North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Scalzo, M., Biava, M., Porretta, G. C. & Cerreto, F. (1991). *QSAR: Rational Approaches on the Design of Bioactive Compounds*, pp. 389–392. Amsterdam: Elsevier.
Toupet, L., Mazari, M., Texier, F. & Carrie, R. (1991). *Acta Cryst.* C**47**, 1528–1531.

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(*Z*)-2-(4-Fluorophenyl)-3-(3-nitrophenyl)-5-phenylisoxazolidine: the Major Isomer Formed by 1,3-Dipolar Addition of an Arylnitrone to Styrene

R. E. Banks, R. A. DuBoisson, R. G. Pritchard and A. E. Tipping

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

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Abstract

In the title molecule, $C_{21}H_{17}FN_2O_3$, the five-membered ring adopts an envelope conformation, folded between sites 2 and 5, with a flap angle of 42.0°. This configuration places the three substituents in axial positions with the fluorophenyl group attached to the N atoms *trans* to the other two aromatic rings.

Comment

The structure determination reported here was carried out as part of a detailed investigation into 1,3-dipolar cycloadditions of fluorine-containing arylnitrones to alkenes to afford isoxazolidines (DuBoisson, 1986). Such additions involving nitrones of type ArCH=N$^+$(O$^-$)C$_6$H$_4$F-*p* (1) (Ar = C$_6$H$_5$, 4-HOC$_6$H$_4$, 4-MeOC$_6$H$_4$) gave mixtures of (*E*)- and (*Z*)-isoxazolidines which are oils. In order to determine which isomer was formed as the major product in these cycloadditions, the reaction with the nitrone (1*a*) (Ar = 3-O$_2$NC$_6$H$_4$) was carried out. This gave a solid mixture of the (*E*)- and (*Z*)-isoxazolidines, (2), from which the major isomer was obtained on crystallization and identified as the (*Z*)-isomer, (2*a*).

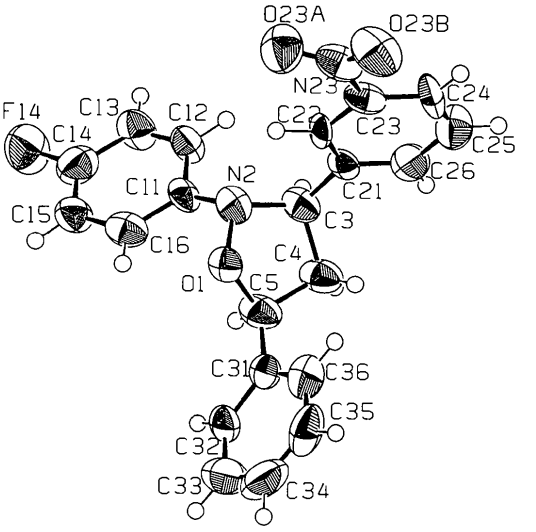
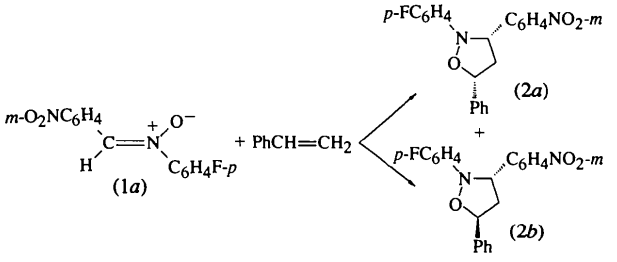


Fig. 1. The title molecule, including atomic numbering scheme, drawn using *ORTEP*II (Johnson, 1976). Displacement ellipsoids are plotted at the 50% probability level.

Experimental

A mixture of *N*-(4-fluorophenyl)-*C*-(3-nitrophenyl)nitrone (1*a*) (1.30 g, 5.00 mmol), toluene (20 ml) and styrene (0.52 g, 5.00 mol) was heated under reflux for 4 h. The solvent was removed from the hot mixture under reduced pressure and the residue was stored at 263 K for 3 d. Trituration of the resulting solid with light petroleum (b.p. 313–333 K) gave a pale buff, amorphous solid which was identified as a mixture of (*E*)- and (*Z*)-2-(4-fluorophenyl)-3-(3-nitrophenyl)-5-phenylisoxazolidine, (2) (ratio 8:94, ^{19}F NMR) (1.75 g, 4.81 mmol, 96%; found C 69.4, H 4.7, F 5.1, N 7.7%, M^+ 364: $C_{21}H_{17}FN_2O_3$ requires C 69.2, H 4.7, F 5.2, N 7.7%, *M* 364; m.p. 378–380 K). The major isomer was isolated by fractional crystallization (CCl_4/light petroleum 1:1 *v*/*v*) and identified as (*Z*)-2-(4-fluorophenyl)-3-(3-nitrophenyl)-5-phenylisoxazolidine, (2*a*) (found: C 69.2, H 4.7, F 5.2, N 7.6%, M^+ 364; m.p. 383–384 K).



Crystal data

$C_{21}H_{17}FN_2O_3$
M_r = 364.38
Monoclinic
$P2_1$
a = 5.556 (2) Å
b = 15.332 (4) Å
c = 10.427 (4) Å
β = 90.47 (2)°

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 20 reflections
θ = 6.00–12.50°
μ = 0.0929 mm^{-1}
T = 296 K
Needle

$V = 888\,(1)$ Å^3
$Z = 2$
$D_x = 1.362$ Mg m^{-3}
0.5 × 0.1 × 0.05 mm
Colourless
Crystal source: synthesized at UMIST

Data collection

CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1887 measured reflections
1638 independent reflections
856 observed reflections $[F > 3\sigma(F)]$
$R_{int} = 1.36$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 18$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 150 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.0617$
$wR = 0.0701$
$S = 1.649$
856 reflections
244 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max} = 0.0775$
$\Delta\rho_{max} = 0.179$ e Å^{-3}
$\Delta\rho_{min} = -0.191$ e Å^{-3}
Extinction correction: Zachariasen type 2 Gaussian isotropic
Extinction coefficient: $38\,(1) \times 10^{-7}$
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	0.719 (1)	0.6692	0.5323 (6)	0.051 (3)
N2	0.890 (2)	0.7282 (7)	0.4764 (8)	0.058 (5)
C3	1.111 (2)	0.7185 (9)	0.553 (1)	0.059 (6)
C4	1.096 (2)	0.6271 (8)	0.614 (1)	0.071 (7)
C5	0.863 (2)	0.5898 (8)	0.559 (1)	0.069 (7)
C11	0.899 (2)	0.7191 (8)	0.343 (1)	0.051 (5)
C12	1.079 (2)	0.7620 (9)	0.271 (1)	0.060 (6)
C13	1.076 (2)	0.7613 (10)	0.141 (1)	0.068 (7)
C14	0.886 (3)	0.7182 (10)	0.075 (1)	0.076 (7)
C15	0.720 (2)	0.6748 (9)	0.140 (1)	0.064 (6)
C16	0.721 (2)	0.6765 (9)	0.275 (1)	0.064 (6)
C21	1.143 (2)	0.7896 (8)	0.6487 (10)	0.045 (5)
C22	0.978 (2)	0.8575 (8)	0.6619 (9)	0.048 (5)
C23	1.014 (2)	0.9175 (8)	0.760 (1)	0.058 (6)
C24	1.209 (2)	0.9144 (10)	0.844 (1)	0.067 (7)
C25	1.375 (2)	0.8497 (10)	0.829 (1)	0.075 (7)
C26	1.341 (2)	0.7858 (9)	0.732 (1)	0.069 (7)
C31	0.706 (2)	0.5343 (8)	0.643 (1)	0.057 (6)
C32	0.594 (2)	0.4629 (9)	0.588 (1)	0.061 (6)
C33	0.432 (3)	0.4165 (10)	0.662 (2)	0.088 (9)
C34	0.392 (3)	0.436 (1)	0.783 (2)	0.10 (1)
C35	0.510 (3)	0.504 (1)	0.839 (1)	0.089 (8)
C36	0.661 (3)	0.553 (1)	0.769 (1)	0.086 (9)
F14	0.884 (1)	0.7200 (7)	−0.0539 (7)	0.106 (5)
N23	0.841 (2)	0.9878 (8)	0.769 (1)	0.070 (6)
O23A	0.712 (2)	1.0065 (8)	0.678 (1)	0.097 (6)
O23B	0.823 (2)	1.0234 (8)	0.873 (1)	0.100 (6)

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

O1—N2	1.44 (1)	C3—C4	1.54 (2)
O1—C5	1.49 (1)	C3—C21	1.49 (1)
N2—C3	1.47 (1)	C4—C5	1.52 (2)
N2—C11	1.40 (1)	C5—C31	1.50 (2)
N2—O1—C5	103.4 (7)	N2—C3—C4	105.6 (9)
O1—N2—C3	105.7 (7)	C3—C4—C5	104 (1)
O1—N2—C11	111.6 (8)	O1—C5—C4	102.8 (8)
C3—N2—C11	119.8 (9)		

Data collection: *CAD-4 Diffractometer Control Software* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Diffractometer Control Software*. Data reduction: *PROCESS TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *LS TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *FINISH TEXSAN*. Literature search: *CSSR* (1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

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 Manual*. Enraf–Nonius, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEPII*. 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). *SHELXS86*. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

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4-(9,10-Dihydro-4*H*-benzo[4,5]cyclohepta[1,2-*b*]thien-4-ylidene)-1-methylpiperidinium Hydrogen Malate†

Oswald M. Peeters, Norbert M. Blaton and Camiel J. De Ranter

Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Faculteit Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium

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Abstract

The title compound, $C_{19}H_{22}NS^+.C_4H_5O_5^-$, is a drug used in the treatment of migraine. The tricyclic moiety is asymmetrically folded with a dihedral angle of

† Internal code of the Janssen Research Foundation: R21448.