Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dinaphtho[2,3-*a*,2′,3′-*h*]phenazine

Chitoshi Kitamura,* Chika Sakamoto, Nobuhiro Kawatsuki and Akio Yoneda

Department of Materials Science and Chemistry, Graduate School of Engineering, University of Hyogo, 2167 Shosha, Himeji, Hyogo 671-2280, Japan
Correspondence e-mail: kitamura@eng.u-hyogo.ac.jp

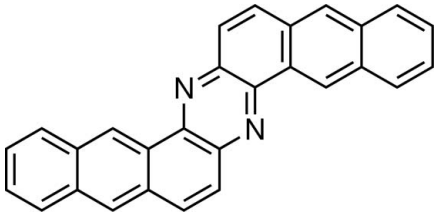
Received 22 August 2007; accepted 27 August 2007

Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.003 Å; R factor = 0.072; wR factor = 0.162; data-to-parameter ratio = 12.0.

The title compound, $C_{28}H_{16}N_2$, also known as anthrazine, possesses a crystallographic center of inversion; thus half of the formula unit is crystallographically independent. The heptacyclic ring system is essentially planar. In the crystal structure, the molecular packing is characterized by a combination of a columnar stacking and a herringbone arrangement. Along the *a* axis, the molecules in one column form face-to-face slipped π-stacks. The interplanar face-to-face distance between neighboring molecules is 3.495 (3) Å.

Related literature

An improved synthesis of the title compound was described by Kawabata *et al.* (1964). The photoconductivity was reported by Inokuchi (1953). For related literature, see: Scholl & Berblinger (1903); Wolak *et al.* (2004).



Experimental

Crystal data

$C_{28}H_{16}N_2$
M_r = 380.43
Monoclinic, $P2_1/n$
a = 4.515 (3) Å
b = 10.325 (6) Å
c = 19.28 (1) Å
β = 95.127 (7)°
V = 895.2 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 223 K
0.6 × 0.03 × 0.03 mm

Data collection

Rigaku/MSC Mercury CCD area-detector diffractometer
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
T_{min} = 0.994, T_{max} = 0.998
6727 measured reflections
2015 independent reflections
1606 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.162
S = 1.23
2015 reflections
168 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We thank the Research Center for Molecular-Scale Nanoscience, the Institute for Molecular Science, for the collection of the X-ray data. This work was partially supported by a Grant-in-Aid (No. 18750123) for Scientific Research from the Ministry of Education, Culture, Sports, Science and Technology, Japan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2032).

References

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
Inokuchi, H. (1953). *Bull. Chem. Soc. Jpn*, **27**, 22–27.
Kawabata, T., Tanimoto, S. & Oda, R. (1964). *Kogyo Kagaku Zasshi*, **67**, 1151–1152.
Rigaku/MSC (2001). *CrystalClear*. Version 1.3. Rigaku Corporation, Tokyo, Japan.
Scholl, R. & Berblinger, H. (1903). *Chem. Ber.* **36**, 3427–3445.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Wolak, M. A., Jang, B.-B., Palilis, L. C. & Kafafi, H. (2004). *J. Phys. Chem. B*, **108**, 5492–5499.

  doi:10.1107/S1600536807041992

supplementary materials

Acta Cryst. (2007). E**63**, o3952 [**doi:10.1107/S1600536807041992**]

Dinaphtho[2,3-*a*,2',3'-*h*]phenazine

C. Kitamura, C. Sakamoto, N. Kawatsuki and A. Yoneda

Comment

The title compound, dinaphtho[2,3 − *a*,2',3'-*h*]phenazine, (I), is an old member of the acene family. The preparation of (I) was first reported by Scholl & Berblinger (1903) in the early 20 t h century. Several decades later, a highly simple synthetic method using the mild oxygen-oxidation of 2-aminoanthracene was developed by Oda's group (Kawabata *et al.*, 1964). Since then, the procurement of the material is easy. From the viewpoint of organic semiconducting materials, (I) may be of potential use because of the large π-system. The photoconductivity of (I) was investigated by Inokuchi (1953), who showed the relation between the spectral response of photoconductivity and the optical energy gap. In the physical properites, intermolecular interactions are critical factors. However, the molecular arrangement of (I) in the solid state has not been unveiled yet. Here we report the crystal structure of (I).

The molecular structure of (I) is shown in Fig. 1. The structure is confirmed to be heptacyclic. The bond lengths and angles are all within the expected ranges. The molecule possesses a crystallographic center of inversion, and then half of the formula unit is crystallographically independent. In addition, the molecule is essentially planar. The maximum deviation from the mean plane of the aromatic ring is 0.048 (2) Å for C10. As shown in Fig. 2, the crystal structure is characterized by the column-by- column stacking mode, which contains two symmetry-independent stacks along the *a* axis. The interplanar tilt angle between the aromatic rings in two adjacent columns is 71.13°. The columns are seen to pack in a herringbone arrangement, when the crystal is viewed along the *c* axis. Along the *a* axis, the molecules form face-to-face slipped π-stacks. The interplanar face-to-face distance between the neighboring molecules is 3.495 (3) Å. There are neither C—H···N nor C—H···π hydrogen bonds in the crystal structure.

Experimental

The title compound was prepared according to the modified method described by Kawabata *et al.* (1964). To a solution of KO^{t}Bu (183 mg, 1.64 mmol) in DMSO (4 ml) and ^{t}BuOH (1 ml), 2-aminoanthracene (155 mg, 0.80 mmol) was added under air, and then the reaction mixture was stirred at room temperature overnight. The mixture was pourd into cold water, and acidified with conc. HCl. The resulting brown precipitate was filtered off, washed with water, and dried under vacuum to produce a crude product (135 mg, 88%). Vacuum sublimation in gradient-temperature tube-furnace as described by Wolak *et al.* (2004) gave brown needle-shaped crystals suitable for X-ray analysis.

Refinement

All the H atoms were located from the difference Fourier map and refined isotropically. The C—H distances are in the range 0.96 (2)–0.99 (3) Å.

Figures

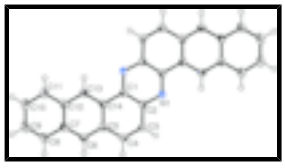


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids for non-H atoms. The unlabeled stoms are related to the labeled ones by the symmetry code (−*x*, 1 − *y*, 1 − *z*).

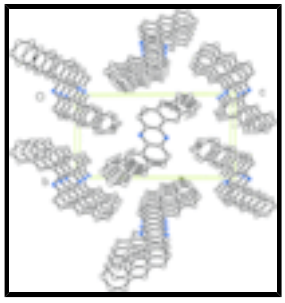


Fig. 2. The packing diagram of (I), viewed down the *a* axis. Hydrogen atoms are omitted for clarity.

Dinaphtho[2,3 − *a*,2',3'-*h*]phenazine

Crystal data

$C_{28}H_{16}N_2$	F_{000} = 396
M_r = 380.43	D_x = 1.411 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1843 reflections
a = 4.515 (3) Å	θ = 3.2–27.5º
b = 10.325 (6) Å	μ = 0.08 mm^{-1}
c = 19.28 (1) Å	*T* = 223 K
β = 95.127 (7)º	Needle, brown
V = 895.2 (9) Å^3	0.6 × 0.03 × 0.03 mm
Z = 2	

Data collection

Rigaku/MSC Mercury CCD area-detector diffractometer	2015 independent reflections
Radiation source: rotating-anode X-ray tube	1606 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
Detector resolution: 14.6199 pixels mm^{-1}	θ_{max} = 27.5º
T = 223 K	θ_{min} = 4.3º
ω scans	*h* = −5→3
Absorption correction: numerical (NUMABS; Higashi, 1999)	*k* = −13→11
T_{min} = 0.994, T_{max} = 0.998	*l* = −23→24
6727 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.072$	$w = 1/[\sigma^2(FO^2) + (0.0645P)^2 + 0.2734P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.162$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.23$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
2015 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
168 parameters	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2051 (4)	0.41851 (18)	0.53097 (9)	0.0220 (4)
C2	0.0906 (4)	0.40351 (19)	0.46010 (9)	0.0227 (4)
C3	0.1953 (4)	0.2991 (2)	0.41947 (10)	0.0276 (5)
C4	0.3987 (4)	0.2143 (2)	0.44742 (11)	0.0283 (5)
C5	0.5201 (4)	0.22354 (19)	0.51910 (10)	0.0247 (4)
C6	0.7253 (4)	0.1343 (2)	0.54795 (11)	0.0275 (5)
C7	0.8404 (4)	0.14200 (18)	0.61788 (11)	0.0255 (4)
C8	1.0472 (4)	0.0495 (2)	0.64880 (12)	0.0333 (5)
C9	1.1514 (4)	0.0595 (2)	0.71718 (12)	0.0364 (5)
C10	1.0542 (4)	0.1604 (2)	0.75901 (12)	0.0361 (5)
C11	0.8577 (4)	0.2512 (2)	0.73117 (11)	0.0312 (5)
C12	0.7453 (4)	0.24478 (18)	0.65992 (10)	0.0252 (4)
C13	0.5393 (4)	0.33602 (19)	0.63034 (10)	0.0250 (4)
C14	0.4242 (4)	0.32624 (18)	0.56151 (10)	0.0228 (4)
N1	−0.1132 (3)	0.48528 (16)	0.42973 (8)	0.0239 (4)
H1	0.120 (4)	0.292 (2)	0.3702 (12)	0.032 (6)*
H2	0.473 (5)	0.139 (2)	0.4197 (12)	0.039 (6)*
H3	0.788 (4)	0.065 (2)	0.5195 (12)	0.030 (6)*
H4	1.119 (5)	−0.019 (2)	0.6190 (13)	0.038 (6)*
H5	1.294 (5)	−0.003 (3)	0.7385 (14)	0.048 (7)*
H6	1.126 (5)	0.163 (2)	0.8083 (13)	0.043 (7)*
H7	0.783 (5)	0.319 (2)	0.7588 (13)	0.037 (6)*
H8	0.471 (5)	0.406 (2)	0.6576 (12)	0.033 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0215 (8)	0.0214 (10)	0.0232 (10)	−0.0019 (6)	0.0019 (6)	0.0014 (7)
C2	0.0230 (8)	0.0227 (10)	0.0219 (10)	−0.0013 (7)	0.0001 (6)	0.0014 (8)
C3	0.0319 (9)	0.0297 (11)	0.0207 (10)	0.0021 (8)	−0.0001 (7)	−0.0038 (8)
C4	0.0320 (9)	0.0280 (11)	0.0252 (11)	0.0029 (8)	0.0043 (7)	−0.0051 (8)
C5	0.0240 (8)	0.0238 (10)	0.0263 (11)	−0.0009 (7)	0.0029 (7)	0.0007 (8)

C6	0.0300 (9)	0.0235 (10)	0.0297 (11)	0.0047 (7)	0.0066 (7)	−0.0021 (9)
C7	0.0236 (8)	0.0236 (10)	0.0296 (10)	0.0007 (7)	0.0039 (7)	0.0046 (8)
C8	0.0308 (10)	0.0316 (12)	0.0374 (12)	0.0076 (8)	0.0033 (8)	0.0061 (10)
C9	0.0315 (10)	0.0390 (13)	0.0380 (12)	0.0065 (9)	−0.0007 (8)	0.0122 (10)
C10	0.0349 (10)	0.0425 (13)	0.0297 (12)	0.0022 (9)	−0.0045 (8)	0.0075 (10)
C11	0.0310 (9)	0.0337 (12)	0.0285 (11)	0.0010 (8)	−0.0003 (8)	0.0024 (9)
C12	0.0234 (8)	0.0241 (10)	0.0281 (11)	−0.0022 (7)	0.0016 (7)	0.0031 (8)
C13	0.0275 (9)	0.0224 (10)	0.0252 (10)	0.0016 (7)	0.0024 (7)	0.0000 (8)
C14	0.0230 (8)	0.0204 (10)	0.0251 (10)	−0.0026 (7)	0.0018 (7)	0.0006 (8)
N1	0.0261 (7)	0.0218 (8)	0.0234 (8)	0.0008 (6)	0.0002 (6)	−0.0002 (7)

Geometric parameters (Å, °)

C1—N1^{i}	1.337 (2)	C7—C8	1.428 (3)
C1—C2	1.425 (3)	C8—C9	1.364 (3)
C1—C14	1.459 (2)	C8—H4	0.99 (3)
C2—N1	1.344 (2)	C9—C10	1.411 (3)
C2—C3	1.437 (3)	C9—H5	0.98 (3)
C3—C4	1.346 (3)	C10—C11	1.367 (3)
C3—H1	0.98 (2)	C10—H6	0.98 (2)
C4—C5	1.444 (3)	C11—C12	1.423 (3)
C4—H2	1.01 (2)	C11—H7	0.96 (2)
C5—C6	1.387 (3)	C12—C13	1.408 (3)
C5—C14	1.430 (3)	C13—C14	1.385 (3)
C6—C7	1.403 (3)	C13—H8	0.96 (2)
C6—H3	0.96 (2)	N1—C1^{i}	1.337 (2)
C7—C12	1.425 (3)		
N1^{i}—C1—C2	121.30 (16)	C9—C8—H4	121.0 (14)
N1^{i}—C1—C14	119.32 (16)	C7—C8—H4	118.4 (14)
C2—C1—C14	119.37 (17)	C8—C9—C10	120.7 (2)
N1—C2—C1	121.66 (17)	C8—C9—H5	120.8 (16)
N1—C2—C3	118.64 (17)	C10—C9—H5	118.5 (16)
C1—C2—C3	119.70 (16)	C11—C10—C9	120.4 (2)
C4—C3—C2	121.03 (18)	C11—C10—H6	120.5 (15)
C4—C3—H1	120.3 (13)	C9—C10—H6	119.1 (15)
C2—C3—H1	118.7 (13)	C10—C11—C12	120.7 (2)
C3—C4—C5	121.83 (18)	C10—C11—H7	122.0 (14)
C3—C4—H2	122.0 (13)	C12—C11—H7	117.2 (14)
C5—C4—H2	116.1 (13)	C13—C12—C11	121.71 (18)
C6—C5—C14	119.26 (18)	C13—C12—C7	119.31 (18)
C6—C5—C4	121.51 (18)	C11—C12—C7	118.97 (18)
C14—C5—C4	119.22 (17)	C14—C13—C12	121.13 (18)
C5—C6—C7	121.61 (18)	C14—C13—H8	117.9 (13)
C5—C6—H3	119.1 (13)	C12—C13—H8	120.9 (13)
C7—C6—H3	119.2 (13)	C13—C14—C5	119.70 (17)
C6—C7—C12	118.97 (17)	C13—C14—C1	121.46 (17)
C6—C7—C8	122.36 (19)	C5—C14—C1	118.84 (17)
C12—C7—C8	118.66 (19)	C1^{i}—N1—C2	117.04 (16)

C9—C8—C7	120.6 (2)		
N1^{i}—C1—C2—N1	0.2 (3)	C10—C11—C12—C7	0.1 (3)
C14—C1—C2—N1	179.48 (16)	C6—C7—C12—C13	−0.1 (3)
N1^{i}—C1—C2—C3	179.84 (17)	C8—C7—C12—C13	−179.40 (18)
C14—C1—C2—C3	−0.9 (3)	C6—C7—C12—C11	178.81 (17)
N1—C2—C3—C4	−179.81 (18)	C8—C7—C12—C11	−0.4 (3)
C1—C2—C3—C4	0.5 (3)	C11—C12—C13—C14	−177.86 (17)
C2—C3—C4—C5	0.2 (3)	C7—C12—C13—C14	1.1 (3)
C3—C4—C5—C6	178.72 (19)	C12—C13—C14—C5	−1.3 (3)
C3—C4—C5—C14	−0.6 (3)	C12—C13—C14—C1	178.43 (17)
C14—C5—C6—C7	0.2 (3)	C6—C5—C14—C13	0.7 (3)
C4—C5—C6—C7	−179.07 (18)	C4—C5—C14—C13	−179.99 (17)
C5—C6—C7—C12	−0.5 (3)	C6—C5—C14—C1	−179.10 (17)
C5—C6—C7—C8	178.73 (19)	C4—C5—C14—C1	0.2 (3)
C6—C7—C8—C9	−179.26 (19)	N1^{i}—C1—C14—C13	0.0 (3)
C12—C7—C8—C9	0.0 (3)	C2—C1—C14—C13	−179.28 (16)
C7—C8—C9—C10	0.9 (3)	N1^{i}—C1—C14—C5	179.79 (16)
C8—C9—C10—C11	−1.2 (3)	C2—C1—C14—C5	0.5 (2)
C9—C10—C11—C12	0.7 (3)	C1—C2—N1—C1^{i}	−0.2 (3)
C10—C11—C12—C13	179.02 (19)	C3—C2—N1—C1^{i}	−179.83 (16)

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

Fig. 1

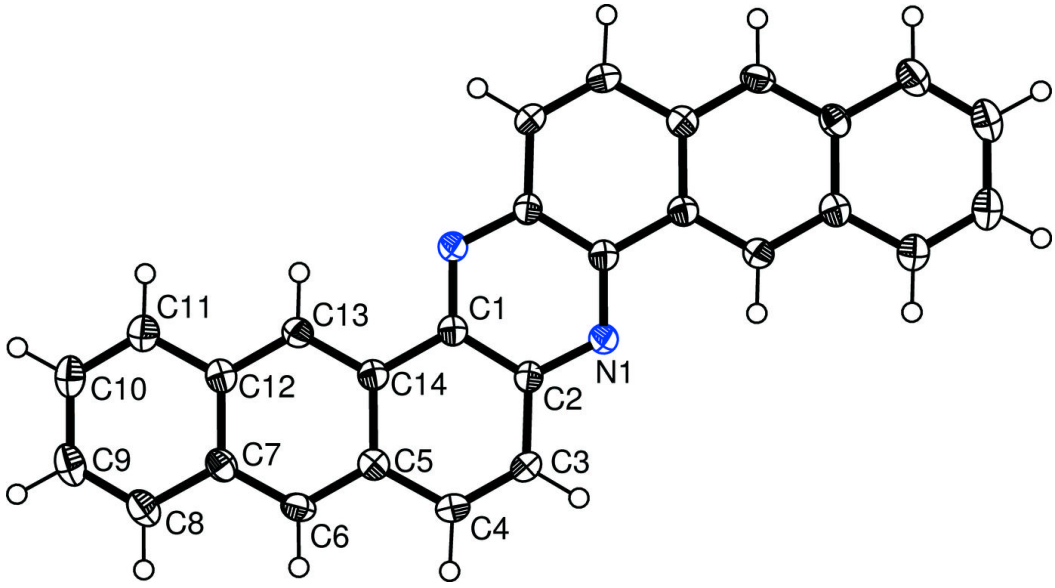


Fig. 2