Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.102$
$S = 0.808$
3388 reflections
252 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Cl—C1	1.739 (2)	N4—C14	1.330 (3)
N1—C7	1.267 (2)	N4—C15	1.345 (3)
N1—N2	1.406 (2)	C6—C7	1.460 (3)
N2—C8	1.286 (2)	C8—C18	1.474 (3)
N3—C13	1.329 (2)	C8—C9	1.484 (3)
N3—C12	1.341 (3)		
C7—N1—N2	111.0 (2)	N1—C7—C6	121.3 (2)
C8—N2—N1	113.9 (2)	N2—C8—C18	120.3 (2)
C13—N3—C12	114.2 (2)	N2—C8—C9	133.2 (2)
C14—N4—C15	114.2 (2)	C18—C8—C9	106.5 (2)

The title structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from difference Fourier maps and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Program used for geometrical calculations: *PARST* (Nardelli, 1983). Software used to prepare material for publication: *SHELXL93*.

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Two Substituted [1,2,4]Triazole Derivatives

Ramasamy Velavan,[a] Kandasamy Sivakumar,[a]† Hoong-Kun Fun,[b] Urvish S. Pathak,[c] Kishor S. Jain[c] and Sanjay Singh[c]

[a]*Department of Physics, Anna University, Madras 600 025, India,* [b]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [c]*Department of Pharmaceutical Chemistry, LM College of Pharmacy, Ahmedabad 380 009, India. E-mail: auphys@imsc.ernet.in*

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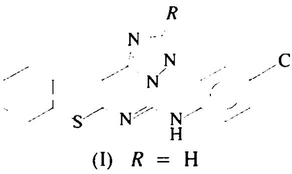
Abstract

The structures of two 1,2,4-triazole derivatives, namely 5-(4-chlorophenyl)amino-8,9,10,11-tetrahydro[1]benzothieno[3,2-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine, $C_{17}H_{14}ClN_5S$, and its 2-methyl derivative, $C_{18}H_{16}ClN_5S$, have been determined. The cyclohexene ring in each compound shows disorder. Both molecules are nearly planar, with the chlorophenyl rings lying almost in the same plane as the rest of the structure. Both crystals are stabilized by N—H···N hydrogen bonds.

Comment

Condensed [1,2,4]triazoles are biologically important compounds (Kottke, Kuesmstedt, Hagen, Renner & Schnitzler, 1983; Francis & Gelette, 1988; Francis *et al.*, 1988). Our interest in the synthesis and characterization of these heterocyclic compounds, especially of thienopyrimidines, has led us to prepare a series of thieno[4,3-*a*][1,2,4]triazolo[3,2-*e*]pyrimidine derivatives. Some of these have been shown to exhibit excellent central-nervous-system depressant and skeletal-muscle relaxant activities through experiments on Swiss albino mice by the rota-rod method and by photoactometer. Previously, we reported the first crystal structure of a triazolo derivative (Velavan *et al.*, 1995); we now present two related structures, 5-(4-chlorophenyl)amino-8,9,10,11-tetrahydro[1]benzothieno[3,2-*e*][1,2,4]triazolo-[1,5-*c*]-pyrimidine, (I), and its 2-methyl derivative, (II).



(I) R = H
(II) R = Me

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† Visiting Post-Doctoral Research Fellow, School of Physics, Universiti Sains Malaysia, Malaysia.

As can be seen from Fig. 1, both (I) and (II) adopt nearly planar conformations. Bond lengths and angles are generally normal. In both structures, the cyclohexene ring is disordered, adopting two different conformations. In (I), the alternative sites for C3 suggest that the ring has conformational flexibility between half-chair and sofa. In (II), both C3 and C4 are disordered: the major conformer is intermediate between half-chair and sofa, while the minor conformation is a half-chair. Similar disorder in related compounds has been attributed to the presence of energetically similar conformers in the crystal (Kapor, Stajer & Bernath, 1993).

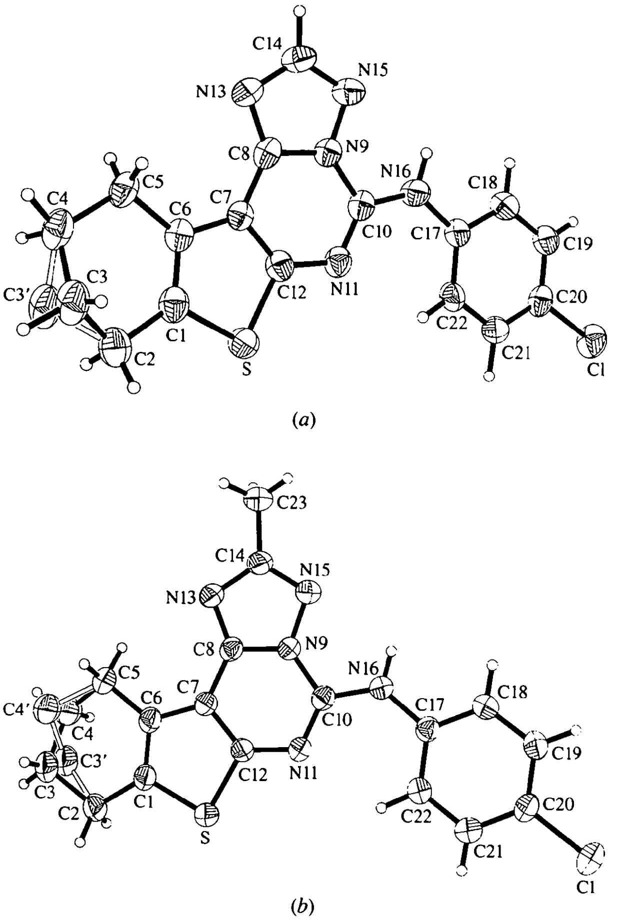

Fig. 1. View of (*a*) molecule (I) and (*b*) molecule (II) with the atom-numbering schemes. Displacement ellipsoids are drawn at the 50% probability level.

The part of each molecule made up of the thiene, triazolo and pyrimidine rings is planar [maximum deviations: −0.074 (4) Å for N15 in (I) and 0.061 (2) Å for C1 in (II)]. The dihedral angle between this plane and that of the chlorophenyl ring is 1.01 (1)° in (I) and 4.63 (4)° in (II). Thus, the molecule as a whole is planar in both (I) and (II), but the degree of planarity is somewhat affected by the substitution at C14.

In both structures, the lone NH group (N16—H) is involved in intermolecular N—H···N hydrogen bonds, but only with N13 even though three possible acceptor atoms are available (N11, N13 and N15) [(I): N16···N13^{i} 3.245 (6), H16···N13^{i} 2.48 (6) Å and N16—H16···N13^{i} 143 (5)°; (II): N16···N13^{ii} 3.132 (2), H16···N13^{ii} 2.37 (2) Å and N16—H16···N13^{ii} 146 (2)°; symmetry codes: (i) $-x+2$, $y+\frac{1}{2}$, $-z+\frac{1}{2}$; (ii) $-x+1$, $y-\frac{1}{2}$, $-z+\frac{1}{2}$]. In (II), short S···S contacts are also observed [S···S^{iii} 3.646 (1) Å; symmetry code: (iii) $1-x$, $-y$, $-z$].

Experimental

Single crystals of (I) and (II) suitable for X-ray studies were obtained by slow evaporation of an ethanol/chloroform solution of the respective compound.

Compound (I)

Crystal data

$C_{17}H_{14}ClN_5S$
$M_r = 355.84$
Orthorhombic
$P2_12_12_1$
$a = 7.354$ (1) Å
$b = 8.477$ (1) Å
$c = 25.511$ (3) Å
$V = 1590.4$ (3) Å^3
$Z = 4$
$D_x = 1.486$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 35 reflections
$\theta = 8$–25°
$\mu = 0.380$ mm^{-1}
$T = 293$ (2) K
Needle
0.62 × 0.24 × 0.12 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2255 measured reflections
2062 independent reflections
1764 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.081$

$\theta_{\rm max} = 25.0°$
$h = -1 \rightarrow 8$
$k = -1 \rightarrow 10$
$l = -1 \rightarrow 30$
3 standard reflections every 97 reflections intensity decay: <4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.184$
$S = 1.046$
2057 reflections
279 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.1218P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.42$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.25$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.00 (14)

Compound (II)

Crystal data

$C_{18}H_{16}ClN_5S$
$M_r = 369.87$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Monoclinic
$P2_1/c$
$a = 8.814$ (1) Å
$b = 8.718$ (1) Å
$c = 22.125$ (2) Å
$\beta = 90.80$ (1)°
$V = 1699.9$ (3) Å^3
$Z = 4$
$D_x = 1.445$ Mg m^{-3}
D_m not measured

Cell parameters from 32 reflections
$\theta = 8$–25°
$\mu = 0.359$ mm^{-1}
$T = 293$ (2) K
Needle
0.58 × 0.38 × 0.08 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5267 measured reflections
3908 independent reflections
2924 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$

$\theta_{\rm max} = 27.5°$
$h = -1 \rightarrow 11$
$k = -1 \rightarrow 11$
$l = -28 \rightarrow 28$
3 standard reflections every 97 reflections intensity decay: <4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.111$
$S = 0.962$
3908 reflections
300 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.30$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

	(I)	(II)
Cl—C20	1.749 (5)	1.741 (2)
S—C12	1.747 (4)	1.733 (2)
S—C1	1.752 (5)	1.740 (2)
C6—C7	1.413 (7)	1.434 (2)
C7—C12	1.379 (6)	1.383 (2)
C7—C8	1.422 (6)	1.420 (2)
C8—N13	1.335 (6)	1.326 (2)
C8—N9	1.377 (6)	1.374 (2)
N9—N15	1.376 (5)	1.374 (2)
N9—C10	1.385 (6)	1.382 (2)
C10—N11	1.303 (6)	1.306 (2)
C10—N16	1.349 (6)	1.358 (2)
N11—C12	1.363 (6)	1.365 (2)
N13—C14	1.352 (7)	1.372 (2)
C14—N15	1.323 (7)	1.317 (2)
C14—C23		1.488 (2)
N16—C17	1.413 (6)	1.412 (2)
C12—S—C1	91.1 (2)	91.20 (8)
C10—N16—C17	129.7 (4)	128.27 (14)

Disorder in the cyclohexene rings was detected during refinement from the large displacement ellipsoids of C3 in (I) and of C3 and C4 in (II). The positional and occupancy parameters of the alternative sites were refined. The displacement parameters of the alternative sites were set equal during the final stages of the refinement. The populations of the major conformers are 94 (1) and 92 (1)% for (I) and (II), respectively. The bond lengths associated with the minor component atoms [C3′ in (I), and C3′ and C4′ in (II)] were made equal to those for the major component atoms using *DFIX* (Sheldrick, 1993).

For both compounds, data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXTL/PC* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993) and *PARST* (Nardelli, 1983); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1295). Services for accessing these data are described at the back of the journal.

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2,12-Dibromo-5,6-dihydrodibenz[*c*,*h*]acridine

JAYANTA KUMAR RAY,[a] MANASH KUMAR HALDAR,[a] KANDASAMY CHINNAKALI[b]† AND HOONG-KUN FUN[b]

[a]*Department of Chemistry, Indian Institute of Technology, Kharagpur 721 302, India, and* [b]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: hkfun@usm.my*

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Abstract

The butterfly-like title molecule, $C_{21}H_{13}Br_2N$, is almost planar except for the cyclohexadiene ring which is in a half-chair conformation.

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† On leave from: Department of Physics, Anna University, Chennai 600 025, India.