

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-Methylsulfanyl-5-phenyl-1,2,4-triazine

Salha Hamri,^a Abderrafia Hafid,^a Mostafa Khouili,^{a*}
Lahcen El Ammari^b and El Mostafa Ketatni^c^aLaboratoire de Chimie Organique et Analytique, Université Sultan Moulay Slimane, Faculté des Sciences et Techniques, BP 523, 23000 Béni-Mellal, Morocco,^bLaboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Battouta, BP 1014, Rabat, Morocco, and^cLaboratoire de Spectrochimie Appliquée et Environnement, Université Sultan Moulay Slimane, Faculté des Sciences et Techniques, BP 523, 23000 Béni-Mellal, Morocco
Correspondence e-mail: m.khouili@usms.ma

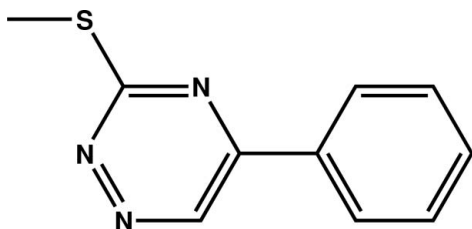
Received 14 May 2014; accepted 21 May 2014

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.037; wR factor = 0.110; data-to-parameter ratio = 22.5.

In the molecule of the title compound, $\text{C}_{10}\text{H}_9\text{N}_3\text{S}$, the dihedral angle between the triazine and phenyl rings is $11.77(7)^\circ$. In the crystal, molecules are linked by π - π stacking interactions [centroid-centroid distances = $3.7359(3)$ and $3.7944(4)$ Å], forming layers parallel to the bc plane.

Related literature

For the biological activity of sulfonamides, see: Abd el-Samii (1992); Kidwai *et al.* (1998); Holla *et al.* (2001); Abdel-Rahman *et al.* (1999); Hay *et al.* (2004); Sztanke *et al.* (2005). For the structure of a similar compound, see: Wen *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{10}\text{H}_9\text{N}_3\text{S}$ $M_r = 203.26$

Monoclinic, $P2_1/c$
 $a = 7.7513(3)$ Å
 $b = 12.9191(5)$ Å
 $c = 9.8262(3)$ Å
 $\beta = 94.584(2)^\circ$
 $V = 980.85(6)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 296$ K
 $0.41 \times 0.35 \times 0.29$ mm

Data collection

Bruker X8 APEX diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2009)
 $T_{\min} = 0.693$, $T_{\max} = 0.747$

15588 measured reflections
 2859 independent reflections
 2544 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.110$
 $S = 1.10$
 2859 reflections

127 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.32$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: PLATON (Spek, 2009) and publCIF (Westrip, 2010).

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements, and the University Sultan Moulay Slimane, Beni-Mellal, for financial support.

Supporting information for this paper is available from the IUCr electronic archives (Reference: RZ5126).

References

- Abdel-Rahman, R. M., Morsy, J. M., Hanafy, F. & Amene, H. A. (1999). *Pharmazie*, **54**, 347–351.
 Abd el-Samii, Z. K. (1992). *J. Chem. Technol. Biotechnol.* **53**, 143–146.
 Bruker (2009). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
 Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
 Hay, M. P., Prujin, F. B., Gamage, S. A., Liyanage, H. D. & Wilson, W. R. (2004). *J. Med. Chem.* **47**, 475–488.
 Holla, B. S., Gonsalves, R., Rao, B. S. & Gopala Krishna, H. N. (2001). *II Farmaco*, **56**, 899–903.
 Kidwai, M., Goel, Y. & Kumar, R. (1998). *Indian J. Chem. Sect. B*, **37**, 174–179.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Sztanke, K., Fidecka, S., Kedzierska, E., Karczmarzyk, Z., Pihlaja, K. & Matosiuk, D. (2005). *Eur. J. Med. Chem.* **40**, 127–134.
 Wen, L.-R., Zhou, J.-X. & Liu, P. (2006). *Acta Cryst.* **E62**, o4704–o4705.
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2014). E70, o720 [doi:10.1107/S1600536814011830]

3-Methylsulfanyl-5-phenyl-1,2,4-triazine

Salha Hamri, Abderrafia Hafid, Mostafa Khouili, Lahcen El Ammari and El Mostafa Ketatni

S1. Comment

1,2,4-Triazine derivatives have been widely studied in terms of their synthetic methodologies and reactivity since some of these compounds were reported to have promising biological activities, including antimicrobial, inflammatory, analgesic, antiviral and anthelmintic activities (Abd el-Samii, 1992; Kidwai *et al.*, 1998; Holla *et al.*, 2001; Abdel-Rahman *et al.*, 1999; Hay *et al.*, 2004; Sztanke *et al.*, 2005). We synthesized the title compound and describe its structure here.

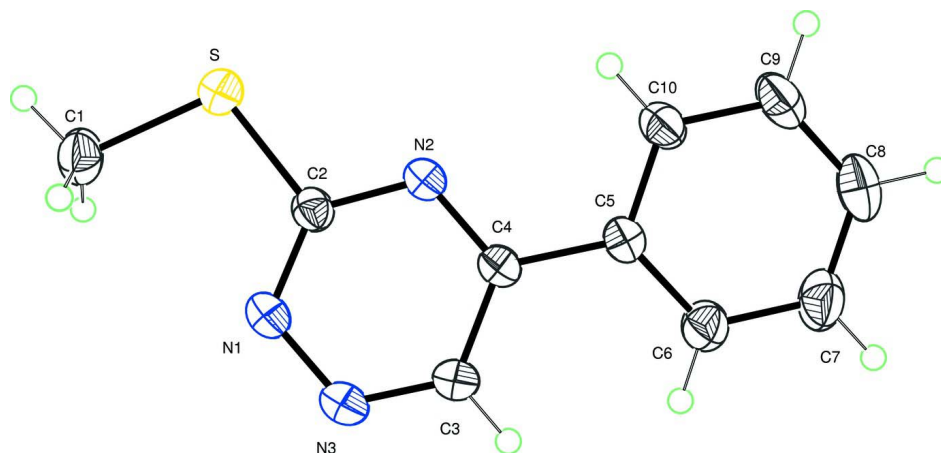
The molecular structure of 3-methylsulfanyl-5-phenyl-1,2,4-triazine is shown in Fig. 1. The triazine ring (N1–N3/C2–C4) forms a dihedral angle of 11.77 (7)° with the phenyl ring. Bond lengths and angles are compatible with those found in a related compound (Wen *et al.*, 2006). The cohesion of the crystal structure is ensured by $\pi\cdots\pi$ stacking interaction between triazine and phenyl rings [intercentroid distance = 3.7944 (4) Å] and between centrosymmetrically related triazine rings [intercentroid distance = 3.7359 (3) Å], forming molecular layers parallel to the *bc* plane.

S2. Experimental

To a solution of thiosemicarbazide (50 g, 0.55 mol) in absolute ethanol (500 ml) was added iodomethane (34.1 ml, 0.55 mol). The reaction mixture was stirred at reflux for 3 h then cooled overnight in the refrigerator. The crystals formed were filtered on a Buchner funnel, washed with ethanol and dried *in vacuo* to give the *S*-methylthiosemicarbazide iodohydrate beige powder (123 g). An aqueous solution (200 ml) of *S*-methylthiosemicarbazide iodohydrate (20 g, 85.8 mmol) was added to an aqueous solution (120 ml) of phenyl glyoxal (15.65 g, 103 mmol) and sodium bicarbonate (10 g, 94.4 mmol) at 5°C. The temperature was maintained at 5°C for 6 h then the reaction medium was extracted with dichloromethane. The organic phases are combined, dried over magnesium sulfate, filtered and evaporated under reduced pressure. The crude material was purified by column on silica (hexane/EtOAc 9:1 *v/v*) as eluent. The title compound (13.95 g) was obtained as yellow crystals (79% yield) on slow evaporation of the solvent.

S3. Refinement

H atoms were located in a difference Fourier map and treated as riding, with C–H = 0.93–0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms. One outlier (-1 7 1) was omitted in the last cycles of refinement.

**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small circles.

3-Methylsulfonyl-5-phenyl-1,2,4-triazine

Crystal data

$C_{10}H_9N_3S$
 $M_r = 203.26$
 Monoclinic, $P2_1/c$
 Hall symbol: $-P\ 2ybc$
 $a = 7.7513\ (3)\ \text{\AA}$
 $b = 12.9191\ (5)\ \text{\AA}$
 $c = 9.8262\ (3)\ \text{\AA}$
 $\beta = 94.584\ (2)^\circ$
 $V = 980.85\ (6)\ \text{\AA}^3$
 $Z = 4$

$F(000) = 424$
 $D_x = 1.376\ \text{Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$
 Cell parameters from 2859 reflections
 $\theta = 2.6\text{--}30.0^\circ$
 $\mu = 0.29\ \text{mm}^{-1}$
 $T = 296\ \text{K}$
 Block, yellow
 $0.41 \times 0.35 \times 0.29\ \text{mm}$

Data collection

Bruker X8 APEX
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2009)
 $T_{\min} = 0.693$, $T_{\max} = 0.747$

15588 measured reflections
 2859 independent reflections
 2544 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -10 \rightarrow 10$
 $k = -18 \rightarrow 16$
 $l = -11 \rightarrow 13$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.110$
 $S = 1.10$
 2859 reflections
 127 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
 $w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.210P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.32\ \text{e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.24\ \text{e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S	0.23977 (4)	0.83039 (3)	0.60310 (3)	0.04391 (12)
N1	0.55958 (14)	0.87884 (9)	0.70043 (10)	0.0411 (2)
N2	0.49883 (12)	0.86358 (7)	0.45807 (9)	0.0321 (2)
N3	0.72612 (15)	0.90131 (11)	0.68242 (11)	0.0492 (3)
C1	0.2219 (2)	0.83401 (12)	0.78375 (15)	0.0478 (3)
H1A	0.1053	0.8176	0.8025	0.072*
H1B	0.2509	0.9020	0.8177	0.072*
H1C	0.2997	0.7844	0.8278	0.072*
C2	0.45673 (14)	0.86159 (8)	0.58822 (11)	0.0315 (2)
C3	0.77637 (16)	0.90344 (12)	0.55773 (13)	0.0434 (3)
H3	0.8915	0.9188	0.5461	0.052*
C4	0.66358 (14)	0.88349 (8)	0.44103 (11)	0.0308 (2)
C5	0.72027 (15)	0.88557 (8)	0.30105 (11)	0.0324 (2)
C6	0.89529 (17)	0.88858 (11)	0.27791 (14)	0.0439 (3)
H6	0.9785	0.8887	0.3515	0.053*
C7	0.9461 (2)	0.89133 (13)	0.14577 (16)	0.0537 (4)
H7	1.0631	0.8939	0.1311	0.064*
C8	0.8234 (2)	0.89027 (12)	0.03593 (15)	0.0539 (4)
H8	0.8576	0.8917	-0.0526	0.065*
C9	0.6501 (2)	0.88713 (12)	0.05779 (13)	0.0499 (3)
H9	0.5677	0.8863	-0.0164	0.060*
C10	0.59717 (17)	0.88521 (10)	0.18942 (12)	0.0403 (3)
H10	0.4799	0.8837	0.2032	0.048*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.03440 (17)	0.0619 (2)	0.03562 (17)	-0.00433 (12)	0.00418 (12)	0.00196 (13)
N1	0.0413 (5)	0.0550 (6)	0.0266 (4)	-0.0031 (4)	0.0003 (4)	0.0005 (4)
N2	0.0327 (4)	0.0367 (5)	0.0264 (4)	-0.0001 (3)	0.0000 (3)	0.0010 (3)
N3	0.0410 (6)	0.0750 (8)	0.0305 (5)	-0.0083 (5)	-0.0042 (4)	-0.0019 (5)
C1	0.0498 (7)	0.0555 (8)	0.0397 (7)	-0.0018 (6)	0.0137 (5)	0.0043 (5)
C2	0.0339 (5)	0.0325 (5)	0.0280 (5)	0.0015 (4)	0.0020 (4)	0.0023 (4)
C3	0.0328 (5)	0.0640 (8)	0.0324 (6)	-0.0063 (5)	-0.0023 (4)	-0.0007 (5)
C4	0.0330 (5)	0.0318 (5)	0.0271 (5)	0.0006 (4)	-0.0003 (4)	0.0008 (4)
C5	0.0374 (5)	0.0321 (5)	0.0280 (5)	0.0004 (4)	0.0033 (4)	-0.0002 (4)

C6	0.0389 (6)	0.0537 (7)	0.0396 (6)	0.0064 (5)	0.0060 (5)	0.0013 (5)
C7	0.0509 (8)	0.0642 (9)	0.0485 (8)	0.0080 (7)	0.0197 (6)	0.0016 (7)
C8	0.0753 (10)	0.0539 (8)	0.0350 (6)	0.0041 (7)	0.0192 (6)	-0.0014 (6)
C9	0.0675 (9)	0.0546 (8)	0.0271 (5)	-0.0046 (6)	-0.0002 (5)	-0.0023 (5)
C10	0.0443 (6)	0.0467 (6)	0.0294 (5)	-0.0060 (5)	-0.0006 (4)	-0.0012 (5)

Geometric parameters (Å, °)

S—C2	1.7469 (11)	C4—C5	1.4773 (15)
S—C1	1.7921 (14)	C5—C6	1.3939 (17)
N1—C2	1.3268 (15)	C5—C10	1.3951 (16)
N1—N3	1.3486 (16)	C6—C7	1.3872 (18)
N2—C4	1.3263 (14)	C6—H6	0.9300
N2—C2	1.3451 (14)	C7—C8	1.381 (2)
N3—C3	1.3153 (17)	C7—H7	0.9300
C1—H1A	0.9600	C8—C9	1.378 (2)
C1—H1B	0.9600	C8—H8	0.9300
C1—H1C	0.9600	C9—C10	1.3880 (17)
C3—C4	1.4091 (15)	C9—H9	0.9300
C3—H3	0.9300	C10—H10	0.9300
C2—S—C1	103.19 (6)	C6—C5—C10	119.03 (11)
C2—N1—N3	116.42 (10)	C6—C5—C4	121.21 (10)
C4—N2—C2	115.65 (9)	C10—C5—C4	119.76 (11)
C3—N3—N1	118.99 (10)	C7—C6—C5	120.43 (13)
S—C1—H1A	109.5	C7—C6—H6	119.8
S—C1—H1B	109.5	C5—C6—H6	119.8
H1A—C1—H1B	109.5	C8—C7—C6	120.11 (14)
S—C1—H1C	109.5	C8—C7—H7	119.9
H1A—C1—H1C	109.5	C6—C7—H7	119.9
H1B—C1—H1C	109.5	C9—C8—C7	119.87 (12)
N1—C2—N2	127.71 (10)	C9—C8—H8	120.1
N1—C2—S	119.19 (9)	C7—C8—H8	120.1
N2—C2—S	113.10 (8)	C8—C9—C10	120.68 (13)
N3—C3—C4	122.93 (11)	C8—C9—H9	119.7
N3—C3—H3	118.5	C10—C9—H9	119.7
C4—C3—H3	118.5	C9—C10—C5	119.88 (13)
N2—C4—C3	118.28 (10)	C9—C10—H10	120.1
N2—C4—C5	118.82 (10)	C5—C10—H10	120.1
C3—C4—C5	122.89 (10)		