Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Allyl-1,5-dibenzyl-1,5-benzodiazepine-2,4-dione

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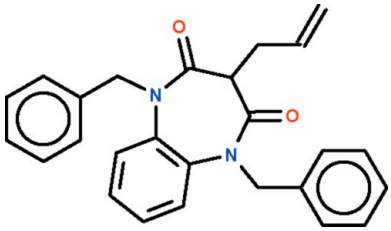
Received 12 November 2009; accepted 15 November 2009

Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.117; data-to-parameter ratio = 17.8.

The title compound, $C_{26}H_{24}N_2O_2$, features a benzene ring fused with a seven-membered diazepine ring; the latter ring adopts a boat conformation (with the allyldimethylamino-methyl-bearing C atom as the prow and the fused-ring C atoms as the stern).

Related literature

For the crystal structure of benzodiazepin-2,4-dione, see: Négrier *et al.* (2006).



Experimental

Crystal data

$C_{26}H_{24}N_2O_2$
M_r = 396.47
Monoclinic, $P2_1/c$
a = 9.2603 (4) Å
b = 14.0037 (6) Å
c = 16.2249 (7) Å
β = 91.996 (1)°
V = 2102.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 193 K
0.40 × 0.40 × 0.07 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
27620 measured reflections
4820 independent reflections
2934 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.117
S = 1.00
4820 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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, 2009).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2680).

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supporting information

Acta Cryst. (2009). E**65**, o3150 [doi:10.1107/S160053680904851X]

3-Allyl-1,5-dibenzyl-1,5-benzodiazepine-2,4-dione

Hind Jabli, Y. Kandri Rodi, Natalie Saffon, El Mokhtar Essassi and Seik Weng Ng

S1. Experimental

To a solution of the potassium *t*-butoxide (0.24 g, 2.13 mmol) in THF (15 ml) was added 1,5-dibenzyl-1,5-benzodiazepine-2,4-dione (0.50 g, 1.40 mmol) and allyl bromide (0.20 ml, 1.88 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.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 1.00 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U(C).

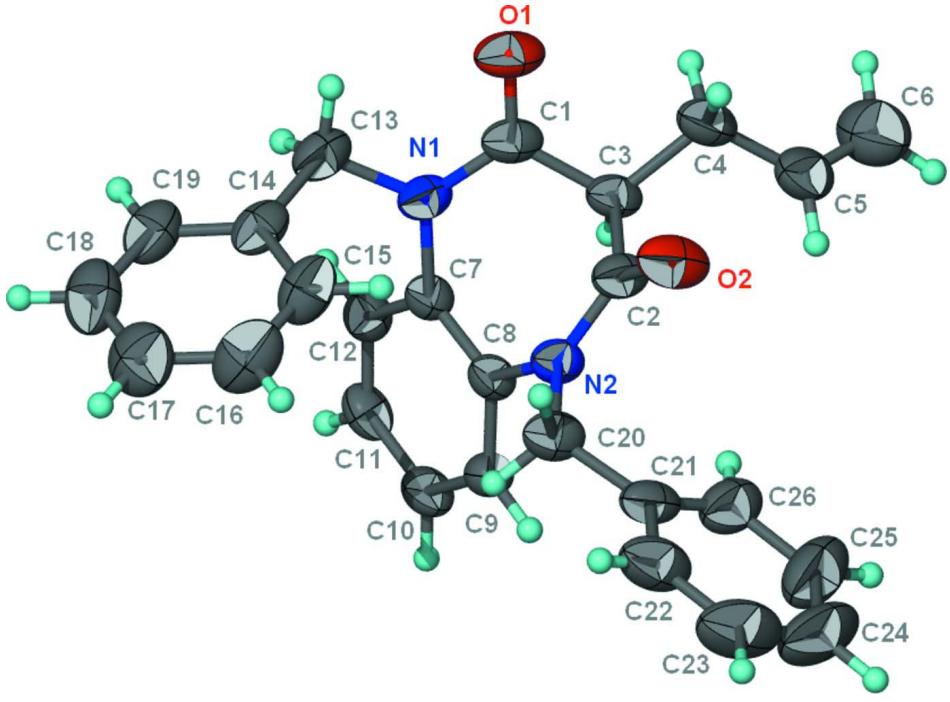

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{26}H_{24}N_2O_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

3-Allyl-1,5-dibenzyl-1,5-benzodiazepine-2,4-dione

Crystal data

$C_{26}H_{24}N_2O_2$
$M_r = 396.47$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.2603$ (4) Å
$b = 14.0037$ (6) Å
$c = 16.2249$ (7) Å
$\beta = 91.996$ (1)°
$V = 2102.7$ (2) Å^3
$Z = 4$
$F(000) = 840$
$D_x = 1.252$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4269 reflections
$\theta = 2.5$–25.0°
$\mu = 0.08$ mm^{-1}
$T = 193$ K
Plate, colorless
0.40 × 0.40 × 0.07 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
27620 measured reflections
4820 independent reflections
2934 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.9°$
$h = -11 \rightarrow 12$
$k = -18 \rightarrow 18$
$l = -21 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.117$
$S = 1.00$
4820 reflections
271 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
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.414P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.49018 (14)	0.26136 (9)	0.33899 (7)	0.0470 (3)
O2	0.33507 (16)	0.40157 (8)	0.16934 (7)	0.0498 (4)
N1	0.50477 (15)	0.15481 (9)	0.23332 (8)	0.0330 (3)
N2	0.37563 (15)	0.26484 (9)	0.09974 (7)	0.0320 (3)
C1	0.43555 (19)	0.22301 (11)	0.27747 (10)	0.0348 (4)
C2	0.33193 (19)	0.31439 (11)	0.16711 (9)	0.0350 (4)
C3	0.28998 (18)	0.25375 (11)	0.24091 (9)	0.0336 (4)
H3	0.2349	0.1963	0.2210	0.040*
C4	0.2006 (2)	0.30886 (12)	0.30184 (10)	0.0409 (4)
H4A	0.1979	0.2725	0.3541	0.049*
H4B	0.2479	0.3709	0.3139	0.049*
C5	0.0492 (2)	0.32639 (13)	0.27024 (11)	0.0485 (5)
H5	0.0365	0.3419	0.2135	0.058*
C6	−0.0667 (3)	0.32235 (16)	0.31310 (14)	0.0658 (6)

H6A	−0.0595	0.3071	0.3701	0.079*
H6B	−0.1585	0.3347	0.2874	0.079*
C7	0.43855 (17)	0.10939 (11)	0.16252 (9)	0.0294 (3)
C8	0.37894 (16)	0.16300 (10)	0.09694 (9)	0.0285 (3)
C9	0.32362 (17)	0.11561 (12)	0.02736 (9)	0.0332 (4)
H9	0.2852	0.1515	−0.0180	0.040*
C10	0.32381 (18)	0.01744 (12)	0.02331 (10)	0.0371 (4)
H10	0.2853	−0.0140	−0.0245	0.045*
C11	0.38017 (18)	−0.03554 (12)	0.08892 (10)	0.0361 (4)
H11	0.3788	−0.1033	0.0866	0.043*
C12	0.43813 (17)	0.01011 (11)	0.15748 (10)	0.0331 (4)
H12	0.4784	−0.0265	0.2019	0.040*
C13	0.65246 (19)	0.12564 (13)	0.25940 (10)	0.0407 (4)
H13A	0.6512	0.0580	0.2772	0.049*
H13B	0.6853	0.1648	0.3073	0.049*
C14	0.75779 (18)	0.13696 (12)	0.19117 (10)	0.0382 (4)
C15	0.7462 (2)	0.21035 (14)	0.13427 (12)	0.0483 (5)
H15	0.6699	0.2554	0.1376	0.058*
C16	0.8450 (2)	0.21874 (16)	0.07231 (13)	0.0603 (6)
H16	0.8353	0.2689	0.0331	0.072*
C17	0.9570 (2)	0.15453 (16)	0.06759 (14)	0.0606 (6)
H17	1.0243	0.1602	0.0250	0.073*
C18	0.9714 (2)	0.08237 (15)	0.12436 (14)	0.0564 (5)
H18	1.0498	0.0389	0.1218	0.068*
C19	0.8714 (2)	0.07300 (13)	0.18536 (12)	0.0459 (5)
H19	0.8808	0.0220	0.2238	0.055*
C20	0.4289 (2)	0.32016 (12)	0.02996 (10)	0.0380 (4)
H20A	0.4780	0.2757	−0.0073	0.046*
H20B	0.5023	0.3659	0.0516	0.046*
C21	0.3168 (2)	0.37497 (12)	−0.02019 (9)	0.0385 (4)
C22	0.3657 (3)	0.43780 (13)	−0.07970 (11)	0.0553 (5)
H22	0.4664	0.4466	−0.0858	0.066*
C23	0.2682 (4)	0.48731 (17)	−0.12977 (13)	0.0764 (8)
H23	0.3021	0.5301	−0.1702	0.092*
C24	0.1224 (4)	0.47491 (18)	−0.12138 (15)	0.0839 (9)
H24	0.0558	0.5084	−0.1566	0.101*
C25	0.0723 (3)	0.41385 (16)	−0.06193 (15)	0.0704 (7)
H25	−0.0286	0.4058	−0.0557	0.084*
C26	0.1706 (2)	0.36411 (13)	−0.01120 (11)	0.0487 (5)
H26	0.1363	0.3224	0.0299	0.058*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0684 (9)	0.0398 (7)	0.0320 (6)	−0.0053 (6)	−0.0109 (6)	−0.0058 (5)
O2	0.0876 (10)	0.0251 (7)	0.0365 (7)	−0.0007 (6)	0.0008 (7)	−0.0003 (5)
N1	0.0420 (8)	0.0286 (7)	0.0280 (7)	−0.0001 (6)	−0.0064 (6)	0.0014 (6)
N2	0.0451 (8)	0.0256 (7)	0.0254 (7)	0.0015 (6)	0.0000 (6)	0.0024 (5)

C1	0.0534 (11)	0.0253 (8)	0.0255 (8)	−0.0046 (8)	−0.0026 (7)	0.0035 (7)
C2	0.0494 (11)	0.0275 (9)	0.0277 (8)	0.0007 (8)	−0.0044 (7)	0.0003 (7)
C3	0.0496 (10)	0.0258 (8)	0.0251 (8)	0.0005 (7)	−0.0003 (7)	−0.0025 (6)
C4	0.0616 (12)	0.0328 (9)	0.0283 (9)	0.0048 (9)	0.0038 (8)	−0.0028 (7)
C5	0.0664 (14)	0.0411 (11)	0.0381 (10)	0.0129 (10)	0.0013 (9)	−0.0056 (8)
C6	0.0641 (15)	0.0742 (16)	0.0593 (13)	−0.0053 (12)	0.0039 (11)	−0.0084 (12)
C7	0.0322 (9)	0.0282 (8)	0.0277 (8)	−0.0006 (7)	0.0018 (7)	−0.0014 (6)
C8	0.0338 (9)	0.0250 (8)	0.0268 (8)	0.0005 (7)	0.0042 (7)	−0.0010 (6)
C9	0.0353 (9)	0.0374 (10)	0.0267 (8)	0.0040 (7)	−0.0012 (7)	−0.0031 (7)
C10	0.0361 (10)	0.0390 (10)	0.0363 (9)	−0.0002 (8)	0.0008 (7)	−0.0131 (8)
C11	0.0394 (10)	0.0270 (8)	0.0425 (10)	0.0000 (7)	0.0090 (8)	−0.0064 (7)
C12	0.0365 (9)	0.0287 (9)	0.0343 (9)	0.0029 (7)	0.0040 (7)	0.0029 (7)
C13	0.0466 (11)	0.0368 (10)	0.0379 (9)	0.0003 (8)	−0.0112 (8)	0.0053 (8)
C14	0.0378 (10)	0.0328 (9)	0.0430 (10)	−0.0051 (8)	−0.0135 (8)	0.0008 (8)
C15	0.0398 (11)	0.0435 (11)	0.0613 (12)	−0.0011 (9)	−0.0048 (9)	0.0144 (9)
C16	0.0472 (12)	0.0683 (15)	0.0654 (14)	−0.0086 (11)	0.0005 (10)	0.0253 (11)
C17	0.0401 (12)	0.0711 (15)	0.0707 (14)	−0.0050 (11)	0.0047 (10)	0.0036 (12)
C18	0.0400 (11)	0.0509 (13)	0.0779 (15)	0.0021 (9)	−0.0041 (11)	−0.0037 (11)
C19	0.0426 (11)	0.0368 (10)	0.0572 (12)	−0.0012 (9)	−0.0127 (9)	0.0023 (9)
C20	0.0502 (11)	0.0339 (9)	0.0302 (8)	−0.0051 (8)	0.0038 (8)	0.0057 (7)
C21	0.0631 (13)	0.0280 (9)	0.0243 (8)	0.0036 (8)	−0.0005 (8)	−0.0006 (7)
C22	0.0937 (16)	0.0383 (11)	0.0345 (10)	0.0081 (11)	0.0103 (10)	0.0069 (8)
C23	0.141 (3)	0.0484 (14)	0.0396 (12)	0.0187 (16)	0.0015 (14)	0.0142 (10)
C24	0.132 (3)	0.0580 (16)	0.0587 (15)	0.0313 (17)	−0.0366 (17)	0.0071 (12)
C25	0.0779 (16)	0.0554 (14)	0.0756 (16)	0.0177 (12)	−0.0285 (13)	−0.0023 (12)
C26	0.0633 (14)	0.0394 (11)	0.0425 (10)	0.0056 (10)	−0.0105 (9)	0.0016 (8)

Geometric parameters (Å, º)

O1—C1	1.2271 (19)	C13—C14	1.509 (2)
O2—C2	1.2216 (19)	C13—H13A	0.9900
N1—C1	1.367 (2)	C13—H13B	0.9900
N1—C7	1.4321 (19)	C14—C15	1.383 (2)
N1—C13	1.475 (2)	C14—C19	1.388 (2)
N2—C2	1.3678 (19)	C15—C16	1.388 (3)
N2—C8	1.4273 (19)	C15—H15	0.9500
N2—C20	1.4710 (19)	C16—C17	1.377 (3)
C1—C3	1.516 (2)	C16—H16	0.9500
C2—C3	1.529 (2)	C17—C18	1.371 (3)
C3—C4	1.521 (2)	C17—H17	0.9500
C3—H3	1.0000	C18—C19	1.385 (3)
C4—C5	1.496 (3)	C18—H18	0.9500
C4—H4A	0.9900	C19—H19	0.9500
C4—H4B	0.9900	C20—C21	1.506 (2)
C5—C6	1.300 (3)	C20—H20A	0.9900
C5—H5	0.9500	C20—H20B	0.9900
C6—H6A	0.9500	C21—C26	1.376 (3)
C6—H6B	0.9500	C21—C22	1.393 (2)

C7—C12	1.393 (2)	C22—C23	1.380 (3)
C7—C8	1.400 (2)	C22—H22	0.9500
C8—C9	1.392 (2)	C23—C24	1.372 (4)
C9—C10	1.376 (2)	C23—H23	0.9500
C9—H9	0.9500	C24—C25	1.381 (4)
C10—C11	1.385 (2)	C24—H24	0.9500
C10—H10	0.9500	C25—C26	1.392 (3)
C11—C12	1.376 (2)	C25—H25	0.9500
C11—H11	0.9500	C26—H26	0.9500
C12—H12	0.9500		

C1—N1—C7	122.36 (14)	N1—C13—C14	112.15 (13)
C1—N1—C13	119.35 (13)	N1—C13—H13A	109.2
C7—N1—C13	118.29 (13)	C14—C13—H13A	109.2
C2—N2—C8	122.66 (13)	N1—C13—H13B	109.2
C2—N2—C20	117.67 (13)	C14—C13—H13B	109.2
C8—N2—C20	119.58 (12)	H13A—C13—H13B	107.9
O1—C1—N1	123.00 (16)	C15—C14—C19	118.41 (17)
O1—C1—C3	121.99 (15)	C15—C14—C13	122.04 (16)
N1—C1—C3	114.85 (13)	C19—C14—C13	119.55 (16)
O2—C2—N2	121.55 (15)	C14—C15—C16	120.56 (18)
O2—C2—C3	122.56 (14)	C14—C15—H15	119.7
N2—C2—C3	115.74 (13)	C16—C15—H15	119.7
C1—C3—C4	112.72 (13)	C17—C16—C15	120.13 (19)
C1—C3—C2	102.56 (13)	C17—C16—H16	119.9
C4—C3—C2	112.70 (13)	C15—C16—H16	119.9
C1—C3—H3	109.5	C18—C17—C16	120.0 (2)
C4—C3—H3	109.5	C18—C17—H17	120.0
C2—C3—H3	109.5	C16—C17—H17	120.0
C5—C4—C3	112.65 (14)	C17—C18—C19	119.90 (19)
C5—C4—H4A	109.1	C17—C18—H18	120.1
C3—C4—H4A	109.1	C19—C18—H18	120.1
C5—C4—H4B	109.1	C18—C19—C14	120.98 (18)
C3—C4—H4B	109.1	C18—C19—H19	119.5
H4A—C4—H4B	107.8	C14—C19—H19	119.5
C6—C5—C4	126.20 (19)	N2—C20—C21	116.17 (14)
C6—C5—H5	116.9	N2—C20—H20A	108.2
C4—C5—H5	116.9	C21—C20—H20A	108.2
C5—C6—H6A	120.0	N2—C20—H20B	108.2
C5—C6—H6B	120.0	C21—C20—H20B	108.2
H6A—C6—H6B	120.0	H20A—C20—H20B	107.4
C12—C7—C8	119.37 (14)	C26—C21—C22	119.19 (18)
C12—C7—N1	119.39 (14)	C26—C21—C20	123.27 (15)
C8—C7—N1	121.20 (14)	C22—C21—C20	117.52 (18)
C9—C8—C7	119.02 (14)	C23—C22—C21	120.2 (2)
C9—C8—N2	119.64 (14)	C23—C22—H22	119.9
C7—C8—N2	121.34 (13)	C21—C22—H22	119.9
C10—C9—C8	120.93 (15)	C24—C23—C22	120.2 (2)

C10—C9—H9	119.5	C24—C23—H23	119.9
C8—C9—H9	119.5	C22—C23—H23	119.9
C9—C10—C11	119.97 (15)	C23—C24—C25	120.2 (2)
C9—C10—H10	120.0	C23—C24—H24	119.9
C11—C10—H10	120.0	C25—C24—H24	119.9
C12—C11—C10	119.91 (15)	C24—C25—C26	119.6 (3)
C12—C11—H11	120.0	C24—C25—H25	120.2
C10—C11—H11	120.0	C26—C25—H25	120.2
C11—C12—C7	120.77 (15)	C21—C26—C25	120.5 (2)
C11—C12—H12	119.6	C21—C26—H26	119.8
C7—C12—H12	119.6	C25—C26—H26	119.8

C7—N1—C1—O1	178.80 (15)	C7—C8—C9—C10	1.6 (2)
C13—N1—C1—O1	−0.6 (2)	N2—C8—C9—C10	−179.23 (15)
C7—N1—C1—C3	−5.8 (2)	C8—C9—C10—C11	−0.3 (2)
C13—N1—C1—C3	174.81 (13)	C9—C10—C11—C12	−1.1 (2)
C8—N2—C2—O2	177.22 (16)	C10—C11—C12—C7	1.2 (2)
C20—N2—C2—O2	0.8 (2)	C8—C7—C12—C11	0.1 (2)
C8—N2—C2—C3	1.5 (2)	N1—C7—C12—C11	−177.57 (14)
C20—N2—C2—C3	−174.87 (14)	C1—N1—C13—C14	−124.15 (16)
O1—C1—C3—C4	−19.6 (2)	C7—N1—C13—C14	56.45 (19)
N1—C1—C3—C4	164.96 (14)	N1—C13—C14—C15	34.5 (2)
O1—C1—C3—C2	101.85 (17)	N1—C13—C14—C19	−146.39 (15)
N1—C1—C3—C2	−73.59 (16)	C19—C14—C15—C16	0.8 (3)
O2—C2—C3—C1	−99.46 (19)	C13—C14—C15—C16	179.95 (18)
N2—C2—C3—C1	76.21 (17)	C14—C15—C16—C17	−0.8 (3)
O2—C2—C3—C4	22.0 (2)	C15—C16—C17—C18	−0.3 (3)
N2—C2—C3—C4	−162.32 (14)	C16—C17—C18—C19	1.4 (3)
C1—C3—C4—C5	−171.39 (14)	C17—C18—C19—C14	−1.3 (3)
C2—C3—C4—C5	73.10 (19)	C15—C14—C19—C18	0.2 (3)
C3—C4—C5—C6	140.7 (2)	C13—C14—C19—C18	−178.88 (17)
C1—N1—C7—C12	−129.21 (16)	C2—N2—C20—C21	−71.74 (19)
C13—N1—C7—C12	50.2 (2)	C8—N2—C20—C21	111.77 (17)
C1—N1—C7—C8	53.2 (2)	N2—C20—C21—C26	−9.8 (2)
C13—N1—C7—C8	−127.46 (16)	N2—C20—C21—C22	171.75 (15)
C12—C7—C8—C9	−1.5 (2)	C26—C21—C22—C23	−1.0 (3)
N1—C7—C8—C9	176.16 (14)	C20—C21—C22—C23	177.51 (17)
C12—C7—C8—N2	179.35 (14)	C21—C22—C23—C24	−0.1 (3)
N1—C7—C8—N2	−3.0 (2)	C22—C23—C24—C25	1.0 (4)
C2—N2—C8—C9	134.46 (16)	C23—C24—C25—C26	−0.8 (4)
C20—N2—C8—C9	−49.2 (2)	C22—C21—C26—C25	1.2 (3)
C2—N2—C8—C7	−46.4 (2)	C20—C21—C26—C25	−177.19 (17)
C20—N2—C8—C7	129.94 (16)	C24—C25—C26—C21	−0.4 (3)