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10-(2-Bromo-4,5-dimethoxybenzyl)-7-chloro-5-cyclopropyl-9-methyl-5,10-dihydro-4,5,6,10-tetraazadibenzo[*a,d*]cyclohepten-11-one

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.155
Data-to-parameter ratio = 13.4

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

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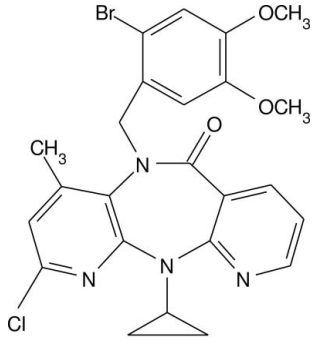
In the crystal structure of the title compound, $C_{24}H_{22}BrClN_4O_3$, there are C—H···O and C—H···Br intermolecular hydrogen bonds, and also C—H···O, C—H···Br and C—H···N intramolecular hydrogen bonds.

Comment

There has been widespread interest in the chemistry of azepines due to their biological activities and their applications as anti-HIV drugs (Dyatkin *et al.*, 1998). This is the first human immuno-deficiency virus type 1 (HIV-1) non-nucleoside reverse transcriptase (RT) inhibitor to obtain regulatory approval (Cywin *et al.*, 1998). It prevents the damage to the immune system and reduces the risk of developing AIDS-related illness (Campiani *et al.*, 1999). The compound 7-chloro-5-cyclopropyl-9-methyl-5,10-dihydro-4,5,6,10-tetraazadibenzo[*a,d*]cyclohepten-11-one is an intermediate of a potent anti-HIV drug, Nevirapine. The *N*-alkylation of the compound by different alkyl and aryl halides leads to novel molecules of biological interest.



(I)

In the title compound (I), $C_{24}H_{22}BrClN_4O_3$, the central seven-membered ring is puckered. Atoms N5 and N13 deviate from the plane defined by the atoms N3/C2/C16–C8/N7/C6–C4 by 1.091 (3) and −0.952 (3) Å, respectively. This deviation may be attributed to the substitution of the 5-cyclopropyl and 10-(2-bromo-4,5-dimethoxybenzyl) groups. The total puckering amplitude Q_T (Cremer & Pople, 1975) is 2.606 (4) Å.

The structure exhibits intermolecular hydrogen bonding of the types C—H···O and C—H···Br (Table 1 and Fig. 2). The packing of the molecules, when viewed down the *a* axis (Fig. 2), indicates that the molecules are interlinked by hydrogen bonds.

Experimental

To a solution of 7-chloro-5-cyclopropyl-9-methyl-5,10-dihydro-4,5,6,10-tetraazadibenzo[*a,d*]cyclohepten-11-one (0.5 g, 1.66 mmol)

in 5 ml of DMF, a mixture of 1-bromo-2-bromomethyl-4,5-dimethoxybenzene (0.51 g, 1.66 mmol) and anhydrous powdered potassium carbonate (0.688 g, 4.98 mmol) were added, and the mixture was heated to 333 K for 6 h. The reaction was monitored by TLC. After completion of the reaction, the solvent was removed under reduced pressure and the product extracted with ethyl acetate. The organic layer was dried with anhydrous sodium sulfate. The solvent was evaporated to obtain the crude product which was purified over silica gel using hexane and ethyl acetate (8:2) as an eluent for column chromatography (yield: 80%). The pure product thus obtained was recrystallized by slow evaporation of an acetonitrile solution. After five days, pale-yellow crystals were obtained. Melting point: 461–463 K. Elemental analysis data in (%) for $C_{24}H_{22}BrClN_4O_3$, calculated: C 54.41, H 4.19, N 10.57; found: C 54.43, H 4.17, N 10.54.

Crystal data

$C_{24}H_{22}BrClN_4O_3$	$Z = 4$
$M_r = 529.82$	$D_x = 1.495$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.0680$ (9) Å	$\mu = 1.89$ mm^{-1}
$b = 16.7840$ (9) Å	$T = 295$ (2) K
$c = 16.1960$ (16) Å	Block, pale yellow
$\beta = 107.260$ (2)°	0.30 × 0.25 × 0.25 mm
$V = 2354.0$ (4) Å^3	

Data collection

MacScience DIPLabo 32001 diffractometer	4048 independent reflections
	2933 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.032$
Absorption correction: none	$\theta_{max} = 25.0°$
7627 measured reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0944P)^2 + 0.2553P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	
$wR(F^2) = 0.155$	
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
4048 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
301 parameters	$\Delta\rho_{min} = -0.68$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
C21—H13B···Br29^{i}	0.97	2.72	3.514 (5)	139
C22—H22A···O18	0.97	2.33	2.730 (6)	104
C22—H22B···Br29	0.97	2.71	3.239 (5)	115
C27—H27···O18^{ii}	0.93	2.47	3.390 (6)	170
C17—H33B···N13	0.96	2.56	2.961 (5)	105

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

H atoms were placed at idealized positions and allowed to ride on their parent atoms with C—H distances in the range 0.93–0.98 Å; U_{iso}(H) = 1.2U_{eq}(carrier atom) for all H atoms.

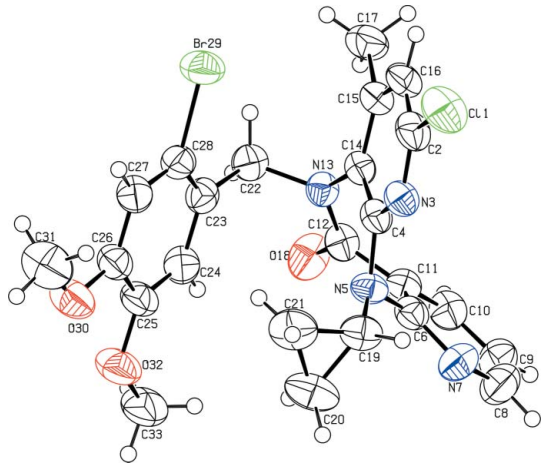


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids.

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski and Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *PLATON*.

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