Table 2. *Selected geometric parameters* (Å, °)

(*A*)			
O13—C12	1.223 (8)	C2—C10	1.52 (1)
N1—C2	1.48 (1)	C2—C11	1.52 (1)
N1—C6	1.352 (9)	C4—C5	1.44 (1)
N1—H1	0.86 (6)	C5—C6	1.352 (9)
N3—C2	1.48 (1)	C6—C12	1.50 (1)
N3—C4	1.291 (9)	C15—C16	1.38 (1)
N7—C5	1.408 (9)	C15—C20	1.378 (9)
N7—C8	1.29 (1)	C16—C17	1.38 (1)
N9—C4	1.411 (9)	C17—C18	1.37 (1)
N9—C8	1.39 (1)	C18—C19	1.37 (1)
N9—C15	1.42 (1)	C19—C20	1.38 (1)
N14—C12	1.332 (9)		
C2—N1—C6	124.0 (7)	C4—C5—C6	117.8 (7)
C2—N3—C4	113.1 (7)	N1—C6—C5	115.7 (8)
C5—N7—C8	104.1 (8)	N1—C6—C12	116.5 (7)
C4—N9—C8	106.2 (6)	C5—C6—C12	127.4 (7)
C4—N9—C15	128.7 (6)	N7—C8—N9	115.5 (7)
C8—N9—C15	125.0 (7)	N9—C15—C16	120.5 (7)
N1—C2—N3	111.1 (6)	N9—C15—C20	118.6 (7)
N1—C2—C10	107.2 (6)	C16—C15—C20	120.9 (8)
N1—C2—C11	109.1 (6)	C15—C16—C17	119.7 (8)
N3—C2—C10	108.5 (7)	C16—C17—C18	119.9 (8)
N3—C2—C11	108.9 (8)	C17—C18—C19	120.2 (8)
C10—C2—C11	112.1 (7)	C18—C19—C20	120.9 (8)
N3—C4—N9	128.2 (7)	C15—C20—C19	118.4 (8)
N3—C4—C5	128.6 (8)	O13—C12—N14	126.0 (9)
N9—C4—C5	103.2 (6)	O13—C12—C6	119.5 (7)
N7—C5—C4	111.1 (7)	N14—C12—C6	114.5 (8)
N7—C5—C6	129.6 (8)		
(*B*)			
O13—C12	1.212 (6)	C2—C10	1.501 (7)
N1—C2	1.505 (6)	C2—C11	1.537 (7)
N1—C6	1.299 (6)	C4—C5	1.370 (6)
N3—C2	1.483 (6)	C5—C6	1.420 (7)
N3—C4	1.376 (6)	C6—C12	1.543 (6)
N3—H3	0.98 (5)	C15—C16	1.393 (7)
N7—C5	1.373 (6)	C15—C20	1.324 (8)
N7—C8	1.313 (7)	C16—C17	1.425 (8)
N9—C4	1.335 (6)	C17—C18	1.35 (1)
N9—C8	1.404 (6)	C18—C19	1.359 (9)
N9—C15	1.459 (6)	C19—C20	1.393 (8)
N14—C12	1.316 (6)		
C2—N1—C6	116.1 (4)	N7—C5—C6	131.7 (4)
C2—N3—C4	112.0 (4)	C4—C5—C6	117.6 (4)
C2—N3—H3	110 (3)	N1—C6—C5	122.6 (4)
C4—N3—H3	114 (3)	N1—C6—C12	114.0 (5)
C5—N7—C8	105.8 (4)	C5—C6—C12	123.0 (5)
C4—N9—C8	106.8 (4)	N7—C8—N9	110.3 (5)
C4—N9—C15	129.7 (4)	N9—C15—C16	116.6 (5)
C8—N9—C15	123.3 (4)	N9—C15—C20	120.7 (5)
N1—C2—N3	114.3 (4)	C16—C15—C20	122.7 (5)
N1—C2—C10	107.9 (4)	C15—C16—C17	115.0 (6)
N1—C2—C11	106.6 (4)	C16—C17—C18	121.6 (6)
N3—C2—C10	108.0 (4)	C17—C18—C19	121.1 (6)
N3—C2—C11	108.0 (4)	C18—C19—C20	118.3 (6)
C10—C2—C11	112.1 (4)	C15—C20—C19	121.2 (6)
N3—C4—N9	129.3 (4)	O13—C12—N14	124.8 (5)
N3—C4—C5	123.7 (5)	O13—C12—C6	121.1 (5)
N9—C4—C5	107.0 (4)	N14—C12—C6	114.0 (5)
N7—C5—C4	110.0 (5)		

The data for the orange tautomer were collected on the basis of a monoclinic unit cell of twice the volume of the current one and Miller indices were transformed to conform with the smaller cell during data reduction. During refinement, H atoms were fixed in calculated positions except H1, whose coordinates were refined. During refinement of the yellow tautomer, H atoms were fixed in calculated positions except H3, whose coordinates were refined. Thus, the same number of variables was refined for both tautomers.

For both compounds, data collection: *TEXSAN* (Molecular Structure Corporation, 1985); cell refinement: *TEXSAN*; data reduction: *TEXSAN*; program(s) used to solve structures: *MITHRIL* (Gilmore, 1984); program(s) used to refine structures: *TEXSAN*; molecular graphics: *PLUTO* (Motherwell & Clegg, 1978); software used to prepare material for publication: *CIF* (Hall, Allen & Brown, 1991).

The EPSRC is thanked for funding the Chemical Databank Service *CSSR* used for crystallographic literature searches.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Alves, M. J., Booth, B. L., Carvalho, A., Eastwood, P. R., Nezhat, L., Pritchard, R. G. & Proença, M. F. J. R. P. (1994). *J. Chem. Soc. Perkin Trans.* 2, pp. 1949–1956.

Beagley, B., Booth, B. L., Pritchard, R. G. & Proença, M. F. (1982). *Acta Cryst.* B**38**, 2921–2924.

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Hall, S. R., Allen, F. H. & Brown, I. D. (1991). *Acta Cryst.* A**47**, 655–685.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.

Acta Cryst. (1995). C**51**, 1470–1472

(*Z*)-*N*-4-Fluorophenyl-*C*-phenylnitrone. Confirmation that the Exclusive Product of the Reaction Between Benzaldehyde and 4-Fluorophenylhydroxylamine is the *Z* Isomer

Ronald E. Banks, Richard A. DuBoisson, Robin G. Pritchard and Anthony E. Tipping

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

(*Received* 4 *August* 1994; *accepted* 19 *December* 1994)

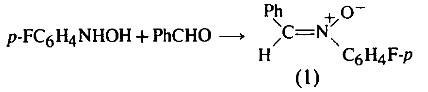
Abstract

The benzaldehyde nitrone moiety in the title molecule, *N*-benzylidene-4-fluoroaniline *N*-oxide, $C_{13}H_{10}FNO$, like that in *N*-(benzylidene)methylamine *N*-oxide [Bed-

ford, Chaloner & Hitchcock (1991). *Acta Cryst.* C**47**, 2484–2485] approaches planarity [C_{benz}—N═C—C_{benz} 179.5 (5) and C_{benz}—C_{benz}—C═N −13 (1)° *cf* 176.0 (2) and 6.8 (2)°, respectively, in the methyl compound]. However, the *p*-fluorobenzene substituent is substantially twisted out of the molecular plane [C_{benz}—C_{benz}—N═C −42.8 (8)°] in order to avoid a non-bonded O···H clash.

Comment

The structure determination of (*Z*)-*N*-4-fluorophenyl-*C*-phenylnitrone, (1), was carried out as part of a general investigation into the preparation of fluorine-containing aryl nitrones and their reaction with alkenes to give isoxazolidines (DuBoisson, 1986).



$$p\text{-}FC_6H_4NHOH + PhCHO \longrightarrow \overset{Ph}{\underset{H}{>}}C{=}\overset{+}{N}\overset{O^-}{\underset{C_6H_4F\text{-}p}{<}}$$

(1)

It was necessary to confirm that the nitrone which resulted from the reaction of benzaldehyde with 4-fluorophenylhydroxylamine was the *Z* isomer so that the stereochemical outcome of 1,3-dipolar cycloaddition reactions involving (1) could be interpreted mechanistically. The parameters for compound (1) as determined by X-ray analysis could then be compared with those obtained previously for *N*-(4-fluorophenyl)-*C*,*C*-diphenylnitrone (Banks, DuBoisson, Pritchard & Tipping, 1991).

As expected, greater steric congestion of the *C*,*C*-diphenylnitrone led to both C-attached phenyl rings being twisted out of the nitrone plane [C_{benz}—C_{benz}—C═N 33.0 (3) and 57.8 (3)° *cf* −13 (1)° for the title compound]. The higher substitution is also reflected in longer bonds to the phenyl substituents [1.469 (4) and 1.477 (4) Å *cf* 1.450 (8) Å in the title molecule].

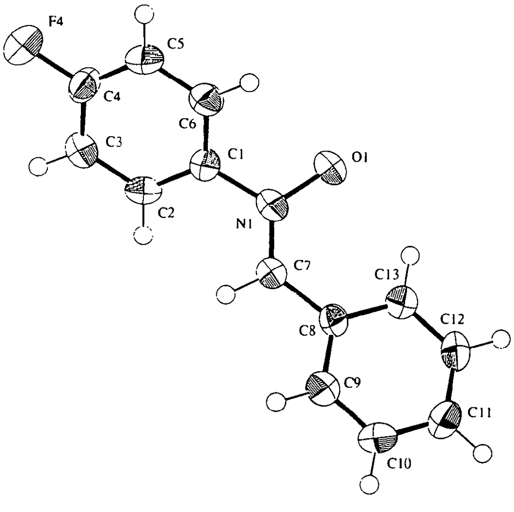


Fig. 1. The title molecule, including the atomic numbering scheme, drawn using *ORTEP*II (Johnson, 1976). Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Benzaldehyde (0.33 g, 3.10 mmol) was stirred under N_2 and treated with a solution of 4-fluorophenylhydroxylamine (0.40 g, 3.10 mmol) in ethanol (10 ml) and stirring was continued in the dark for 16 h. The resulting material was warmed to dissolve the precipitate, then cooled and the resulting solid removed by filtration, washed with ethanol and dried *in vacuo* to afford the title compound (1) (0.64 g, 3.0 mmol, 96%; m.p. 445–446 K). Analysis found C 72.7, H 4.8, F 8.8, N 6.8%, *M* = 215. $C_{13}H_{10}FNO$ requires C 72.6, H 4.7, F 8.8, N 6.5%, *M* = 215. The product was recrystallized from aqueous ethanol.

Crystal data

$C_{13}H_{10}FNO$
$M_r = 215.23$
Monoclinic
$P2_1/n$
$a = 5.590 (2)$ Å
$b = 25.299 (4)$ Å
$c = 7.310 (2)$ Å
$\beta = 90.90 (2)°$
$V = 1034 (1)$ Å^3
$Z = 4$
$D_x = 1.383$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 5.19–12.75°$
$\mu = 0.0943$ mm^{-1}
$T = 296$ K
Plate
0.35 × 0.35 × 0.10 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2097 measured reflections
1861 independent reflections
740 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.0313$
$\theta_{max} = 25.0°$
$h = -6 \rightarrow 0$
$k = -30 \rightarrow 0$
$l = -8 \rightarrow 8$
3 standard reflections monitored every 150 reflections
intensity decay: none

Refinement

Refinement on *F*
$R = 0.0561$
$wR = 0.0695$
$S = 1.478$
740 reflections
145 parameters
H-atom parameters not refined

Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max} = 0.0101$
$\Delta\rho_{max} = 0.304$ e Å^{-3}
$\Delta\rho_{min} = -0.215$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
O1	0.1337 (6)	0.2323 (2)	0.2906 (7)	0.070 (2)
N1	0.3547 (8)	0.2435 (2)	0.2532 (6)	0.044 (2)
C1	0.4103 (9)	0.2999 (2)	0.2437 (7)	0.040 (2)

C2	0.6197 (9)	0.3194 (2)	0.3173 (8)	0.047 (3)
C3	0.6646 (9)	0.3728 (3)	0.3125 (8)	0.048 (3)
C4	0.495 (1)	0.4047 (2)	0.2295 (8)	0.049 (3)
C5	0.2889 (10)	0.3863 (2)	0.1550 (8)	0.050 (3)
C6	0.2447 (9)	0.3327 (2)	0.1631 (8)	0.046 (3)
C7	0.5193 (9)	0.2086 (2)	0.2211 (8)	0.043 (3)
C8	0.4902 (9)	0.1517 (2)	0.2271 (7)	0.037 (2)
C9	0.6765 (9)	0.1222 (2)	0.1550 (8)	0.048 (3)
C10	0.668 (1)	0.0679 (2)	0.1543 (9)	0.055 (3)
C11	0.475 (1)	0.0417 (3)	0.2283 (9)	0.057 (3)
C12	0.2919 (10)	0.0708 (3)	0.3028 (9)	0.055 (3)
C13	0.2973 (9)	0.1250 (2)	0.3022 (8)	0.045 (3)
F4	0.5392 (6)	0.4577 (1)	0.2259 (5)	0.074 (2)

Table 2. *Selected geometric parameters* (Å, °)

O1—N1	1.300 (5)	C5—C6	1.381 (9)
N1—C1	1.463 (6)	C7—C8	1.450 (8)
N1—C7	1.298 (6)	C8—C9	1.392 (7)
C1—C2	1.373 (7)	C8—C13	1.393 (7)
C1—C6	1.369 (7)	C9—C10	1.373 (8)
C2—C3	1.373 (8)	C10—C11	1.383 (8)
C3—C4	1.378 (8)	C11—C12	1.382 (8)
C4—C5	1.351 (8)	C12—C13	1.371 (8)
C4—F4	1.363 (7)		
O1—N1—C1	115.1 (4)	N1—C1—C6	118.0 (5)
O1—N1—C7	124.7 (4)	N1—C7—C8	126.1 (5)
C1—N1—C7	120.2 (4)	C7—C8—C9	115.9 (5)
N1—C1—C2	120.9 (5)	C7—C8—C13	125.6 (5)

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*. Literature Survey: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: HU1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Banks, R. E., DuBoisson, R. A., Pritchard, R. G. & Tipping, A. E. (1991). *Acta Cryst.* C**47**, 230–233.

Bedford, R. B., Chaloner, P. A. & Hitchcock, P. B. (1991). *Acta Cryst.* C**47**, 2484–2485.

CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual.* SERC Daresbury Laboratory, Warrington, England.

DuBoisson, R. A. (1986). PhD thesis, Univ. of Manchester, England.

Enraf–Nonius (1977). *CAD*-4 *Operations Manual.* Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1985). *SHELXS*86. *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Acta Cryst. (1995). C**51**, 1472–1474

5,5′-Di(anthracenecarboxylic) Anhydride

ALEXANDER J. BLAKE, RACHEL S. GRIMDITCH AND MARTIN SCHRÖDER

Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

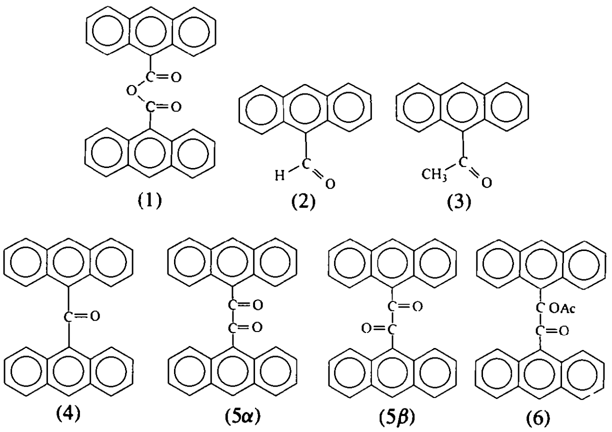
(*Received* 6 *March* 1995; *accepted* 23 *March* 1995)

Abstract

The title compound, $C_{30}H_{18}O_3$, has a dihedral angle of 76.24 (5)° between the two anthracene units and a mean dihedral angle of 64.0 (2)° between an anthracene unit and the plane defined by C5 and the C and O atoms of the carbonyl group.

Comment

It has been shown that in 5-substituted anthracenes the torsion angle between the aromatic π system and the substituent is governed by steric requirements (Trotter, 1959). The least bulky anthracene carbonyl derivative, 5-anthraldehyde, (2), exhibits a torsion of 27° (Trotter, 1959), while the maximum value of 88° is found for 5-acetylanthracene, (3) (Andersson, Becker, Engelhardt, Hansen & White, 1984), with torsion angles for related molecules falling between these values. The value of 64.0 (2)° for the title compound, (1), compares with those of 50.5° for dianthril ketone, (4) (Becker, Langer, Skelton & White, 1989), 75.0° for 5,5-anthroin acetate, (6) (Becker, Kildea, Patrick, Skelton & White, 1991), and 54.0 and 86.15° for the α (5α) and β (5β) forms, respectively, of 5,5′-anthril (Becker, Skelton & White, 1991).



(1) (2) (3)

(4) (5α) (5β) (6)

The dihedral angle between the two anthracene units is also of interest, the value of 76.24 (5)° in (1) falling between those of 87.3° in (4) and 74.8° in (5α).

Acta Crystallographica Section C
ISSN 0108-2701 ©1995