Monoclinic
Cc
$a = 11.325\,(2)$ Å
$b = 16.996\,(2)$ Å
$c = 17.193\,(2)$ Å
$\beta = 99.780\,(10)°$
$V = 3261.2\,(8)$ Å^3
$Z = 4$
$D_x = 1.172$ Mg m^{-3}
D_m not measured

Cell parameters from 28 reflections
$\theta = 2.68–17.07°$
$\mu = 0.071$ mm^{-1}
$T = 290\,(2)$ K
Chunk
0.5 × 0.4 × 0.4 mm
Brown–yellow

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: none
4700 measured reflections
4147 independent reflections
2362 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$

$\theta_{\rm max} = 28°$
$h = 0 \rightarrow 14$
$k = -1 \rightarrow 22$
$l = -22 \rightarrow 22$
3 standard reflections every 97 reflections intensity decay: 3.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.063$
$S = 0.82$
4147 reflections
398 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.01$

$\Delta\rho_{\rm max} = 0.12$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.09$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0083 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C12	1.365 (3)	B—C29	1.634 (4)
O1—C15	1.432 (3)	B—C35	1.638 (3)
O2—C13	1.379 (3)	B—C23	1.647 (3)
O2—C16	1.424 (3)	B—C17	1.651 (4)
N—C2	1.334 (3)	C4—C7	1.457 (3)
N—C6	1.335 (3)	C7—C8	1.312 (3)
N—C1	1.464 (3)	C8—C9	1.471 (4)
C12—O1—C15	117.5 (2)	C35—B—C23	112.5 (2)
C13—O2—C16	117.2 (2)	C29—B—C17	111.6 (2)
C2—N—C6	119.7 (2)	C35—B—C17	113.7 (2)
C2—N—C1	119.6 (2)	C23—B—C17	103.3 (2)
C6—N—C1	120.7 (2)	C8—C7—C4	125.0 (2)
C29—B—C35	104.8 (2)	C7—C8—C9	127.6 (2)
C29—B—C23	111.2 (2)		
C3—C4—C7—C8	166.7 (3)	C7—C8—C9—C14	163.7 (3)
C5—C4—C7—C8	−13.1 (4)	C15—O1—C12—C11	0.1 (4)
C4—C7—C8—C9	179.8 (2)	C16—O2—C13—C14	−11.8 (3)
C7—C8—C9—C10	−16.4 (4)		

Table 2. *Contact distances (Å)*

N···C37$^{\rm i}$	3.418 (4)	N···C36$^{\rm i}$	3.677 (3)
C2···C36$^{\rm i}$	3.326 (4)	C2···C37$^{\rm i}$	3.381 (5)
C27···C15$^{\rm ii}$	3.351 (4)		

Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $x - 1, 1 - y, z - \frac{1}{2}$.

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1$^{\rm i}$	2.56	3.388 (4)	149
C3—H3···O2$^{\rm i}$	2.52	3.346 (4)	148
C21—H21···O1$^{\rm ii}$	2.65	3.512 (3)	155

Symmetry codes: (i) $x, 1 - y, z - \frac{1}{2}$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$.

Data collection: *XSCANS* (Siemens, 1994*a*). Cell refinement: *XSCANS*. Data reduction: *SHELXTL* (Siemens, 1994*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1469). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1939–1941

6-Benzylidene-2-(2-chlorophenyl)thiazolo[3,2-*b*]-1,2,4-triazol-5(6*H*)-one

SÜHEYLA ÖZBEY,[a] ENGIN KENDI,[a] BIRSEN TOZKOPARAN[b] AND MEVLÜT ERTAN[b]

[a]*Hacettepe University, Department of Physics Engineering, Beytepe 06532, Ankara, Turkey, and* [b]*Hacettepe University, Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Sıhhiye 06100, Ankara, Turkey. E-mail: sozbey@lidya.cc.hun.edu.tr*

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Abstract

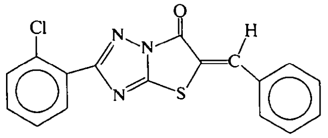
In the title compound, $C_{17}H_{10}ClN_3OS$, the fused thiazolo[3,2-*b*]-1,2,4-triazole system is almost planar. The phenyl substituents are planar within experimental error and make dihedral angles of 16.5 (1) and 10.9 (1)° with the thiazolo–triazole system. There are three intramolec-

ular hydrogen-bond interactions of types C—H···N, C—H···O and C—H···S.

Comment

Compounds containing the 1,2,4-triazole ring possess a broad pharmacological activity spectrum encompassing anti-inflammatory (Prasad *et al.*, 1989), sedative, smooth-muscle relaxation (Gall *et al.*, 1976), anticonvulsant (Kane *et al.*, 1990), antituberculosis (Mir *et al.*, 1970) and platelet-aggregation inhibitory activities (Lagorce *et al.*, 1992).

In a previous study, we described the synthesis and *in vitro* platelet-aggregation inhibitory activities of some thiazolo[3,2-*b*]-1,2,4-triazole-5(6*H*)-ones. Compounds with a double bond showed geometric isomerism. Among these compounds, the molecular structure of 6-benzylidene-2-methylthiazolo[3,2-*b*]-1,2,4-triazol-5(6*H*)-one was investigated using a molecular-modelling system (Tozkoparan *et al.*, 1995). In a continuation of this work on thiazolo[3,2-*b*]-1,2,4-triazoles, we have prepared some new compounds. In the present work, with the aim of further clarifying the molecular structure of this type of compound, the single-crystal X-ray analysis of 6-benzylidene-2-(2-chlorophenyl)thiazolo[3,2-*b*]-1,2,4-triazole-5(6*H*)-one, (I), has been carried out.



(I)

The title compound consists of a fused thiazolo[3,2-*b*]-1,2,4-triazole system and two phenyl groups, one of which bears a 2-chloro substituent (Fig. 1). The three rings do not share a common plane. The fused thiazolo–triazole system is nearly planar; the displacements of the eight atoms contained in the rings are less than 0.020 (2) Å from their least-squares plane. The phenyl groups at C7 and C11 are essentially planar [maximum deviations of 0.005 (2) and 0.004 (2) Å for C5 and C16, respectively] and twisted slightly out of the plane of the thiazolo[3,2-*b*]-1,2,4-triazole ring, with torsion angles of −165.6 (2) and 172.2 (3)° for C1—C6—C7—N2 and C10—C11—C12—C13, respectively. The dihedral angles between the phenyl groups (C1–C6 and C12–C17) and the fused thiazolo–triazole ring are 16.5 (1) and 10.9 (1)°, respectively, while the dihedral angle between these two phenyl groups is 8.3 (1)°. The phenyl ring at the C11 position is in the *cis* (*Z*) configuration with respect to the S atom of the fused thiazolo[3,2-*b*]-1,2,4-triazole system.

The C10—C11 bond is a double bond [1.339 (2) Å]. The C11—C12 [1.453 (3) Å] and C6—C7 [1.475 (2) Å] bonds are found to have normal single-bond lengths. The lengthening of the S1—C10 bond distance *versus* the S1—C8 bond distance may be described in terms of the steric effect of the phenyl substituent at C11 and strain. The N1—N3 bond length [1.374 (2) Å] is very similar to corresponding distances in structures containing the 1,2,4-triazole ring (Borbulevych *et al.*, 1998; Wang *et al.*, 1998).

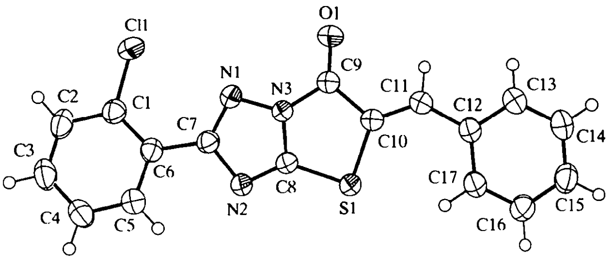


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles of arbitrary radii.

In compound (I), there are three intramolecular hydrogen-bond interactions; details are given in Table 2. The minimum intermolecular distances conform to normal van der Waals interactions.

Experimental

Compound (I) was synthesized previously by Mohamed I. Ali (Ali *et al.*, 1976). In the present study, the title compound was obtained as described in Tozkoparan *et al.* (1999) and recrystallized from tetrahydrofuran.

Crystal data

$C_{17}H_{10}ClN_3OS$
$M_r = 339.80$
Monoclinic
$P2_1/c$
$a = 10.741\ (1)$ Å
$b = 12.387\ (1)$ Å
$c = 12.196\ (2)$ Å
$\beta = 112.61\ (1)°$
$V = 1497.9\ (3)$ Å^3
$Z = 4$
$D_x = 1.507$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10.11–18.13°$
$\mu = 0.401$ mm^{-1}
$T = 295$ K
Prismatic
0.48 × 0.30 × 0.24 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Fair, 1990)
$T_{min} = 0.832$, $T_{max} = 0.911$
5830 measured reflections
3848 independent reflections

2864 reflections with $I > 3\sigma(I)$
$R_{int} = 0.02$
$\theta_{max} = 25.5°$
$h = -11 \rightarrow 13$
$k = 0 \rightarrow 14$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: 0.8%

Refinement

Refinement on F
$R = 0.035$
$wR = 0.045$
$S = 0.90$
2742 reflections
211 parameters
H atoms constrained
$w = 1/[\sigma(F^2) + (0.02F)^2 + 0.6]$
$(\Delta/\sigma)_{\max} < 0.01$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

Cl1—C1	1.731 (2)	N2—C8	1.308 (2)
S1—C8	1.734 (2)	N3—C8	1.346 (2)
S1—C10	1.772 (2)	N3—C9	1.399 (2)
O1—C9	1.199 (3)	C6—C7	1.475 (2)
N1—N3	1.374 (2)	C9—C10	1.491 (3)
N1—C7	1.321 (2)	C10—C11	1.339 (2)
N2—C7	1.393 (2)	C11—C12	1.453 (3)
C8—S1—C10	89.60 (8)	N3—C9—C10	107.0 (2)
N3—N1—C7	101.3 (1)	S1—C10—C9	112.1 (1)
C7—N2—C8	101.7 (1)	C9—C10—C11	120.0 (2)
N1—N3—C8	110.1 (1)	C10—C11—C12	131.3 (2)
C8—N3—C9	117.7 (1)	C12—C11—H11	114.5 (2)
Cl1—C1—C6	122.2 (1)	C11—C12—C13	118.2 (2)
S1—C8—N3	113.6 (1)	C11—C12—C17	123.9 (2)
O1—C9—C10	128.2 (2)	C12—C13—H13	119.6
N3—N1—C7—C6	177.5 (2)	S1—C10—C11—C12	−2.4 (4)
C1—C6—C7—N2	−165.6 (2)	C10—C11—C12—C13	172.2 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···N2	0.94	2.44	2.815 (2)	104
C11—H11···O1	0.93	2.55	2.910 (2)	104
C17—H17···S	0.95	2.50	3.218 (2)	133

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIR* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN* (Fair, 1990) and *PARST* (Nardelli, 1995).

The authors acknowledge the purchase of the CAD-4 diffractometer under grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1204). Services for accessing these data are described at the back of the journal.

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Configurational analysis of 14,14-dimethyl-12-oxatricyclo[9.2.1.0^{3,8}]tetradeca-3,5,7-trien-13-one

REINHOLD ZIMMER,[a] GERNOT ZAHN,[b] FAIZ AHMED KHAN[a] AND HANS-ULRICH REISSIG[a]

[a]*Institut für Organische Chemie, Technische Universität Dresden, D-01062 Dresden, Germany, and* [b]*Institut für Kristallographie und Festkörperphysik, Technische Universität Dresden, D-01062 Dresden, Germany. E-mail: zahn@physik.tu-dresden.de*

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Abstract

The title compound, $C_{15}H_{18}O_2$, is shown to contain a *cis*-fused, five-membered lactone ring and bears some resemblance to the ring system of palitaxel (Taxol^{TM}).

Comment

The taxane diterpenes are a group of substances isolated from various yew (*Taxus*) species that, with few exceptions, share the carbon skeleton (1) (Nicolaou *et al.*, 1994). A well known diterpene natural product containing this framework is Taxol (Wani *et al.*, 1971). Recently we described a practical procedure to construct the *BC*-ring system of the taxane framework by a novel reductive coupling reaction using samarium(II) diiodide (Khan *et al.*, 1997). As part of our current research program we are aiming at the subsequent formation of the *A* ring using the lactone moiety in key intermediate (3). Compound (3) was obtained in isomerically pure form, but the relative configuration of the stereocentres could not be determined unambiguously by NMR analysis. The X-ray analysis led to the determination of the structural features of (3) with the relative stereochemistry as