Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-Phenyl-2-nitrobenzamide, an acridone alkaloid precursor

Roxanne Jean Jackson,[a] Masood Parvez,[a]* Shaikh S. Nizami[b] and Reem Y. Khan[b]

[a]Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4, and [b]Department of Chemistry, University of Karachi, Karachi 75270, Pakistan

Correspondence e-mail: parvez@ucalgary.ca

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.128
Data-to-parameter ratio = 16.1

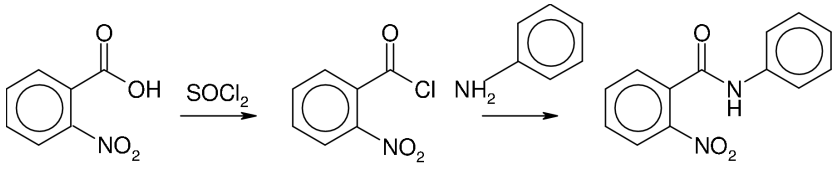
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of *N*-phenyl-2-nitrobenzamide, $C_{13}H_{10}N_2O_3$, likely to be an intermediate in the biosynthesis of acridone alkaloids, is composed of strongly hydrogen-bonded molecules *via* amido H and carbonyl O atoms [H···O 1.99, N···O 2.8364 (13) Å and N—H···O 167°], thus forming chains along the *b* axis. The molecular dimensions are normal and the phenyl rings are inclined at right angles [89.41 (5)°] to each other.

Received 28 January 2002
Accepted 6 February 2002
Online 22 February 2002

Comment

2,4,6-Trimethoxy-*N*-methylaminobenzophenone (tecleanone; Waterman, 1975; Khalid & Waterman, 1981) and 1,3-dimethoxy-*N*-methylacridone (Tillequim *et al.*, 1980) have been isolated from different plants. These compounds have very similar structures and the former is thought to be a precursor in the biosynthesis of the latter. The isolation of both of these compounds from the same plants, *Teclea verdoorniana* and *Oricio suareolens*, has led to the proposal that aminobenzophenones are probable intermediates in the biosynthesis of acridone alkaloids (Adams *et al.*, 1981). This step is of great importance in biological systems as acridone alkaloids have reported success as anticancer drugs (Adams *et al.*, 1981). The conclusion that aminobenzophenones are likely intermediates in the synthesis of acridone alkaloids is further supported by the biomimetic synthesis of acronycine from a benzophenone precursor. For this purpose, *N*-substituted-2-nitrobenzamide has been prepared from 2-nitrobenzoic acid as an intermediate. In this preparation, 2-nitrobenzoic acid, (1), was converted to its acid chloride, (2), followed by nucleophillic replacement of the chloride by the nitrogen of the aniline molecule, resulting in *N*-phenyl-2-nitrobenzamide, (3). The crystal structure of (3) has been determined by X-ray crystallography and is reported in this paper.



(1) (2) (3)

The structure of (3) is presented in Fig. 1. The molecular dimensions in (3) are normal and lie within expected values (Orpen *et al.*, 1994) for the corresponding bond distances and angles, with bond distances as follows: mean C—$C_{aromatic}$ 1.381 (2), N—O 1.217 (4), N—$C_{aromatic}$ (nitro) 1.470 (2), N—$C_{aromatic}$ (amino) 1.4142 (17), Csp^2–N 1.3436 (17), Csp^2—Csp^2

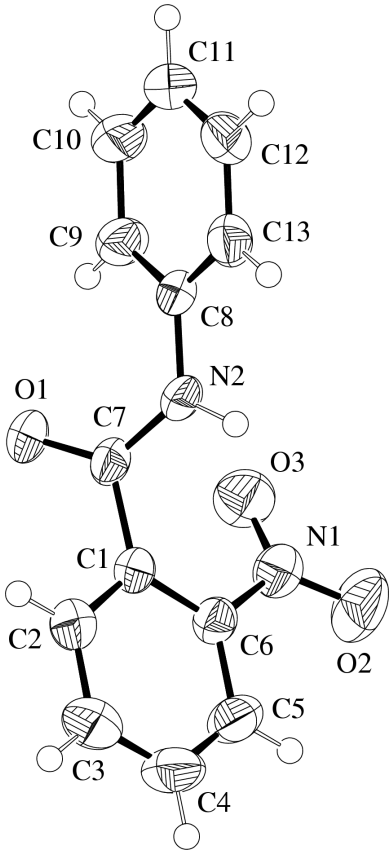


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (3), with displacement ellipsoids plotted at the 50% probability level.

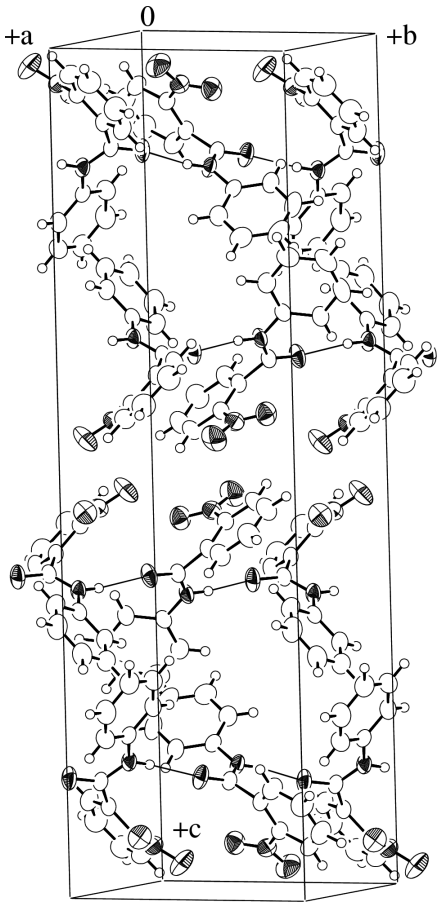


Figure 2
Hydrogen-bonding pattern in (3), showing a hydrogen-bonded polymeric chain along the *b* axis.

1.5056 (19) and C═O 1.2212 (15) Å. Both phenyl rings are essentially planar, as expected, and their mean planes are inclined at right angles, 89.41 (5)°, to each other. The mean plane of the nitro group is oriented at 21.40 (10)° to the plane of the aromatic ring (C1–C6) to which it is attached. The atoms lying in between the two aromatic rings, *i.e.* atoms O1/N2/C1/C7/C8 are almost coplanar, with a maximum deviation of 0.103 (2) Å for C7, and the mean planes of the aromatic rings C1–C6 and C8–C13 form dihedral angles of 58.44 (6) and 31.94 (6)°, respectively, with the mean plane of these atoms.

The structure is stabilized by a strong hydrogen bond between the amido H and carbonyl O atoms [H2···O1 1.99, N2···O1 2.8364 (13) Å and N2—H2···O1 167°], thus linking the molecules into chains along the *b* axis (Fig. 2). A search of the Cambridge Structural Database (Allen & Kennard, 1993) for similar structures revealed a dozen or so phenylbenzamide derivatives closely related to the structure of (3).

Experimental

2-Nitrobenzoic acid, (1) (10 g), was added to benzene (10 ml) and thionyl chloride (10 ml). The mixture was refluxed on a steam bath for 1 h, after which more thionyl chloride (10 ml) was added. The mixture was further refluxed for 2 h. The benzene was removed under reduced pressure. The resulting oil was treated with benzene (10 ml) in order to remove excess thionyl chloride. Benzene was removed under reduced pressure and the residue was cooled, leaving a dark brown oil, 2-nitrobenzoyl chloride, (2) (10.5 g). Aniline (10 ml) was added dropwise to (2) and the reaction mixture was left at room temperature for 1 h. Cold water (150 ml) was added and the mixture allowed to stand for half an hour. The solid was filtered, washed with water, dried and crystallized from ethanol to give *N*-phenyl-2-nitrobenzamide, (3) (12.04 g, 87.69%), in the form of colourless prisms suitable for X-ray diffraction analysis.

Crystal data

$C_{13}H_{10}N_2O_3$
M_r = 242.23
Orthorhombic, *Pbca*
a = 7.9406 (1) Å
b = 9.4695 (2) Å
c = 31.1671 (5) Å
V = 2343.56 (7) Å^3
Z = 8
D_x = 1.373 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2949 reflections
θ = 1.0–27.5°
μ = 0.10 mm^{-1}
T = 293 (2) K
Prismatic, colorless
0.32 × 0.17 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*: Blessing, 1995, 1997)
T_{min} = 0.97, T_{max} = 0.99
4857 measured reflections
2639 independent reflections
1945 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -40 \rightarrow 40$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.31P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.128$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.16$ e Å^{-3}
2639 reflections	Extinction correction: *SHELXL*
164 parameters	Extinction coefficient: 0.035 (6)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.2212 (15)	N1—C6	1.470 (2)
O2—N1	1.2124 (16)	N2—C7	1.3436 (17)
O3—N1	1.2205 (17)	N2—C8	1.4142 (17)
O2—N1—O3	123.65 (14)	O3—N1—C6	118.05 (12)
O2—N1—C6	118.28 (13)	C7—N2—C8	126.45 (11)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.86	1.99	2.8364 (13)	167

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

The H atoms were located from difference Fourier syntheses and were included in the refinement at geometrically idealized positions with C—H = 0.93 Å and N—H = 0.86 Å, utilizing a riding model.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1994); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

SSN and RYK thank the Dean, Faculty of Science, University of Karachi, for financial support.

References

Adams, J. H., Brown, P. M., Gupta, P., Khan, M. S. & Lewis, H. R. (1981). *Tetrahedron*, **37**, 209–217.
Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Fan, H.-F. (1991). *SAPI*91. Rigaku Corporation, Tokyo, Japan.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Khalid, S. A. & Waterman, P. G. (1981). *Phytochemistry*, **20**, 2761–2763.
Molecular Structure Corporation (1994). *TEXSAN*. MSC. 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G. & Taylor, R. (1994). *Structure Correlation*, Vol. 2, edited by H.-B. Bürgi and J. D. Dunitz, pp. 751–858. New York: VCH.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter and R. M. Sweet, pp. 307–326. London: Academic Press.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Tillequim, F., Baudouin, G., Koch, M. & Severnet, T. (1980). *J. Nat. Prod.* **43**, 498–502.
Waterman, P. G. (1975). *Phytochemistry*, **14**, 2092–2093.