Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
2432 measured reflections
1726 independent reflections
1163 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 25.02°$
$h = -9 \rightarrow 0$
$k = -13 \rightarrow 3$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.143$
$S = 1.018$
1726 reflections
142 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.5865P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.232$ e Å^{-3}
$\Delta\rho_{\min} = -0.199$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C5	1.364 (3)	C2—C3	1.387 (4)
N1—C1	1.367 (3)	C3—C4	1.385 (4)
C1—C6	1.405 (4)	C4—C5	1.385 (4)
C1—C2	1.418 (3)		
C5—N1—C1	126.3 (2)	C4—C3—C2	123.1 (2)
N1—C1—C2	114.5 (2)	C5—C4—C3	116.9 (2)
C3—C2—C1	119.9 (2)	N1—C5—C4	119.1 (3)

For the H-atom treatment, H1 was refined freely, the methyl H atoms were kept rigid and H3 was refined as riding.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

We thank Professor L. Ernst, Dr H.-M. Schiebel and assistants for recording NMR and mass spectra, the Deutsche Forschungsgemeinschaft for supporting our collaboration, the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauf for technical assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1411). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
Bernstein, J. (1988). *Acta Cryst.* C**44**, 900–902.
Dupont, L., Masereel, B., de Tullio, P., Pirotte, B. & Delarge, J. (1995). *Acta Cryst.* C**51**, 505–507.
Elgemeie, G. E. H., Alnaimi, I. S. & Alarab, H. F. (1992). *Heterocycles*, **34**, 1721–1728.
Elgemeie, G. E. H., Elzanate, A. M. & Mansour, A. K. (1992). *J. Chem. Soc. Perkin Trans.* 1, pp. 1073–1074.
Elgemeie, G. E. H. & Hussain, B. A. W. (1994). *Tetrahedron*, **50**, 199–204.
Elgemeie, G. E. H., Regaila, H. H. & Shehata, N. (1990). *J. Chem. Soc. Perkin Trans.* 1, pp. 1267–1270.
Gangjee, A., Vasudevan, A., Queener, S. F. & Kisliuk, R. L. (1996). *J. Med. Chem.* **39**, 1438–1446.
Newkome, G. R., Joo, Y. J., Evans, D. W., Pappalardo, S. & Fronczek, F. R. (1988). *J. Org. Chem.* **53**, 786–790.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1994). *XP. Molecular Graphics Program.* Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Stoe & Cie (1992*a*). *DIF4. Diffractometer Control Program.* Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1992*b*). *REDU4. Data Reduction Program.* Stoe & Cie, Darmstadt, Germany.
Taylor, E. C., Hamby, J. M., Shih, C., Grindey, G. B., Rinzel, S. M., Beardsley, P. G. & Moran, R. G. (1989). *J. Med. Chem.* **32**, 1517–1522.

Acta Cryst. (1998). C**54**, 822–824

2-Methyl-2-(10-phenoxazinyl)propiononitrile

Didier Gigmes,[a] Didier Siri,[a] Jean-Pierre Reboul,[b] Nourredine Redouane,[a] Paul Tordo[a] and Gerard Pèpe[c]

[a]*UMR 6517-CNRS Université d'Aix-Marseille I et III, Av. Esc. Normandie Niemen, Case 521, 13397 Marseille CEDEX 20, France,* [b]*Groupe d'Enseignement et de Recherche en Chimie Thérapeutique, Organique et Physique, Faculté de Pharmacie, 27 boulevard Jean-Moulin, 13385 Marseille CEDEX 5, France, and* [c]*Centre de Recherches sur les Mécanismes de la Croissance Cristalline, Université d'Aix-Marseille III, Campus Luminy, Case 913, 13288 Marseille CEDEX 2, France. E-mail: siri@srepir2.univ-mrs.fr*

(*Received 27 May 1997; accepted 15 December 1997*)

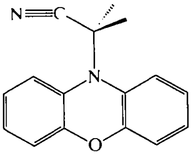
Abstract

The title compound, $C_{16}H_{14}N_2O$, adopts a folded conformation, with a dihedral angle of 145.0 (1)° between the phenyl-ring mean planes, similar to other molecules belonging to the same family.

Comment

Phenoxazine derivatives have been studied particularly for their therapeutic activities (Ionescu & Mantsch, 1967; Mylari *et al.*, 1990; Palmer *et al.*, 1988). Besides their potential pharmacological applications, phenoxazine derivatives have also been used for their antioxidant and stabilizant actions (Fukuzumi *et al.*,

1976) for various substrates. Related to one of our research programs dealing with acrylic monomer stabilization by heterocyclic compounds, the title compound, (I), was obtained during studies of the reactivity of phenoxazine with free radicals.



(I)

The conformation of the tricycle is folded, with an angle of 145.0 (1)° between the phenyl groups (C1–C6 and C9, C8, C11–C14). It is interesting to note that this value is equivalent to the value observed in phenothiazines of 142 (10)° [average value calculated from 69 selected phenothiazines found in the Cambridge Structural Database (Allen & Kennard, 1993)]. The title molecule is quasi-symmetric with respect to a plane orthogonal to the tricycle and passing through atoms O7 and N10. The non-perfect symmetry is induced by the electrostatic repulsion between heteroatoms O7 and N19. The 1-cyano-1-methylethyl group is rotated by 7° around the N10—C15 bond with respect to the symmetric position.

The stability of the crystal is essentially governed by van der Waals interactions in order to optimize the intermolecular contacts between the phenyl groups.

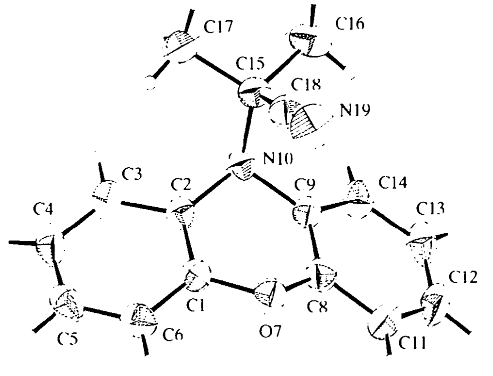


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with displacement ellipsoids of 50% probability.

Experimental

The title compound was obtained by reaction of 2,2′-azobis-(isobutyronitrile) (0.5 equivalents) with phenoxazine over a period of 48 h at 333 K in benzene solution. Chromatography of the crude product on silica gel, eluting with a 1:5 mixture of diethyl ether and pentane, gave the title compound in 30% yield. The compound was recrystallized as colourless parallelepipedic crystals from acetone solution.

Crystal data

$C_{16}H_{14}N_2O$
$M_r = 250.29$
Monoclinic
$C2/c$
$a = 25.281 (4)$ Å
$b = 6.741 (1)$ Å
$c = 15.559 (3)$ Å
$\beta = 101.92 (3)°$
$V = 2594.4 (8)$ Å^3
$Z = 8$
$D_x = 1.282$ Mg m^{-3}
$D_m = 1.29 (2)$ Mg m^{-3}
D_m measured by flotation in benzene/chloroform

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–14°$
$\mu = 0.082$ mm^{-1}
$T = 293 (2)$ K
Parallelepiped
0.42 × 0.38 × 0.23 mm
Colourless

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3587 measured reflections
3587 independent reflections
2523 reflections with $I > 2\sigma(I)$

$\theta_{max} = 30.16°$
$h = -34 \rightarrow 34$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0440$
$wR(F^2) = 0.0896$
$S = 0.961$
3587 reflections
214 parameters
H atoms refined with $U = 1.2U_{eq}$(bonded atom)

$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.211$ e Å^{-3}
$\Delta\rho_{min} = -0.153$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—C2	1.3760 (15)	C14—H14	0.889 (14)
C1—O7	1.3953 (13)	C15—C18	1.489 (2)
C2—N10	1.4440 (13)	C15—C17	1.531 (2)
O7—C8	1.3862 (13)	C17—H173	0.90 (2)
C8—C9	1.3837 (15)	C18—N19	1.143 (2)
C9—N10	1.4425 (12)		
C2—C1—O7	120.72 (9)	N10—C15—C17	111.19 (9)
C8—O7—C1	113.41 (8)	C18—C15—C16	108.04 (10)
C8—C9—N10	120.51 (9)	N10—C15—C16	109.66 (9)
C9—N10—C2	109.04 (8)	C17—C15—C16	108.30 (11)
C18—C15—N10	110.22 (9)	N19—C18—C15	179.40 (12)
C18—C15—C17	109.34 (10)		

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *DATARED* (Pèpe, 1979). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1513). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.

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

Fukuzumi, K., Ikeda, N. & Egawa, M. (1976). *J. Am. Oil Chem. Soc.* **53**, 623–627.

Ionescu, M. & Mantsch, H. (1967). *Adv. Heterocycl. Chem.* **8**, 83–113.

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

Mylari, B. L., Carty, T. J., Moore, P. F. & Zembrowski, W. J. (1990). *J. Med. Chem.* **33**, 2019–2024.

Palmer, B. D., Rewcastle, G. W., Atwell, G. J., Baguley, B. C. & Denny, W. A. (1988). *J. Med. Chem.* **31**, 707–712.

Pèpe, G. (1979). *DATARED. Programs for X-ray Diffraction Data Reduction.* CRMC2–CNRS, Marseille, France.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Acta Cryst. (1998). C**54**, 824–825

5′-[2-(*N*-*tert*-Butylethanal nitrone)]-1,3,3-trimethylspiro[indoline-2,3′-naphtho[2,1-*b*][1,4]oxazine]†

Barbara Luccioni-Houzé,[a] Gerard Giusti[a] and Gérard Pèpe[b]

[a]*LCMOM, Université d'Aix-Marseille II, Campus de Luminy, Case 901, 13288 Marseille CEDEX 9, France, and* [b]*Centre de Recherche sur les Mécanismes de la Croissance Cristalline, Universités d'Aix-Marseille II et III, Campus de Luminy, Case 913, 13288 Marseille CEDEX 9, France. E-mail: genmol@crmc2.univ-mrs.fr*

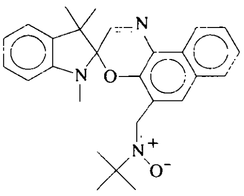
(*Received 19 June 1997; accepted 1 October 1997*)

Abstract

The title compound, $C_{27}H_{29}N_3O_2$, is photochromic with spin-trapping properties.

Comment

Photochromic compounds are able to undergo light-induced reversible colour changes (Bertelson, 1971). One of the major problems encountered in their use is their irreversible degradation (Baillet *et al.*, 1993), probably involving radical species. In order to increase the life of these compounds, a spin trap (nitrone) is added to the molecule (Campredon *et al.*, 1997).



(1)

All geometric parameters of the title compound, (1), correspond to the expected values. The orientation of the nitrone with respect to the phenyl ring is characterized by the torsion angle C8—C9—C11—N12 [168.6 (2)°].

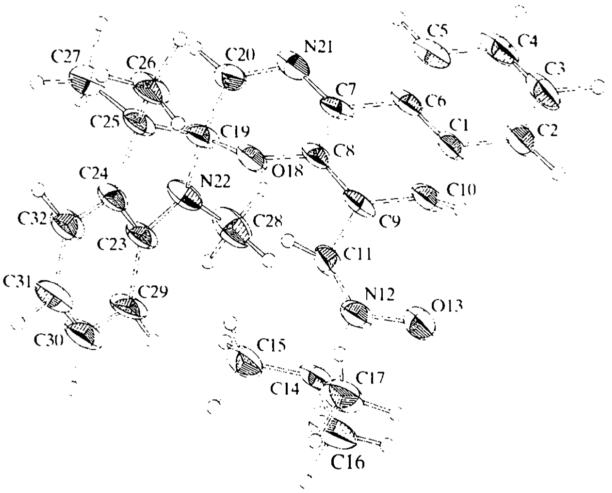


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1) with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as small circles of arbitrary radii.

Experimental

Nitrone (1) was prepared (Campredon *et al.*, 1997) by oxidation of the corresponding hydroxymethyl derivative with 1,1,1-triacetoxy-1,1-dihydro-1,2-benzodioxol-3(1*H*)-one according to the Dess–Martin procedure (Dess & Martin, 1983) followed by condensation of the resulting aldehyde with *tert*-butylhydroxylamine (yield 41%). Colourless pseudo-hexagonal prismatic crystals were obtained by dissolving (1) in CH_2Cl_2 and saturating this solution with pentane to give a 5:95 mixed solvent.

Crystal data

$C_{27}H_{29}N_3O_2$
$M_r = 427.53$
Monoclinic
$P2_1/c$
$a = 8.580 (1)$ Å
$b = 13.901 (2)$ Å
$c = 19.977 (3)$ Å
$\beta = 98.57 (2)°$
$V = 2356.1 (6)$ Å^3
$Z = 4$
$D_x = 1.205$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 9–15°$
$\mu = 0.077$ mm^{-1}
$T = 293 (2)$ K
Pseudo-hexagonal prism
0.5 × 0.3 × 0.3 mm
Colourless

---

† Alternative name: spiro[1,3,3-trimethylindoline-2,3′-naphtho[2,1-*b*]-[1,4]oxazine]-5′-methylidene-*tert*-butylamine *N*-oxide.