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5-(4-Chlorophenyl)-6-ethoxycarbonyl-3,7-dimethyl-5*H*-thiazolo[3,2-*a*]-pyrimidin-8-ium bromide

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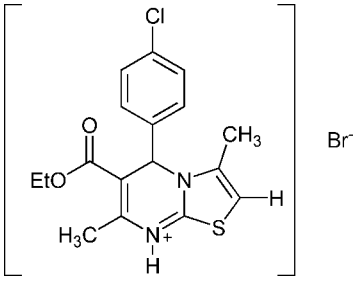
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.094; data-to-parameter ratio = 17.9.

In the title salt, $C_{17}H_{18}ClN_2O_2S^+\cdot Br^-$, the benzene ring is roughly perpendicular to the heterocyclic ring system.

Related literature

For the synthesis, see Ranu *et al.* (2002). For related literature, see: Holla *et al.* (2004); Sayed *et al.* (2006); Tozkoparan *et al.* (1998).



Experimental

Crystal data

$C_{17}H_{18}ClN_2O_2S^+\cdot Br^-$
M_r = 429.75
Monoclinic, $C2/c$
a = 25.457 (2) Å
b = 11.697 (2) Å
c = 14.231 (3) Å
β = 106.8080 (16)°
V = 4056.5 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.27 mm^{-1}
T = 291 (2) K
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.505, T_{max} = 0.584
10641 measured reflections
3986 independent reflections
2937 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.094
S = 1.04
3986 reflections
223 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2207).

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supplementary materials

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5-(4-Chlorophenyl)-6-ethoxycarbonyl-3,7-dimethyl-5*H*-thiazolo[3,2-*a*]pyrimidin-8-ium bromide

T. Shang

Comment

A number of thiazolo[3,2-*a*]pyrimidine derivatives have been reported in the literature. These heterocyclic compounds are pharmacologically interesting systems with remarkable biological activities such as anti-inflammatory, anti-cancer and anti-microbial properties (Tozkoparan *et al.*, 1998; Holla *et al.*, 2004; Sayed *et al.*, 2006).

The structure of the newly synthesized compound, (I), was confirmed by its elemental analysis, and by IR, NMR and mass spectroscopic studies. The crystallographic analysis (Fig. 1) shows the aryl and oxazolidine rings to be essentially co-planar

Experimental

Ethyl 5-(4-chlorophenyl)-3,7-dimethyl-5*H*-thiazolo[3,2-*a*]pyrimidine-6-carboxylate was prepared according to the literature method (Ranu *et al.*, 2002). This compound (1 mmol) and 1-bromopropan-2-one (1 mmol) were successively added to water (5 ml) in a test tube and the reaction mixture was vigorously refluxed for 24 h. The system was cooled to room temperature and filtered to isolate the crude product. The analytical sample was obtained by recrystallization of the crude product from ethanol. The solid was isolated, washed three times with ethanol and dried in a vacuum desiccator; yield 92%, m.p. 513–514 K. IR: 3110, 2980, 1688, 1630, 1600, 1511, 1500 cm^{-1}; ^1H NMR: 1.22 (t, J = 7.2 Hz, 3H), 2.00 (s, 3H), 2.55 (s, 3H), 4.10–4.21 (m, 2H), 6.27 (s, 1H), 7.10 (s, 1H), 7.14–7.24 (m, 2H), 7.26–7.29 p.p.m. (m, 2H). Analysis found: C 47.51, H 4.22, N 6.52%; $C_{17}H_{18}BrN_2O_2S$ requires: C 47.60, H 4.29, N 6.48%. Single crystals suitable for crystallography were obtained by the slow evaporation of an ethanol solution of (I).

Refinement

The H atom bonded to N atom was located in a difference map and refined with $U_{iso}(H) = 1.2U_{eq}(N)$ so that N—H = 0.86 (1) Å. Other H atoms were positioned geometrically and refined using a riding model approximation with C—H = 0.93–0.98 Å and with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$.

Figures

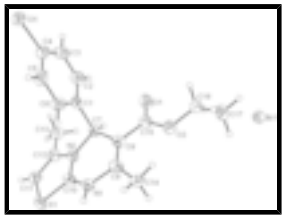

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

5-(4-Chlorophenyl)-6-ethoxycarbonyl-3,7-dimethyl-5*H*- thiazolo[3,2-*a*]pyrimidin-8-ium bromide

Crystal data

$C_{17}H_{18}ClN_2O_2S^+\cdot Br^-$	F_{000} = 1744
M_r = 429.75	D_x = 1.407 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 513-514 K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 25.457 (2) Å	Cell parameters from 2973 reflections
b = 11.697 (2) Å	θ = 2.3–25.5º
c = 14.231 (3) Å	μ = 2.27 mm^{-1}
β = 106.8080 (16)º	*T* = 291 (2) K
V = 4056.5 (12) Å^3	Block, colourless
Z = 8	0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer	3986 independent reflections
Radiation source: sealed tube	2937 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 291(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	*h* = −29→31
T_{min} = 0.505, T_{max} = 0.584	*k* = −14→13
10641 measured reflections	*l* = −17→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.094	$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 1.5186P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
3986 reflections	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
223 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.193130 (15)	0.08363 (3)	0.11668 (3)	0.06287 (14)
C1	0.05124 (13)	0.2550 (3)	0.5811 (2)	0.0529 (8)
C2	0.00250 (15)	0.2082 (3)	0.5272 (3)	0.0606 (9)
H2	0.0025	0.1379	0.4966	0.073*
C3	−0.04725 (15)	0.2644 (3)	0.5177 (3)	0.0681 (10)
H3	−0.0803	0.2330	0.4808	0.082*
C4	−0.04561 (15)	0.3696 (3)	0.5654 (3)	0.0621 (9)
C5	0.00383 (16)	0.4186 (3)	0.6183 (3)	0.0685 (10)
H5	0.0042	0.4902	0.6469	0.082*
C6	0.05217 (14)	0.3609 (3)	0.6283 (3)	0.0603 (9)
H6	0.0853	0.3916	0.6659	0.072*
C7	0.10663 (12)	0.1866 (3)	0.5932 (2)	0.0456 (7)
H7	0.0983	0.1127	0.5597	0.055*
C8	0.14678 (12)	0.2529 (3)	0.5513 (2)	0.0467 (7)
C9	0.19109 (13)	0.3174 (3)	0.6071 (2)	0.0527 (8)
S1	0.19598 (4)	0.18989 (8)	0.88153 (6)	0.0567 (2)
C10	0.17799 (13)	0.2276 (3)	0.7561 (2)	0.0530 (8)
C11	0.14183 (14)	0.0864 (3)	0.8572 (3)	0.0602 (9)
H11	0.1333	0.0420	0.9050	0.072*
C12	0.11427 (14)	0.0811 (3)	0.7576 (3)	0.0560 (8)
C13	0.06841 (13)	0.0014 (3)	0.7070 (3)	0.0637 (10)
H13A	0.0659	−0.0591	0.7511	0.096*
H13B	0.0756	−0.0303	0.6498	0.096*
H13C	0.0344	0.0429	0.6880	0.096*
C14	0.23141 (13)	0.3942 (3)	0.5837 (3)	0.0580 (9)
H14A	0.2168	0.4228	0.5180	0.087*
H14B	0.2647	0.3528	0.5886	0.087*
H14C	0.2391	0.4570	0.6290	0.087*
C15	0.13101 (13)	0.2443 (3)	0.4422 (2)	0.0489 (7)
C16	0.14414 (14)	0.3068 (3)	0.2878 (2)	0.0578 (8)
H16A	0.1052	0.3235	0.2631	0.069*
H16B	0.1504	0.2310	0.2656	0.069*
C17	0.17647 (16)	0.3939 (3)	0.2484 (3)	0.0656 (10)

H17C	0.2148	0.3753	0.2708	0.098*
H17D	0.1707	0.4687	0.2715	0.098*
H17E	0.1643	0.3930	0.1780	0.098*
Cl1	−0.10751 (4)	0.44589 (8)	0.55951 (7)	0.0653 (2)
N1	0.20339 (11)	0.3071 (3)	0.7143 (2)	0.0524 (7)
H1A	0.2272 (15)	0.352 (3)	0.751 (3)	0.063*
N2	0.13435 (11)	0.1668 (2)	0.70220 (18)	0.0512 (6)
O1	0.09178 (10)	0.1876 (2)	0.39502 (17)	0.0640 (6)
O2	0.16151 (9)	0.31004 (19)	0.39710 (16)	0.0576 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0658 (2)	0.0651 (2)	0.0608 (2)	0.02571 (17)	0.02317 (17)	0.01386 (17)
C1	0.058 (2)	0.063 (2)	0.0365 (16)	0.0073 (16)	0.0130 (14)	0.0039 (15)
C2	0.066 (2)	0.0456 (18)	0.062 (2)	−0.0010 (16)	0.0062 (18)	−0.0170 (16)
C3	0.056 (2)	0.063 (2)	0.071 (2)	−0.0100 (17)	−0.0033 (18)	−0.0028 (19)
C4	0.067 (2)	0.057 (2)	0.063 (2)	0.0246 (17)	0.0194 (18)	0.0080 (18)
C5	0.071 (2)	0.066 (2)	0.076 (2)	0.0059 (18)	0.032 (2)	−0.016 (2)
C6	0.0563 (19)	0.0502 (19)	0.067 (2)	0.0073 (16)	0.0069 (16)	−0.0057 (17)
C7	0.0529 (17)	0.0482 (17)	0.0347 (15)	0.0020 (13)	0.0110 (13)	−0.0028 (13)
C8	0.0483 (17)	0.0397 (16)	0.0513 (17)	0.0009 (13)	0.0130 (14)	0.0051 (13)
C9	0.0566 (18)	0.0418 (17)	0.0519 (18)	0.0098 (14)	0.0037 (15)	0.0119 (14)
S1	0.0619 (5)	0.0638 (5)	0.0379 (4)	0.0063 (4)	0.0039 (4)	−0.0014 (4)
C10	0.0530 (18)	0.0535 (19)	0.0436 (17)	0.0054 (15)	0.0000 (14)	0.0027 (14)
C11	0.059 (2)	0.055 (2)	0.067 (2)	0.0177 (16)	0.0182 (17)	0.0049 (17)
C12	0.060 (2)	0.0515 (19)	0.056 (2)	−0.0008 (15)	0.0154 (16)	0.0025 (15)
C13	0.0506 (19)	0.061 (2)	0.075 (2)	−0.0112 (16)	0.0112 (17)	0.0335 (18)
C14	0.0406 (17)	0.064 (2)	0.063 (2)	0.0054 (14)	0.0052 (15)	0.0018 (17)
C15	0.0542 (18)	0.0412 (16)	0.0512 (17)	0.0069 (14)	0.0153 (15)	−0.0062 (14)
C16	0.0500 (18)	0.074 (2)	0.0516 (19)	0.0026 (16)	0.0178 (15)	0.0031 (17)
C17	0.078 (2)	0.062 (2)	0.062 (2)	0.0046 (18)	0.0274 (19)	0.0238 (18)
Cl1	0.0674 (5)	0.0663 (6)	0.0663 (5)	0.0288 (4)	0.0256 (4)	0.0153 (4)
N1	0.0506 (16)	0.0581 (17)	0.0463 (15)	−0.0042 (12)	0.0104 (12)	−0.0016 (13)
N2	0.0575 (16)	0.0523 (16)	0.0432 (15)	0.0045 (12)	0.0137 (12)	0.0031 (12)
O1	0.0707 (15)	0.0666 (16)	0.0556 (14)	−0.0212 (12)	0.0198 (12)	−0.0160 (12)
O2	0.0584 (14)	0.0594 (14)	0.0568 (14)	0.0008 (11)	0.0193 (11)	0.0104 (11)

Geometric parameters (Å, °)

C1—C2	1.370 (5)	C10—N1	1.363 (4)
C1—C6	1.405 (5)	C11—C12	1.389 (5)
C1—C7	1.587 (4)	C11—H11	0.9300
C2—C3	1.398 (5)	C12—N2	1.456 (4)
C2—H2	0.9300	C12—C13	1.505 (5)
C3—C4	1.401 (5)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.389 (5)	C13—H13C	0.9600
C4—Cl1	1.791 (3)	C14—H14A	0.9600

C5—C6	1.375 (5)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C6—H6	0.9300	C15—O1	1.225 (4)
C7—N2	1.524 (4)	C15—O2	1.376 (4)
C7—C8	1.534 (4)	C16—O2	1.489 (4)
C7—H7	0.9800	C16—C17	1.516 (5)
C8—C9	1.398 (4)	C16—H16A	0.9700
C8—C15	1.492 (4)	C16—H16B	0.9700
C9—N1	1.471 (4)	C17—H17C	0.9600
C9—C14	1.473 (5)	C17—H17D	0.9600
S1—C10	1.766 (3)	C17—H17E	0.9600
S1—C11	1.792 (4)	N1—H1A	0.86 (4)
C10—N2	1.355 (4)		
C2—C1—C6	120.5 (3)	C11—C12—N2	111.7 (3)
C2—C1—C7	119.5 (3)	C11—C12—C13	127.5 (3)
C6—C1—C7	120.0 (3)	N2—C12—C13	120.8 (3)
C1—C2—C3	121.0 (3)	C12—C13—H13A	109.5
C1—C2—H2	119.5	C12—C13—H13B	109.5
C3—C2—H2	119.5	H13A—C13—H13B	109.5
C2—C3—C4	117.8 (3)	C12—C13—H13C	109.5
C2—C3—H3	121.1	H13A—C13—H13C	109.5
C4—C3—H3	121.1	H13B—C13—H13C	109.5
C5—C4—C3	121.3 (3)	C9—C14—H14A	109.5
C5—C4—Cl1	117.9 (3)	C9—C14—H14B	109.5
C3—C4—Cl1	120.8 (3)	H14A—C14—H14B	109.5
C6—C5—C4	119.8 (3)	C9—C14—H14C	109.5
C6—C5—H5	120.1	H14A—C14—H14C	109.5
C4—C5—H5	120.1	H14B—C14—H14C	109.5
C5—C6—C1	119.5 (3)	O1—C15—O2	121.9 (3)
C5—C6—H6	120.3	O1—C15—C8	122.6 (3)
C1—C6—H6	120.3	O2—C15—C8	115.4 (3)
N2—C7—C8	107.9 (2)	O2—C16—C17	109.9 (3)
N2—C7—C1	108.8 (2)	O2—C16—H16A	109.7
C8—C7—C1	111.9 (2)	C17—C16—H16A	109.7
N2—C7—H7	109.4	O2—C16—H16B	109.7
C8—C7—H7	109.4	C17—C16—H16B	109.7
C1—C7—H7	109.4	H16A—C16—H16B	108.2
C9—C8—C15	123.9 (3)	C16—C17—H17C	109.5
C9—C8—C7	124.8 (3)	C16—C17—H17D	109.5
C15—C8—C7	111.2 (3)	H17C—C17—H17D	109.5
C8—C9—N1	116.1 (3)	C16—C17—H17E	109.5
C8—C9—C14	134.5 (3)	H17C—C17—H17E	109.5
N1—C9—C14	109.4 (3)	H17D—C17—H17E	109.5
C10—S1—C11	90.78 (17)	C10—N1—C9	121.5 (3)
N2—C10—N1	121.7 (3)	C10—N1—H1A	119 (2)
N2—C10—S1	111.8 (2)	C9—N1—H1A	119 (2)
N1—C10—S1	126.5 (2)	C10—N2—C12	114.4 (3)
C12—C11—S1	111.1 (3)	C10—N2—C7	124.0 (3)
C12—C11—H11	124.4	C12—N2—C7	121.6 (3)

S1—C11—H11	124.4	C15—O2—C16	115.8 (2)

Fig. 1