4-(4-Bromophenyl)-3-(2,5-dioxopyrrolidin-1-yl)-1-(4-methyloxyphenyl)azetidin-2-one

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.077
Data-to-parameter ratio = 13.0

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

The title compound, $C_{20}H_{17}BrN_2O_4$, contains a four-membered β-lactam ring; the 2,5-dioxopyrrolidin-1-yl and 4-bromophenyl groups are in *cis* positions.

Comment

2-Azetidinones are an important class of organic compounds having a wide range of biological and antibacterial activities. (Durckheimer *et al.*, 1985; Palomo *et al.*, 2004; Khadsan *et al.*, 2005). It is well known that the ketene–imine cycloaddition reaction (the Staudinger reaction) is one of the most versatile procedures for the synthesis of 2-azetidinones; in this reaction ketenes are usually generated by the elimination of acyl chlorides (Delpiccolo *et al.*, 2004; Rosenblum *et al.*, 2000). During our efforts at synthesizing 2-azetidinones using *N*-(chloroformyl)methylsuccinimide as the source of the ketene, we obtained the title compound, (I).

(I)
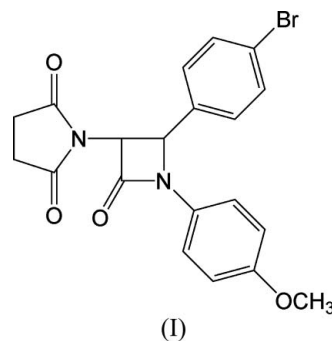
A view of the molecular structure is shown in Fig.1. The C5—C7 bond in the four-membered ring [1.574 (3) Å] is much longer than that of the normal C—C bond (1.54 Å), indicating that there is steric hindrance between the substituent groups on C5 and C7. The 2,5-dioxopyrrolidin-1-yl and bromophenyl groups attached to C5 and C7 are oriented away from the plane and are in *cis* positions.

Experimental

A mixture of *N*-(chloroformyl)methyl succinimide (5 mmol) and *N*-[(4-bromophenyl)methylene]-4-methoxybenzenamine(3.3 mmol), benzene (30 ml) and Et_3N (1.6 ml) was stirred at room temperature for 6 h. The product was filtered off and washed with 10% HCl, then dried over anhydrous Na_2SO_4. The benzene solution was evaporated and the residue was purified by column chromatography (ethyl acetate/petroleum ether = 1:1) to give the title product. Crystals suitable for X-ray analysis were obtained by slow evaporation of the solvent in air. Analysis calculated for $C_{20}H_{17}BrN_2O_4$: C 55.91, H 3.96, N 6.52%; found: C 55.93, H 3.98, N 6.50%.

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Crystal data

$C_{20}H_{17}BrN_2O_4$
$M_r = 429.27$
Triclinic, $P\bar{1}$
$a = 9.1715\ (11)$ Å
$b = 10.1286\ (12)$ Å
$c = 10.6487\ (12)$ Å
$\alpha = 111.211\ (1)°$
$\beta = 92.045\ (2)°$
$\gamma = 94.645\ (1)°$

$V = 916.91\ (19)$ Å^3
$Z = 2$
$D_x = 1.555$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.27$ mm^{-1}
$T = 293\ (2)$ K
Block, colorless
$0.24 \times 0.20 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector
  diffractometer
φ and ω scans
Absorption correction: multi-scan
  (SADABS; Sheldrick, 1996)
  $T_{min} = 0.572$, $T_{max} = 0.728$

5015 measured reflections
3189 independent reflections
2608 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.077$
$S = 1.04$
3189 reflections
245 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.036P)^2$
  $+ 0.3352P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ e Å^{-3}

H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and refined in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

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: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.
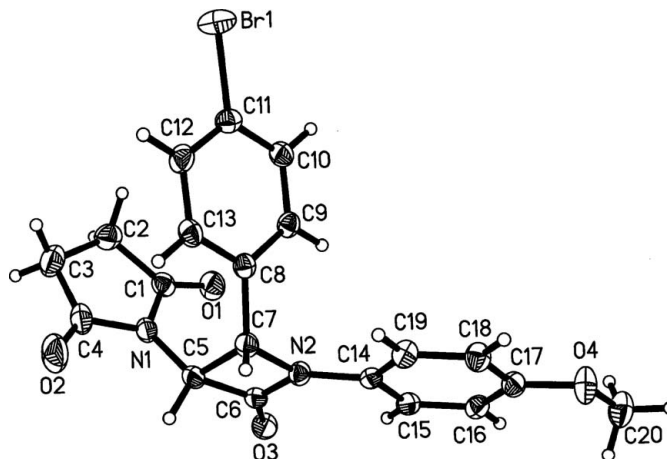

Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

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