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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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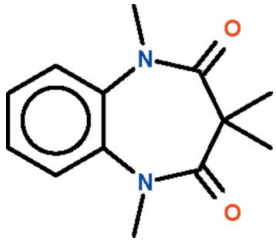
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.108; data-to-parameter ratio = 17.2.

The seven-membered ring in the title compound, $C_{13}H_{16}N_2O_2$, 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

$C_{13}H_{16}N_2O_2$
M_r = 232.28
Monoclinic, $P2_1/c$
a = 7.5112 (1) Å
b = 10.1731 (2) Å
c = 15.8697 (3) Å
β = 103.675 (1)°
V = 1178.26 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.32 × 0.20 × 0.18 mm

Data collection

Bruker X8 APEXII diffractometer
20993 measured reflections
2719 independent reflections
2130 reflections with $I > 2\sigma(I)$
R_{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
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −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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supporting information

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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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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 haloalkane to form 3-substituted derivatives. Previous studies have largely described the mono-substituted derivatives only. 1,12-Dibromo-docane 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).

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 methyl iodide (0.68 g, 4.80 mmol). Stirring was continued for 24 h. The reaction was monitored by thin layer ch romatography. 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 0.93–0.96 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C).

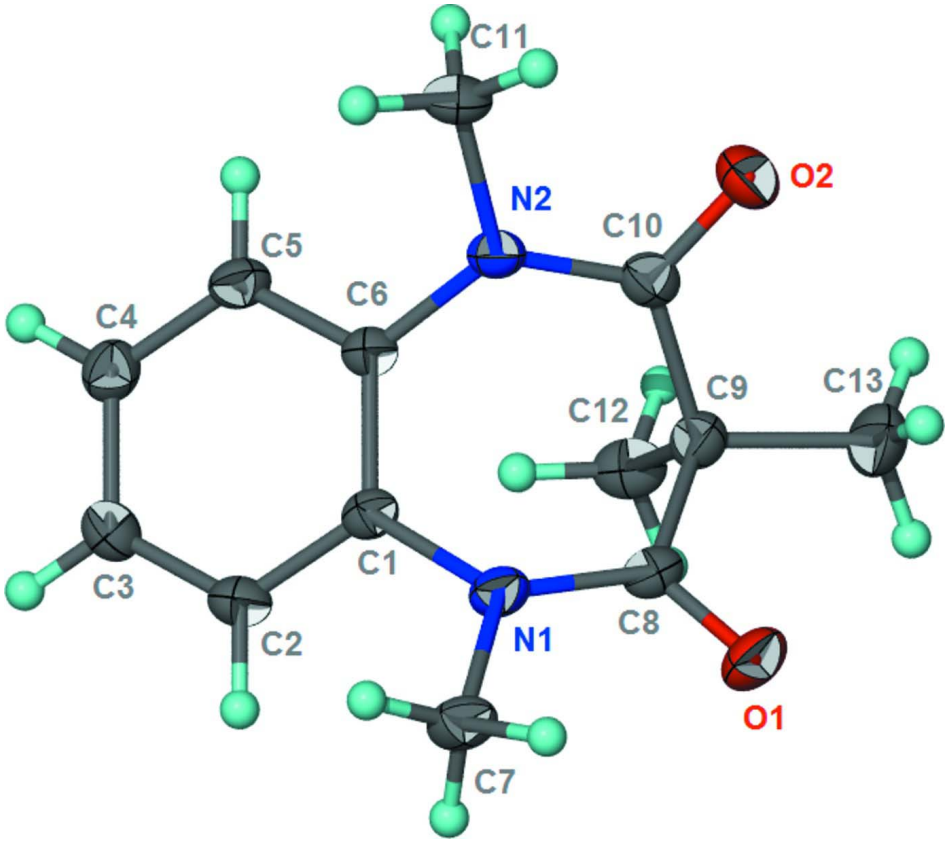

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{13}H_{16}N_2O_2$ at the 50% probability level; hydrogen atoms are drawn as arbitrary radius.

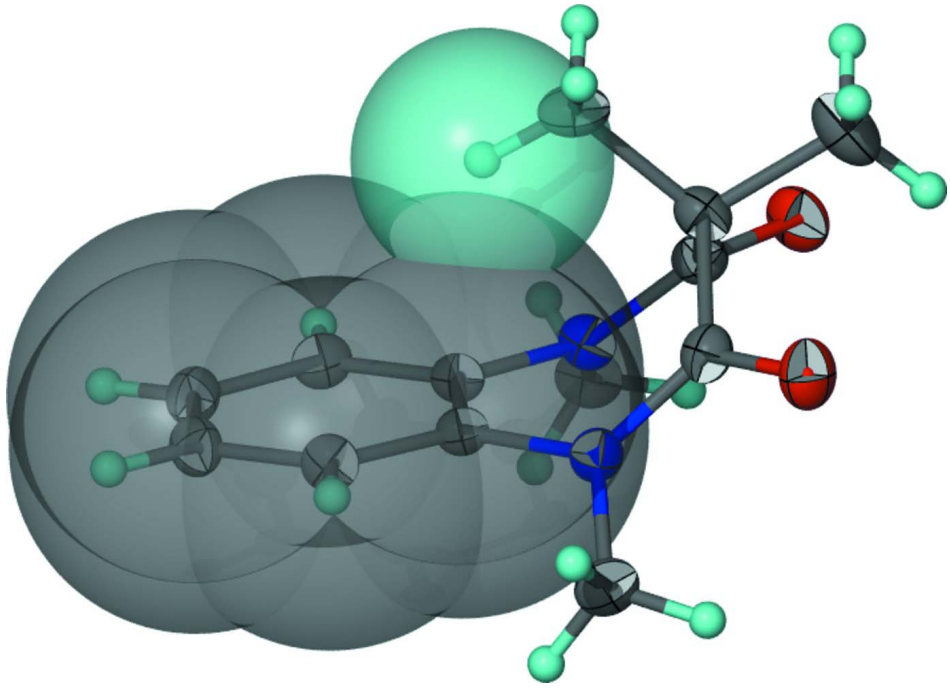

Figure 2

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

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

Data collection

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

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.04$
2719 reflections
158 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
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.280P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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)
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, º)

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)		