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3-(6-Bromohexyl)-1,5-dimethyl-1*H*-1,5-benzodiazepine-2,4(3*H*,5*H*)-dione

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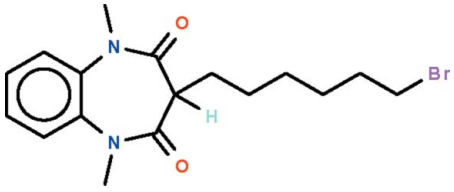
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.153; data-to-parameter ratio = 24.4.

The seven-membered ring in the title compound, $C_{17}H_{23}BrN_2O_2$, adopts a boat-shaped conformation (with the C atoms of the fused-ring as the stern and the methine C atom as the prow). The bromohexyl substituent occupies an equatorial position, with the hexyl chain exhibiting an extended conformation. Weak intermolecular C—H···O hydrogen bonding is present in the crystal structure.

Related literature

For the crystal structure of 1,5-dimethyl-1,5-benzodiazepin-2,4-dione, see: Mondieig *et al.* (2005).



Experimental

Crystal data

$C_{17}H_{23}BrN_2O_2$ M_r = 367.28
Monoclinic, $P2_1/n$
a = 7.5214 (1) Å
b = 9.3693 (2) Å
c = 23.8686 (5) Å
β = 91.750 (1)°
V = 1681.24 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.45 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.526, T_{max} = 0.791
25590 measured reflections
4897 independent reflections
3478 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.153
S = 1.01
4897 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.92 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7B···O1^{i}	0.96	2.58	3.430 (3)	147
C7—H7C···O2^{ii}	0.96	2.51	3.471 (3)	174
C11—H11B···O1^{ii}	0.96	2.60	3.551 (3)	173

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5049).

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supporting information

Acta Cryst. (2010). E**66**, o2804 [https://doi.org/10.1107/S1600536810040122]

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S1. Comment

The methylene part of 1,5-dimethyl-1,5-benzodiazepine-2,4-dione is relatively acidic, and one proton can be abstracted by using potassium *t*-butoxide; the resulting carbanion can undergo a nucleophlilic subsitution with a dibromoalkane to form 3-substituted derivatives. In this study, the compound is reacted with 1,6-dibromohexane the title compound (Scheme I, Fig. 1).

S2. Experimental

To a solution of the potassium *t*-butoxide (0.42 g, 3.6 mmol) in DMF (15 ml) was added 1,5-dimethyl-1,5-benzodiazepine-2,4-dione (0.50 g, 2.4 mmol) and 1,6-dibromodohexane (0.40 ml, 2.88 mmol). Stirring was continued for 24 h. The reaction was monitored by thin layer chromatography. The mixture was filtered and the solution evaporated to give colorless crystals.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C).

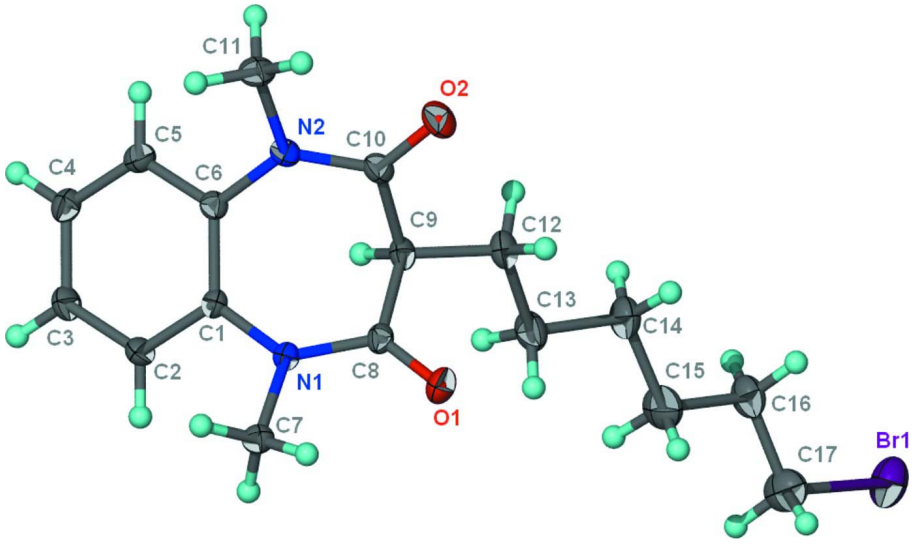

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{17}H_{23}BrN_2O_2$ at the 50% probability level; hydrogen atoms are drawn as arbitrary radius.

3-(6-Bromohexyl)-1,5-dimethyl-1*H*-1,5-benzodiazepine- 2,4(3*H*,5*H*)-dione

Crystal data

$C_{17}H_{23}BrN_2O_2$
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Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.5214$ (1) Å
$b = 9.3693$ (2) Å
$c = 23.8686$ (5) Å
$\beta = 91.750$ (1)°
$V = 1681.24$ (6) Å^3
$Z = 4$
$F(000) = 760$
$D_x = 1.451$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5411 reflections
$\theta = 2.3$–$26.0°$
$\mu = 2.45$ mm^{-1}
$T = 293$ K
Prism, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Bruker X8 APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.526$, $T_{max} = 0.791$
25590 measured reflections
4897 independent reflections
3478 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 30.0°$, $\theta_{min} = 2.8°$
$h = -10 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -28 \rightarrow 33$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.153$
$S = 1.01$
4897 reflections
201 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
$w = 1/[\sigma^2(F_o^2) + (0.082P)^2 + 1.8005P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.92$ e Å^{-3}
$\Delta\rho_{min} = -0.68$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.30982 (5)	0.07856 (4)	0.564211 (15)	0.04567 (14)
O1	0.5859 (3)	0.4826 (2)	0.28329 (9)	0.0297 (4)
O2	0.1496 (3)	0.4797 (2)	0.35848 (10)	0.0351 (5)
N1	0.5074 (3)	0.7166 (2)	0.27741 (9)	0.0211 (4)
N2	0.1710 (3)	0.7124 (2)	0.33415 (9)	0.0231 (4)
C1	0.4344 (3)	0.8410 (3)	0.30158 (10)	0.0199 (5)
C2	0.5247 (4)	0.9710 (3)	0.29605 (11)	0.0247 (5)
H2	0.6331	0.9729	0.2783	0.030*
C3	0.4550 (4)	1.0961 (3)	0.31659 (12)	0.0278 (6)
H3	0.5153	1.1818	0.3119	0.033*
C4	0.2944 (4)	1.0944 (3)	0.34434 (12)	0.0266 (5)
H4	0.2476	1.1784	0.3585	0.032*
C5	0.2056 (4)	0.9665 (3)	0.35053 (11)	0.0251 (5)

H5	0.0991	0.9651	0.3693	0.030*
C6	0.2725 (3)	0.8394 (3)	0.32918 (10)	0.0203 (5)
C7	0.5848 (3)	0.7301 (3)	0.22189 (11)	0.0244 (5)
H7A	0.5875	0.6381	0.2042	0.037*
H7B	0.7036	0.7667	0.2259	0.037*
H7C	0.5137	0.7942	0.1992	0.037*
C8	0.5209 (3)	0.5879 (3)	0.30474 (11)	0.0215 (5)
C9	0.4425 (4)	0.5856 (3)	0.36297 (11)	0.0233 (5)
H9	0.4770	0.6735	0.3827	0.028*
C10	0.2407 (4)	0.5863 (3)	0.35245 (11)	0.0237 (5)
C11	−0.0223 (3)	0.7209 (3)	0.32308 (12)	0.0281 (6)
H11A	−0.0647	0.6313	0.3084	0.042*
H11B	−0.0476	0.7951	0.2962	0.042*
H11C	−0.0807	0.7419	0.3573	0.042*
C12	0.5012 (4)	0.4579 (3)	0.39872 (12)	0.0291 (6)
H12A	0.4395	0.4608	0.4338	0.035*
H12B	0.4662	0.3709	0.3794	0.035*
C13	0.6995 (4)	0.4532 (3)	0.41145 (14)	0.0363 (7)
H13A	0.7388	0.5469	0.4239	0.044*
H13B	0.7603	0.4312	0.3772	0.044*
C14	0.7533 (4)	0.3430 (3)	0.45629 (14)	0.0372 (7)
H14A	0.7202	0.3782	0.4927	0.045*
H14B	0.6884	0.2551	0.4491	0.045*
C15	0.9504 (5)	0.3118 (4)	0.45750 (15)	0.0462 (8)
H15A	1.0143	0.4018	0.4588	0.055*
H15B	0.9794	0.2646	0.4228	0.055*
C16	1.0164 (4)	0.2199 (3)	0.50614 (13)	0.0360 (7)
H16A	1.0014	0.2709	0.5411	0.043*
H16B	0.9465	0.1331	0.5074	0.043*
C17	1.2095 (5)	0.1827 (5)	0.50011 (14)	0.0469 (8)
H17A	1.2768	0.2699	0.4953	0.056*
H17B	1.2219	0.1253	0.4666	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0511 (2)	0.0394 (2)	0.0454 (2)	0.00836 (15)	−0.01604 (15)	−0.00333 (14)
O1	0.0329 (10)	0.0220 (9)	0.0342 (10)	0.0060 (8)	0.0014 (8)	−0.0021 (8)
O2	0.0311 (11)	0.0260 (10)	0.0482 (13)	−0.0106 (8)	0.0014 (9)	0.0044 (9)
N1	0.0222 (10)	0.0170 (9)	0.0243 (10)	0.0011 (8)	0.0034 (8)	−0.0001 (8)
N2	0.0189 (10)	0.0223 (10)	0.0283 (11)	−0.0039 (8)	0.0017 (8)	0.0011 (8)
C1	0.0204 (11)	0.0177 (11)	0.0215 (11)	0.0023 (9)	−0.0003 (9)	0.0006 (9)
C2	0.0231 (12)	0.0213 (11)	0.0299 (13)	−0.0023 (10)	0.0051 (10)	0.0017 (10)
C3	0.0310 (14)	0.0196 (12)	0.0329 (14)	−0.0041 (10)	0.0022 (11)	0.0000 (10)
C4	0.0279 (13)	0.0209 (12)	0.0309 (13)	0.0027 (10)	−0.0004 (10)	−0.0027 (10)
C5	0.0220 (12)	0.0259 (12)	0.0276 (13)	0.0017 (10)	0.0024 (10)	−0.0006 (10)
C6	0.0187 (11)	0.0195 (11)	0.0227 (11)	−0.0019 (9)	−0.0011 (9)	0.0008 (9)
C7	0.0242 (12)	0.0257 (12)	0.0234 (12)	0.0011 (10)	0.0033 (9)	−0.0011 (10)

C8	0.0193 (11)	0.0171 (11)	0.0281 (12)	−0.0003 (9)	−0.0020 (9)	−0.0003 (9)
C9	0.0266 (12)	0.0166 (11)	0.0264 (12)	−0.0020 (9)	−0.0009 (10)	0.0013 (9)
C10	0.0258 (12)	0.0218 (12)	0.0237 (12)	−0.0042 (10)	0.0029 (9)	−0.0007 (9)
C11	0.0183 (11)	0.0328 (14)	0.0330 (14)	−0.0036 (10)	−0.0004 (10)	−0.0013 (11)
C12	0.0359 (15)	0.0201 (11)	0.0311 (14)	−0.0012 (11)	−0.0019 (11)	0.0030 (10)
C13	0.0343 (15)	0.0339 (15)	0.0403 (16)	−0.0016 (13)	−0.0050 (12)	0.0139 (13)
C14	0.0436 (17)	0.0312 (15)	0.0365 (16)	0.0045 (13)	−0.0029 (13)	0.0103 (12)
C15	0.0474 (19)	0.054 (2)	0.0369 (17)	0.0059 (17)	−0.0025 (14)	0.0178 (15)
C16	0.0428 (17)	0.0348 (15)	0.0298 (14)	−0.0013 (13)	−0.0064 (12)	0.0079 (12)
C17	0.0480 (19)	0.059 (2)	0.0334 (16)	0.0098 (17)	−0.0005 (14)	0.0101 (15)

Geometric parameters (Å, º)

Br1—C17	1.947 (3)	C9—C12	1.527 (4)
O1—C8	1.221 (3)	C9—C10	1.531 (4)
O2—C10	1.222 (3)	C9—H9	0.9800
N1—C8	1.373 (3)	C11—H11A	0.9600
N1—C1	1.419 (3)	C11—H11B	0.9600
N1—C7	1.469 (3)	C11—H11C	0.9600
N2—C10	1.359 (3)	C12—C13	1.514 (4)
N2—C6	1.420 (3)	C12—H12A	0.9700
N2—C11	1.472 (3)	C12—H12B	0.9700
C1—C6	1.402 (3)	C13—C14	1.533 (4)
C1—C2	1.402 (4)	C13—H13A	0.9700
C2—C3	1.380 (4)	C13—H13B	0.9700
C2—H2	0.9300	C14—C15	1.511 (5)
C3—C4	1.396 (4)	C14—H14A	0.9700
C3—H3	0.9300	C14—H14B	0.9700
C4—C5	1.382 (4)	C15—C16	1.517 (4)
C4—H4	0.9300	C15—H15A	0.9700
C5—C6	1.395 (4)	C15—H15B	0.9700
C5—H5	0.9300	C16—C17	1.505 (5)
C7—H7A	0.9600	C16—H16A	0.9700
C7—H7B	0.9600	C16—H16B	0.9700
C7—H7C	0.9600	C17—H17A	0.9700
C8—C9	1.526 (4)	C17—H17B	0.9700
C8—N1—C1	123.5 (2)	N2—C11—H11A	109.5
C8—N1—C7	118.6 (2)	N2—C11—H11B	109.5
C1—N1—C7	117.7 (2)	H11A—C11—H11B	109.5
C10—N2—C6	123.5 (2)	N2—C11—H11C	109.5
C10—N2—C11	118.3 (2)	H11A—C11—H11C	109.5
C6—N2—C11	118.0 (2)	H11B—C11—H11C	109.5
C6—C1—C2	118.9 (2)	C13—C12—C9	113.7 (2)
C6—C1—N1	122.3 (2)	C13—C12—H12A	108.8
C2—C1—N1	118.7 (2)	C9—C12—H12A	108.8
C3—C2—C1	120.9 (2)	C13—C12—H12B	108.8
C3—C2—H2	119.5	C9—C12—H12B	108.8

C1—C2—H2	119.5	H12A—C12—H12B	107.7
C2—C3—C4	120.2 (2)	C12—C13—C14	113.4 (3)
C2—C3—H3	119.9	C12—C13—H13A	108.9
C4—C3—H3	119.9	C14—C13—H13A	108.9
C5—C4—C3	119.3 (2)	C12—C13—H13B	108.9
C5—C4—H4	120.4	C14—C13—H13B	108.9
C3—C4—H4	120.4	H13A—C13—H13B	107.7
C4—C5—C6	121.4 (2)	C15—C14—C13	112.4 (3)
C4—C5—H5	119.3	C15—C14—H14A	109.1
C6—C5—H5	119.3	C13—C14—H14A	109.1
C5—C6—C1	119.4 (2)	C15—C14—H14B	109.1
C5—C6—N2	118.9 (2)	C13—C14—H14B	109.1
C1—C6—N2	121.7 (2)	H14A—C14—H14B	107.9
N1—C7—H7A	109.5	C14—C15—C16	115.0 (3)
N1—C7—H7B	109.5	C14—C15—H15A	108.5
H7A—C7—H7B	109.5	C16—C15—H15A	108.5
N1—C7—H7C	109.5	C14—C15—H15B	108.5
H7A—C7—H7C	109.5	C16—C15—H15B	108.5
H7B—C7—H7C	109.5	H15A—C15—H15B	107.5
O1—C8—N1	122.4 (2)	C17—C16—C15	110.6 (3)
O1—C8—C9	122.8 (2)	C17—C16—H16A	109.5
N1—C8—C9	114.8 (2)	C15—C16—H16A	109.5
C12—C9—C8	114.0 (2)	C17—C16—H16B	109.5
C12—C9—C10	111.3 (2)	C15—C16—H16B	109.5
C8—C9—C10	105.0 (2)	H16A—C16—H16B	108.1
C12—C9—H9	108.8	C16—C17—Br1	113.1 (2)
C8—C9—H9	108.8	C16—C17—H17A	109.0
C10—C9—H9	108.8	Br1—C17—H17A	109.0
O2—C10—N2	122.4 (3)	C16—C17—H17B	109.0
O2—C10—C9	122.3 (2)	Br1—C17—H17B	109.0
N2—C10—C9	115.3 (2)	H17A—C17—H17B	107.8

C8—N1—C1—C6	−47.6 (3)	C1—N1—C8—C9	2.9 (3)
C7—N1—C1—C6	137.3 (2)	C7—N1—C8—C9	177.9 (2)
C8—N1—C1—C2	134.5 (3)	O1—C8—C9—C12	17.7 (4)
C7—N1—C1—C2	−40.6 (3)	N1—C8—C9—C12	−164.4 (2)
C6—C1—C2—C3	−1.0 (4)	O1—C8—C9—C10	−104.4 (3)
N1—C1—C2—C3	177.0 (2)	N1—C8—C9—C10	73.5 (3)
C1—C2—C3—C4	1.3 (4)	C6—N2—C10—O2	177.1 (3)
C2—C3—C4—C5	−0.6 (4)	C11—N2—C10—O2	2.0 (4)
C3—C4—C5—C6	−0.6 (4)	C6—N2—C10—C9	−5.0 (4)
C4—C5—C6—C1	0.9 (4)	C11—N2—C10—C9	179.9 (2)
C4—C5—C6—N2	−176.7 (2)	C12—C9—C10—O2	−18.6 (4)
C2—C1—C6—C5	−0.2 (4)	C8—C9—C10—O2	105.2 (3)
N1—C1—C6—C5	−178.1 (2)	C12—C9—C10—N2	163.5 (2)
C2—C1—C6—N2	177.4 (2)	C8—C9—C10—N2	−72.7 (3)
N1—C1—C6—N2	−0.5 (4)	C8—C9—C12—C13	62.5 (3)
C10—N2—C6—C5	−133.0 (3)	C10—C9—C12—C13	−178.9 (2)

C11—N2—C6—C5	42.1 (3)	C9—C12—C13—C14	168.8 (3)
C10—N2—C6—C1	49.5 (4)	C12—C13—C14—C15	164.8 (3)
C11—N2—C6—C1	−135.5 (3)	C13—C14—C15—C16	171.7 (3)
C1—N1—C8—O1	−179.2 (2)	C14—C15—C16—C17	174.1 (3)
C7—N1—C8—O1	−4.1 (4)	C15—C16—C17—Br1	175.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···O1^{i}	0.96	2.58	3.430 (3)	147
C7—H7*C*···O2^{ii}	0.96	2.51	3.471 (3)	174
C11—H11*B*···O1^{ii}	0.96	2.60	3.551 (3)	173

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+1/2; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2.