Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[2-(4-Methylpiperazin-1-yl)ethyl]isoindoline-1,3-dione

Mi Zhou, Ying Shao,* Yong-an Xia, Xiao-Long Liu and Xiao-Qiang Sun

Key Laboratory of Fine Petrochemical Engineering, Changzhou University, Changzhou 213164, Jiangsu, People's Republic of China
Correspondence e-mail: shaoying810724@163.com

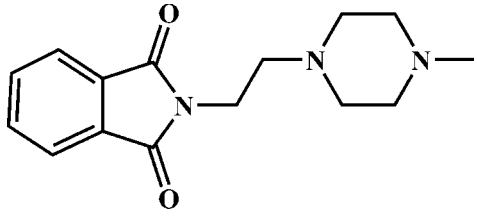
Received 15 April 2013; accepted 30 January 2014

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.154; data-to-parameter ratio = 14.5.

In the title compound, $C_{15}H_{19}N_3O_2$, the piperazine ring adopts a chair conformation, with its N—C bonds in pseudo-equatorial orientations. The dihedral angle between the C atoms of the piperazine ring and the phthalamide ring system (r.m.s. deviaiton = 0.008 Å) is 89.30 (8)°. In the crystal, molecules are linked by C—H···O hydrogen bonds, generating a three-dimensional network and aromatic π–π interactions also occur [centroid–centroid distances = 3.556 (1)–3.716 (1) Å].

Related literature

For background to piperazine derivatives, see: Tian *et al.* (2011); Stauffer (2011). For the preparation, see: Ghosh *et al.* (2010). For a similar structure, see: Shao *et al.* (2012).



Experimental

Crystal data

$C_{15}H_{19}N_3O_2$
M_r = 273.33
Triclinic, $P\overline{1}$
a = 6.9537 (12) Å
b = 8.4410 (15) Å
c = 12.563 (2) Å
α = 96.260 (4)°
β = 98.381 (4)°
γ = 92.647 (3)°
V = 723.7 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.30 × 0.28 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.975, T_{max} = 0.979
4194 measured reflections
2654 independent reflections
2206 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.154
S = 1.01
2654 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.57	3.406 (3)	149
C15—H15B···O2^{ii}	0.96	2.53	3.343 (3)	142

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+1, -y, -z$.

Data collection: *APEX2* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge the Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD) and the Natural Science Foundation of Jiangsu Education Department (No. SCZ1212400004) for financial support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7071).

References

Bruker (2000). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ghosh, B., Antonio, T., Zhen, J., Kharkar, P., Reith, M. E. A. & Dutta, A. K. (2010). *J. Med. Chem.* **53**, 1023–1037.
Shao, Y., An, D., Zhou, M., Liu, L. & Sun, X.-Q. (2012). *Acta Cryst.* E**68**, o173.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stauffer, S. R. (2011). *ACS Chem. Neurosci.* **2**, 450–470.
Tian, Z., Wei, X., Liang, J., Liu, R. & Zhang, Y. (2011). *Yingyong Huagong*, **40**, 1648–1652.

supplementary materials

Acta Cryst. (2014). E**70**, o287 [doi:10.1107/S1600536814002232]

2-[2-(4-Methylpiperazin-1-yl)ethyl]isoindoline-1,3-dione

Mi Zhou, Ying Shao, Yong-an Xia, Xiao-Long Liu and Xiao-Qiang Sun

1. Comment

Piperazine derivatives have received much attention for their pharmaceutical activities (Tian, *et al.*, 2011; Stauffer, 2011). The title compound was synthesized from *N*-(2-bromoethyl)phthalimide and *N*-methylpiperazine (Ghosh, *et al.*, 2010), as a drug intermediate. In the molecule, Fig. 1, the six-membered piperazine ring adopts the chair conformation. The phthalimide ring is coplanar [r.m.s. deviations = 0.01 Å]. In the crystal, Fig. 2, molecules are linked *via* intermolecular C–H···O hydrogen bonding interactions (Table 1) and $\pi\cdots\pi$ stacking interactions involving the benzene and maleinimide rings (Table 2), which is different form the similar compound (Shao, *et al.*, 2012).

2. Experimental

A suspension of *N*-(2-bromoethyl)phthalimide (2.54 g, 10.0 mmol), *N*-methylpiperazine (1.10 g, 11.1 mmol) and K_2CO_3 (2.70 g, 19.6 mmol) in 20 ml CH_3CN was stirred at room temperature for 0.5 h, and then heated to reflux for over 20 h. After cooling and filtration, the filter residue was washed with CH_3CN. And the filtrate and washing were combined prior to removing the solvent under vacuum. The title compound (2.15 g, 7.9 mmol) was obtained as colorless needles with m.p. 102.2–103.0 °C, after recrystallization from n-hexane. Colourless blocks were obtained by slow evaporation of CH_3OH.

3. Refinement

All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances of 0.93–0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Computing details

Data collection: *APEX2* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

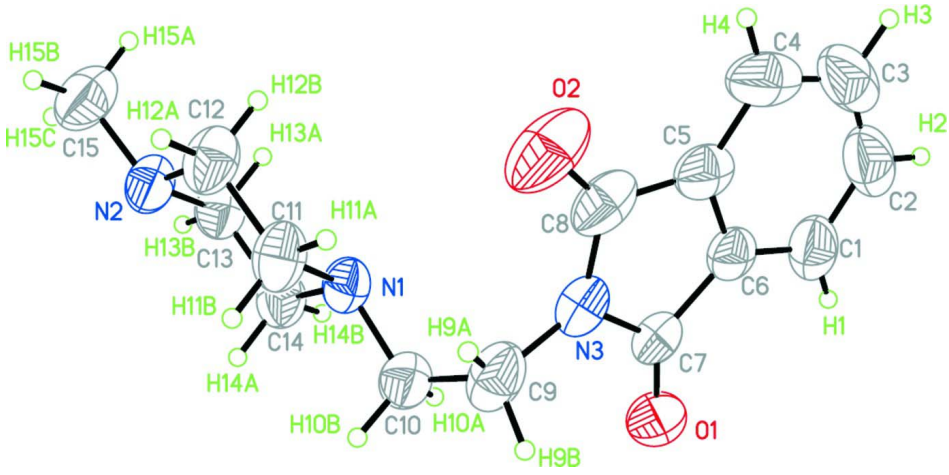


Figure 1

View of the title compound, showing 50% probability ellipsoids.

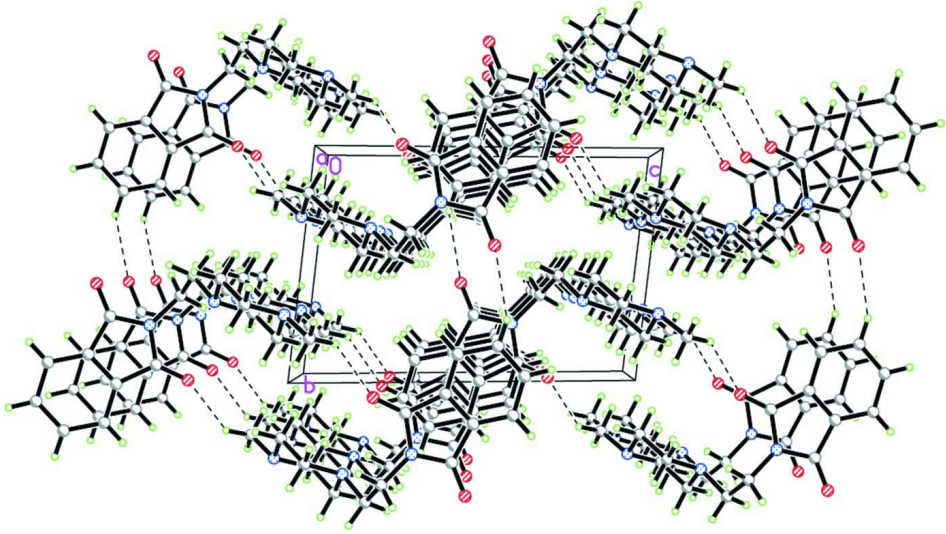


Figure 2

Perspective view of the title compound along a direction. Labels of atoms have been omitted for clarity.

2-[2-(4-Methylpiperazin-1-yl)ethyl]isoindoline-1,3-dione

Crystal data

$C_{15}H_{19}N_3O_2$
$M_r = 273.33$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.9537\ (12)$ Å
$b = 8.4410\ (15)$ Å
$c = 12.563\ (2)$ Å
$\alpha = 96.260\ (4)°$
$\beta = 98.381\ (4)°$
$\gamma = 92.647\ (3)°$
$V = 723.7\ (2)$ Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.254$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2417 reflections
$\theta = 2.8–29.0°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Block, colorless
0.30 × 0.28 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.975, T_{max} = 0.979
4194 measured reflections
2654 independent reflections
2206 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.5°, θ_{min} = 1.7°
h = −8→8
k = −10→9
l = −7→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.154
S = 1.01
2654 reflections
183 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0987P)^2 + 0.097P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.074 (13)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.7472 (2)	0.1040 (2)	0.64462 (16)	0.0663 (5)
H1	0.7495	0.1864	0.7004	0.080*
C2	0.7384 (3)	−0.0535 (3)	0.6639 (2)	0.0853 (7)
H2	0.7335	−0.0780	0.7339	0.102*
C3	0.7366 (3)	−0.1743 (3)	0.5820 (3)	0.0952 (9)
H3	0.7321	−0.2795	0.5976	0.114*
C4	0.7415 (3)	−0.1437 (2)	0.4756 (2)	0.0866 (8)
H4	0.7398	−0.2263	0.4200	0.104*
C5	0.7489 (2)	0.0147 (2)	0.45574 (15)	0.0583 (5)
C6	0.75250 (19)	0.13529 (18)	0.53922 (13)	0.0490 (4)
C7	0.7637 (2)	0.28929 (18)	0.49368 (12)	0.0470 (4)
C8	0.7547 (2)	0.0895 (2)	0.35403 (15)	0.0623 (5)
C9	0.7949 (2)	0.3733 (3)	0.31185 (14)	0.0682 (5)
H9A	0.8471	0.3229	0.2500	0.082*
H9B	0.8920	0.4539	0.3501	0.082*

C10	0.6130 (3)	0.4551 (2)	0.27092 (14)	0.0618 (5)
H10A	0.5547	0.4975	0.3328	0.074*
H10B	0.6512	0.5447	0.2348	0.074*
C11	0.5329 (2)	0.3035 (2)	0.09326 (13)	0.0585 (4)
H11A	0.6436	0.2385	0.1059	0.070*
H11B	0.5750	0.3973	0.0624	0.070*
C12	0.3737 (3)	0.2097 (2)	0.01439 (13)	0.0613 (5)
H12A	0.4212	0.1801	−0.0534	0.074*
H12B	0.3373	0.1124	0.0431	0.074*
C13	0.1346 (2)	0.3462 (2)	0.09674 (13)	0.0534 (4)
H13A	0.0968	0.2504	0.1267	0.064*
H13B	0.0211	0.4083	0.0848	0.064*
C14	0.2917 (2)	0.44238 (19)	0.17559 (12)	0.0532 (4)
H14A	0.3254	0.5402	0.1467	0.064*
H14B	0.2430	0.4712	0.2432	0.064*
C15	0.0515 (3)	0.2147 (3)	−0.08443 (17)	0.0777 (6)
H15A	0.0143	0.1167	−0.0585	0.117*
H15B	0.0996	0.1915	−0.1519	0.117*
H15C	−0.0595	0.2779	−0.0951	0.117*
N1	0.46613 (18)	0.35309 (15)	0.19655 (9)	0.0484 (3)
N2	0.20302 (19)	0.30264 (16)	−0.00548 (10)	0.0541 (4)
N3	0.76325 (17)	0.25325 (17)	0.38367 (10)	0.0521 (4)
O1	0.77364 (19)	0.42351 (14)	0.54040 (10)	0.0671 (4)
O2	0.7541 (2)	0.0268 (2)	0.26330 (12)	0.0968 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0513 (9)	0.0830 (13)	0.0639 (11)	0.0043 (8)	−0.0028 (8)	0.0210 (9)
C2	0.0537 (11)	0.0913 (16)	0.1136 (18)	0.0011 (10)	−0.0070 (11)	0.0526 (15)
C3	0.0492 (11)	0.0704 (14)	0.164 (3)	0.0016 (9)	−0.0154 (13)	0.0502 (17)
C4	0.0445 (9)	0.0536 (11)	0.149 (2)	0.0062 (7)	−0.0133 (11)	−0.0109 (12)
C5	0.0338 (7)	0.0555 (9)	0.0795 (12)	0.0077 (6)	−0.0046 (7)	−0.0040 (8)
C6	0.0329 (7)	0.0546 (9)	0.0564 (9)	0.0042 (6)	−0.0025 (6)	0.0048 (7)
C7	0.0382 (7)	0.0555 (9)	0.0436 (8)	0.0009 (6)	−0.0003 (6)	−0.0014 (6)
C8	0.0384 (8)	0.0796 (12)	0.0612 (10)	0.0113 (7)	0.0005 (7)	−0.0197 (8)
C9	0.0503 (9)	0.1015 (14)	0.0504 (9)	−0.0138 (9)	0.0030 (7)	0.0131 (9)
C10	0.0672 (10)	0.0666 (10)	0.0485 (9)	−0.0100 (8)	0.0004 (7)	0.0109 (7)
C11	0.0535 (9)	0.0802 (11)	0.0439 (8)	0.0120 (8)	0.0104 (7)	0.0087 (8)
C12	0.0636 (10)	0.0726 (11)	0.0466 (9)	0.0210 (8)	0.0065 (7)	−0.0027 (8)
C13	0.0515 (9)	0.0584 (9)	0.0511 (9)	0.0121 (7)	0.0080 (7)	0.0067 (7)
C14	0.0598 (9)	0.0564 (9)	0.0442 (8)	0.0118 (7)	0.0090 (7)	0.0040 (7)
C15	0.0771 (13)	0.0777 (13)	0.0673 (12)	0.0170 (10)	−0.0150 (10)	−0.0104 (10)
N1	0.0503 (7)	0.0565 (8)	0.0385 (7)	0.0033 (5)	0.0062 (5)	0.0066 (5)
N2	0.0588 (8)	0.0588 (8)	0.0423 (7)	0.0119 (6)	0.0009 (6)	0.0015 (6)
N3	0.0424 (7)	0.0677 (9)	0.0434 (7)	0.0035 (5)	0.0020 (5)	−0.0005 (6)
O1	0.0850 (9)	0.0542 (7)	0.0564 (7)	0.0002 (6)	0.0027 (6)	−0.0058 (5)
O2	0.0825 (10)	0.1228 (13)	0.0717 (9)	0.0219 (9)	0.0034 (7)	−0.0439 (9)

Geometric parameters (Å, º)

C1—C2	1.377 (3)	C10—H10A	0.9700
C1—C6	1.384 (3)	C10—H10B	0.9700
C1—H1	0.9300	C11—N1	1.466 (2)
C2—C3	1.366 (4)	C11—C12	1.504 (2)
C2—H2	0.9300	C11—H11A	0.9700
C3—C4	1.394 (4)	C11—H11B	0.9700
C3—H3	0.9300	C12—N2	1.459 (2)
C4—C5	1.386 (3)	C12—H12A	0.9700
C4—H4	0.9300	C12—H12B	0.9700
C5—C6	1.376 (2)	C13—N2	1.450 (2)
C5—C8	1.490 (3)	C13—C14	1.503 (2)
C6—C7	1.480 (2)	C13—H13A	0.9700
C7—O1	1.2120 (19)	C13—H13B	0.9700
C7—N3	1.382 (2)	C14—N1	1.464 (2)
C8—O2	1.202 (2)	C14—H14A	0.9700
C8—N3	1.388 (2)	C14—H14B	0.9700
C9—N3	1.458 (2)	C15—N2	1.451 (2)
C9—C10	1.519 (3)	C15—H15A	0.9600
C9—H9A	0.9700	C15—H15B	0.9600
C9—H9B	0.9700	C15—H15C	0.9600
C10—N1	1.457 (2)		

C2—C1—C6	117.6 (2)	C12—C11—H11A	109.4
C2—C1—H1	121.2	N1—C11—H11B	109.4
C6—C1—H1	121.2	C12—C11—H11B	109.4
C3—C2—C1	121.1 (2)	H11A—C11—H11B	108.0
C3—C2—H2	119.4	N2—C12—C11	111.05 (14)
C1—C2—H2	119.4	N2—C12—H12A	109.4
C2—C3—C4	121.6 (2)	C11—C12—H12A	109.4
C2—C3—H3	119.2	N2—C12—H12B	109.4
C4—C3—H3	119.2	C11—C12—H12B	109.4
C5—C4—C3	117.5 (2)	H12A—C12—H12B	108.0
C5—C4—H4	121.3	N2—C13—C14	110.46 (13)
C3—C4—H4	121.3	N2—C13—H13A	109.6
C6—C5—C4	120.36 (19)	C14—C13—H13A	109.6
C6—C5—C8	107.93 (15)	N2—C13—H13B	109.6
C4—C5—C8	131.71 (19)	C14—C13—H13B	109.6
C5—C6—C1	121.86 (17)	H13A—C13—H13B	108.1
C5—C6—C7	107.87 (15)	N1—C14—C13	111.53 (13)
C1—C6—C7	130.27 (15)	N1—C14—H14A	109.3
O1—C7—N3	124.58 (15)	C13—C14—H14A	109.3
O1—C7—C6	128.68 (14)	N1—C14—H14B	109.3
N3—C7—C6	106.73 (13)	C13—C14—H14B	109.3
O2—C8—N3	124.7 (2)	H14A—C14—H14B	108.0
O2—C8—C5	129.28 (19)	N2—C15—H15A	109.5
N3—C8—C5	106.01 (14)	N2—C15—H15B	109.5
N3—C9—C10	114.32 (13)	H15A—C15—H15B	109.5
N3—C9—H9A	108.7	N2—C15—H15C	109.5

C10—C9—H9A	108.7	H15A—C15—H15C	109.5
N3—C9—H9B	108.7	H15B—C15—H15C	109.5
C10—C9—H9B	108.7	C10—N1—C14	108.35 (13)
H9A—C9—H9B	107.6	C10—N1—C11	112.08 (13)
N1—C10—C9	114.94 (15)	C14—N1—C11	108.84 (12)
N1—C10—H10A	108.5	C13—N2—C15	111.53 (14)
C9—C10—H10A	108.5	C13—N2—C12	108.61 (12)
N1—C10—H10B	108.5	C15—N2—C12	111.25 (13)
C9—C10—H10B	108.5	C7—N3—C8	111.45 (15)
H10A—C10—H10B	107.5	C7—N3—C9	123.36 (15)
N1—C11—C12	111.26 (13)	C8—N3—C9	124.69 (15)
N1—C11—H11A	109.4		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.93	2.57	3.406 (3)	149
C15—H15*B*···O2^{ii}	0.96	2.53	3.343 (3)	142

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*+1, −*y*, −*z*.

Table 2 π-π hydrogen-bond geometry (Å)

*Cg*I	*Cg*J	*Cg*I···*Cg*J
*Cg*1	*Cg*3^{i}	3.607 (1)
*Cg*1	*Cg*3^{ii}	3.716 (1)
*Cg*1	*Cg*3^{i}	3.556 (1)

Symmetry codes: (i) 1-*x*, -*y*, 1-*z*; (ii) 2-*x*, -*y*, 1-*z*; *Cg*1 and *Cg*3 are the centroids of the N3–C7–C6–C5–C8 and C1–C6 rings.