

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1177). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Eichhorn, E. L. (1956). *Acta Cryst.* **9**, 787–793.  
 Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.  
 Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Determination. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft University Press.  
 Johnson, C. K. (1976). *ORTEPII*. Report ORLN-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 Lai, T. F. & Marsh, R. E. (1967). *Acta Cryst.* **22**, 885–893.  
 Moreno Fuquen, R., de Castro, E. V. R. & Lechat, J. R. (1992). *Rev. Colomb. Quím.* **21**, 7–14.  
 Moreno Fuquen, R., Lechat, J. R. & de Almeida Santos, R. H. (1991). *Acta Cryst.* **C47**, 2388–2391.  
 Panunto, T. W., Urbánczyk-Lipkowska, Z., Johnson, R. & Etter, M. C. (1987). *J. Am. Chem. Soc.* **109**, 7786–7797.  
 Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement*. University of Göttingen, Germany.  
 Wang, Y., Blessing, R. H., Ross, F. K. & Coppens, P. (1976). *Acta Cryst.* **B32**, 572–578.

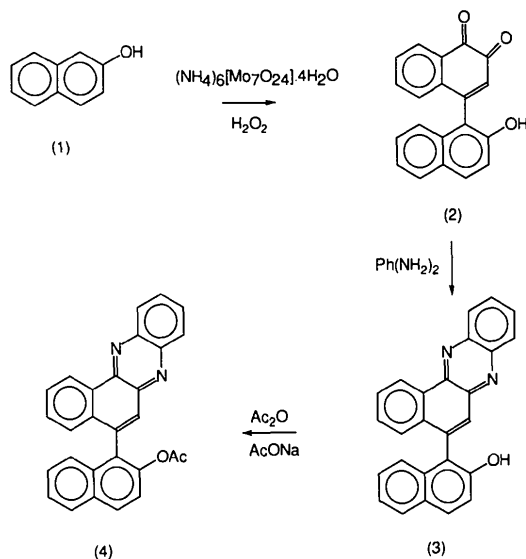


Fig. 1. As expected, both the benzo[*a*]phenazin-5-yl and naphthyl ring systems are planar (r.m.s. deviations 0.028 and 0.015 Å, respectively). The mean planes of the two ring systems are inclined at 80.42 (7)° and the normal to the plane of the acetate group forms an angle of 76.6 (1)° with that of the naphthyl ring. None of the

*Acta Cryst.* (1996). **C52**, 222–224

## 1-(Benzo[*a*]phenazin-5-yl)-2-acetyloxynaphthalene,† C<sub>28</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>

VICKIE MCKEE,<sup>a</sup> MALACHY McCANN,<sup>b</sup> NIGEL C. CONNAUGHTON<sup>b</sup> AND MICHAEL G. KENNEDY<sup>b</sup>

<sup>a</sup>School of Chemistry, Queen's University, Belfast BT9 5AG, Northern Ireland, and <sup>b</sup>Chemistry Department, St Patrick's College, Maynooth, Co Kildare, Ireland

(Received 18 September 1995; accepted 10 October 1995)

### Abstract

The angle between the benzo[*a*]phenazin-5-yl and naphthyl rings is 80.42 (7)° and the benzo[*a*]phenazin-5-yl rings exhibit π–π stacking.

### Comment

The title compound, (4), has been prepared previously (Bader, 1951) but the structure has not been reported. A perspective view of a molecule of (4) is shown in

† Alternative nomenclature: 1-(benzo[*a*]phenazin-5-yl)naphthalen-2-yl acetate.

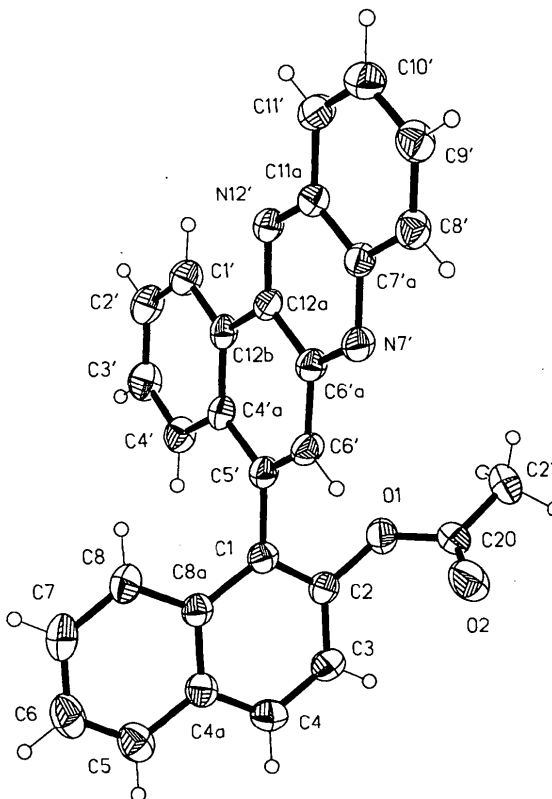


Fig. 1. Perspective views of the title molecule showing 30% probability displacement ellipsoids. The H atoms are shown as small spheres for clarity.

bond lengths or angles are particularly unusual, those of the benzo[*a*]phenazin-5-yl ring being similar to those of phenazine itself (Wozniak, Kariuki & Jones, 1991), as well as to substituted systems such as tetrabenzo[*a,c,h,j*]phenazine (Sato, Oya & Otani, 1980).

The packing diagram (Fig. 2) shows the  $\pi$ - $\pi$  stacking of the benzo[*a*]phenazin-5-yl rings. The interplanar distance between the rings is 3.4 Å and, as expected, they are offset to allow efficient overlap (Hunter & Sanders, 1990). There are no similar interactions involving the naphthyl ring system and no orthogonal 'edge-to-face' interactions.

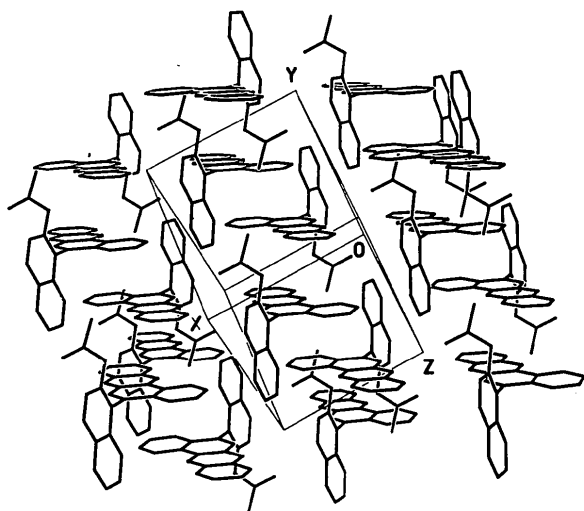


Fig. 2. Unit-cell plot showing the  $\pi$ - $\pi$  stacking of the phenazine rings.

## Experimental

The naphthoquinone, (2), was prepared as a racemic mixture from  $\beta$ -naphthol, (1), using a literature method (Raake-Fels, Wang, Robins & Christensen, 1950). Compound (2) was converted to the phenazine, (3) and then to (4) using a slight modification of the methods used by Bader (1951).

1-(Benzo[*a*]phenazin-5-yl)-2-naphthol, (3), was prepared when compound (2) (0.50 g, 1.67 mmol) and *o*-phenylenediamine (0.225 g, 2.08 mmol) were dissolved in 40 ml of acetic acid at room temperature. A yellow solid precipitated immediately on agitation of the mixture. Concentrating the mixture to dryness *in vacuo* afforded the crude product (0.554 g). Chromatography on silica with chloroform as eluant afforded (3) as a yellow solid (yield 0.426 g, 69%); m.p. 565–568 K (lit. 566–567 K; Bader, 1951);  $\nu_{\max}$  (KBr disk) 3050, 2940, 2850, 1630, 1580, 1490, 1380, 1170, 1120, 760  $\text{cm}^{-1}$ ;  $^1\text{H NMR}$  ( $\delta$  p.p.m.,  $\text{CDCl}_3$ ) 8.9–8.8 (1H, *m*), 8.3–7.0 (15H, *m*);  $m/z$  372 ( $M^+$ ), 355.

Compound (4) was prepared when compound (3) (0.20 g, 0.54 mmol) and sodium acetate (2 g) were dissolved in a mixture of glacial acetic acid (20 ml) and acetic anhydride (20 ml). The mixture was heated at reflux for 2 h to give a dark yellow solution. Concentrating the mixture to dryness *in vacuo* afforded a yellow semi-solid which was dissolved

in dichloromethane and washed with water to give the crude product (0.145 g). Chromatography on silica with chloroform as eluant afforded (4) as a yellow powder (yield 0.120 g, 54%). Crystals suitable for X-ray analysis were obtained by taking the powder up in the minimum volume of hot methanol and then placing the solution in a refrigerator at 277 K for 72 h. M.p. 454–455 K (lit. 454–455 K; Bader, 1951);  $\nu_{\max}$  (KBr disk) 3390, 3010, 2880, 2810, 1750, 1190, 740  $\text{cm}^{-1}$ ;  $^1\text{H NMR}$  ( $\delta$  p.p.m.,  $\text{CDCl}_3$ ) 9.7–9.9 (1H, *dd*), 8.5–7.2 (14H, *m*) 1.9 (3H, *s*);  $m/z$  414 ( $M^+$ ) 371, 355.

## Crystal data

$\text{C}_{28}\text{H}_{18}\text{N}_2\text{O}_2$   
 $M_r = 414.44$   
 Triclinic  
 $P\bar{1}$   
 $a = 9.006(2)$  Å  
 $b = 9.655(3)$  Å  
 $c = 12.468(3)$  Å  
 $\alpha = 97.16(2)^\circ$   
 $\beta = 97.22(2)^\circ$   
 $\gamma = 97.22(2)^\circ$   
 $V = 1056.1(5)$  Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.303$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 33 reflections

$\theta = 5\text{--}11^\circ$

$\mu = 0.083$  mm<sup>-1</sup>

$T = 293(2)$  K

Block

$0.68 \times 0.25 \times 0.13$  mm

Yellow

## Data collection

Siemens P4 diffractometer

From ( $K_{\alpha_1} - 0.9^\circ$ ) to ( $K_{\alpha_2} + 0.9^\circ$ )  $\omega$  scans

Absorption correction: none

2966 measured reflections

2755 independent reflections

1492 observed reflections

[ $I > 2\sigma(I)$ ]

$R_{\text{int}} = 0.0410$

$\theta_{\text{max}} = 22.50^\circ$

$h = 0 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -13 \rightarrow 13$

3 standard reflections

monitored every 97

reflections

intensity decay: none

## Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.0599$

$wR(F^2) = 0.1633$

$S = 1.044$

2753 reflections

290 parameters

H-atom parameters not

refined

$w = 1/[\sigma^2(F_o^2) + (0.076P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.15$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992),

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

	$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
C1	0.7163 (4)	-0.0259 (4)	0.1795 (3)	0.0466 (10)
C2	0.7102 (4)	0.0171 (4)	0.0789 (3)	0.0495 (11)
C3	0.7706 (5)	-0.0501 (5)	-0.0077 (4)	0.0632 (13)
C4	0.8383 (5)	-0.1660 (5)	0.0064 (4)	0.0629 (12)
C4A	0.8439 (5)	-0.2199 (4)	0.1063 (4)	0.0535 (11)
C5	0.9097 (5)	-0.3437 (5)	0.1215 (4)	0.0693 (14)

C6	0.9131 (6)	-0.3943 (5)	0.2188 (5)	0.078 (2)
C7	0.8542 (5)	-0.3279 (5)	0.3043 (4)	0.0744 (14)
C8	0.7903 (5)	-0.2080 (4)	0.2936 (4)	0.0623 (13)
C8A	0.7835 (4)	-0.1505 (4)	0.1942 (3)	0.0489 (11)
O1	0.6297 (3)	0.1307 (3)	0.0594 (2)	0.0601 (8)
O2	0.8465 (4)	0.2761 (3)	0.0720 (3)	0.1012 (14)
C20	0.7126 (6)	0.2584 (5)	0.0551 (4)	0.0636 (13)
C21	0.6102 (6)	0.3612 (4)	0.0286 (4)	0.083 (2)
C1'	0.3086 (5)	0.0501 (4)	0.4077 (4)	0.0596 (12)
C2'	0.2211 (5)	-0.0689 (5)	0.3494 (4)	0.0670 (13)
C3'	0.2758 (5)	-0.1479 (5)	0.2665 (4)	0.0651 (13)
C4'	0.4176 (5)	-0.1090 (4)	0.2418 (3)	0.0579 (12)
C4'A	0.5097 (4)	0.0126 (4)	0.2995 (3)	0.0478 (11)
C5'	0.6592 (5)	0.0556 (4)	0.2709 (3)	0.0492 (11)
C6'	0.7454 (5)	0.1734 (4)	0.3280 (3)	0.0539 (11)
C6'A	0.6970 (5)	0.2563 (4)	0.4162 (3)	0.0481 (11)
N7'	0.7897 (4)	0.3699 (3)	0.4682 (3)	0.0513 (9)
C7'A	0.7405 (5)	0.4439 (4)	0.5521 (3)	0.0468 (10)
C8'	0.8338 (5)	0.5674 (4)	0.6103 (3)	0.0578 (12)
C9'	0.7865 (5)	0.6421 (4)	0.6951 (4)	0.0651 (13)
C10'	0.6446 (6)	0.6004 (5)	0.7273 (4)	0.0700 (13)
C11'	0.5528 (5)	0.4844 (4)	0.6728 (3)	0.0628 (13)
C11A	0.5972 (5)	0.4035 (4)	0.5837 (3)	0.0493 (11)
N12'	0.5020 (4)	0.2881 (3)	0.5290 (3)	0.0503 (9)
C12A	0.5511 (4)	0.2161 (4)	0.4465 (3)	0.0448 (10)
C12B	0.4552 (4)	0.0913 (4)	0.3844 (3)	0.0466 (10)

Table 2. Selected geometric parameters (Å, °)

C4'A—C12B	1.401 (5)	N7'—C7'A	1.345 (4)
C4'A—C5'	1.460 (5)	C7'A—C11A	1.419 (5)
C5'—C6'	1.356 (5)	C11A—N12'	1.365 (5)
C6'—C6'A	1.424 (5)	N12'—C12A	1.323 (4)
C6'A—N7'	1.334 (4)	C12A—C12B	1.462 (5)
C6'A—C12A	1.433 (5)		

Refinement on  $F^2$  for all reflections except for 2 with very negative  $F^2$ .

Data collection: XSCANS (Fait, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1035). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Bader, A. R. (1951). *J. Am. Chem. Soc.* **73**, 3731–3732.  
 Fait, J. (1994). *XSCANS Users Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Hunter, C. A. & Sanders, J. K. M. (1990). *J. Am. Chem. Soc.* **112**, 5525–5534.  
 Raacke-Fels, I. D., Wang, C. H., Robins, R. K. & Christensen, B. E. (1950). *J. Org. Chem.* **15**, 627–630.  
 Sato, M., Oya, A. & Otani, S. (1980). *Cryst. Struct. Commun.* **9**, 811–813.  
 Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1990b). *SHELXTL/PC Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Wozniak, K., Kariuki, B. & Jones, W. (1991). *Acta Cryst.* **C47**, 1113–1114.

*Acta Cryst.* (1996). **C52**, 224–226

## 3,3-Dimethylglutaronitrile

ANNIE HÉROUX AND FRANÇOIS BRISSE\*

Département de Chimie, Université de Montréal, CP 6128, Succ. Centre-ville, Montréal, Québec, Canada H3C 3J7.  
 E-mail: brisse@tornado.ere.umontreal.ca

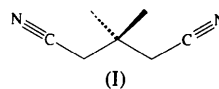
(Received 15 March 1995; accepted 25 August 1995)

## Abstract

The X-ray determination of the title compound, 3,3-dimethylpentanedinitrile, C<sub>7</sub>H<sub>10</sub>N<sub>2</sub>, was performed in order to characterize an intermediate product. The compound crystallizes in the  $C2/c$  space group, with one molecule in a general position and another molecule on a crystallographic twofold rotation axis.

## Comment

The title compound, (I), was synthesized following the procedure previously described by Equi, Brown, Cooper, Ner, Watson & Robins (1991). The compound is an intermediate in the procedure used to obtain 3,3-dimethylpentanediamine from 3,3-dimethylglutaric acid. In the first reaction step, the acid function was protected with a mesylate (phenyl methanesulfonate) group (yield 83%). The product was then reacted with NaCN in DMSO to give the dinitrile compound. This last product was isolated by extraction according to Equi *et al.* (1991). A yellow liquid was obtained, from which a white solid appeared after distillation on a Kugelrohr apparatus (80 K/0.5 mmHg).



There are 12 molecules in the monoclinic unit cell of the  $C2/c$  space group, eight of which occupy general positions, while the other four possess a crystallographic twofold rotation axis passing through the central atom, C33. The bond distances and angles within the molecules are in good agreement with expected values. All C—H distances are in the range 0.96 (2)–1.02 (2) Å, while the C—C—H bond angles range from 105.7 (12) to 112.8 (10)° and the H—C—H angles range from 102.4 (16) to 114.3 (16)°. The environment of the central C atom deviates slightly from tetrahedral; the C—CH<sub>2</sub> and C—CH<sub>3</sub> bond lengths average 1.538 and 1.520 Å, respectively, while the CH<sub>3</sub>—C—CH<sub>3</sub> and CH<sub>2</sub>—C—CH<sub>2</sub> angles also show a marked difference, with values of 110.5 and 105.1°, respectively. The two groups of atoms N11, C11, C12, C13, C22, C21, N21 (r.m.s.