Instrumental Achievements

Crystal Structure of 4-(4-Bromophenyl)-1,7,7-trimethyl-1,2,3,4,5,6,7,8-octahydroquinazoline-2,5-dione

M. Murat CANDAN,*† Engin KENDİ,* Mine YARİM, Selma SARAÇ** and Mevlüt ERTAN****

**Department of Engineering Physics, Hacettepe University, Beytepe 06532, Ankara, Turkey*
***Hacettepe University, Faculty of Pharmacy, Department of Pharmaceutical Chemistry, 06100 Sıhhiye, Ankara, Turkey*

(Received December 25, 2000; Accepted April 16, 2001)

4-(4-Bromophenyl)-1,7,7-trimethyl-1,2,3,4,5,6,7,8-octahydroquinazoline-2,5-dione was obtained from Biginelli-type cyclocondensation[1] of 5,5-dimethylcyclohexane-1,3-dione with urea and 4-bromobenzaldehyde: yield 1.16 g (62.19%), m.p. 174 – 175°C. A summary of the key crystallographic information is given in Table 1. The atomic coordinates and equivalent isotropic displacement parameters with estimated standard deviations for atoms except H are listed in Table 2, and selected bond lengths and bond angles in Table 3. A perspective view of the title molecule showing the atom-numbering scheme is presented in Fig. 2.

The phenyl ring at C4 is nearly perpendicular to the quinazoline ring system, with the dihedral angle of 84.4(1)°. The observed bond lengths of both C–O in these structures are normal. The differences between the lenghts of C2–N1 and C2–N3 are 0.065 Å, similar to the value of 0.065 Å found in 1,7,7-trimethyl-4-(4-methylphenyl)-1,2,3,4,5,6,7,8-octahydro-

Table 1 Crystal and experimental data

Formula: $C_{17}H_{19}BrN_2O_2$
Formula weight = 363.25
Crystal system: monoclinic
Space group: $P2_1/c$ $Z = 4$
a = 11.233(1)Å
b = 8.043(1)Å β = 95.62(1)°
c = 17.594(2)Å
V = 1581.9(3)Å^3
D_c = 1.525 g/cm^3
μ (Cu K$_\alpha$) = 3.614 mm^{-1}
T = 295 K
Yellow
F(0 0 0) = 744
Crystal size: 0.40 × 0.40 × 0.12 mm
$2\theta_{max}$ = 148.5°
R = 0.051
Rw = 0.136
No. of reflection used = 2745 ($I > 2\sigma(I)$)
No. of parameters = 203
Goodness-of-fit = 1.13
$(\Delta/\sigma)_{max}$ = 0.003
$(\Delta\rho)_{max}$ = 0.76 eÅ^{-3}
$(\Delta\rho)_{min}$ = –0.44 eÅ^{-3}
Measurements: Enraf-Nonius CAD-4 diffractometer
Refinement: full matrix least-squares (SHELXL-97)
Program system: CAD-4 EXPRESS software
Structure determination: SHELXS-97
Treatment of hydrogen atoms: geometric calculation

† To whom correspondence should be addressed.

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters (U_{eq}) for non-hydrogen atoms

Atom	x	y	z	U_{eq}/Å^2
Br1	0.1834(1)	0.8698(9)	0.0588(5)	0.0894(4)
O1	0.5042(3)	1.5551(5)	0.1276(2)	0.0583(9)
O2	0.8420(3)	1.0267(5)	0.2941(2)	0.0620(9)
N1	0.8420(3)	1.2179(5)	0.1981(2)	0.0479(8)
N3	0.6763(3)	1.1812(5)	0.2661(2)	0.0492(9)
C2	0.7878(4)	1.1329(5)	0.2555(3)	0.0461(10)
C4	0.5963(3)	1.2890(5)	0.2173(2)	0.0415(9)
C4a	0.6678(4)	1.3838(5)	0.1636(2)	0.0427(9)
C5	0.6062(4)	1.5108(6)	0.1170(3)	0.0455(9)
C6	0.6695(4)	1.5815(7)	0.0519(3)	0.0562(11)
C7	0.8035(4)	1.6041(6)	0.0733(3)	0.0477(10)
C8	0.8575(4)	1.4392(6)	0.1037(2)	0.0450(9)
C8a	0.7846(4)	1.3459(5)	0.1568(2)	0.0414(9)
C9	0.8655(5)	1.6531(8)	0.0023(3)	0.0654(14)
C10	0.8259(5)	1.7404(7)	0.1335(3)	0.0637(13)
C11	0.9629(4)	1.1645(7)	0.1846(3)	0.0588(12)
C12	0.4958(4)	1.1875(5)	0.1765(2)	0.0421(9)
C13	0.3791(4)	1.2153(5)	0.1908(3)	0.0464(9)
C14	0.2865(4)	1.1208(6)	0.1556(3)	0.0540(11)
C15	0.3117(4)	1.0005(6)	0.1054(3)	0.0540(11)
C16	0.4270(5)	0.9683(7)	0.0889(3)	0.0605(12)
C17	0.5181(4)	1.0633(6)	0.1238(3)	0.0531(11)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

Table 3 Selected geometric parameters (Å, °)

Br1 – C15	1.903 (5)	N3 – C2	1.342 (6)
O1 – C5	1.232 (5)	N3 – C4	1.465 (6)
O2 – C2	1.216 (6)	C4a – C8a	1.364 (6)
N1 – C2	1.407 (6)	C4 – C12	1.516 (6)
N1 – C8a	1.382 (6)	C7 – C9	1.540 (6)
N1 – C11	1.466 (6)	C7 – C10	1.527 (7)
C2-N1-C8a	121.8 (4)	C4a-C5-C6	117.4 (4)
C2-N3-C4	128.2 (3)	C5-C6-C7	112.4 (4)
N1-C2-N3	115.7 (3)	C6-C7-C8	109.3 (4)
N3-C4-C4a	109.5 (3)	N1-C8a-C8	116.4 (4)
C6-C7-C10	110.4 (4)	C8-C7-C9	108.0 (4)

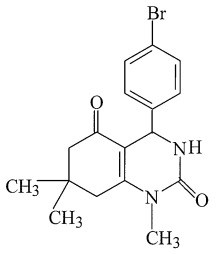

Fig. 1 Chemical structure.

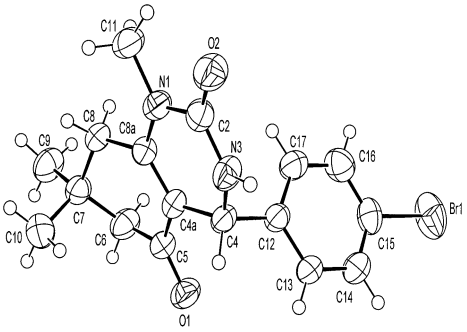

Fig. 2 The ORTEP drawing of the title compound with atom labeling.

Table 4 Selected geometric parameters (Å, °)

D–H···A	D···A	D–H	H···A	∠D–H···A
C11-H11b...O2	2.700 (7)	0.960	2.255	107.3
N3-H3...O1^{i}	3.062 (5)	0.860	2.274	152.3

Symmetry codes: (i) -x+1,+y-1/2,-z+1/2

quinazoline-2,5-dione.[2] This situation can be attributed to the difference in the hybridization of the adjacent carbon atoms C8a and C4.

An intermolecular hydrogen bond is found between N and O; geometric details are given in Table 4.

References

1. P. Biginelli, *Chem. Ber*., **1991**, *24*, 1317.
2. M. M. Candan, E. Kendi, M. Yarim, S. Saraç, and M. Ertan, *Anal. Sci*., **2000**, submitted.