salt; a modification of the method used by Skibo & Islam (1991) was incorporated. The iminoquinone product was recrystallized over a period of one week from hexane.

Crystal data

$C_{14}H_9NO$
$M_r = 207.22$
Monoclinic
$C2/c$
$a = 11.5121\ (3)$ Å
$b = 8.5186\ (3)$ Å
$c = 20.4454\ (7)$ Å
$\beta = 100.768\ (2)°$
$V = 1969.71\ (11)$ Å^3
$Z = 8$
$D_x = 1.398$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 7403 reflections
$\theta = 4.45–26.37°$
$\mu = 0.089$ mm^{-1}
$T = 150.0\ (1)$ K
Fragment cut from needle
$0.30 \times 0.26 \times 0.25$ mm
Purple

Data collection

Nonius Kappa-CCD diffractometer
1° φ scans
Absorption correction: none
7403 measured reflections
1996 independent reflections
1682 reflections with $I > 2\sigma(I)$

$R_{int} = 0.049$
$\theta_{max} = 26.37°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 10$
$l = -25 \rightarrow 25$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.099$
$S = 1.035$
1996 reflections
146 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 1.2397P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.190$ e Å^{-3}
$\Delta\rho_{min} = -0.139$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.013 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C2	1.2402 (17)	N6—C7	1.3921 (16)
C2—C16	1.451 (2)	C7—C12	1.4177 (18)
C2—C3	1.456 (2)	C12—C13	1.4427 (18)
C3—C4	1.3361 (19)	C13—C14	1.3450 (19)
C4—C5	1.4585 (18)	C14—C15	1.4420 (19)
C5—N6	1.3040 (16)	C15—C16	1.3616 (18)
C5—C15	1.4747 (18)		
C16—C2—C3	116.20 (12)	C7—C12—C13	125.45 (12)
C4—C3—C2	120.86 (13)	C14—C13—C12	130.32 (13)
C3—C4—C5	123.22 (13)	C13—C14—C15	130.13 (13)
N6—C5—C4	112.60 (11)	C16—C15—C14	117.80 (12)
N6—C5—C15	130.57 (12)	C16—C15—C5	118.57 (12)
C4—C5—C15	116.82 (11)	C14—C15—C5	123.64 (12)
C5—N6—C7	130.74 (11)	C15—C16—C2	124.13 (13)
N6—C7—C12	128.91 (12)		

Data collection: *Kappa-CCD Server Software* (Nonius, 1997). Cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997). Data reduction: *DENZO–SMN*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1517). Services for accessing these data are described at the back of the journal.

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2-Amino-4-phenylthiazole

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Abstract

The title compound, $C_9H_8N_2S$, is almost planar, with an angle of 6.2 (3)° between the planes of the phenyl and thiazole rings. Molecules are linked by an intermolecular

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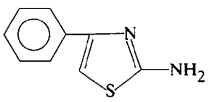
hydrogen bond between one of the amino H atoms and the N atom of the thiazole ring.

Comment

The title compound, (I), and some of its derivatives are used for the preparation of intermediates in the synthesis of pharmaceutical compounds or in the preparation of industrial colouring materials. Moreover, these compounds have tuberculostatic properties (Allen *et al.*, 1954) and others show good bactericide activity against *Staphylococcus aureos* (Tajika *et al.*, 1951). This study was undertaken in order to ascertain the crystal structure of (I).



(I)

A search of the Cambridge Structural Database (Allen & Kennard, 1993) located two chemically related compounds, namely 2-amino-4-phenylthiazole hydrobromine monohydrate (Form *et al.*, 1974) and 2-aminothiazole (Caroni & Capella, 1982). Comparing (I) with the latter reveals that all bond distances of the thiazole ring in (I) are greater, except for the S1—C5 bond, and that the bond angles of the thiazole ring are similar.

Comparing (I) with 2-amino-4-phenylthiazole hydrobromine monohydrate shows that all bond distances of the thiazole ring in (I) are smaller, except for the S1—C2 and C4—C5 bonds. The C2—S—C5 angle is smaller than that in 2-amino-4-phenylthiazole hydrobromide monohydrate [88.7 (2) *versus* 90.17°].

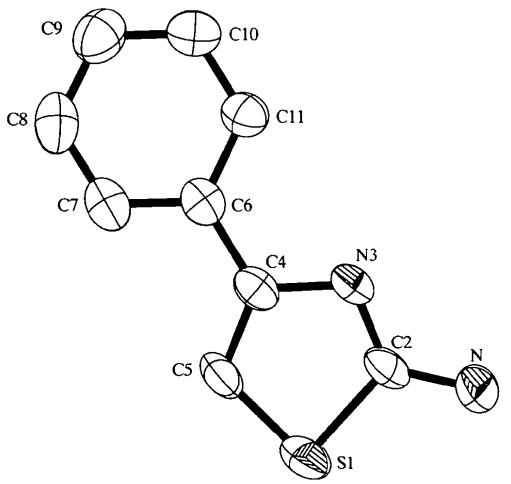


Fig. 1. *ORTEP*-3 (Farrugia, 1997) drawing of (I) with 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Compound (I) is almost planar, with an angle of 6.2 (3)° between the planes of the phenyl and thiazole rings. 2-Amino-4-phenylthiazole hydrobromine monohydrate is less planar than (I), as the angle between the phenyl and thiazole rings is 19.23°. The planarity of (I) is related to the shorter C4—C6 bond distance [1.473 (5) Å] in (I) compared with the value of 1.506 Å found in 2-amino-4-phenylthiazole hydrobromine monohydrate; there is a greater double-bond character of the C4—C6 bond in (I).

The structure of (I) contains an intermolecular hydrogen bond between one of the H atoms of the amino group and the N atom of the thiazole ring, as shown in Table 2.

Experimental

The synthesis of (I) was carried out by refluxing acetophenone (12 g), iodine (24.5 g) and thiourea (15.2 g) for 4 h. The solid which formed was cooled and then stirred with 100 ml of ether. After filtering, washing with ether and drying, the solid obtained was dissolved in hot water and concentrated NH_4OH was added until an alkaline pH was reached. Recrystallization from methyl alcohol gave good crystals for X-ray analysis (m.p. 423–424 K).

Crystal data

$C_9H_8N_2S$
M_r = 176.23
Tetragonal
$P4_3$
a = 12.105 (3) Å
c = 5.776 (3) Å
V = 846.4 (5) Å^3
Z = 4
D_x = 1.383 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 23 reflections
θ = 3.04–11.55°
μ = 0.321 mm^{-1}
T = 293 (2) K
Lath-shaped
0.25 × 0.20 × 0.08 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: none
2631 measured reflections
818 independent reflections (plus 139 Friedel-related reflections)
748 reflections with $I > 2\sigma(I)$
R_{int} = 0.071
θ_{max} = 24.95°
h = −14 → 14
k = −8 → 14
l = −4 → 6
3 standard reflections
frequency: 60 min
intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.105
S = 1.072
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.199 e Å^{-3}
$\Delta\rho_{min}$ = −0.362 e Å^{-3}
Extinction correction: none

957 reflections
111 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.01 (15)

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

S1—C5	1.708 (5)	N3—C4	1.397 (5)
S1—C2	1.749 (4)	C4—C5	1.354 (5)
N3—C2	1.299 (5)	C4—C6	1.473 (5)
C5—S1—C2	88.7 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—HB···N3^{i}	0.86	2.18	2.990 (5)	156.0

Symmetry code: (i) $y - 1, 1 - x, \frac{1}{4} + z$.

Since (I) crystallizes in a polar space group, polar-axis restraints were applied according to the method of Flack & Schwarzenbach (1988), and the absolute structure of the crystal was established according to Flack (1983).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *PROFIT* (Strel'tsov & Zavodnik, 1989). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1045). Services for accessing these data are described at the back of the journal.

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A functionalized dimethyl maleate (maleic acid dimethyl ester)

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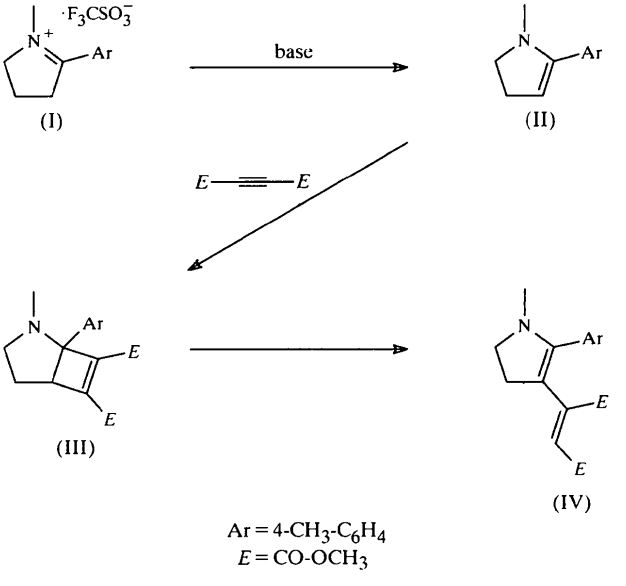
Abstract

In the title compound, dimethyl (*Z*)-2-[1-methyl-2-(*p*-tolyl)-4,5-dihydro-1*H*-pyrrol-3-yl]but-2-enedioate ($C_{18}H_{21}NO_4$), the central butadienyl segment is in a *trans* conformation. The terminal enone segment is in-plane with the butadienyl segment and participates in the conjugated system, whereas the lateral methoxycarbonyl group and the phenyl ring are orientated perpendicular to the butadienyl segment.

Comment

Specifically substituted 1-methyl-5-aryl-3,4-dihydro-2*H*-pyrrolium salts, (I), react with dimethyl acetylene dicarboxylate under basic conditions. The functionalization of the 4-position can be explained as a result of consecutive transformations. Deprotonation of (I) by ethyl diisopropylamine at the 4-position generates the reactive enamine, (II), followed by a [2+2] cycloaddition with dimethyl acetylene dicarboxylate. The resulting bicyclic intermediate, (III), isomerizes in a cycloreversion to the title compound, (IV). These consecutive reac-



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