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5-Chloro-2-methylsulfonyl-1,2,4-triazolo[1,5-*a*]quinazoline

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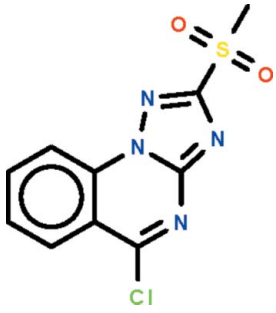
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.113; data-to-parameter ratio = 14.4.

The triazoloquinazole fused-ring system of the title compound, $C_{10}H_7ClN_4O_2S$, is essentially planar (r.m.s. deviation = 0.009 Å). In the methylsulfonyl substituent, the two S—O bonds are of equal length [1.402 (2) Å]. In the crystal, adjacent molecules interact weakly through Cl···N contacts [*ca* 3.197 (2) Å].

Related literature

For the synthesis of the precursor, see: Al-Salahi & Geffken (2011).



Experimental

Crystal data

$C_{10}H_7ClN_4O_2S$
M_r = 282.71
Monoclinic, $P2_1/c$
a = 12.6386 (3) Å
b = 10.7464 (3) Å
c = 8.6317 (3) Å
β = 102.459 (3)°
V = 1144.74 (6) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 4.69 mm^{-1}
T = 294 K
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.334, T_{max} = 0.454
10130 measured reflections
2383 independent reflections
2168 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.113
S = 1.03
2383 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO* program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5916).

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supplementary materials

Acta Cryst. (2012). E**68**, o1809 [doi:10.1107/S1600536812021770]

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Comment

In this study, 2-(methylsulfanyl)-[1,2,4]triazolo[1,5-*a*]quinazolin-5-one (Al-Salahi & Geffken, 2011) was first treated with phosphorus oxychloride to yield the chlorinated compound, whose sulfur linkage was then oxidized by hydrogen peroxide. Chlorination took place at the carbon atom bearing the ketonic oxygen in the title compound (Scheme I). The triazoloquinazole fused-ring system is planar; in the methanesulfonyl substitutent, the two S–O bonds are of equal length (1.402 (2) Å). Adjacent molecules interact weakly through Cl···N contacts (*ca*. 3.20 Å).

Experimental

Under ice-cold conditions, 2-hydrazinobenzoic acid (10 mmol, 1.52 g) was added to a solution of dimethyl *N*-cyano-dithioimidocarbonate (10 mmol, 1.46 g) in ethanol (20 ml). Triethylamine (30 mmol, 3.03 g) was added. The reaction mixture was stirred overnight at room temperature. Concentrated hydrochloric acid was added; the acidified mixture for heated for an hour. The mixture was poured into ice water; the solid that formed was collected and recrystallized from ethanol to give colorless crystals of 2-(methylsulfanyl)-[1,2,4]triazolo[1,5-*a*]quinazolin-5-one. The procedure was that reported earlier (Al-Salahi & Geffken, 2011).

The above compound (1 mmol, 0.232 g) was heated with phosphorus oxychloride (1 ml) in benzene (10 ml) for 2 h. The solvent was evaporated and the residue was treated with saturated potassium carbonate to give the chlorinated [1,2,4]triazoloquinazoline.

To the boiling mixture of chlorinated [1,2,4]triazoloquinazoline (1 mmol, 0.25 g) in glacial acetic acid (5 ml) was added hydrogen peroxide. Colorless crystals of the oxidized product were obtained when the solution was allowed to cool.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.93 to 0.96 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

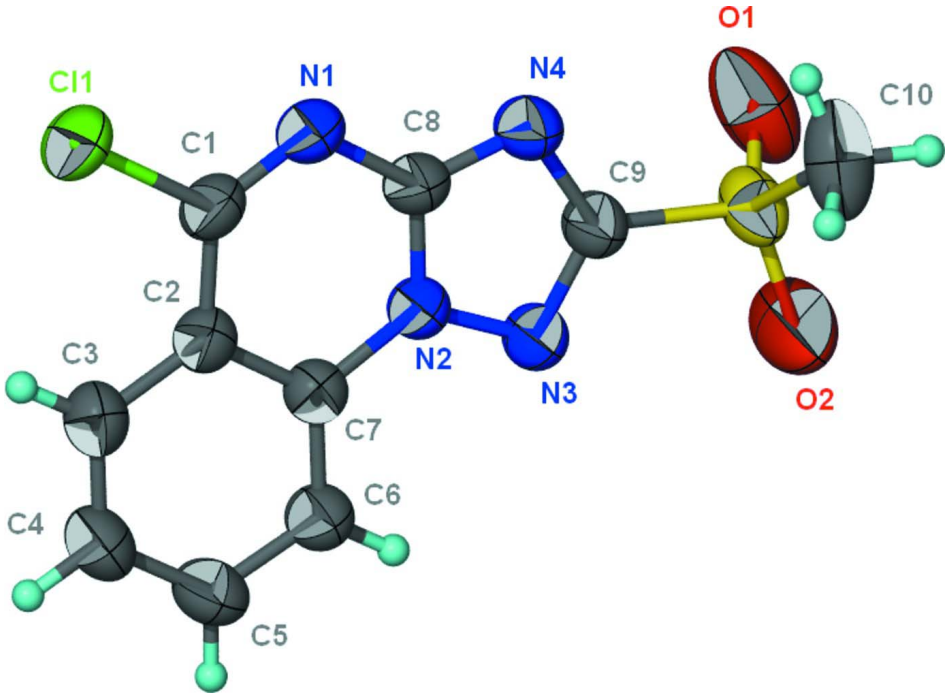

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{10}H_7ClN_4O_2S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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Crystal data

$C_{10}H_7ClN_4O_2S$
$M_r = 282.71$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 12.6386$ (3) Å
$b = 10.7464$ (3) Å
$c = 8.6317$ (3) Å
$\beta = 102.459$ (3)°
$V = 1144.74$ (6) Å^3
$Z = 4$
$F(000) = 576$
$D_x = 1.640$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 4932 reflections
$\theta = 3.6$–$76.5°$
$\mu = 4.69$ mm^{-1}
$T = 294$ K
Prism, colorless
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.334$, $T_{max} = 0.454$
10130 measured reflections
2383 independent reflections
2168 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 76.7°$, $\theta_{min} = 3.6°$
$h = -15\rightarrow12$
$k = -13\rightarrow12$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.113$
$S = 1.03$
2383 reflections
165 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.3878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0033 (6)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.45911 (4)	0.43675 (5)	0.75576 (7)	0.05379 (19)
S1	0.88914 (4)	0.86294 (4)	0.54205 (6)	0.04933 (19)
O1	0.82482 (19)	0.94586 (18)	0.4366 (2)	0.0828 (7)
O2	0.9760 (2)	0.8096 (2)	0.4887 (4)	0.1080 (10)
N1	0.58990 (12)	0.60611 (16)	0.6966 (2)	0.0445 (4)
N2	0.74566 (12)	0.56267 (13)	0.59166 (18)	0.0365 (3)
N3	0.82959 (12)	0.62371 (15)	0.55072 (19)	0.0404 (3)
N4	0.71600 (13)	0.75914 (15)	0.6396 (2)	0.0449 (4)
C1	0.57202 (14)	0.48755 (18)	0.6911 (2)	0.0412 (4)
C2	0.63661 (14)	0.39385 (17)	0.6353 (2)	0.0388 (4)
C3	0.61415 (16)	0.26559 (18)	0.6285 (3)	0.0467 (4)
H3A	0.5539	0.2353	0.6620	0.056*
C4	0.68132 (17)	0.18528 (19)	0.5721 (3)	0.0518 (5)
H4	0.6663	0.1005	0.5680	0.062*
C5	0.77164 (17)	0.22883 (19)	0.5209 (3)	0.0498 (5)
H5	0.8158	0.1727	0.4826	0.060*
C6	0.79670 (16)	0.35396 (17)	0.5261 (2)	0.0431 (4)
H6	0.8573	0.3829	0.4924	0.052*
C7	0.72873 (14)	0.43547 (16)	0.5832 (2)	0.0368 (4)
C8	0.67944 (14)	0.64414 (16)	0.6455 (2)	0.0394 (4)
C9	0.80569 (15)	0.74008 (17)	0.5817 (2)	0.0408 (4)
C10	0.9299 (2)	0.9347 (3)	0.7255 (3)	0.0708 (8)
H10A	0.9786	1.0017	0.7171	0.106*
H10B	0.9662	0.8752	0.8018	0.106*
H10C	0.8676	0.9668	0.7591	0.106*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0401 (3)	0.0539 (3)	0.0736 (4)	−0.00281 (18)	0.0258 (2)	0.0039 (2)
S1	0.0629 (3)	0.0383 (3)	0.0549 (3)	−0.0106 (2)	0.0306 (2)	−0.00469 (19)
O1	0.1214 (18)	0.0528 (10)	0.0643 (11)	−0.0205 (10)	−0.0021 (11)	0.0108 (8)
O2	0.1003 (16)	0.0681 (13)	0.189 (3)	−0.0195 (11)	0.1040 (18)	−0.0260 (15)
N1	0.0364 (7)	0.0418 (8)	0.0590 (10)	0.0016 (6)	0.0187 (7)	−0.0013 (7)

N2	0.0350 (7)	0.0340 (7)	0.0427 (8)	−0.0004 (5)	0.0135 (6)	−0.0003 (6)
N3	0.0413 (8)	0.0373 (8)	0.0470 (8)	−0.0019 (6)	0.0194 (6)	−0.0006 (6)
N4	0.0438 (8)	0.0358 (8)	0.0602 (10)	0.0010 (6)	0.0224 (7)	−0.0011 (7)
C1	0.0319 (8)	0.0437 (10)	0.0497 (10)	0.0000 (7)	0.0126 (7)	0.0031 (8)
C2	0.0352 (8)	0.0378 (9)	0.0435 (9)	−0.0012 (7)	0.0088 (7)	0.0013 (7)
C3	0.0438 (9)	0.0404 (10)	0.0566 (11)	−0.0060 (8)	0.0124 (8)	0.0012 (8)
C4	0.0567 (12)	0.0351 (10)	0.0645 (13)	−0.0044 (8)	0.0154 (10)	−0.0030 (8)
C5	0.0534 (11)	0.0378 (10)	0.0606 (12)	0.0038 (8)	0.0177 (9)	−0.0055 (8)
C6	0.0418 (9)	0.0392 (9)	0.0512 (10)	0.0004 (7)	0.0165 (8)	−0.0006 (7)
C7	0.0361 (8)	0.0349 (9)	0.0396 (9)	0.0000 (6)	0.0082 (7)	0.0005 (7)
C8	0.0358 (8)	0.0372 (9)	0.0475 (10)	0.0026 (7)	0.0140 (7)	−0.0007 (7)
C9	0.0422 (9)	0.0357 (9)	0.0480 (10)	−0.0018 (7)	0.0176 (8)	−0.0005 (7)
C10	0.0859 (18)	0.0761 (17)	0.0471 (12)	−0.0406 (14)	0.0070 (11)	0.0010 (11)

Geometric parameters (Å, º)

Cl1—C1	1.7288 (19)	C2—C3	1.406 (3)
S1—O1	1.402 (2)	C2—C7	1.408 (2)
S1—O2	1.402 (2)	C3—C4	1.371 (3)
S1—C10	1.737 (2)	C3—H3A	0.9300
S1—C9	1.7689 (19)	C4—C5	1.391 (3)
N1—C1	1.293 (3)	C4—H4	0.9300
N1—C8	1.363 (2)	C5—C6	1.380 (3)
N2—N3	1.357 (2)	C5—H5	0.9300
N2—C8	1.360 (2)	C6—C7	1.389 (3)
N2—C7	1.383 (2)	C6—H6	0.9300
N3—C9	1.327 (2)	C10—H10A	0.9600
N4—C8	1.324 (2)	C10—H10B	0.9600
N4—C9	1.349 (2)	C10—H10C	0.9600
C1—C2	1.443 (3)		
O1—S1—O2	115.62 (17)	C5—C4—H4	119.5
O1—S1—C10	109.07 (14)	C6—C5—C4	121.05 (19)
O2—S1—C10	112.43 (17)	C6—C5—H5	119.5
O1—S1—C9	108.29 (11)	C4—C5—H5	119.5
O2—S1—C9	107.48 (11)	C5—C6—C7	118.07 (18)
C10—S1—C9	103.11 (10)	C5—C6—H6	121.0
C1—N1—C8	115.74 (16)	C7—C6—H6	121.0
N3—N2—C8	110.54 (14)	N2—C7—C6	122.78 (17)
N3—N2—C7	125.77 (15)	N2—C7—C2	115.21 (16)
C8—N2—C7	123.68 (15)	C6—C7—C2	122.01 (17)
C9—N3—N2	100.26 (14)	N4—C8—N2	109.99 (16)
C8—N4—C9	101.57 (15)	N4—C8—N1	127.82 (17)
N1—C1—C2	126.35 (17)	N2—C8—N1	122.18 (16)
N1—C1—Cl1	116.69 (14)	N3—C9—N4	117.62 (16)
C2—C1—Cl1	116.95 (14)	N3—C9—S1	119.54 (14)
C3—C2—C7	118.11 (17)	N4—C9—S1	122.82 (14)
C3—C2—C1	125.06 (17)	S1—C10—H10A	109.5
C7—C2—C1	116.83 (16)	S1—C10—H10B	109.5
C4—C3—C2	119.81 (18)	H10A—C10—H10B	109.5

C4—C3—H3A	120.1	S1—C10—H10C	109.5
C2—C3—H3A	120.1	H10A—C10—H10C	109.5
C3—C4—C5	120.94 (19)	H10B—C10—H10C	109.5
C3—C4—H4	119.5		

C8—N2—N3—C9	−0.9 (2)	C3—C2—C7—C6	0.0 (3)
C7—N2—N3—C9	179.96 (17)	C1—C2—C7—C6	−179.65 (17)
C8—N1—C1—C2	−0.6 (3)	C9—N4—C8—N2	−0.5 (2)
C8—N1—C1—Cl1	179.85 (14)	C9—N4—C8—N1	179.41 (19)
N1—C1—C2—C3	−179.0 (2)	N3—N2—C8—N4	0.9 (2)
Cl1—C1—C2—C3	0.5 (3)	C7—N2—C8—N4	−179.93 (17)
N1—C1—C2—C7	0.7 (3)	N3—N2—C8—N1	−178.95 (16)
Cl1—C1—C2—C7	−179.81 (13)	C7—N2—C8—N1	0.2 (3)
C7—C2—C3—C4	0.0 (3)	C1—N1—C8—N4	−179.7 (2)
C1—C2—C3—C4	179.63 (19)	C1—N1—C8—N2	0.2 (3)
C2—C3—C4—C5	−0.2 (3)	N2—N3—C9—N4	0.7 (2)
C3—C4—C5—C6	0.4 (3)	N2—N3—C9—S1	−177.85 (13)
C4—C5—C6—C7	−0.3 (3)	C8—N4—C9—N3	−0.2 (2)
N3—N2—C7—C6	−1.7 (3)	C8—N4—C9—S1	178.32 (15)
C8—N2—C7—C6	179.27 (18)	O1—S1—C9—N3	122.07 (19)
N3—N2—C7—C2	178.86 (16)	O2—S1—C9—N3	−3.5 (2)
C8—N2—C7—C2	−0.1 (3)	C10—S1—C9—N3	−122.44 (19)
C5—C6—C7—N2	−179.21 (19)	O1—S1—C9—N4	−56.4 (2)
C5—C6—C7—C2	0.2 (3)	O2—S1—C9—N4	178.1 (2)
C3—C2—C7—N2	179.43 (17)	C10—S1—C9—N4	59.1 (2)
C1—C2—C7—N2	−0.2 (2)		