rules. Intensities for the structure determination and refinement were collected from the Guinier photographs in 0.01° steps using a Johannson LS18 line scanner. The structure was solved by two different methods, namely a grid search procedure (Chernyshev & Schenk, 1998) and the Patterson method used in *DIRDIF*96 (Beurskens *et al.*, 1996), on the basis of 439 intensities derived from the powder pattern with *MRIA* (Zlokazov & Chernyshev, 1992). The diffraction profile and the difference between the measured and calculated profiles after the final bond-restrained Rietveld refinement are shown in Fig. 2. All non-H atoms were refined isotropically. H atoms were placed in geometrically calculated positions and allowed to refine using bond restraints, with a common isotropic displacement parameter $U_{\rm iso}$ fixed to 0.05 Å^2. March–Dollase texture formalism (Dollase, 1986) with various directions of preferred orientation had been applied; however, the specimen turned out to be texture-free. The observed anisotropy of diffraction-line broadening was approximated by a quartic form in *hkl* (Popa, 1998).

Data collection: Johannson LS18 line-scanner data collection program. Cell refinement: *LSPAID*. Data reduction: *Profit for Windows* (Philips, 1996). Program(s) used to solve structure: *MRIA* and *DIRDIF*96. Program(s) used to refine structure: *MRIA*. Molecular graphics: *PLUTON* (Spek, 1992). Software used to prepare material for publication: *MRIA*, *SHELXL*93 (Sheldrick, 1993) and *PARST* (Nardelli, 1983).

This work has been supported by the Netherlands Organization for Scientific Research, NWO (VVC, AVY and VAT) and the Bundesministerium Forschung und Technologie, Bonn, Germany, project ⟨⟨Transform⟩⟩ 01 KX9812 (VAM).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1236). Services for accessing these data are described at the back of the journal.

References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., García-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1996). *The DIRDIF96 Program System*. Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

Chernyshev, V. V. & Schenk, H. (1998). *Z. Kristallogr.* **213**, 1–3.

Dollase, W. A. (1986). *J. Appl. Cryst.* **19**, 267–272.

Karapetyan, H. A., Struchkov, Yu. T., Andrianov, V. G., Zakharova, G. N., Dvoryantseva, G. G. & Chernov, V. A. (1981). *Cryst. Struct. Commun.* **10**, 271–279.

Makarov, V. A. (1996). 211th Am. Chem. Soc. Natl Meet., New Orleans, March 24–28. *Division of Medicinal Chemistry*, Abstracts, p. 177.

Makarov, V. A., Schmidtke, V. G. & Granik, V. G. (1999). 12th Int. Conf. Antivir. Res., Jerusalem, March 26–29. Abstracts, p. 125.

Makarov, V. A., Sedov, A. L., Nemeriuk, M. P. & Safonova, T. S. (1994). *Chem. Heterocycl. Comput.* pp. 971–976.

Mikhalev, V. A., Chernov, V. A., Dorokhova, M. I., Smolina, N. E. & Tikhonova, O. Ya. (1972). *Khim. Farm. Zh.* **6**, 9–12.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Philips (1996). *PROFIT for Windows*. Release 1.0b. Philips Analytical X-ray BV, Almelo, The Netherlands.

Popa, N. C. (1998). *J. Appl. Cryst.* **31**, 176–180.

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

Spek, A. L. (1992). *PLUTON92. Molecular Graphics Program*. University of Utrecht, The Netherlands.

Visser, J. W. (1969). *J. Appl. Cryst.* **2**, 89–95.

Visser, J. W. (1986). *Powder Diffr.* **1**, 66–76.

Zlokazov, V. B. & Chernyshev, V. V. (1992). *J. Appl. Cryst.* **25**, 447–451.

Acta Cryst. (1999). C**55**, 1101–1103

6-(2-Nitroprop-2-yl)quinoline-5-carbonitrile

JÁN LOKAJ,[a] VIKTOR KETTMANN,[b] VLADIMÍR MACHÁČEK[c] AND ALEŠ HALAMA[d]

[a]*Faculty of Chemical Technology, Slovak Technical University, Radlinskeho 9, Bratislava, Slovakia 81237,* [b]*Faculty of Pharmacy, Comenius University, Odbojarov 10, Bratislava, Slovakia 83232,* [c]*Faculty of Chemical Technology, University of Pardubice, Nám. Českoslov. légií 565, Pardubice, Czech Republic 53210, and* [d]*Research Institute of Pharmacy and Biochemistry, Výskumná 421, Pardubice-Rosice, Czech Republic 53351.*
E-mail: kettmann@fpharm.uniba.sk

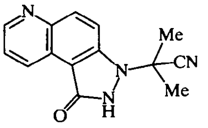
(*Received 26 August 1998; accepted 15 February 1999*)

Abstract

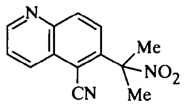
The title compound, $C_{13}H_{11}N_3O_2$, was unexpectedly formed as a minor product from the reaction of 6-nitroquinoline with 2-nitropropane and potassium cyanide, and was selected for crystal structure determination in order to clarify its molecular structure unambiguously. The bonding pattern of the heterocyclic ring agrees with that found in other quinoline derivatives. The nitro group of the 2-nitropropyl substituent is oriented *cis* with respect to the aromatic ring. The principal packing interaction seems to be π–π stacking of the heterocyclic rings.

Comment

As part of our on-going studies on quinoline derivatives, we attempted to prepare, by treatment of 6-nitroquinoline with 2-nitropropane and potassium cyanide, the corresponding pyrrolo-quinoline derivative, (1). However, analytical data from the reaction mixture indicated, besides (1) as the main product, the presence of another compound whose structure could not be resolved unequivocally from spectroscopic methods (Macháček & Halama, 1999). This minor product is the title compound, (2), and in order to identify the structure of (2), and hence to help in clarifying the reaction mechanism, its crystal structure was determined by X-ray diffraction. Another reason for the interest in (2) was to obtain precise structural data for the future study of the relationship between the structural and electronic properties of quinoline derivatives.



(1)



(2)

The molecular structure of (2) is shown in Fig. 1. As expected, the quinoline ring is planar within experimental error [r.m.s. deviation 0.011 (3) Å] and atoms C11 and C13 are displaced by 0.035 (3) and 0.071 (3) Å, respectively, on the same side of this ring plane. The distribution of aromatic bond lengths is in good agreement with that calculated for quinoline (Dewar & Gleicher, 1966) and with those observed in 2,3′- and 2,6′-biquinolines (Chiang *et al.*, 1991). As revealed by a search of the Cambridge Structural Database (Allen *et al.*, 1983), a similar pattern of bond lengths and angles has also been observed in other quinoline derivatives containing substituents not involved in conjugation with the aromatic system. The 2-nitropropyl substituent is rotated on the C6—C13 bond in such a manner that, with respect to C5—C6, one of the methyl groups (C15) is antiperiplanar, while the arrangement of the nitro group is +synclinal [C5—C6—C13—N16 torsion angle = 53.0 (2)°].

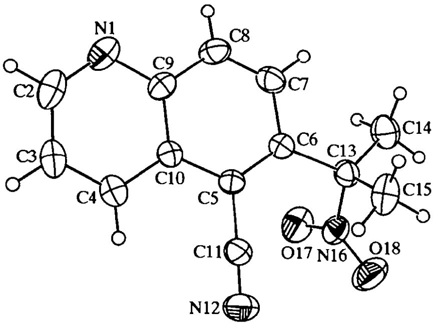


Fig. 1. *ORTEP*II view (Johnson, 1976) of the title molecule, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 35% probability level and H atoms are drawn as small spheres of an arbitrary radius.

The crystal packing is dominated by stacks of centrosymmetrically related molecules, the stacking axes being parallel to the *a* axis and passing through $(0,0,\frac{1}{2})$ and $(0,\frac{1}{2},0)$. As the molecules lie approximately in the (100) plane, the stacking separation is *ca* 3.421 Å (**a**/2). The overlapping geometry of the adjacent molecules along the stack is in line with the model of π–π interactions presented by Hunter & Saunders (1990). The stacks are packed by electrostatic and van der Waals interactions.

Experimental

The title compound was obtained as a minor product from the reaction of a mixture of 6-nitroquinoline, 2-nitropropane and potassium cyanide in dimethyl sulfoxide. Details of the synthetic work will be published elsewhere (Macháček & Halama, 1999). Crystals were obtained by recrystallization from chloroform.

Crystal data

$C_{13}H_{11}N_3O_2$
$M_r = 241.25$
Monoclinic
$P2_1/c$
$a = 6.842\ (2)$ Å
$b = 12.156\ (3)$ Å
$c = 14.184\ (3)$ Å
$\beta = 92.32\ (3)°$
$V = 1178.7\ (5)$ Å^3
$Z = 4$
$D_x = 1.359$ Mg m^{-3}
$D_m = 1.36\ (1)$ Mg m^{-3}
D_m measured by flotation in bromoform–hexane
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 15 reflections
$\theta = 8$–$19°$
$\mu = 0.095$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.40 × 0.25 × 0.20 mm
Colourless

Data collection

Syntex $P2_1$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2954 measured reflections
2715 independent reflections
1092 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 27.54°$
$h = -8 \rightarrow 0$
$k = 0 \rightarrow 15$
$l = -18 \rightarrow 18$
2 standard reflections frequency: 100 min intensity decay: none

Refinement

Refinement on F^2
$R = 0.047$
$wR = 0.094$
$S = 1.125$
2715 reflections
164 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.003$
$\Delta\rho_{\max} = 0.282$ e Å^{-3}
$\Delta\rho_{\min} = -0.221$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.030 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C2	1.307 (3)	C6—C13	1.525 (3)
N1—C9	1.362 (2)	C7—C8	1.350 (3)
C2—C3	1.386 (3)	C8—C9	1.395 (3)
C3—C4	1.359 (3)	C9—C10	1.404 (3)
C4—C10	1.407 (3)	C11—N12	1.130 (2)
C5—C6	1.374 (3)	C13—N16	1.541 (3)
C5—C10	1.422 (2)	N16—O18	1.205 (2)
C5—C11	1.441 (3)	N16—O17	1.209 (2)
C6—C7	1.411 (3)		
C2—N1—C9	116.6 (2)	N12—C11—C5	178.0 (2)
C5—C6—C13—C15	−66.6 (2)	C5—C6—C13—N16	53.0 (2)
C5—C6—C13—C14	167.1 (2)	C6—C13—N16—O17	41.9 (2)

All H atoms were located in a difference map and fixed at these positions, with $U_{\rm iso}$ set to $1.2U_{\rm eq}$ of the associated atom.

Data collection: $P2_1$ diffractometer software. Cell refinement: $P2_1$ diffractometer software. Data reduction: *XP*21 (Pavelčík, 1987). 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: GS1022). Services for accessing these data are described at the back of the journal.

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.

Chiang, L. Y., Swirczewski, J. W., Kastrup, R., Hsu, C. S. & Upasani, R. B. (1991). *J. Am. Chem. Soc.* **113**, 6574–6584.

Dewar, M. J. S. & Gleicher, G. J. (1966). *J. Chem. Phys.* **44**, 759–773.

Hunter, C. A. & Saunders, J. K. M. (1990). *J. Am. Chem. Soc.* **112**, 5525–5534.

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

Macháček, V. & Halama, A. (1999). *J. Chem. Soc. Perkin Trans.* Submitted.

Pavelčík, F. (1987). *XP*21. *Program for Syntex* $P2_1$ *Data Reduction.* Comenius University, Bratislava, Slovakia.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures.* University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Acta Cryst. (1999). C**55**, 1103-1105

4,6,7,8,8a,9-Hexahydrothieno[3,2-*f*]indolizine-6,9-dione

JÁN LOKAJ,[a] VIKTOR KETTMANN[b] AND STEPHAN MARCHALIN[a]

[a]*Faculty of Chemical Technology, Slovak Technical University, Radlinskeho 9, Bratislava, Slovakia 81237, and* [b]*Faculty of Pharmacy, Comenius University, Odbojarov 10, Bratislava, Slovakia 83232. E-mail: kettmann@fpharm.uniba.sk*

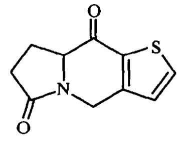
(*Received 18 August 1998; accepted 20 January 1999*)

Abstract

The title compound, $C_{10}H_9NO_2S$, belongs to a series of new heterocyclic compounds and was selected for structure determination in order to study some aspects of the relationship between conformational and electronic properties. The conformation of the individual rings and the bonding pattern of the molecule are analysed in detail and compared with those of analogous compounds.

Comment

As part of our on-going study of the relationship between the molecular and electronic structures of new heterocyclic compounds, we report here on the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The ring system of the molecule consists of an oxo-substituted tetrahydrothienopyridine moiety joined to an oxo-pyrrolidine ring. The thiophene ring is exactly planar, with an average deviation of the ring atoms from the least-squares plane of 0.006 (2) Å. Bond lengths and angles within the thiophene ring agree well with the corresponding values in other compounds containing this molecular fragment (Gilmore *et al.*, 1983; Bak *et al.*, 1961).

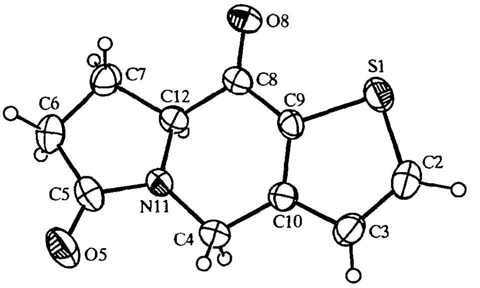


Fig. 1. *ORTEP*II (Johnson, 1976) view of the title molecule, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 35% probability level and H atoms are drawn as small spheres of an arbitrary radius.

The central six-membered ring is not planar. A calculation of the least-squares planes has shown that this ring is puckered in such a manner that the four atoms C8, C9, C10 and C4 are planar to within 0.010 (3) Å, while atoms N11 and C12 are unequally displaced from this plane on opposite sides, with out-of-plane displacements of −0.356 (2) and 0.168 (2) Å, respectively. Using the terminology of Cremer & Pople (1975), the conformation of the ring can be described as intermediate between an envelope form, ^{N}E, with N11 as the out-of-plane atom, and a half-chair form, $^{12}H_N$, with C12 and N11 as the out-of-plane atoms [Q =

Acta Crystallographica Section C
ISSN 0108-2701 © 1999