

Chu-Yi Yu,* Hong-Yun Gao and
Yan QiaoBeijing National Laboratory for Molecular
Science (BNLMS), Laboratory for Chemical
Biology, Institute of Chemistry, Chinese
Academy of Sciences, Beijing 100080, People's
Republic of China

Correspondence e-mail: yucy@iccas.ac.cn

Key indicators

Single-crystal X-ray study
 $T = 294\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.010\text{ \AA}$
 R factor = 0.041
 wR factor = 0.093
Data-to-parameter ratio = 10.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Methyl 3-[(*E*)-2-(4-bromobenzoylmethyl-
idene)imidazolidin-1-yl]acrylateThe title compound, $\text{C}_{15}\text{H}_{15}\text{BrN}_2\text{O}_3$, contains two molecules in the asymmetric unit, which differ in conformation. Both exhibit intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. Intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link molecules into dimers and short $\text{Br}\cdots\text{O}$ contacts exist between dimers.Received 8 October 2006
Accepted 2 November 2006

Comment

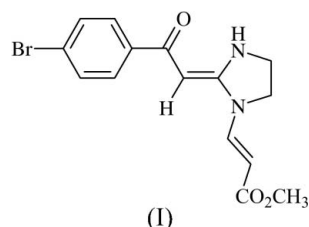
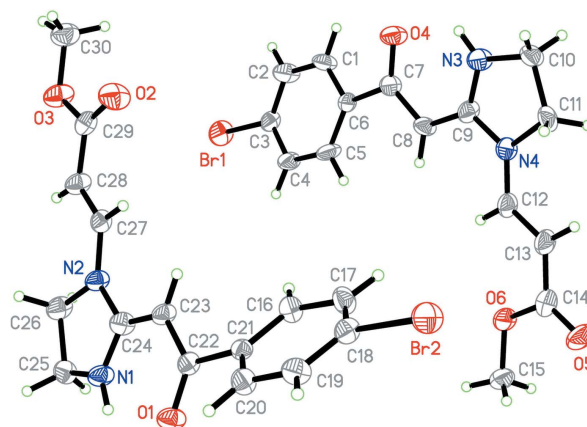
Heterocyclic ketene amins (HKAs) are ambidentate nucleophiles (Huang & Wang, 2002). The title compound, (I), is an *N*-alkylation product of an HKA. The crystal structure of (I) was determined in order to provide information regarding its electronic conjugation properties and to examine a possible intramolecular hydrogen bond (Wang *et al.*, 1987), which may be correlated with the reactivity of the secondary amine at the αC atom (Huang & Wamhoff, 1984).There are two molecules in the asymmetric unit of (I) (Fig. 1), which differ principally in the orientation of the benzene ring with respect to the rest of the molecule. The dihedral angle between the benzene ring and the mean plane of the remainder of the molecule is $13.4(1)$ and $50.0(1)^\circ$ in the two independent molecules.

Figure 1

The structure of the asymmetric unit of (I), showing displacement ellipsoids at the 30% probability level (arbitrary spheres for H atoms).

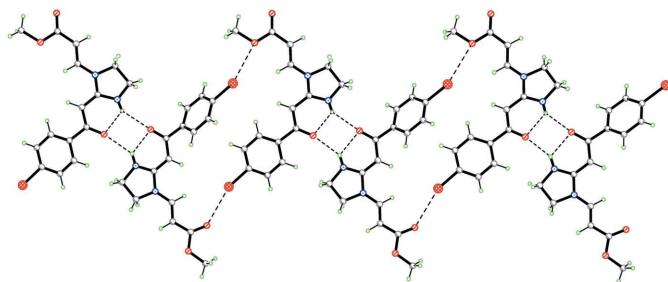


Figure 2
The hydrogen-bonded dimers in the structure of (I), with Br \cdots O contacts shown between them. Both types of intermolecular contacts are indicated by dashed lines.

Intramolecular hydrogen bonds are observed between the N–H group and the adjacent C=O group in both independent molecules (Table 1 and Fig. 2). Intermolecular N–H \cdots O hydrogen bonds (Table 1) link the molecules into dimers, and short Br \cdots O contacts exist between dimers [Br1 \cdots O2ⁱ = 3.074 (7) Å; symmetry code: (i) $x, y, -1 + z$].

Experimental

Compound (I) was prepared according to the procedure of Wang *et al.* (1994) and recrystallized from chloroform in 75% yield (m.p. 388 K). Elemental analysis, found: C 51.32, H 4.32, N 7.95%; calculated: C 51.30, H 4.31, N 7.98%.

Crystal data

C ₁₅ H ₁₅ BrN ₂ O ₃	$Z = 8$
$M_r = 351.20$	$D_x = 1.589 \text{ Mg m}^{-3}$
Monoclinic, Cc	Mo $K\alpha$ radiation
$a = 15.819 (4) \text{ \AA}$	$\mu = 2.81 \text{ mm}^{-1}$
$b = 20.899 (5) \text{ \AA}$	$T = 294 (2) \text{ K}$
$c = 9.994 (2) \text{ \AA}$	Block, colourless
$\beta = 117.307 (4)^\circ$	$0.28 \times 0.26 \times 0.22 \text{ mm}$
$V = 2936.0 (12) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	8187 measured reflections
φ and ω scans	4154 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 1997)	2440 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.473, T_{\max} = 0.539$	$R_{\text{int}} = 0.047$
	$\theta_{\text{max}} = 26.4^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.093$
 $S = 0.97$
 4154 reflections
 381 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 with 1147 Friedel pairs
 Flack parameter: 0.012 (10)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 \cdots O1	0.86	2.14	2.709 (7)	123
N3–H3 \cdots O4	0.86	2.07	2.638 (7)	123
N1–H1 \cdots O4 ⁱ	0.86	2.30	3.007 (7)	140
N3–H3 \cdots O1 ⁱⁱ	0.86	2.29	3.030 (7)	145

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

All H atoms were placed in geometrically idealized positions, with C–H = 0.93–0.97 Å and N–H = 0.86 Å, and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$, except for the methyl groups, for which $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. Each methyl group was allowed to rotate about its local threefold axis.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

The authors are grateful to Dr Hai-Bing Song and Hong-Gen Wang of Nankai University for collecting the intensity data.

References

- Bruker (1997). SMART (Version 5.611), SAINT (Version 6.0), SADABS (Version 2.03) and SHELXTL (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Huang, Z. T. & Wamhoff, H. (1984). *Chem. Ber.* **117**, 1856–1867.
- Huang, Z.-T. & Wang, M.-X. (2002). *Prog. Nat. Sci.* **12**, 249–257.
- Wang, L.-B., Yu, C. Y. & Huang, Z.-T. (1994). *Synthesis*, **12**, 1441–1444.
- Wang, X. J., Zhu, N. J., Guo, F., Liu, Z. R. & Huang, Z. T. (1987). *J. Struct. Chem.* **6**, 62–65.