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ISSN 2414-3146

2,4,6-Triphenyl-*N*-{(3*E*)-3-[(2,4,6-triphenylphenyl)-imino]butan-2-ylidene}aniline

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Received 13 April 2020
Accepted 16 April 2020

Edited by E. R. T. Tiekink, Sunway University, Malaysia

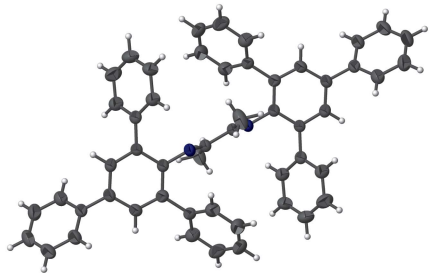
Keywords: crystal structure; C_i symmetry; 1,4-diazabutadiene.

CCDC reference: 1949863

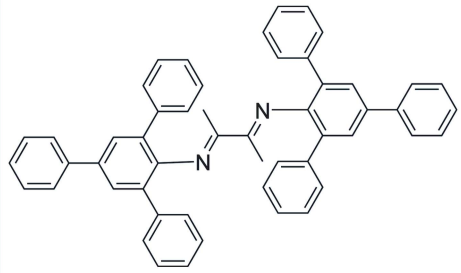
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{52}H_{40}N_2$, is disposed about a centre of inversion and the conformation about the imine bond [1.268 (3) Å] is *E*. The terminal benzene ring is approximately perpendicular to the central 1,4-diazabutadiene mean plane, forming a dihedral angle of 81.2 (3)°. Weak C—H···π and π–π [inter-centroid distance = 4.021 (5) Å] interactions help to consolidate the packing.

3D view



Chemical scheme



Structure description

The seminal studies by Brookhart and co-workers leading to the discovery of cationic α-diimine-based Ni and Pd catalysts marked the start of a new era in olefin polymerization studies (Killian *et al.*, 1996). Branched polyolefins are generally produced using these catalysed ethylene polymerizations through a characteristic chain-walking process (Wang & Chen, 2019). More importantly, these α-diimine Ni and Pd catalysts are able to co-polymerize olefins with polar co-monomers to afford co-polymers containing functional groups without the pre-protection of the polar groups (Chen *et al.*, 2018). For the synthesis of the α-diimine molecules and background to the applications of the olefin polymerization catalysts ligated by α-diimine, see: Wang *et al.* (2016, 2018, 2019).

In this study, we designed and synthesized the title compound (Fig. 1) as a potential bidentate ligand, and its molecular structure was characterized by X-ray diffraction. In the solid state, the molecule exhibits C_i symmetry, being disposed about a centre of inversion. The single bond of the 1,4-diazabutadiene fragment [1.491 (4) Å] has an anti-disposition and the imine bonds [1.268 (3) Å] are *E*-configured. The dihedral angle between the pendent benzene ring and the 1,4-diazabutadiene least-squares plane is 81.2 (3)°, consistent with an almost perpendicular relationship. In the crystal, C—H···π, Table 1, and π–π interactions are noted. For the latter, the closest approach of 4.021 (5) Å

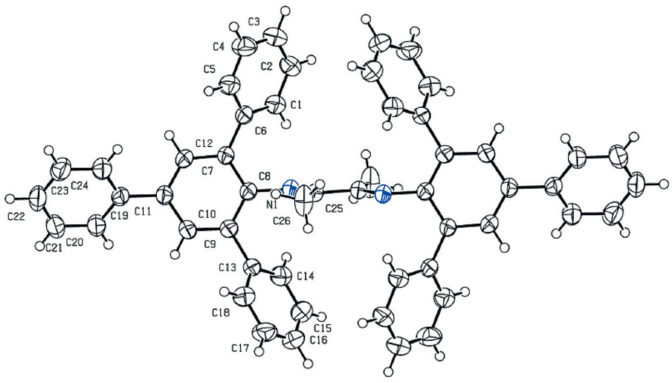


Figure 1
Molecular structure of the title compound showing the atom-labelling scheme and displacement ellipsoids at the 30% probability level. Unlabelled atoms are related by the symmetry operation $-x, 2-y, 1-z$.

occurs between centrosymmetrically related (C13–C18)-phenyl rings with the off-set distance being 1.86 Å; symmetry operation $-x, 1-y, 1-z$.

Synthesis and crystallization

After the protection of the amino group by acetic acid, the aniline was brominated. The Suzuki coupling reaction of the aniline and phenylboronic acid catalysed by a Pd catalyst in PEG-400 /H_2O led to the corresponding triphenyl-substituted aniline (Fig. 2). The title compound was prepared by the condensation of two equivalents of the appropriate aniline with one equivalent of 2,3-butanedione, in the presence of formic acid or *p*-toluenesulfonic acid, as a catalyst at 81% yield.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

This work was supported by the National Natural Science Foundation of China (grant No. 21801002), the Natural Science Foundation of Anhui Province (grant No. 1808085MB47) and the Postgraduate Research & Practice Innovation Program of Jiangsu Province (grant No. SJCX18–0953).

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

*Cg*1 is the centroid of the (C19–C24) phenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C17—H17···$Cg1^{i}$	0.93	2.89	3.737 (5)	152

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{52}H_{40}N_2$
M_r	692.86
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	296
a, b, c (Å)	6.383 (8), 12.498 (15), 12.814 (16)
α, β, γ (°)	68.718 (11), 86.988 (12), 81.397 (12)
V (Å^3)	942 (2)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.07
Crystal size (mm)	0.23 × 0.21 × 0.20
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 1996)
T_{min}, T_{max}	0.984, 0.986
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6847, 3456, 2023
R_{int}	0.049
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.060, 0.158, 1.02
No. of reflections	3456
No. of parameters	245
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.20

Computer programs: *APEX2* and *SAINT* (Bruker, 2002), *SHELXS97* (Sheldrick, 2015*a*), *SHELXL97* (Sheldrick, 2015*b*) and *SHELXTL* (Sheldrick, 2008).

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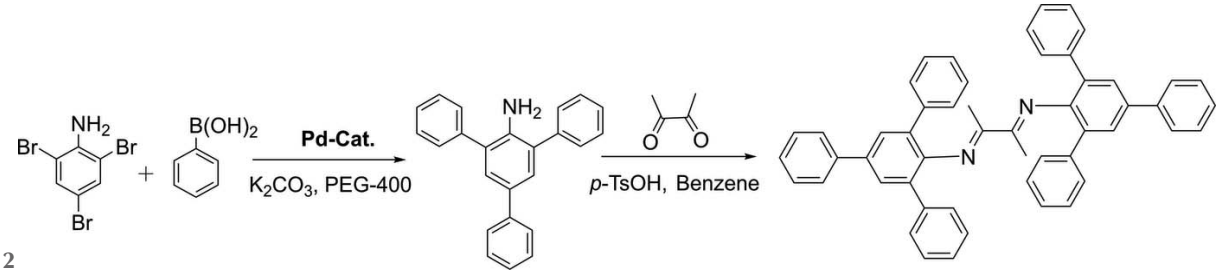

Figure 2
Reaction scheme.

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full crystallographic data

IUCrData (2020). **5**, x200531 [https://doi.org/10.1107/S2414314620005313]

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Crystal data

$C_{52}H_{40}N_2$
M_r = 692.86
Triclinic, $P\overline{1}$
a = 6.383 (8) Å
b = 12.498 (15) Å
c = 12.814 (16) Å
α = 68.718 (11)°
β = 86.988 (12)°
γ = 81.397 (12)°
V = 942 (2) Å^3
Z = 1
$F(000)$ = 366
D_x = 1.222 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 936 reflections
θ = 2.9–21.8°
μ = 0.07 mm^{-1}
T = 296 K
Block, yellow
0.23 × 0.21 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.984, T_{max} = 0.986
6847 measured reflections
3456 independent reflections
2023 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 25.5°, θ_{min} = 2.9°
h = −7→7
k = −15→15
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.158
S = 1.02
3456 reflections
245 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
$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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. All hydrogen atoms were placed in calculated positions with C—H distances of 0.93 and 0.96 Å for aryl and methyl type H-atoms. They were included in the refinement in a riding model approximation, respectively. The H-atoms were assigned *U*iso = 1.2 times *U*eq of the aryl C atoms and 1.5 times *U*eq of the methyl C atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1450 (4)	0.9506 (2)	0.8008 (2)	0.0489 (7)
H1	0.0143	0.9357	0.7848	0.059*
C2	0.1537 (5)	1.0422 (2)	0.8349 (2)	0.0596 (8)
H2	0.0291	1.0883	0.8417	0.071*
C3	0.3434 (5)	1.0660 (2)	0.8588 (2)	0.0632 (8)
H3	0.3483	1.1280	0.8821	0.076*
C4	0.5269 (5)	0.9980 (2)	0.8481 (2)	0.0616 (8)
H4	0.6569	1.0137	0.8641	0.074*
C5	0.5181 (4)	0.9065 (2)	0.8138 (2)	0.0500 (7)
H5	0.6435	0.8612	0.8065	0.060*
C6	0.3275 (4)	0.87991 (19)	0.78990 (18)	0.0401 (6)
C7	0.3258 (4)	0.77723 (18)	0.75853 (18)	0.0384 (6)
C8	0.1961 (4)	0.77693 (19)	0.67287 (18)	0.0382 (6)
C9	0.2085 (4)	0.67797 (19)	0.64472 (19)	0.0408 (6)
C10	0.3516 (4)	0.5816 (2)	0.7011 (2)	0.0448 (7)
H10	0.3601	0.5162	0.6817	0.054*
C11	0.4824 (4)	0.57853 (19)	0.78513 (19)	0.0422 (6)
C12	0.4641 (4)	0.6772 (2)	0.81216 (19)	0.0442 (6)
H12	0.5491	0.6763	0.8693	0.053*
C13	0.0786 (4)	0.67611 (19)	0.5518 (2)	0.0415 (6)
C14	−0.1360 (4)	0.7119 (2)	0.5430 (2)	0.0552 (8)
H14	−0.2056	0.7381	0.5966	0.066*
C15	−0.2493 (5)	0.7095 (2)	0.4553 (3)	0.0704 (10)
H15	−0.3942	0.7352	0.4498	0.085*
C16	−0.1511 (6)	0.6698 (2)	0.3769 (3)	0.0705 (10)
H16	−0.2285	0.6688	0.3179	0.085*
C17	0.0605 (6)	0.6317 (2)	0.3851 (2)	0.0648 (9)
H17	0.1276	0.6035	0.3321	0.078*
C18	0.1756 (5)	0.6345 (2)	0.4715 (2)	0.0539 (7)
H18	0.3203	0.6083	0.4764	0.065*
C19	0.6392 (4)	0.4760 (2)	0.84286 (19)	0.0424 (6)
C20	0.5946 (4)	0.3643 (2)	0.8704 (2)	0.0502 (7)
H20	0.4625	0.3527	0.8526	0.060*
C21	0.7426 (5)	0.2696 (2)	0.9238 (2)	0.0565 (8)
H21	0.7098	0.1949	0.9416	0.068*
C22	0.9375 (5)	0.2857 (2)	0.9504 (2)	0.0613 (8)
H22	1.0369	0.2219	0.9869	0.074*

C23	0.9862 (5)	0.3965 (3)	0.9231 (2)	0.0620 (8)
H23	1.1186	0.4076	0.9410	0.074*
C24	0.8376 (4)	0.4909 (2)	0.8691 (2)	0.0543 (7)
H24	0.8716	0.5654	0.8502	0.065*
C25	0.0839 (4)	0.94943 (18)	0.52404 (19)	0.0386 (6)
C26	0.2771 (5)	0.9412 (2)	0.4569 (2)	0.0703 (9)
H26A	0.3871	0.8873	0.5042	0.105*
H26B	0.3228	1.0161	0.4244	0.105*
H26C	0.2467	0.9148	0.3984	0.105*
N1	0.0470 (3)	0.87680 (15)	0.61974 (15)	0.0412 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0513 (18)	0.0484 (15)	0.0515 (16)	−0.0026 (14)	−0.0047 (13)	−0.0245 (13)
C2	0.062 (2)	0.0522 (17)	0.072 (2)	0.0061 (15)	−0.0097 (16)	−0.0359 (15)
C3	0.076 (2)	0.0555 (18)	0.071 (2)	−0.0078 (17)	−0.0073 (18)	−0.0380 (15)
C4	0.059 (2)	0.0664 (19)	0.073 (2)	−0.0183 (16)	−0.0041 (16)	−0.0367 (16)
C5	0.0514 (18)	0.0520 (16)	0.0505 (16)	−0.0042 (13)	−0.0021 (13)	−0.0238 (13)
C6	0.0505 (17)	0.0379 (13)	0.0292 (13)	−0.0020 (12)	−0.0051 (12)	−0.0096 (10)
C7	0.0475 (16)	0.0332 (13)	0.0324 (13)	−0.0002 (11)	−0.0063 (12)	−0.0104 (10)
C8	0.0453 (15)	0.0364 (13)	0.0309 (13)	−0.0007 (11)	−0.0049 (11)	−0.0112 (10)
C9	0.0497 (16)	0.0380 (13)	0.0349 (13)	−0.0042 (12)	−0.0040 (12)	−0.0136 (11)
C10	0.0569 (17)	0.0335 (13)	0.0420 (15)	0.0005 (12)	−0.0070 (13)	−0.0132 (11)
C11	0.0501 (16)	0.0370 (14)	0.0356 (14)	0.0035 (12)	−0.0063 (12)	−0.0114 (11)
C12	0.0525 (16)	0.0440 (14)	0.0346 (14)	0.0010 (12)	−0.0111 (12)	−0.0141 (11)
C13	0.0530 (17)	0.0331 (13)	0.0382 (14)	−0.0057 (12)	−0.0090 (13)	−0.0114 (11)
C14	0.0527 (18)	0.0529 (17)	0.0656 (19)	−0.0036 (14)	−0.0094 (15)	−0.0284 (14)
C15	0.061 (2)	0.0627 (19)	0.094 (3)	0.0026 (16)	−0.0332 (19)	−0.0357 (18)
C16	0.095 (3)	0.0565 (18)	0.064 (2)	−0.0099 (19)	−0.036 (2)	−0.0221 (16)
C17	0.091 (3)	0.066 (2)	0.0473 (18)	−0.0151 (18)	−0.0050 (17)	−0.0297 (15)
C18	0.0596 (19)	0.0569 (17)	0.0500 (17)	−0.0068 (14)	−0.0041 (15)	−0.0249 (13)
C19	0.0501 (17)	0.0432 (15)	0.0331 (13)	0.0049 (13)	−0.0060 (12)	−0.0164 (11)
C20	0.0566 (18)	0.0431 (15)	0.0452 (15)	0.0042 (13)	−0.0052 (13)	−0.0127 (12)
C21	0.071 (2)	0.0423 (15)	0.0488 (17)	0.0076 (14)	−0.0019 (15)	−0.0132 (13)
C22	0.066 (2)	0.0560 (18)	0.0494 (17)	0.0208 (16)	−0.0120 (15)	−0.0141 (14)
C23	0.0544 (19)	0.071 (2)	0.0600 (18)	0.0113 (16)	−0.0145 (15)	−0.0282 (15)
C24	0.0589 (19)	0.0503 (16)	0.0515 (17)	0.0052 (14)	−0.0088 (14)	−0.0194 (13)
C25	0.0428 (16)	0.0349 (13)	0.0367 (14)	0.0002 (11)	−0.0064 (12)	−0.0125 (11)
C26	0.060 (2)	0.0629 (18)	0.0619 (19)	0.0136 (16)	0.0013 (16)	−0.0005 (15)
N1	0.0475 (13)	0.0365 (11)	0.0378 (12)	0.0040 (10)	−0.0101 (10)	−0.0136 (9)

Geometric parameters (Å, º)

C1—C2	1.374 (4)	C14—H14	0.9300
C1—C6	1.386 (3)	C15—C16	1.361 (4)
C1—H1	0.9300	C15—H15	0.9300
C2—C3	1.365 (4)	C16—C17	1.360 (4)

C2—H2	0.9300	C16—H16	0.9300
C3—C4	1.373 (4)	C17—C18	1.375 (4)
C3—H3	0.9300	C17—H17	0.9300
C4—C5	1.375 (4)	C18—H18	0.9300
C4—H4	0.9300	C19—C20	1.380 (4)
C5—C6	1.385 (4)	C19—C24	1.383 (4)
C5—H5	0.9300	C20—C21	1.381 (3)
C6—C7	1.479 (4)	C20—H20	0.9300
C7—C12	1.388 (3)	C21—C22	1.369 (4)
C7—C8	1.411 (3)	C21—H21	0.9300
C8—C9	1.399 (4)	C22—C23	1.379 (4)
C8—N1	1.422 (3)	C22—H22	0.9300
C9—C10	1.384 (3)	C23—C24	1.383 (3)
C9—C13	1.495 (4)	C23—H23	0.9300
C10—C11	1.384 (3)	C24—H24	0.9300
C10—H10	0.9300	C25—N1	1.268 (3)
C11—C12	1.384 (3)	C25—C26	1.479 (4)
C11—C19	1.482 (3)	C25—C25^{i}	1.491 (4)
C12—H12	0.9300	C26—H26A	0.9600
C13—C14	1.373 (4)	C26—H26B	0.9600
C13—C18	1.391 (4)	C26—H26C	0.9600
C14—C15	1.379 (4)		

C2—C1—C6	121.2 (3)	C15—C14—H14	119.7
C2—C1—H1	119.4	C16—C15—C14	120.6 (3)
C6—C1—H1	119.4	C16—C15—H15	119.7
C3—C2—C1	120.6 (3)	C14—C15—H15	119.7
C3—C2—H2	119.7	C15—C16—C17	119.8 (3)
C1—C2—H2	119.7	C15—C16—H16	120.1
C2—C3—C4	119.5 (3)	C17—C16—H16	120.1
C2—C3—H3	120.3	C16—C17—C18	120.2 (3)
C4—C3—H3	120.3	C16—C17—H17	119.9
C3—C4—C5	119.8 (3)	C18—C17—H17	119.9
C3—C4—H4	120.1	C17—C18—C13	120.9 (3)
C5—C4—H4	120.1	C17—C18—H18	119.5
C4—C5—C6	121.8 (3)	C13—C18—H18	119.5
C4—C5—H5	119.1	C20—C19—C24	118.2 (2)
C6—C5—H5	119.1	C20—C19—C11	121.8 (3)
C1—C6—C5	117.0 (2)	C24—C19—C11	119.9 (2)
C1—C6—C7	123.3 (2)	C19—C20—C21	121.2 (3)
C5—C6—C7	119.7 (2)	C19—C20—H20	119.4
C12—C7—C8	117.7 (2)	C21—C20—H20	119.4
C12—C7—C6	119.0 (2)	C22—C21—C20	119.9 (3)
C8—C7—C6	123.2 (2)	C22—C21—H21	120.0
C9—C8—C7	120.2 (2)	C20—C21—H21	120.0
C9—C8—N1	121.0 (2)	C21—C22—C23	119.9 (3)
C7—C8—N1	118.7 (2)	C21—C22—H22	120.0
C10—C9—C8	118.9 (2)	C23—C22—H22	120.0

C10—C9—C13	119.4 (2)	C22—C23—C24	119.8 (3)
C8—C9—C13	121.5 (2)	C22—C23—H23	120.1
C9—C10—C11	122.7 (2)	C24—C23—H23	120.1
C9—C10—H10	118.7	C23—C24—C19	120.9 (3)
C11—C10—H10	118.7	C23—C24—H24	119.5
C12—C11—C10	117.0 (2)	C19—C24—H24	119.5
C12—C11—C19	120.8 (2)	N1—C25—C26	125.5 (2)
C10—C11—C19	122.2 (2)	N1—C25—C25^{i}	116.6 (3)
C11—C12—C7	123.5 (2)	C26—C25—C25^{i}	118.0 (3)
C11—C12—H12	118.2	C25—C26—H26A	109.5
C7—C12—H12	118.2	C25—C26—H26B	109.5
C14—C13—C18	117.8 (2)	H26A—C26—H26B	109.5
C14—C13—C9	122.6 (3)	C25—C26—H26C	109.5
C18—C13—C9	119.6 (3)	H26A—C26—H26C	109.5
C13—C14—C15	120.7 (3)	H26B—C26—H26C	109.5
C13—C14—H14	119.7	C25—N1—C8	121.1 (2)

C6—C1—C2—C3	0.1 (4)	C10—C9—C13—C14	133.0 (3)
C1—C2—C3—C4	0.3 (4)	C8—C9—C13—C14	−50.0 (3)
C2—C3—C4—C5	−0.1 (4)	C10—C9—C13—C18	−45.6 (3)
C3—C4—C5—C6	−0.4 (4)	C8—C9—C13—C18	131.3 (3)
C2—C1—C6—C5	−0.6 (4)	C18—C13—C14—C15	−1.8 (4)
C2—C1—C6—C7	177.1 (2)	C9—C13—C14—C15	179.6 (2)
C4—C5—C6—C1	0.7 (4)	C13—C14—C15—C16	1.0 (4)
C4—C5—C6—C7	−177.1 (2)	C14—C15—C16—C17	0.3 (5)
C1—C6—C7—C12	−139.5 (3)	C15—C16—C17—C18	−0.8 (4)
C5—C6—C7—C12	38.1 (3)	C16—C17—C18—C13	0.1 (4)
C1—C6—C7—C8	43.3 (4)	C14—C13—C18—C17	1.2 (4)
C5—C6—C7—C8	−139.1 (3)	C9—C13—C18—C17	180.0 (2)
C12—C7—C8—C9	0.3 (4)	C12—C11—C19—C20	142.3 (3)
C6—C7—C8—C9	177.6 (2)	C10—C11—C19—C20	−39.0 (4)
C12—C7—C8—N1	177.5 (2)	C12—C11—C19—C24	−38.7 (3)
C6—C7—C8—N1	−5.2 (4)	C10—C11—C19—C24	140.0 (3)
C7—C8—C9—C10	−0.9 (4)	C24—C19—C20—C21	0.7 (4)
N1—C8—C9—C10	−178.0 (2)	C11—C19—C20—C21	179.8 (2)
C7—C8—C9—C13	−177.9 (2)	C19—C20—C21—C22	0.1 (4)
N1—C8—C9—C13	5.0 (4)	C20—C21—C22—C23	−0.5 (4)
C8—C9—C10—C11	0.5 (4)	C21—C22—C23—C24	0.1 (4)
C13—C9—C10—C11	177.6 (2)	C22—C23—C24—C19	0.7 (4)
C9—C10—C11—C12	0.5 (4)	C20—C19—C24—C23	−1.1 (4)
C9—C10—C11—C19	−178.3 (2)	C11—C19—C24—C23	179.9 (2)
C10—C11—C12—C7	−1.1 (4)	C26—C25—N1—C8	0.2 (4)
C19—C11—C12—C7	177.7 (2)	C25^{i}—C25—N1—C8	179.7 (2)
C8—C7—C12—C11	0.7 (4)	C9—C8—N1—C25	−81.2 (3)
C6—C7—C12—C11	−176.7 (2)	C7—C8—N1—C25	101.6 (3)

Symmetry code: (i) −*x*, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the (C19–C24) phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17···*Cg*1^{ii}	0.93	2.89	3.737 (5)	152

Symmetry code: (ii) −*x*+1, −*y*+1, −*z*+1.