by column chromatography to yield the title compound (yield 0.368 g, 65%) as a pale-yellow solid [m.p. 326–328 K; R_F = 0.55 (hexane–ethyl acetate–triethylamine = 10:5:1)]. Colourless plates of (I) were recrystallized from chloroform. ν_{max} (KBr, cm^{-1}): 810 (CH═C), 1045 (pyridine Br), 1371, 1445 (CH_3), 1501 (aryl, conjugated), 1577 (N–H), 1645 [C═C(CH_3)$_2$], 1684 (C═O amide), 3379 (N—H).

Crystal data

$C_{10}H_{11}BrN_2O$
M_r = 255.12
Monoclinic, $P2_1/n$
a = 6.5351 (12) Å
b = 8.1108 (13) Å
c = 20.222 (4) Å
β = 98.387 (4)°
V = 1060.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.85 mm^{-1}
T = 120 (2) K
0.40 × 0.31 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.308, T_{max} = 0.927
7892 measured reflections
2377 independent reflections
1762 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.06
2377 reflections
132 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.82 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···Br1	0.81 (5)	2.59 (5)	3.072 (3)	119 (4)
C3—H3···O1^{i}	0.95	2.54	3.288 (5)	136
C7—H7···N1^{ii}	0.95	2.56	3.495 (5)	169

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

The N-bound H atom was located in a difference map and its position was freely refined with U_{iso}(H) = 1.2U_{eq}(N). The C-bound H atoms were placed in calculated positions (C—H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl groups were allowed to rotate but not to tip to best fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); 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*.

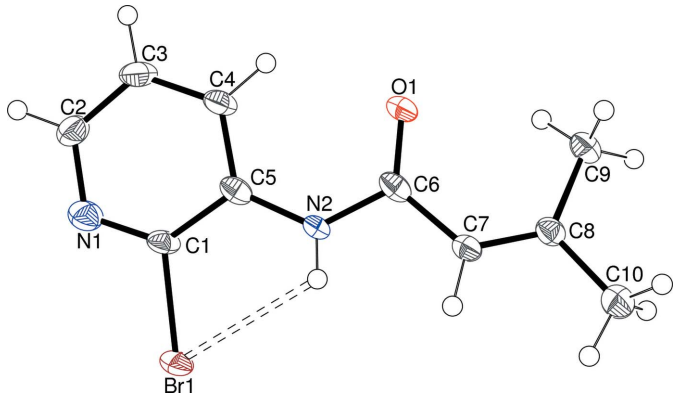


Figure 1
View of the molecular structure of (I), showing 50% displacement ellipsoids. The H atoms are drawn as spheres of arbitrary radius and the intramolecular hydrogen bond is shown as a double-dashed line.

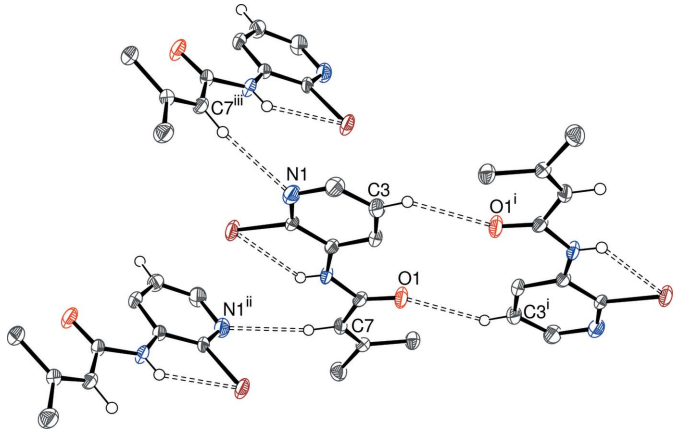


Figure 2
Fragment of the packing of (I) showing the connectivity of molecules *via* C—H···O and C—H···N links (double dashed lines). All H atoms except H1, H3 and H7 have been omitted for clarity. [Symmetry codes as in Table 1; additionally (iii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.]

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