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1,3,3,5-Tetramethyl-1*H*-1,5-benzodiazepine-2,4(3*H*,5*H*)-dioneRachida Dardouri,^a Youssef Kandri Rodi,^a Natalie Saffon,^b El Mokhtar Essassi^c and Seik Weng Ng^{d*}

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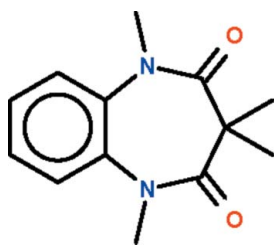
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.108; data-to-parameter ratio = 17.2.

The seven-membered ring in the title compound, $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_2$, adopts a boat-shaped conformation (with the C atoms of the fused-ring as the stern and the C atom bearing two methyl groups) as the prow.

Related literature

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



Experimental

Crystal data

$\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_2$
 $M_r = 232.28$
 Monoclinic, $P2_1/c$
 $a = 7.5112$ (1) Å
 $b = 10.1731$ (2) Å
 $c = 15.8697$ (3) Å
 $\beta = 103.675$ (1)°

$V = 1178.26$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 295$ K
 $0.32 \times 0.20 \times 0.18$ mm

Data collection

Bruker X8 APEXII diffractometer
 20993 measured reflections
 2719 independent reflections

2130 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.108$
 $S = 1.04$
 2719 reflections

158 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

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: BT5483).

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supplementary materials

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1,3,3,5-Tetramethyl-1*H*-1,5-benzodiazepine-2,4(3*H*,5*H*)-dione

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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 nucleophilic substitution with haloalkane to form 3-substituted derivatives. Previous studies have largely described the mono-substituted derivatives only. 1,12-Dibromodocane yielded the mono-substituted 12-bromodeyl derivative. In this study, the compound is reacted with methyl iodide to yield the di-methylated compound (Scheme I). The seven-membered ring adopts a boat-shaped conformation (with the C atoms of the fused-ring as the stern and the C atom bearing two methyl groups) as the prow (Fig. 1). The methyl group occupying the axial position hovers over the seven-membered ring, and the methyl group appears to be stopped from tipping over because of the π -system of the phenylene ring (Fig. 2).

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 methyl iodide (0.68 g, 4.80 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.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.96 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to 1.2–1.5 $U_{\text{eq}}(\text{C})$.

Figures

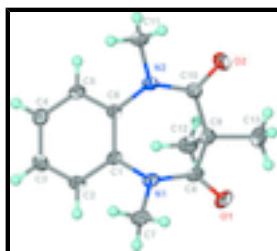


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_2$ at the 50% probability level; hydrogen atoms are drawn as arbitrary radius.

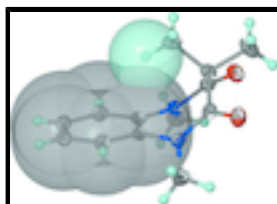


Fig. 2. Thermal ellipsoid plot (Barbour, 2001) showing van der Waals surfaces for the carbon atoms of the phenylene ring as well as the van der Waals surface for one of the methyl hydrogen atoms.

1,3,3,5-Tetramethyl-1*H*-1,5-benzodiazepine-2,4(3*H*,5*H*)-dione

Crystal data

$C_{13}H_{16}N_2O_2$	$F(000) = 496$
$M_r = 232.28$	$D_x = 1.309 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 4653 reflections
$a = 7.5112 (1) \text{ \AA}$	$\theta = 2.4\text{--}32.5^\circ$
$b = 10.1731 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 15.8697 (3) \text{ \AA}$	$T = 295 \text{ K}$
$\beta = 103.675 (1)^\circ$	Block, colorless
$V = 1178.26 (4) \text{ \AA}^3$	$0.32 \times 0.20 \times 0.18 \text{ mm}$
$Z = 4$	

Data collection

Bruker X8 APEXII diffractometer	2130 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.042$
graphite	$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.4^\circ$
φ and ω scans	$h = -9 \rightarrow 9$
20993 measured reflections	$k = -13 \rightarrow 13$
2719 independent reflections	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.108$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.280P]$
2719 reflections	where $P = (F_o^2 + 2F_c^2)/3$
158 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.16162 (14)	0.16773 (10)	0.73262 (7)	0.0395 (3)
O2	0.56946 (14)	0.16400 (10)	0.59503 (7)	0.0400 (3)
N1	0.25294 (15)	0.37740 (11)	0.72907 (7)	0.0288 (3)
N2	0.54245 (14)	0.37284 (11)	0.63540 (7)	0.0275 (2)

C1	0.29905 (16)	0.49020 (12)	0.68579 (8)	0.0253 (3)
C2	0.20477 (18)	0.60726 (13)	0.68935 (9)	0.0307 (3)
H2	0.1123	0.6096	0.7193	0.037*
C3	0.24611 (19)	0.71969 (14)	0.64933 (9)	0.0327 (3)
H3	0.1835	0.7974	0.6535	0.039*
C4	0.38090 (19)	0.71718 (13)	0.60286 (9)	0.0328 (3)
H4	0.4079	0.7924	0.5750	0.039*
C5	0.47450 (18)	0.60146 (13)	0.59851 (9)	0.0303 (3)
H5	0.5643	0.5994	0.5670	0.036*
C6	0.43742 (16)	0.48743 (12)	0.64028 (8)	0.0250 (3)
C7	0.2047 (2)	0.39958 (15)	0.81279 (9)	0.0351 (3)
H7A	0.2283	0.3211	0.8472	0.053*
H7B	0.0771	0.4217	0.8027	0.053*
H7C	0.2771	0.4704	0.8430	0.053*
C8	0.22299 (17)	0.25474 (13)	0.69423 (9)	0.0289 (3)
C9	0.26599 (18)	0.22616 (13)	0.60544 (9)	0.0313 (3)
C10	0.47116 (18)	0.25147 (13)	0.61116 (8)	0.0288 (3)
C11	0.73932 (18)	0.39067 (15)	0.64072 (10)	0.0350 (3)
H11A	0.8050	0.3139	0.6661	0.052*
H11B	0.7832	0.4660	0.6760	0.052*
H11C	0.7582	0.4038	0.5836	0.052*
C12	0.13980 (19)	0.30489 (16)	0.53176 (9)	0.0371 (3)
H12A	0.1699	0.2846	0.4777	0.056*
H12B	0.1566	0.3973	0.5433	0.056*
H12C	0.0144	0.2818	0.5284	0.056*
C13	0.2302 (2)	0.07970 (15)	0.58570 (12)	0.0487 (4)
H13A	0.3049	0.0282	0.6313	0.073*
H13B	0.2599	0.0586	0.5317	0.073*
H13C	0.1033	0.0606	0.5817	0.073*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0423 (6)	0.0378 (6)	0.0421 (6)	-0.0102 (4)	0.0170 (5)	0.0065 (4)
O2	0.0431 (6)	0.0331 (5)	0.0469 (6)	0.0082 (4)	0.0170 (5)	-0.0008 (4)
N1	0.0306 (6)	0.0327 (6)	0.0261 (5)	-0.0038 (4)	0.0128 (5)	0.0013 (4)
N2	0.0234 (5)	0.0299 (6)	0.0312 (6)	0.0015 (4)	0.0104 (4)	0.0019 (4)
C1	0.0250 (6)	0.0291 (6)	0.0226 (6)	-0.0028 (5)	0.0071 (5)	0.0006 (5)
C2	0.0272 (6)	0.0367 (7)	0.0305 (7)	0.0017 (5)	0.0111 (5)	-0.0016 (5)
C3	0.0329 (7)	0.0306 (7)	0.0347 (7)	0.0053 (5)	0.0079 (6)	-0.0004 (5)
C4	0.0375 (7)	0.0294 (7)	0.0323 (7)	-0.0014 (5)	0.0096 (6)	0.0042 (5)
C5	0.0298 (6)	0.0344 (7)	0.0299 (6)	-0.0017 (5)	0.0134 (5)	0.0027 (5)
C6	0.0232 (6)	0.0285 (6)	0.0237 (6)	-0.0003 (5)	0.0064 (5)	-0.0002 (5)
C7	0.0373 (7)	0.0449 (8)	0.0271 (7)	-0.0061 (6)	0.0156 (6)	-0.0004 (6)
C8	0.0243 (6)	0.0331 (7)	0.0300 (6)	-0.0028 (5)	0.0077 (5)	0.0030 (5)
C9	0.0333 (7)	0.0306 (7)	0.0313 (7)	-0.0053 (5)	0.0106 (5)	-0.0029 (5)
C10	0.0326 (7)	0.0302 (7)	0.0252 (6)	0.0036 (5)	0.0102 (5)	0.0039 (5)
C11	0.0241 (6)	0.0429 (8)	0.0401 (8)	0.0028 (5)	0.0119 (6)	0.0001 (6)

supplementary materials

C12	0.0315 (7)	0.0512 (9)	0.0277 (7)	-0.0038 (6)	0.0053 (6)	-0.0045 (6)
C13	0.0576 (10)	0.0360 (8)	0.0571 (10)	-0.0134 (7)	0.0227 (8)	-0.0103 (7)

Geometric parameters (Å, °)

O1—C8	1.2248 (15)	C5—H5	0.9300
O2—C10	1.2214 (15)	C7—H7A	0.9600
N1—C8	1.3619 (17)	C7—H7B	0.9600
N1—C1	1.4217 (15)	C7—H7C	0.9600
N1—C7	1.4749 (16)	C8—C9	1.5457 (18)
N2—C10	1.3645 (17)	C9—C13	1.533 (2)
N2—C6	1.4199 (16)	C9—C10	1.5438 (18)
N2—C11	1.4722 (16)	C9—C12	1.544 (2)
C1—C2	1.3934 (18)	C11—H11A	0.9600
C1—C6	1.3992 (16)	C11—H11B	0.9600
C2—C3	1.3788 (19)	C11—H11C	0.9600
C2—H2	0.9300	C12—H12A	0.9600
C3—C4	1.3866 (19)	C12—H12B	0.9600
C3—H3	0.9300	C12—H12C	0.9600
C4—C5	1.3813 (19)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—C6	1.3964 (17)	C13—H13C	0.9600
C8—N1—C1	125.39 (10)	O1—C8—N1	120.38 (12)
C8—N1—C7	116.99 (11)	O1—C8—C9	120.12 (12)
C1—N1—C7	116.84 (11)	N1—C8—C9	119.50 (11)
C10—N2—C6	124.90 (11)	C13—C9—C10	107.37 (12)
C10—N2—C11	116.72 (11)	C13—C9—C12	107.62 (12)
C6—N2—C11	117.33 (11)	C10—C9—C12	112.55 (11)
C2—C1—C6	118.99 (11)	C13—C9—C8	107.74 (11)
C2—C1—N1	119.09 (10)	C10—C9—C8	109.63 (11)
C6—C1—N1	121.92 (11)	C12—C9—C8	111.70 (11)
C3—C2—C1	121.22 (12)	O2—C10—N2	120.12 (12)
C3—C2—H2	119.4	O2—C10—C9	120.71 (12)
C1—C2—H2	119.4	N2—C10—C9	119.16 (11)
C2—C3—C4	120.13 (13)	N2—C11—H11A	109.5
C2—C3—H3	119.9	N2—C11—H11B	109.5
C4—C3—H3	119.9	H11A—C11—H11B	109.5
C5—C4—C3	119.11 (12)	N2—C11—H11C	109.5
C5—C4—H4	120.4	H11A—C11—H11C	109.5
C3—C4—H4	120.4	H11B—C11—H11C	109.5
C4—C5—C6	121.56 (12)	C9—C12—H12A	109.5
C4—C5—H5	119.2	C9—C12—H12B	109.5
C6—C5—H5	119.2	H12A—C12—H12B	109.5
C5—C6—C1	118.96 (11)	C9—C12—H12C	109.5
C5—C6—N2	118.71 (11)	H12A—C12—H12C	109.5
C1—C6—N2	122.33 (11)	H12B—C12—H12C	109.5
N1—C7—H7A	109.5	C9—C13—H13A	109.5
N1—C7—H7B	109.5	C9—C13—H13B	109.5
H7A—C7—H7B	109.5	H13A—C13—H13B	109.5

N1—C7—H7C	109.5	C9—C13—H13C	109.5
H7A—C7—H7C	109.5	H13A—C13—H13C	109.5
H7B—C7—H7C	109.5	H13B—C13—H13C	109.5
C8—N1—C1—C2	132.49 (13)	C7—N1—C8—O1	-1.24 (19)
C7—N1—C1—C2	-37.06 (17)	C1—N1—C8—C9	8.88 (19)
C8—N1—C1—C6	-48.03 (18)	C7—N1—C8—C9	178.41 (12)
C7—N1—C1—C6	142.42 (12)	O1—C8—C9—C13	-3.72 (18)
C6—C1—C2—C3	-0.31 (19)	N1—C8—C9—C13	176.63 (13)
N1—C1—C2—C3	179.18 (12)	O1—C8—C9—C10	-120.26 (13)
C1—C2—C3—C4	1.4 (2)	N1—C8—C9—C10	60.09 (16)
C2—C3—C4—C5	-1.0 (2)	O1—C8—C9—C12	114.27 (14)
C3—C4—C5—C6	-0.5 (2)	N1—C8—C9—C12	-65.38 (16)
C4—C5—C6—C1	1.5 (2)	C6—N2—C10—O2	167.89 (12)
C4—C5—C6—N2	-178.11 (12)	C11—N2—C10—O2	-0.06 (18)
C2—C1—C6—C5	-1.14 (18)	C6—N2—C10—C9	-12.78 (18)
N1—C1—C6—C5	179.38 (12)	C11—N2—C10—C9	179.28 (11)
C2—C1—C6—N2	178.50 (12)	C13—C9—C10—O2	4.75 (18)
N1—C1—C6—N2	-0.98 (18)	C12—C9—C10—O2	-113.51 (14)
C10—N2—C6—C5	-128.94 (13)	C8—C9—C10—O2	121.52 (13)
C11—N2—C6—C5	38.94 (17)	C13—C9—C10—N2	-174.59 (12)
C10—N2—C6—C1	51.42 (18)	C12—C9—C10—N2	67.16 (16)
C11—N2—C6—C1	-140.70 (12)	C8—C9—C10—N2	-57.81 (15)
C1—N1—C8—O1	-170.77 (12)		

Fig. 1

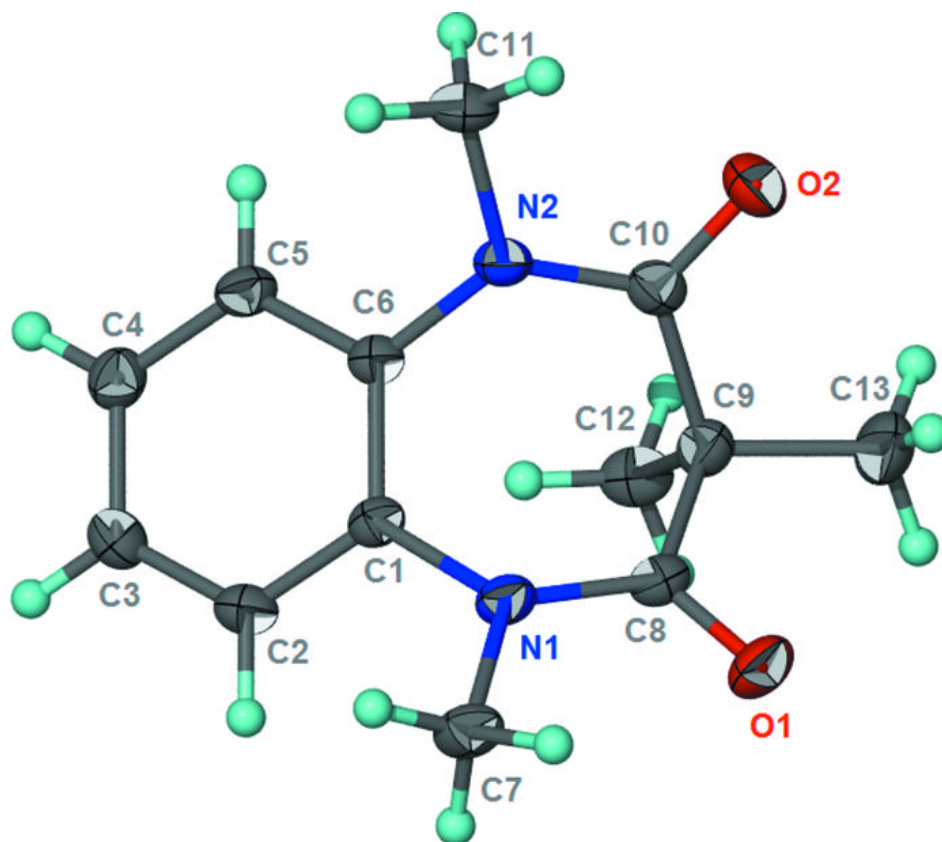


Fig. 2

