Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

10-Ethyl-3-(4-methoxyphenyl)-1,2,4-triazolo[4′,3′:2,3]-1,2,4-triazino[5,6-*b*]indole

Mohammed A. E. Shaban *et al.*

Electronic paper
This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

10-Ethyl-3-(4-methoxyphenyl)-1,2,4-triazolo[4′,3′:2,3]-1,2,4-triazino-[5,6-*b*]indole

Mohammed A. E. Shaban,[a] Ali Morgaan,[a] Hyungphil Chun[b] and Ivan Bernal[b]*

[a]Department of Chemistry, Faculty of Science, Alexandria University, PO Box 426 Ibrahimia, Alexandia 21321, Egypt, and [b]Chemistry Department, University of Houston, Texas 77204-5641, USA
Correspondence e-mail: ibernal@uh.edu

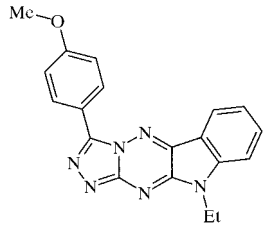
Received 1 August 2000
Accepted 29 August 2000

Data validation number: IUC0000235

Oxidative cyclization of 5-ethyl-3-(4-methoxybenzylidene)-hydrazino-1,2,4-triazino[5,6-*b*]indole gave the linearly annelated title compound, $C_{19}H_{16}N_6O$. The skeleton is approximately planar, except for the ethyl group.

Comment

Valuable medicinal applications and biological activities are known to be associated with the 1,2,4-triazolo-1,2,4-triazino[5,6-*b*]indole structure (Katz & Margalith, 1984). One of the synthetic pathways to obtain the 10-substituted derivatives of these compounds is the oxidative cyclization of 5-substituted-3-alkylidene- and 3-arylidenehydrazino-1,2,4-triazino[5,6-*b*]indoles (Shaban *et al.*, 1993, 1999, 2000; Rashed *et al.*, 1998). Assignment of structures to the obtained products was inconclusive whether they are the linearly annelated 1,2,4-triazolo[4′,3′:2,3]-1,2,4-triazino[5,6-*b*]indoles such as the title compound, (I) (Shaban *et al.*, 1993, 1999, 2000), or the angular-annelated 1,2,4-triazolo[3′,4′:3,4]-1,2,4-triazino[5,6-*b*]indole regioisomers, (II) (Holla & Udupa, 1990; Rashed *et al.*, 1998). The present work has been initiated to settle this contradiction. The title compound, (I), was prepared by oxidative cyclization of 3-(4-methoxybenzylidene)hydrazino-1,2,4-triazino[5,6-*b*]indole with 10% ethanolic iron(III) chloride, thionyl chloride or 30% aqueous sodium hypochlorite in dioxane. The determined molecular structure revealed that the four-ring system corresponds to the linearly annelated structure (I) and not the angularly annelated structure (II). The skeleton is approximately planar except for the ethyl group. Bond distances showed localization of the N2—C8 and N3—C7 π bonds of the 1,2,4-trizole ring, as well as N5—C9 and N6—C10 π bonds of the 1,2,4-triazine ring.



(I)

Experimental

The title compound can be synthesized in three different ways (*a*–*c*): (*a*) a suspension of 5-ethyl-3-(4-methoxybenzylidene)hydrazino-1,2,4-triazino[5,6-*b*]indole (0.664 g, 2 mmol) in toluene (30 ml) was treated with 10% ethanolic iron(III) chloride solution (20 ml) and heated at reflux for 3 h. The mixture was evaporated to dryness and the obtained orange residue was triturated with water, filtered, washed with water, dried and crystallized from ethanol (yield 39%, m.p. 585 K). (*b*) A mixture of the aforementioned hydrazone (0,664 g, 2 mmol) and thionyl chloride (25 ml) was heated at reflux for 5 h. The mixture was evaporated and the obtained residue was crystallized from ethanol (yield 45%, m.p. 585 K). (*c*) A suspension of the same aforementioned hydrazone (0.664 g, 2 mmol) in dioxane (5 ml) was treated with 30% aqueous sodium hypochlorite solution (15 ml) and heated at 373 K for 10 min. After attaining ambient temperature, the product was filtered, washed with water, dried, and crystallized from ethanol (yield 52%, m.p. 585 K).

Crystal data

$C_{19}H_{16}N_6O$	$Z = 2$
$M_r = 344.38$	$D_x = 1.371$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.420$ (3) Å	Cell parameters from 25 reflections
$b = 9.805$ (4) Å	$\theta = 2.97$–$16.45°$
$c = 11.228$ (6) Å	$\mu = 0.091$ mm^{-1}
$\alpha = 114.55$ (4)°	$T = 293$ K
$\beta = 114.67$ (4)°	Plate, orange
$\gamma = 91.03$ (3)°	0.50 × 0.31 × 0.07 mm
$V = 834.0$ (8) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	1005 reflections with $I > 2\sigma(I)$
ω–2θ scans	$\theta_{max} = 24.97°$
Absorption correction: ψ scan (*NRCVAX ABSORP*; Gabe *et al.*, 1989)	$h = -11 \rightarrow 11$
	$k = 0 \rightarrow 11$
$T_{min} = 0.777$, $T_{max} = 0.996$	$l = -13 \rightarrow 13$
2935 measured reflections	3 standard reflections
2935 independent reflections	frequency: 240 min
	intensity decay: 1.27%

Refinement

Refinement on F^2	H-atom parameters constrained
$R(F) = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.0706P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.115$	$(\Delta/\sigma)_{max} = 0.005$
$S = 1.223$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
2935 reflections	$\Delta\rho_{min} = -0.24$ e Å^{-3}
235 parameters	

Table 1
Selected geometric parameters (Å, °).

O1—C14	1.363 (4)	N4—C10	1.384 (4)
O1—C17	1.405 (4)	N4—C9	1.394 (4)
N1—C8	1.357 (4)	N5—C9	1.311 (4)
N1—C1	1.396 (4)	N5—N6	1.398 (4)
N1—C18	1.485 (4)	N6—C10	1.298 (4)
N2—C8	1.311 (4)	C1—C6	1.401 (4)
N2—C9	1.368 (4)	C6—C7	1.439 (4)
N3—C7	1.307 (4)	C7—C8	1.442 (4)
N3—N4	1.368 (4)	C10—C11	1.461 (5)
C14—O1—C17	118.3 (3)	C1—C6—C7	105.4 (3)
C8—N1—C1	108.5 (3)	N3—C7—C6	129.7 (3)
C8—N1—C18	125.0 (3)	N3—C7—C8	123.2 (3)
C1—N1—C18	126.6 (3)	C6—C7—C8	107.0 (3)
C8—N2—C9	111.6 (3)	N2—C8—N1	125.9 (3)
C7—N3—N4	111.6 (3)	N2—C8—C7	125.6 (3)
N3—N4—C10	129.3 (3)	N1—C8—C7	108.5 (3)
N3—N4—C9	125.3 (3)	N5—C9—N2	127.0 (4)
C10—N4—C9	105.3 (3)	N5—C9—N4	110.4 (3)
C9—N5—N6	105.5 (3)	N2—C9—N4	122.6 (3)
C10—N6—N5	110.8 (3)	N6—C10—N4	108.0 (3)
C2—C1—N1	128.3 (4)	N6—C10—C11	125.6 (3)
N1—C1—C6	110.5 (3)	N4—C10—C11	126.4 (3)
C5—C6—C7	133.5 (3)		

The low fraction of observed reflections with $I > 2\sigma(I)$ to the total number of measured reflections, 1005/2935 for $2\theta_{max} = 50°$, was due to the weak high-order reflections. If the original data is cut off at $2\theta = 45°$, the ratio of observed/total improves to 953/2171.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4-*PC Software*; data reduction: *NRCVAX* *DATRD*2 (Le Page & Gabe, 1979); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

We thank the Robert A. Welch Foundation for the fellowship granted (to HC) and the National Science Foundation for the funds (to IB) used in purchasing the diffractometer.

References

Enraf–Nonius (1989). *CAD*-4-*PC Software*. Enraf–Nonius, Delft, The Netherlands.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Holla, B. S. & Udupa, K. V. (1990). *J. Indian Chem. Soc.* **67**, 79–85.
Katz, E. & Margalith, E. (1984). *Antimicrob. Agents Chemother.* **25**, 195–200.
Le Page, Y. & Gabe, E. J. (1979). *J. Appl. Cryst.* **12**, 464–466.
Rashed, N., Abdel Hamid, H., Ramadan, E. & El Ashry, E. H. (1998). *Nucleosides Nucleotides*, **17**, 1373–1384.
Shaban, M. A. E., Nasr, A. Z. & Morgaan, A. E. A. (1999). *Pharamzie*, **54**, 580–587; *Il Farmaco*, **54**, 800–809.
Shaban, M. A. E., Taha, M. A. M. & Morgaan, A. E. A. (1993). First International Conference In Chemistry and Its Applications, Doha, State of Qatar, December 7–9, 1993. Abstract p. 361.
Shaban, M. A. E., Taha, M. A. M. & Morgaan, A. E. A. (2000). *Monatsh. Chem.* In the press.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1993). *SHELXL*93. University of Göttingen, Germany.