Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-(1*H*-1,2,3-Benzotriazol-1-yl)-1,2,5,6-tetrahydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline

Antonio Quesada,[a]† Debbie Cannon,[a] Jairo Quiroga,[b] Braulio Insuasty,[b] Rodrigo Abonia,[b] Andrea Albornoz,[b] Justo Cobo,[c] Manuel Nogueras,[c] Adolfo Sánchez[c] and John Nicolson Low[d]*

[a]Department of Electronic Engineering and Physics, University of Dundee, Dundee DD1 4HN, Scotland, [b]Grupo de Investigación de Compuestos Heterocíclicos, Departamento de Química, Universidad de Valle, AA 25360 Cali, Colombia, [c]Departamento de Química Inorgánica y Orgánica, Universidad de Jaén, 23071 Jaén, Spain, and [d]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland

† Antonio Quesada is a visiting researcher from the Departamento de Química, Inorgánica y Orgánica, Universidad de Jaén, Spain.

Correspondence e-mail: jnlow111@hotmail.com

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.112
Data-to-parameter ratio = 16.2

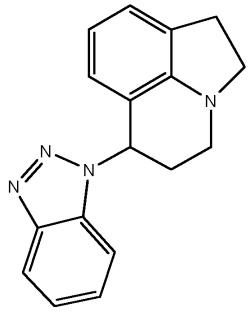
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 January 2001
Accepted 23 January 2001
Online 30 January 2001

The structure of $C_{17}H_{16}N_4$ contains no hydrogen bonds either strong or weak. The only molecular interaction is π–π stacking between the benzotriazole groups in which the perpendicular distance between the triazole group and the benzene group is 3.695 (1) Å, with a distance of 3.822(2) Å between their centroids.

Comment

The title compound, (I), was obtained as a by-product in the preparation of tricyclic heterocycles by annelation using benzotriazole as a synthetic auxiliary (Katritzky *et al.*, 1998). Derivatives of the tricyclic system 1,2,5,6-tetrahydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline, known as lilolidine (Katayama *et al.*, 1985), *i.e.* pyroquilone (Bass *et al.*, 1981; Muecke & Gross, 1986; Nakamura, 1986) and analogues (Bass *et al.*, 1975*a*,*b*, 1981) have shown antifungal applications in rice crops.



(I)

Table 1 lists the geometric parameters, while Fig. 1 shows a view of the molecule. The only molecular interaction is π–π stacking between the benzotriazole groups in which the perpendicular distance between the triazole group at (x, y, z) and the benzene group at ($-x$, $-y$, $-z$) is 3.695 (1) Å, with a distance of 3.822 (2) Å between their centroids (Fig. 2). Examination of the structure with *PLATON* (Spek, 2000) showed that there were no solvent-accessible voids in the crystal lattice.

Experimental

Anhydrous $ZnBr_2$ (50 mg) was added to a mixture of 1-(indolin-1-ylmethyl)benzotriazole (1.99 mmol) and dodecyl vinyl ether (4 mmol) in dry tetrahydrofuran (20 ml) and then stirred at room temperature for 3 h. The solvent was removed under vacuum and the residue was separated by column chromatography (gradient: hexane/ethyl acetate) affording, in addition to the expected compound 6-dodecyloxy-1,2,5,6-tetrahydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline (210 mg), the title compound as a by-product (160 mg, m.p. 422–423K). Compound (I) crystallized directly from the chromatographic

solvents (hexane/ethyl acetate 3:2), affording crystals suitable for X-ray diffraction.

Crystal data

$C_{17}H_{16}N_4$
$M_r = 275.35$
Orthorhombic, *Pbca*
$a = 10.7112$ (3) Å
$b = 10.8297$ (3) Å
$c = 23.6321$ (5) Å
$V = 2741.56$ (12) Å^3
$Z = 8$
$D_x = 1.334$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5825 reflections
$\theta = 1.0$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 150$ (1) K
Block, red
0.30 × 0.20 × 0.20 mm

Data collection

KappaCCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.976$, $T_{max} = 0.984$
10 806 measured reflections
3083 independent reflections
2317 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$
$\theta_{max} = 27.4^\circ$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = -27 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.112$
$S = 1.27$
3083 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N3	1.4654 (17)	N61—N62	1.3575 (14)
N3—C10*B*	1.3948 (16)	N61—C67*A*	1.3652 (16)
N3—C4	1.4539 (16)	N62—N63	1.3028 (16)
C6—N61	1.4598 (16)	N63—C63*A*	1.3771 (18)
C10*B*—N3—C4	114.42 (10)	N62—N61—C6	120.10 (10)
C10*B*—N3—C2	106.20 (10)	C67*A*—N61—C6	130.02 (10)
C4—N3—C2	119.75 (11)	N63—N62—N61	109.17 (10)
N62—N61—C67*A*	109.75 (11)	N62—N63—C63*A*	108.13 (10)

H atoms were treated as riding, with C—H distances in the range 0.95–1.00 Å.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC, X-ray Crystallographic Service, University of Southampton, using an Enraf—Nonius KappaCCD diffractometer. The authors thank the staff for all their help and advice. We are grateful to the Ministerio de Educación y Cultura for the award of a grant to one of the authors (AQ).

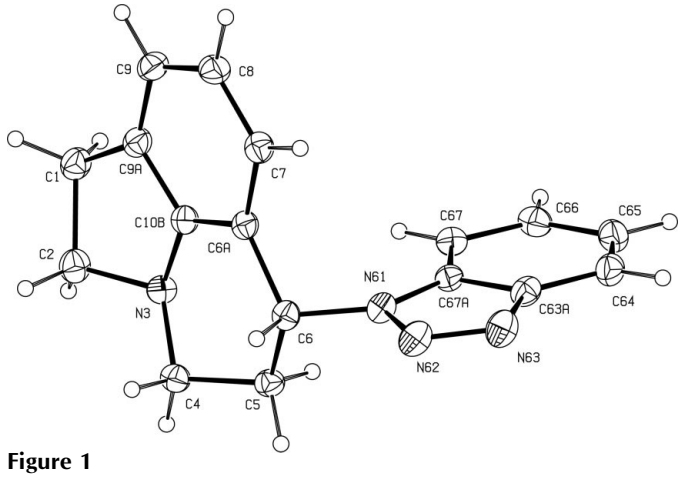


Figure 1
A view of the title molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

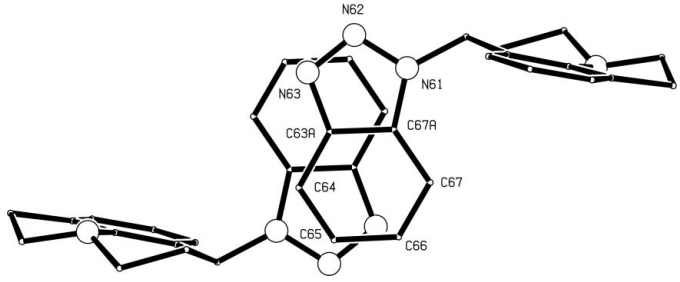


Figure 2
The interaction of two molecules in the crystal structure.

References

Bass, R. J., Koch, R. C., Richards, H. C. & Thorpe, J. E. (1975*a*). British Patent 1,394,373.
Bass, R. J., Koch, R. C., Richards, H. C. & Thorpe, J. E. (1975*b*). US Patent 3,917,838.
Bass, R. J., Koch, R. C., Richards, H. C. & Thorpe, J. E. (1981). *J. Agric. Food Chem.* **29**, 576–579.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Katayama, H., Nakazawa, Y. & Funayama, N. (1985). *Niigata Yakka Daigaku Kenkyu Hokoku*, **5**, 1–3.
Katritzky, A. R., Abonia, R., Yang, B., Qi, M. & Inuasty, B. (1998). *Synthesis*, pp. 469–474.
Muecke, W. & Gross, D. (1986). *Proc. Br. Crop Prot. Conf. Pests Dis*. (2), pp. 469–474.
Nakamura, M. (1986). *Jpn Pestic. Inf.* **48**, 27–30.
Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). Methods Enzymol. **276**, 307–326.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2000). *PLATON*. May 2000 Version. University of Utrecht, The Netherlands.