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4-Anilino-1-benzylpiperidine-4-carbonitrile

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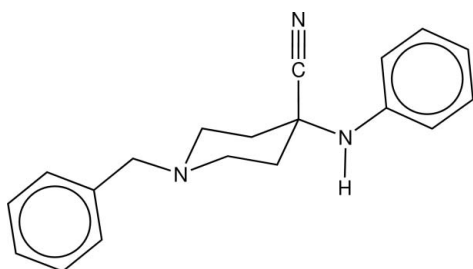
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 Key indicators: single-crystal X-ray study; $T = 90$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.045; wR factor = 0.124; data-to-parameter ratio = 33.7.

The title molecule, $\text{C}_{19}\text{H}_{21}\text{N}_3$, an important precursor in the synthesis of porphyrin-fentanyl conjugates, has its piperidine ring in the chair conformation, with endocyclic torsion-angle magnitudes in the range 53.26 (8)– 60.63 (9)°. The $\text{C}\equiv\text{N}$ group is axial, while the CH_2Ph and NHPh groups are equatorial. The NH group does not engage in strong hydrogen bonding, but forms an intermolecular $\text{N}-\text{H}\cdots\text{N}$ interaction.

Related literature

For background literature, see: Barth *et al.* (2005); Deguchi *et al.* (2004); Henriksen *et al.* (2005); Terasaki *et al.* (2003); Vicente, (2001). For a related structure, see: Brine *et al.* (1994).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{21}\text{N}_3$
 $M_r = 291.39$
 Monoclinic, $P2_1/n$
 $a = 9.7718$ (13) Å
 $b = 10.0415$ (14) Å
 $c = 15.9519$ (15) Å
 $\beta = 94.532$ (9)°

$V = 1560.4$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 90$ K
 $0.37 \times 0.25 \times 0.23$ mm

Data collection

Nonius KappaCCD diffractometer
 with an Oxford Cryosystems
 Cryostream cooler
 Absorption correction: none
 24180 measured reflections
 6842 independent reflections
 5189 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.124$
 $S = 1.03$
 6842 reflections
 203 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.49$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2N}\cdots\text{N3}^i$	0.847 (14)	2.756 (13)	3.5044 (12)	148.2 (11)

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The purchase of the diffractometer was made possible by grant No. LEQSF(1999–2000)-ENH-TR-13, administered by the Louisiana Board of Regents.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2222).

References

- Altomare, A., Burla, M. C., Camalli, M., Casciarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Barth, R. F., Coderre, J. A., Vicente, M. G. H. & Blue, T. E. (2005). *Clin. Cancer Res.* **11**, 3987–4002.
- Brine, G. A., Stark, P. A., Carroll, F. I. & Singh, P. (1994). *J. Heterocycl. Chem.* **31**, 513–520.
- Deguchi, Y., Naito, Y., Ohtsuki, S., Miyakawa, Y., Morimoto, K., Hosoya, K.-I., Sakurada, S. & Terasaki, T. (2004). *J. Pharmacol. Exp. Ther.* **310**, 177–184.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Henriksen, G., Platzter, S., Marton, J., Hauser, A., Berthele, A., Schwaiger, M., Marinelli, L., Lavecchia, A., Novellino, E. & Wester, H.-J. (2005). *J. Med. Chem.* **48**, 7720–7732.
- Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Terasaki, T., Ohtsuki, S., Hori, S., Takanaga, H., Nakashima, E. & Hosoya, K. (2003). *Drug Discovery Today*, **8**, 944–954.
- Vicente, M. G. H. (2001). *Curr. Med. Chem. Anti-Cancer Agents*, **1**, 175–194.

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supplementary materials

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4-Anilino-1-benzylpiperidine-4-carbonitrile

K. K. Allam, F. R. Fronczek and M. G. H. Vicente

Comment

1-Benzyl-4-phenylamino-4-piperidinecarbonitrile is an important precursor in the synthesis of porphyrin-fentanyl conjugates that might be used as sensitizers in photodynamic therapy (PDT) (Vicente, 2001) and/or in boron neutron capture therapy (BNCT) (Barth *et al.*, 2005) of brain tumors. One of the obstacles for treating brain tumors using chemotherapy is the presence of the blood brain barrier (BBB), which protects the central nervous system from drugs and endogenous molecules (Terasaki *et al.*, 2003). Porphyrin-fentanyl conjugates could potentially cross the BBB due to the affinity of fentanyl derivatives for the opioid receptors highly expressed in the BBB (Deguchi *et al.*, 2004), and thus selectively accumulate within brain tumors.

The structure of the title compound is shown in Fig 1. The piperidine ring is in the chair conformation, with the C≡N group axial and the CH₂Ph and NPh groups equatorial. The conformation of the CH₂Ph group with respect to the piperidine is described by torsion angles C5—N1—C6—C7 - 168.52 (6) and N1—C6—C7—C8 62.28 (10)°. The conformation of the NPh group with respect to the piperidine is described by torsion angles C2—C3—N2—C13 179.19 (8) and C3—N2—C13—C18 172.56 (8)°. The pyramidal nature of N1 can be seen by the near-tetrahedral C—N1—C angles, which all fall within the narrow range 110.18 (6) - 110.76 (7)°, such that N1 lies 0.465 (1) Å from the plane defined by C1, C5, and C6.

Despite the presence of both a potential hydrogen-bond donor and potential hydrogen-bond acceptors, the compound exhibits no strong hydrogen bonding, nor any short C—H...N interactions. The nearest distance of the N—H group to a hydrogen-bond acceptor is to nitrile N3ⁱ (at $i = 3/2 - x, 1/2 + y, 3/2 - z$), having N2...N3ⁱ distance 3.5044 (12) Å, H2N...N3ⁱ distance 2.756 (13) Å, and angle about H2N 148.2 (11)°.

Experimental

The title compound was prepared in 89.5% yield from *N*-benzyl-4-piperidone, using an optimized procedure from that previously published (Henriksen *et al.*, 2005), as follows. To a 100 ml round-bottom flask under an argon atmosphere were added *N*-benzyl-4-piperidone (1.89 g, 10 mmol), aniline (3.7 g, 40 mmol), KCN (2.6 g, 40 mmol) and dry dichloromethane (40 ml). The reaction mixture was cooled to 0° C and stirred under argon for 20 minutes. Acetic acid (1.8 g, 30 mmol) was added to the reaction mixture over a period of 10 minutes and the final mixture heated at 50° C for 24 h. After cooling to room temperature the reaction mixture was poured into crushed ice (50 g), neutralized with 25% aqueous NaOH and the pH of the mixture was adjusted to about 10 using 40% aqueous K₂CO₃. The organic phase was collected and the water layer was extracted with dichloromethane (2 x 25 ml). The organic extracts were dried over anhydrous sodium bicarbonate and concentrated under reduced pressure to give a yellow solid. The yellow crystals were purified by re-crystallization from dichloromethane/hexane to give 2.6 g (89.5%) yield of colorless crystals. Spectroscopic analysis, ¹H NMR (250 MHz, CDCl₃): 1.92 (td, J₁ = 3.6 Hz, J₂ = 10.8 Hz, 2H, CH₂), 2.29–2.53 (m, 4H, CH₂), 2.79–2.85 (m, 2H, CH₂), 3.56 (s, 2H,

supplementary materials

CH₂Ph), 3.64 (s, 1H, NH), 6.88–6.95 (m, 3H, ArH), 7.20–7.33 (m, 7H, ArH). ¹³C NMR (CDCl₃): 36.09, 49.27, 59.06, 62.58, 117.78, 120.93, 127.26, 128.96, 129.00, 129.31, 134.02, 138.00, 143.29. MS MALDI-TOF *m/z* 290.9 (*M*⁺).

Refinement

H atoms on C were placed in idealized positions with C—H distances 0.95 - 0.99 Å and thereafter treated as riding. Coordinates of the N—H hydrogen atom were refined. *U*_{iso} for H was assigned as 1.2 times *U*_{eq} of the attached atoms.

Figures



Fig. 1. Ellipsoids at the 50% level, with H atoms having arbitrary radius.

4-Anilino-1-benzylpiperidine-4-carbonitrile

Crystal data

C₁₉H₂₁N₃

M_r = 291.39

Monoclinic, *P*2₁/*n*

Hall symbol: -*P* 2yn

a = 9.7718 (13) Å

b = 10.0415 (14) Å

c = 15.9519 (15) Å

β = 94.532 (9)°

V = 1560.4 (3) Å³

Z = 4

*F*₀₀₀ = 624

D_x = 1.240 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 5228 reflections

θ = 2.5–35.0°

μ = 0.07 mm⁻¹

T = 90 K

Fragment, colorless

0.37 × 0.25 × 0.23 mm

Data collection

Nonius KappaCCD

diffractometer with an Oxford Cryosystems Cryo-stream cooler

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 90 K

ω scans with κ offsets

Absorption correction: none

24180 measured reflections

6842 independent reflections

5189 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.025

θ_{max} = 35.0°

θ_{min} = 2.5°

h = -15→15

k = -12→16

l = -25→25

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.3181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.04$	$\Delta\rho_{\max} = 0.49 \text{ e } \text{\AA}^{-3}$
6842 reflections	$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$
203 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001 \times Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0081 (18)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.43216 (7)	0.37118 (8)	0.73394 (4)	0.01247 (13)
N2	0.68233 (8)	0.66885 (8)	0.63306 (4)	0.01659 (14)
H2N	0.6447 (13)	0.7350 (14)	0.6548 (8)	0.020*
N3	0.86941 (8)	0.40176 (9)	0.70892 (5)	0.01981 (15)
C1	0.53227 (8)	0.44146 (9)	0.79097 (4)	0.01280 (14)
H1A	0.6124	0.3830	0.8050	0.015*
H1B	0.4903	0.4628	0.8438	0.015*
C2	0.57970 (8)	0.56932 (9)	0.75123 (5)	0.01372 (14)
H2A	0.6475	0.6144	0.7910	0.016*
H2B	0.5002	0.6298	0.7403	0.016*
C3	0.64507 (8)	0.54198 (8)	0.66810 (5)	0.01146 (13)
C4	0.54095 (8)	0.45965 (9)	0.61165 (5)	0.01336 (14)
H4A	0.4602	0.5158	0.5947	0.016*
H4B	0.5837	0.4328	0.5600	0.016*
C5	0.49370 (8)	0.33556 (9)	0.65622 (5)	0.01364 (15)

supplementary materials

H5A	0.4256	0.2869	0.6185	0.016*
H5B	0.5731	0.2759	0.6695	0.016*
C6	0.38149 (8)	0.25204 (9)	0.77503 (5)	0.01443 (14)
H6A	0.4604	0.1982	0.7983	0.017*
H6B	0.3273	0.1971	0.7329	0.017*
C7	0.29300 (8)	0.28909 (9)	0.84504 (5)	0.01256 (14)
C8	0.17137 (8)	0.35979 (9)	0.82626 (5)	0.01463 (15)
H8	0.1458	0.3852	0.7698	0.018*
C9	0.08730 (9)	0.39339 (9)	0.88947 (5)	0.01645 (16)
H9	0.0047	0.4414	0.8760	0.020*
C10	0.12395 (9)	0.35679 (10)	0.97276 (5)	0.01752 (16)
H10	0.0655	0.3777	1.0157	0.021*
C11	0.24640 (10)	0.28969 (9)	0.99214 (5)	0.01758 (16)
H11	0.2730	0.2665	1.0488	0.021*
C12	0.33101 (9)	0.25597 (9)	0.92851 (5)	0.01539 (15)
H12	0.4149	0.2102	0.9423	0.018*
C13	0.74564 (8)	0.68867 (8)	0.55887 (5)	0.01178 (14)
C14	0.80105 (8)	0.58537 (8)	0.51281 (5)	0.01315 (14)
H14	0.7935	0.4957	0.5308	0.016*
C15	0.86740 (8)	0.61438 (9)	0.44045 (5)	0.01405 (14)
H15	0.9037	0.5437	0.4094	0.017*
C16	0.88109 (8)	0.74472 (9)	0.41323 (5)	0.01466 (15)
H16	0.9273	0.7636	0.3644	0.018*
C17	0.82588 (8)	0.84752 (9)	0.45882 (5)	0.01452 (15)
H17	0.8344	0.9371	0.4408	0.017*
C18	0.75859 (8)	0.82007 (9)	0.53034 (5)	0.01298 (14)
H18	0.7208	0.8911	0.5604	0.016*
C19	0.77255 (8)	0.46283 (9)	0.68964 (5)	0.01285 (14)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0132 (3)	0.0145 (3)	0.0100 (2)	-0.0015 (2)	0.0032 (2)	-0.0008 (2)
N2	0.0258 (4)	0.0095 (3)	0.0161 (3)	0.0039 (3)	0.0115 (3)	0.0025 (2)
N3	0.0166 (3)	0.0209 (4)	0.0217 (3)	0.0027 (3)	0.0001 (2)	0.0017 (3)
C1	0.0158 (3)	0.0133 (3)	0.0096 (3)	-0.0012 (3)	0.0027 (2)	0.0001 (3)
C2	0.0178 (3)	0.0127 (3)	0.0113 (3)	0.0002 (3)	0.0056 (2)	-0.0002 (3)
C3	0.0131 (3)	0.0106 (3)	0.0111 (3)	0.0017 (3)	0.0034 (2)	0.0015 (2)
C4	0.0125 (3)	0.0182 (4)	0.0095 (3)	0.0011 (3)	0.0016 (2)	0.0012 (3)
C5	0.0137 (3)	0.0168 (4)	0.0106 (3)	-0.0015 (3)	0.0024 (2)	-0.0024 (3)
C6	0.0155 (3)	0.0131 (3)	0.0152 (3)	-0.0009 (3)	0.0050 (2)	-0.0007 (3)
C7	0.0133 (3)	0.0121 (3)	0.0126 (3)	-0.0022 (3)	0.0032 (2)	-0.0003 (3)
C8	0.0135 (3)	0.0170 (4)	0.0134 (3)	-0.0017 (3)	0.0016 (2)	-0.0013 (3)
C9	0.0132 (3)	0.0178 (4)	0.0187 (3)	-0.0017 (3)	0.0036 (3)	-0.0033 (3)
C10	0.0200 (4)	0.0168 (4)	0.0167 (3)	-0.0048 (3)	0.0080 (3)	-0.0025 (3)
C11	0.0250 (4)	0.0146 (4)	0.0136 (3)	-0.0030 (3)	0.0046 (3)	0.0016 (3)
C12	0.0183 (3)	0.0130 (4)	0.0151 (3)	-0.0003 (3)	0.0024 (3)	0.0021 (3)
C13	0.0125 (3)	0.0117 (3)	0.0114 (3)	0.0011 (3)	0.0027 (2)	0.0014 (2)

C14	0.0157 (3)	0.0109 (3)	0.0134 (3)	0.0010 (3)	0.0042 (2)	0.0012 (3)
C15	0.0148 (3)	0.0150 (4)	0.0128 (3)	0.0007 (3)	0.0038 (2)	-0.0007 (3)
C16	0.0150 (3)	0.0163 (4)	0.0131 (3)	0.0002 (3)	0.0038 (2)	0.0022 (3)
C17	0.0152 (3)	0.0132 (4)	0.0154 (3)	0.0002 (3)	0.0032 (2)	0.0039 (3)
C18	0.0141 (3)	0.0115 (3)	0.0135 (3)	0.0012 (3)	0.0027 (2)	0.0009 (3)
C19	0.0139 (3)	0.0126 (3)	0.0123 (3)	-0.0011 (3)	0.0023 (2)	0.0003 (3)

Geometric parameters (Å, °)

N1—C1	1.4636 (10)	C7—C12	1.3940 (11)
N1—C5	1.4644 (10)	C7—C8	1.3964 (12)
N1—C6	1.4688 (11)	C8—C9	1.3915 (11)
N2—C13	1.3924 (10)	C8—H8	0.9500
N2—C3	1.4491 (11)	C9—C10	1.3977 (12)
N2—H2N	0.847 (14)	C9—H9	0.9500
N3—C19	1.1492 (11)	C10—C11	1.3867 (13)
C1—C2	1.5203 (12)	C10—H10	0.9500
C1—H1A	0.9900	C11—C12	1.4004 (12)
C1—H1B	0.9900	C11—H11	0.9500
C2—C3	1.5414 (11)	C12—H12	0.9500
C2—H2A	0.9900	C13—C14	1.4042 (11)
C2—H2B	0.9900	C13—C18	1.4047 (12)
C3—C4	1.4946 (11)	C14—C15	1.3986 (11)
C3—C4	1.5443 (11)	C14—H14	0.9500
C4—C5	1.5242 (12)	C15—C16	1.3887 (12)
C4—H4A	0.9900	C15—H15	0.9500
C4—H4B	0.9900	C16—C17	1.3955 (12)
C5—H5A	0.9900	C16—H16	0.9500
C5—H5B	0.9900	C17—C18	1.3888 (11)
C6—C7	1.5119 (11)	C17—H17	0.9500
C6—H6A	0.9900	C18—H18	0.9500
C6—H6B	0.9900		
C1—N1—C5	110.18 (6)	C7—C6—H6B	109.4
C1—N1—C6	110.34 (6)	H6A—C6—H6B	108.0
C5—N1—C6	110.76 (7)	C12—C7—C8	118.96 (7)
C13—N2—C3	126.55 (7)	C12—C7—C6	121.47 (7)
C13—N2—H2N	118.2 (9)	C8—C7—C6	119.57 (7)
C3—N2—H2N	113.6 (9)	C9—C8—C7	120.60 (7)
N1—C1—C2	111.03 (6)	C9—C8—H8	119.7
N1—C1—H1A	109.4	C7—C8—H8	119.7
C2—C1—H1A	109.4	C8—C9—C10	120.23 (8)
N1—C1—H1B	109.4	C8—C9—H9	119.9
C2—C1—H1B	109.4	C10—C9—H9	119.9
H1A—C1—H1B	108.0	C11—C10—C9	119.43 (8)
C1—C2—C3	111.66 (7)	C11—C10—H10	120.3
C1—C2—H2A	109.3	C9—C10—H10	120.3
C3—C2—H2A	109.3	C10—C11—C12	120.29 (8)
C1—C2—H2B	109.3	C10—C11—H11	119.9
C3—C2—H2B	109.3	C12—C11—H11	119.9

supplementary materials

H2A—C2—H2B	108.0	C7—C12—C11	120.44 (8)
N2—C3—C19	109.02 (7)	C7—C12—H12	119.8
N2—C3—C2	108.01 (7)	C11—C12—H12	119.8
C19—C3—C2	106.94 (6)	N2—C13—C14	123.69 (7)
N2—C3—C4	114.82 (6)	N2—C13—C18	117.84 (7)
C19—C3—C4	110.37 (7)	C14—C13—C18	118.43 (7)
C2—C3—C4	107.35 (6)	C15—C14—C13	120.06 (8)
C5—C4—C3	112.06 (6)	C15—C14—H14	120.0
C5—C4—H4A	109.2	C13—C14—H14	120.0
C3—C4—H4A	109.2	C16—C15—C14	121.12 (8)
C5—C4—H4B	109.2	C16—C15—H15	119.4
C3—C4—H4B	109.2	C14—C15—H15	119.4
H4A—C4—H4B	107.9	C15—C16—C17	118.91 (7)
N1—C5—C4	110.80 (7)	C15—C16—H16	120.5
N1—C5—H5A	109.5	C17—C16—H16	120.5
C4—C5—H5A	109.5	C18—C17—C16	120.59 (8)
N1—C5—H5B	109.5	C18—C17—H17	119.7
C4—C5—H5B	109.5	C16—C17—H17	119.7
H5A—C5—H5B	108.1	C17—C18—C13	120.88 (8)
N1—C6—C7	111.21 (7)	C17—C18—H18	119.6
N1—C6—H6A	109.4	C13—C18—H18	119.6
C7—C6—H6A	109.4	N3—C19—C3	177.73 (8)
N1—C6—H6B	109.4		
C5—N1—C1—C2	60.63 (9)	C12—C7—C8—C9	-1.83 (13)
C6—N1—C1—C2	-176.75 (6)	C6—C7—C8—C9	179.10 (8)
N1—C1—C2—C3	-58.68 (9)	C7—C8—C9—C10	0.10 (13)
C13—N2—C3—C19	63.33 (10)	C8—C9—C10—C11	1.61 (14)
C13—N2—C3—C2	179.19 (8)	C9—C10—C11—C12	-1.57 (14)
C13—N2—C3—C4	-61.08 (11)	C8—C7—C12—C11	1.86 (13)
C1—C2—C3—N2	177.84 (6)	C6—C7—C12—C11	-179.09 (8)
C1—C2—C3—C19	-64.94 (8)	C10—C11—C12—C7	-0.17 (14)
C1—C2—C3—C4	53.51 (8)	C3—N2—C13—C14	-9.65 (13)
N2—C3—C4—C5	-173.35 (7)	C3—N2—C13—C18	172.56 (8)
C19—C3—C4—C5	62.96 (8)	N2—C13—C14—C15	-177.68 (8)
C2—C3—C4—C5	-53.26 (8)	C18—C13—C14—C15	0.09 (12)
C1—N1—C5—C4	-59.98 (8)	C13—C14—C15—C16	0.65 (12)
C6—N1—C5—C4	177.65 (6)	C14—C15—C16—C17	-0.77 (12)
C3—C4—C5—N1	57.77 (8)	C15—C16—C17—C18	0.15 (12)
C1—N1—C6—C7	69.20 (8)	C16—C17—C18—C13	0.59 (12)
C5—N1—C6—C7	-168.52 (6)	N2—C13—C18—C17	177.20 (7)
N1—C6—C7—C12	-116.76 (9)	C14—C13—C18—C17	-0.70 (12)
N1—C6—C7—C8	62.28 (10)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2N \cdots N3 ⁱ	0.847 (14)	2.756 (13)	3.5044 (12)	148.2 (11)

Symmetry codes: (i) $-x+3/2, y+1/2, -z+3/2$.

Fig. 1

