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4-Phenyl-6,7,8,9-tetrahydro-[1]benzothieno-[3,2-*e*][1,2,4]triazolo[4,3-*a*]pyrimidin-5(4*H*)-one

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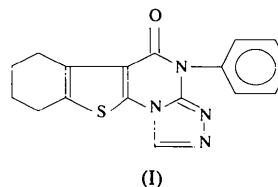
Abstract

The cyclohexane ring of the title compound, C₁₇H₁₄N₄O₃, shows disorder with an 85% major conformer and a 15% minor conformer. Both adopt half-chair conformations. The phenyl ring is tilted by 80.79 (4)° to the plane formed by the rest of the atoms of the mol-

ecule. Molecules of the same orientation are linked by C—H···O hydrogen bonds and short S···O contacts, forming chains along the *b* axis.

Comment

Condensed [1,2,4]triazoles are biologically important compounds (Kottke, Kuesmstedt, Hagen, Renner & Schnitzler, 1983; Francis & Gelotte, 1988; Francis *et al.*, 1988). Since our interest is in the synthesis and characterization of biologically active heterocyclic compounds, especially thienopyrimidines, we have prepared a series of [4,3-*a*]thieno[1,2,4]triazolo[3,2-*e*]pyrimidin-5(4*H*)-one derivatives and studied both their depressant effect on the central nervous system and their analgesic activities (Pathak, Gandhi, Singh, Warde & Jain, 1992). The structure of the title compound, (I), one of these derivatives, has been determined in order to elucidate the conformational features of the molecule. To our knowledge, this is the first crystal structure report of a [1,2,4]triazolo- or thienopyrimidine derivative.



The bond lengths and angles in the title compound have normal values. The part of the molecule comprising the thiene, pyrimidine and triazo rings is planar, with a maximum deviation of 0.050 (2) Å for atom C15. Atoms C2 and C5 also lie close to this plane and the phenyl ring is almost perpendicular to it [dihedral angle

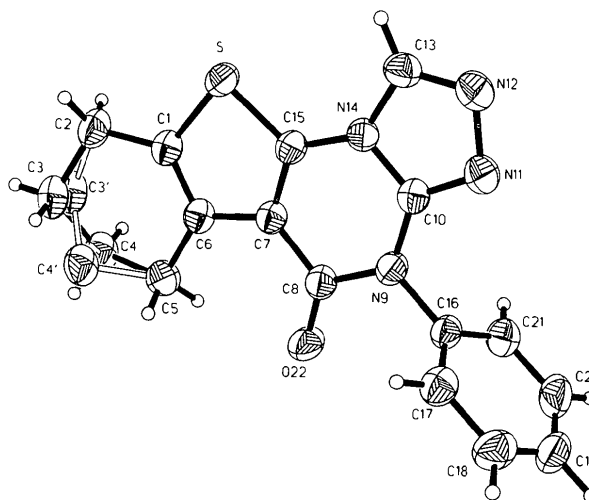


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme. The minor conformer positions are linked by open bonds.

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80.79 (4)°]. Atoms C3 and C4 of the cyclohexane ring (C1–C6) are each disordered over two positions, with 85 and 15% occupancies. The two alternative conformations of this ring are both half chair and differ in the direction of the displacement of atoms C3 and C4 from the plane defined by C1, C2, C5 and C6 (relevant torsion angles are given in Table 2). The carbonyl O22 atom is involved in a C—H···O hydrogen bond and a short S···O contact with a neighbouring molecule [C—H2A 0.98 (3), H2A···O22ⁱ 2.52 (2), C2···O22ⁱ 3.374 (3) Å and C2—H2A···O22ⁱ 146 (2)°; S···O22ⁱ 3.189 (2) Å; symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, z$]. These two contacts link molecules of the same orientation into chains along the *b* axis, as shown in Fig. 2.

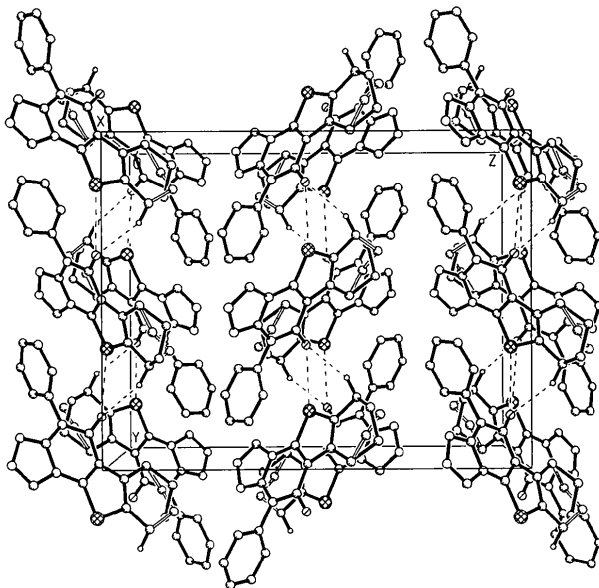


Fig. 2. Packing of the molecules viewed down the *a* axis, with the C—H···O hydrogen bonds and short S···O contacts shown by dashed lines.

Experimental

Details of the synthesis of the title compound are published elsewhere (Pathak, Gandhi, Singh, Warde & Jain, 1992). Single crystals were obtained by recrystallization from an ethanol–chloroform mixture.

Crystal data

C₁₇H₁₄N₄O₅
M_r = 322.38
 Orthorhombic
Pbca
a = 10.668 (1) Å
b = 14.687 (2) Å
c = 18.820 (2) Å
V = 2948.7 (6) Å³
Z = 8
D_x = 1.452 Mg m⁻³

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 8–25°
 μ = 0.230 mm⁻¹
T = 293 (2) K
 Rectangular slab
 0.66 × 0.52 × 0.26 mm
 Transparent

Data collection

Siemens *P4* diffractometer
 $\theta/2\theta$ scans
 Absorption correction: none
 3270 measured reflections
 2560 independent reflections
 2034 observed reflections
 $[I > 2\sigma(I)]$
R_{int} = 0.0193

θ_{\max} = 24.99°
 $h = -1 \rightarrow 12$
 $k = -1 \rightarrow 17$
 $l = -1 \rightarrow 22$
 3 standard reflections monitored every 100 reflections
 intensity decay: < 4%

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.0341$
 $wR(F^2) = 0.0996$
S = 1.037
 2560 reflections
 271 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0582P)^2 + 0.1938P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.22 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{Å}^{-3}$
 Atomic scattering factors from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
S	0.09195 (4)	0.84896 (3)	0.01955 (2)	0.0403 (2)
C1	0.2124 (2)	0.86993 (12)	-0.04086 (9)	0.0384 (4)
C2	0.2512 (2)	0.79996 (14)	-0.09487 (12)	0.0495 (5)
C3 †	0.3777 (3)	0.8243 (2)	-0.1258 (2)	0.0529 (8)
C3' †	0.3400 (19)	0.8467 (13)	-0.1498 (10)	0.0529 (8)
C4 †	0.3862 (3)	0.9243 (2)	-0.1444 (2)	0.0508 (8)
C4' †	0.4378 (17)	0.9049 (12)	-0.1142 (9)	0.0508 (8)
C5	0.3697 (2)	0.98631 (15)	-0.07948 (11)	0.0454 (5)
C6	0.2643 (2)	0.95364 (12)	-0.03269 (9)	0.0359 (4)
C7	0.2056 (2)	1.00408 (12)	0.02394 (8)	0.0333 (4)
C8	0.2411 (2)	1.09378 (12)	0.05058 (9)	0.0366 (4)
N9	0.17003 (14)	1.12454 (10)	0.10892 (7)	0.0360 (3)
C10	0.0741 (2)	1.07456 (12)	0.13839 (9)	0.0345 (4)
N11	0.00326 (14)	1.09584 (11)	0.19227 (8)	0.0425 (4)
N12	-0.07673 (15)	1.02130 (11)	0.20233 (8)	0.0466 (4)
C13	-0.0502 (2)	0.96061 (13)	0.15462 (10)	0.0411 (4)
N14	0.04470 (13)	0.99058 (9)	0.11158 (7)	0.0339 (3)
C15	0.1117 (2)	0.95512 (11)	0.05486 (9)	0.0333 (4)
C16	0.2016 (2)	1.21037 (12)	0.14291 (9)	0.0358 (4)
C17	0.2991 (2)	1.21260 (14)	0.19048 (10)	0.0442 (5)
C18	0.3274 (2)	1.2942 (2)	0.22400 (11)	0.0553 (6)
C19	0.2571 (2)	1.3707 (2)	0.21073 (12)	0.0580 (6)
C20	0.1597 (2)	1.36710 (14)	0.16364 (14)	0.0600 (6)
C21	0.1308 (2)	1.28684 (14)	0.12834 (12)	0.0490 (5)
O22	0.32536 (14)	1.14048 (9)	0.02664 (7)	0.0527 (4)

† Partially occupied (see below).

Table 2. Selected geometric parameters (Å, °)

S—C15	1.708 (2)	C8—N9	1.409 (2)
S—C1	1.743 (2)	N9—C10	1.376 (2)
C1—C6	1.357 (3)	N9—C16	1.453 (2)
C1—C2	1.504 (3)	C10—N11	1.302 (2)
C2—C3	1.513 (4)	C10—N14	1.369 (2)
C2—C3'	1.56 (2)	N11—N12	1.401 (2)
C3—C4	1.512 (5)	N12—C13	1.296 (2)
C3'—C4'	1.51 (3)	C13—N14	1.370 (2)
C4—C5	1.534 (3)	N14—C15	1.386 (2)
C4'—C5	1.54 (2)	C16—C17	1.373 (3)
C5—C6	1.506 (3)	C16—C21	1.381 (3)

C6—C7	1.441 (2)	C17—C18	1.388 (3)
C7—C15	1.364 (2)	C18—C19	1.373 (4)
C7—C8	1.460 (2)	C19—C20	1.367 (4)
C8—O22	1.217 (2)	C20—C21	1.388 (3)
C15—S—C1	90.10 (8)	C10—N9—C16	117.23 (14)
C6—C1—C2	125.7 (2)	C8—N9—C16	119.77 (14)
C6—C1—S	112.76 (13)	N11—C10—N14	111.7 (2)
C2—C1—S	121.54 (15)	N11—C10—N9	128.1 (2)
C1—C2—C3	110.1 (2)	N14—C10—N9	120.14 (15)
C1—C2—C3'	108.3 (7)	C10—N11—N12	105.71 (14)
C4—C3—C2	111.8 (3)	C13—N12—N11	108.12 (15)
C4'—C3'—C2	112.1 (14)	N12—C13—N14	110.5 (2)
C3—C4—C5	112.7 (2)	C10—N14—C13	103.93 (14)
C3'—C4'—C5	107.6 (14)	C10—N14—C15	120.30 (14)
C6—C5—C4	111.2 (2)	C13—N14—C15	135.7 (2)
C6—C5—C4'	110.6 (6)	C7—C15—N14	120.6 (2)
C1—C6—C7	111.9 (2)	C7—C15—S	113.94 (13)
C1—C6—C5	121.8 (2)	N14—C15—S	125.40 (13)
C7—C6—C5	126.4 (2)	C17—C16—C21	121.6 (2)
C15—C7—C6	111.3 (2)	C17—C16—N9	118.9 (2)
C15—C7—C8	121.3 (2)	C21—C16—N9	119.4 (2)
C6—C7—C8	127.3 (2)	C16—C17—C18	118.8 (2)
O22—C8—N9	120.4 (2)	C19—C18—C17	120.3 (2)
O22—C8—C7	125.0 (2)	C20—C19—C18	120.1 (2)
N9—C8—C7	114.67 (15)	C19—C20—C21	120.8 (2)
C10—N9—C8	122.92 (15)	C16—C21—C20	118.3 (2)
C1—C2—C3—C4	-44.7 (3)	C1—C2—C3'—C4'	45.6 (17)
C2—C3—C4—C5	61.2 (4)	C2—C3'—C4'—C5	-67.5 (19)
C3—C4—C5—C6	-43.0 (4)	C3'—C4'—C5—C6	52.5 (16)
C4—C5—C6—C1	13.0 (3)	C4'—C5—C6—C1	-21.0 (8)
C5—C6—C1—C2	0.6 (3)	C6—C1—C2—C3'	-12.0 (9)
C6—C1—C2—C3	15.3 (3)		

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Disordered Structure of 2-Methyl-anthraquinone

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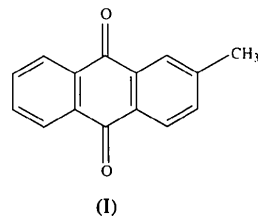
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Abstract

Crystals of the title compound, C₁₅H₁₀O₂, are monoclinic, space group *P*₂₁/*c*. The molecules are disordered around a centre of symmetry. Packing considerations and the calculation of the intermolecular interaction energies show that the disordered structure has occurred by random packing of structures with space groups *P*₂₁ and *Pc*, the maximal subgroups of *P*₂₁/*c*.

Comment

A certain kind of structural disorder has been observed in homomolecular crystals like those of azulene (Robertson, Shearer, Sim & Watson, 1962) and 1-bromo-4-chlorobenzene (Klug, 1947). In these crystals, each molecule is apparently disordered in two orientations related by a $\bar{1}$ symmetry operation. Here, we comment on such a structural disorder as found in 2-methylanthraquinone, (I).



The molecular arrangement is characterized by the formation of a molecular sheet in (10 $\bar{2}$) and molecular stacking along the *a* axis with an interplanar spacing

Disorder of atoms C3 and C4 was observed after anisotropic refinement of the non-H atoms. C3 and C4 were assigned occupancies of *p* and C3' and C4' were assigned occupancies of 1–*p*, with *p* = 0.850 (6) at convergence. All the H atoms belonging to the major conformer 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: *SHELXS86* (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXL93*. Geometric calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1178). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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