

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-(4,6-Dimethylpyrimidin-2-ylsulfanyl)-*N*-phenylacetamide

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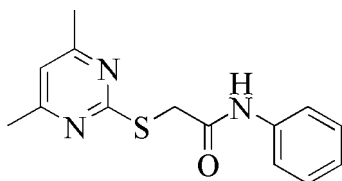
Received 11 March 2008; accepted 24 March 2008

Key indicators: single-crystal X-ray study; $T = 571$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.041; wR factor = 0.116; data-to-parameter ratio = 14.9.

In the title compound, $\text{C}_{14}\text{H}_{15}\text{N}_3\text{OS}$, the phenyl ring is almost perpendicular to the dimethylpyrimidine group, with a dihedral angle of $88.1(3)^\circ$. The Csp^2-S bond of $1.759(3)$ Å is significantly shorter than the Csp^3-S bond of $1.795(3)$ Å due to the $p-\pi$ conjugation.

Related literature

For related literature, see: Koike *et al.* (1999); Liang *et al.* (2008); Wang *et al.* (2004, 2005).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{15}\text{N}_3\text{OS}$	$V = 2952.9(9)$ Å ³
$M_r = 273.35$	$Z = 8$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 9.1691(17)$ Å	$\mu = 0.21$ mm ⁻¹
$b = 15.485(3)$ Å	$T = 571(2)$ K
$c = 20.798(4)$ Å	$0.28 \times 0.24 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	14235 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	2606 independent reflections
$T_{\min} = 0.942$, $T_{\max} = 0.971$	1494 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.073$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	175 parameters
$wR(F^2) = 0.116$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.17$ e Å ⁻³
2606 reflections	$\Delta\rho_{\text{min}} = -0.16$ e Å ⁻³

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge the 05 L003 project supported by the Education Department of Liaoning Province in China and the 2006 SH03 project supported by Anshan Municipal Science and Technology Commission.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2550).

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supporting information

Acta Cryst. (2008). E64, o760 [doi:10.1107/S1600536808007940]

2-(4,6-Dimethylpyrimidin-2-ylsulfanyl)-*N*-phenylacetamide

Li-Xin Gao, Guang-Jun Fang, Jin-Guo Feng, Dong Liang and Wei Wang

S1. Comment

Acetamide is an important class of medical intermidate. Many biologically active compounds are synthesized by using acetamide (Koike *et al.*, 1999). We have reported the synthesis and crystal structure of a acetamide compound, 1,1'-diphenyl-5,5'-[*o*-phenylenebis(methylenethio)]di-1*H*-tetrazole (Liang *et al.*, 2008). Now, a new acetamide derivative, namely 2-(4,6-dimethylpyrimidin-2-ylsulfanyl)-*N*-phenylacetamide (I) are prepared from the reaction of 2-thio-4,6-dimethylpyrimidine with 2-chloro-*N*-phenylacetamide. We present its crystal structure here.

The title compound contains a benzene ring and a dimethylpyrimidine ring. The two methyl groups attached to the pyrimidine ring don't deviate from the pyrimidine ring, with an r.m.s. of 0.0082 (4) Å. The dihedral angle between the benzene ring and dimethylpyrimidine ring is 91.9 (3)°, which indicates that the two aromatic rings are almost perpendicular. The O1—C8—N3—C9 and C8—N3—C9—C14 torsion angles are 2.5 (4) and 164.3 (3)°, indicating that the acetamide is planar with the benzene ring.

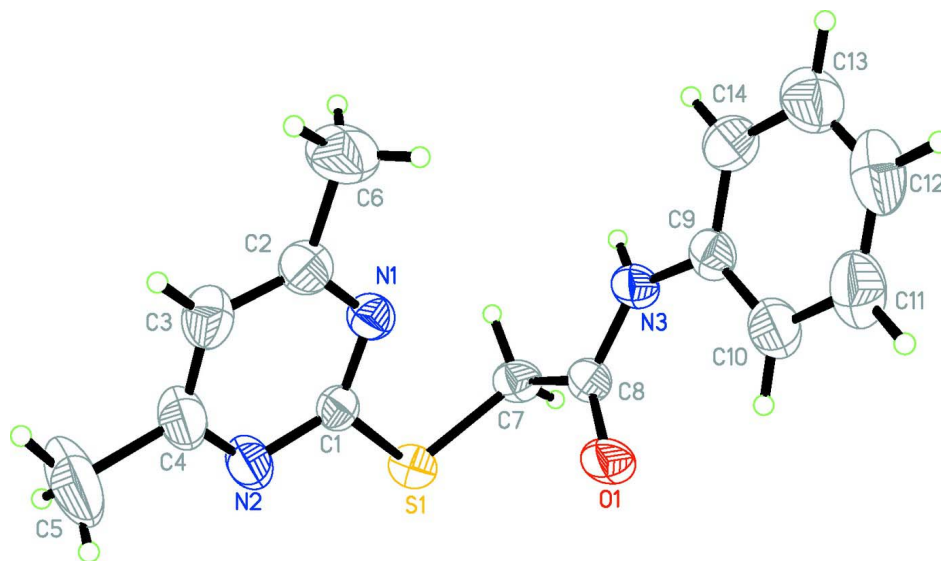
Due to the *p*- π conjugation, the C_{sp^2} —S bond [S1—C1 = 1.759 (3) Å] is significantly shorter than the C_{sp^3} —S bond [C7—S1 = 1.795 (3) Å]. These values compare with the values reported in the literatures (Wang *et al.*, 2004, 2005).

S2. Experimental

The title compound was synthesized by the reaction of 2-thio-4,6-dimethylpyrimidine (2 mmol) with 2-chloro-*N*-phenylacetamide (2 mmol) in refluxing ethanol (40 ml). Single crystals suitable for X-ray analysis were grown by slow evaporation of a chloroform-acetone (1:5 *v/v*) solution.

S3. Refinement

All H atoms were positioned geometrically and refined as riding [N—H = 0.86 Å and C—H = 0.93–0.97 Å]. For the NH, CH and CH₂ groups, $U_{iso}(H)$ values were set equal to 1.2 $U_{eq}(C)$ and for the methyl groups they were set equal to 1.5 $U_{eq}(C)$.

**Figure 1**

View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

2-(4,6-dimethylpyrimidin-2-ylsulfanyl)-*N*-phenylacetamide

Crystal data

$C_{14}H_{15}N_3OS$

$M_r = 273.35$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 9.1691$ (17) Å

$b = 15.485$ (3) Å

$c = 20.798$ (4) Å

$V = 2952.9$ (9) Å³

$Z = 8$

$F(000) = 1152$

$D_x = 1.230$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2395 reflections

$\theta = 2.8$ – 22.1°

$\mu = 0.22$ mm⁻¹

$T = 571$ K

Block, colourless

$0.28 \times 0.24 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.942$, $T_{\max} = 0.971$

14235 measured reflections

2606 independent reflections

1494 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.073$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -8 \rightarrow 10$

$k = -18 \rightarrow 18$

$l = -22 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.116$

$S = 1.01$

2606 reflections

175 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

$$w = 1/[\sigma^2(F_o^2) + (0.044P)^2 + 0.9487P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.17 \text{ e } \text{Å}^{-3}$

$$\Delta\rho_{\min} = -0.16 \text{ e } \text{Å}^{-3}$$

Extinction correction: *SHELXL*,
 $\text{Fc}^* = k\text{Fc}[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0155 (11)

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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.10446 (8)	0.82693 (5)	0.31856 (3)	0.0566 (3)
O1	0.08455 (18)	0.69624 (13)	0.20969 (9)	0.0658 (6)
N1	0.1800 (2)	0.67674 (14)	0.37036 (10)	0.0540 (6)
C8	0.2078 (3)	0.70685 (17)	0.23078 (12)	0.0498 (7)
C1	0.0880 (3)	0.74194 (16)	0.37418 (12)	0.0480 (7)
N3	0.3186 (2)	0.65184 (14)	0.22060 (10)	0.0539 (6)
H3A	0.4015	0.6669	0.2364	0.065*
C7	0.2494 (3)	0.78653 (16)	0.26898 (13)	0.0541 (7)
H7A	0.2793	0.8316	0.2394	0.065*
H7B	0.3324	0.7728	0.2961	0.065*
C9	0.3164 (3)	0.57251 (18)	0.18732 (13)	0.0538 (7)
N2	-0.0195 (3)	0.75292 (15)	0.41631 (11)	0.0665 (7)
C2	0.1643 (3)	0.61439 (19)	0.41447 (15)	0.0643 (8)
C14	0.4321 (4)	0.51737 (19)	0.19623 (14)	0.0703 (9)
H14	0.5079	0.5332	0.2235	0.084*
C10	0.2056 (3)	0.5472 (2)	0.14672 (15)	0.0779 (10)
H10	0.1266	0.5837	0.1396	0.093*
C3	0.0557 (4)	0.6186 (2)	0.45961 (14)	0.0707 (9)
H3	0.0440	0.5748	0.4898	0.085*
C4	-0.0353 (4)	0.6890 (2)	0.45926 (15)	0.0774 (10)
C13	0.4371 (5)	0.4388 (2)	0.16514 (18)	0.0922 (12)
H13	0.5167	0.4025	0.1712	0.111*
C12	0.3259 (6)	0.4141 (2)	0.12546 (18)	0.0939 (12)
H12	0.3289	0.3608	0.1048	0.113*
C6	0.2714 (4)	0.5413 (2)	0.41157 (19)	0.1032 (13)
H6A	0.2749	0.5188	0.3686	0.155*
H6B	0.2415	0.4965	0.4406	0.155*
H6C	0.3663	0.5618	0.4236	0.155*
C11	0.2113 (5)	0.4679 (3)	0.11651 (17)	0.0930 (12)
H11	0.1354	0.4512	0.0896	0.112*
C5	-0.1579 (6)	0.6993 (3)	0.5073 (2)	0.147 (2)

H5A	-0.1525	0.7555	0.5266	0.221*
H5B	-0.1491	0.6559	0.5400	0.221*
H5C	-0.2498	0.6929	0.4857	0.221*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0512 (4)	0.0549 (4)	0.0636 (5)	0.0038 (4)	0.0011 (4)	0.0005 (4)
O1	0.0307 (10)	0.0895 (15)	0.0773 (13)	-0.0027 (10)	-0.0043 (9)	-0.0168 (11)
N1	0.0526 (14)	0.0541 (13)	0.0554 (14)	0.0043 (12)	-0.0011 (11)	-0.0041 (12)
C8	0.0318 (15)	0.0690 (17)	0.0486 (16)	-0.0061 (14)	0.0067 (12)	0.0041 (14)
C1	0.0472 (16)	0.0529 (16)	0.0438 (15)	-0.0019 (14)	-0.0039 (13)	-0.0085 (12)
N3	0.0309 (12)	0.0714 (16)	0.0595 (14)	-0.0028 (11)	0.0009 (10)	-0.0078 (12)
C7	0.0362 (14)	0.0618 (16)	0.0641 (18)	-0.0076 (13)	-0.0001 (13)	0.0049 (14)
C9	0.0445 (16)	0.0656 (18)	0.0514 (17)	-0.0071 (15)	0.0112 (14)	0.0026 (15)
N2	0.0706 (17)	0.0670 (16)	0.0620 (16)	0.0121 (14)	0.0178 (14)	0.0002 (13)
C2	0.072 (2)	0.0547 (18)	0.066 (2)	0.0035 (16)	-0.0021 (18)	-0.0043 (16)
C14	0.075 (2)	0.069 (2)	0.067 (2)	-0.0001 (18)	-0.0013 (17)	0.0073 (16)
C10	0.059 (2)	0.101 (3)	0.073 (2)	-0.0015 (18)	0.0026 (17)	-0.026 (2)
C3	0.092 (2)	0.0622 (19)	0.0583 (19)	-0.0038 (19)	0.0064 (18)	0.0034 (15)
C4	0.089 (2)	0.078 (2)	0.065 (2)	0.009 (2)	0.0241 (19)	0.0003 (18)
C13	0.124 (4)	0.066 (2)	0.087 (3)	0.019 (2)	0.012 (2)	0.007 (2)
C12	0.137 (4)	0.071 (2)	0.074 (3)	-0.017 (3)	0.028 (3)	-0.013 (2)
C6	0.113 (3)	0.075 (2)	0.122 (3)	0.031 (2)	0.010 (3)	0.016 (2)
C11	0.091 (3)	0.109 (3)	0.079 (3)	-0.018 (3)	0.010 (2)	-0.030 (2)
C5	0.175 (5)	0.138 (4)	0.129 (4)	0.045 (3)	0.100 (3)	0.029 (3)

Geometric parameters (Å, °)

S1—C1	1.759 (3)	C14—H14	0.9300
S1—C7	1.795 (3)	C10—C11	1.380 (4)
O1—C8	1.223 (3)	C10—H10	0.9300
N1—C1	1.318 (3)	C3—C4	1.373 (4)
N1—C2	1.340 (3)	C3—H3	0.9300
C8—N3	1.343 (3)	C4—C5	1.512 (5)
C8—C7	1.516 (3)	C13—C12	1.367 (5)
C1—N2	1.330 (3)	C13—H13	0.9300
N3—C9	1.410 (3)	C12—C11	1.354 (5)
N3—H3A	0.8600	C12—H12	0.9300
C7—H7A	0.9700	C6—H6A	0.9600
C7—H7B	0.9700	C6—H6B	0.9600
C9—C14	1.375 (4)	C6—H6C	0.9600
C9—C10	1.378 (4)	C11—H11	0.9300
N2—C4	1.341 (4)	C5—H5A	0.9600
C2—C3	1.370 (4)	C5—H5B	0.9600
C2—C6	1.500 (4)	C5—H5C	0.9600
C14—C13	1.378 (4)		

C1—S1—C7	100.40 (13)	C11—C10—H10	119.9
C1—N1—C2	116.2 (2)	C2—C3—C4	118.4 (3)
O1—C8—N3	123.9 (2)	C2—C3—H3	120.8
O1—C8—C7	122.0 (2)	C4—C3—H3	120.8
N3—C8—C7	114.1 (2)	N2—C4—C3	121.6 (3)
N1—C1—N2	127.7 (2)	N2—C4—C5	116.3 (3)
N1—C1—S1	118.6 (2)	C3—C4—C5	122.1 (3)
N2—C1—S1	113.7 (2)	C12—C13—C14	120.4 (4)
C8—N3—C9	128.2 (2)	C12—C13—H13	119.8
C8—N3—H3A	115.9	C14—C13—H13	119.8
C9—N3—H3A	115.9	C11—C12—C13	119.3 (3)
C8—C7—S1	113.47 (17)	C11—C12—H12	120.4
C8—C7—H7A	108.9	C13—C12—H12	120.4
S1—C7—H7A	108.9	C2—C6—H6A	109.5
C8—C7—H7B	108.9	C2—C6—H6B	109.5
S1—C7—H7B	108.9	H6A—C6—H6B	109.5
H7A—C7—H7B	107.7	C2—C6—H6C	109.5
C14—C9—C10	118.4 (3)	H6A—C6—H6C	109.5
C14—C9—N3	117.6 (3)	H6B—C6—H6C	109.5
C10—C9—N3	124.0 (3)	C12—C11—C10	121.0 (4)
C1—N2—C4	115.1 (2)	C12—C11—H11	119.5
N1—C2—C3	120.9 (3)	C10—C11—H11	119.5
N1—C2—C6	116.5 (3)	C4—C5—H5A	109.5
C3—C2—C6	122.6 (3)	C4—C5—H5B	109.5
C9—C14—C13	120.7 (3)	H5A—C5—H5B	109.5
C9—C14—H14	119.6	C4—C5—H5C	109.5
C13—C14—H14	119.6	H5A—C5—H5C	109.5
C9—C10—C11	120.3 (3)	H5B—C5—H5C	109.5
C9—C10—H10	119.9		
C2—N1—C1—N2	-0.7 (4)	C10—C9—C14—C13	0.1 (4)
C2—N1—C1—S1	178.98 (19)	N3—C9—C14—C13	-179.9 (3)
C7—S1—C1—N1	3.4 (2)	C14—C9—C10—C11	0.6 (5)
C7—S1—C1—N2	-176.94 (19)	N3—C9—C10—C11	-179.3 (3)
O1—C8—N3—C9	-2.5 (4)	N1—C2—C3—C4	-1.0 (4)
C7—C8—N3—C9	179.8 (2)	C6—C2—C3—C4	178.7 (3)
O1—C8—C7—S1	34.4 (3)	C1—N2—C4—C3	1.1 (4)
N3—C8—C7—S1	-147.93 (19)	C1—N2—C4—C5	-179.3 (3)
C1—S1—C7—C8	66.6 (2)	C2—C3—C4—N2	-0.3 (5)
C8—N3—C9—C14	-164.3 (3)	C2—C3—C4—C5	-179.9 (4)
C8—N3—C9—C10	15.6 (4)	C9—C14—C13—C12	-0.8 (5)
N1—C1—N2—C4	-0.6 (4)	C14—C13—C12—C11	0.6 (5)
S1—C1—N2—C4	179.7 (2)	C13—C12—C11—C10	0.1 (6)
C1—N1—C2—C3	1.5 (4)	C9—C10—C11—C12	-0.8 (5)
C1—N1—C2—C6	-178.3 (3)		