organic compounds

N-(2-Chlorophenyl)-2-(4,6-dimethyl-pyrimidin-2-ylsulfanyl)acetamide

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In the title compound, $C_{14}H_{14}ClN_3OS$, the 4,6-dimethyl-pyrimidine ring and the chlorobenzene ring subtend a dihedral angle of 80.0 (2)°. The length of the Csp^2—S bond is significantly shorter than that of the Csp^3—S bond. The crystal structure is stabilized by intermolecular N—H⋯O, C—H⋯O and C—H⋯N hydrogen bonding, and C—H⋯π interactions.

Related literature

For bond-length data, see: Gao *et al.* (2007). For heteroatom-rich compounds as effective precursors for active molecules, see: Huynh *et al.* (2005); Ye *et al.* (2006).
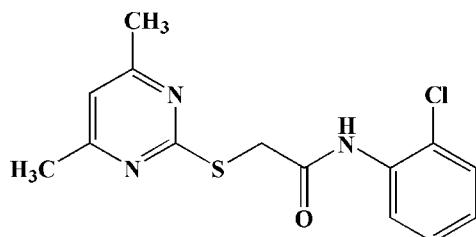
Experimental

Crystal data

$C_{14}H_{14}ClN_3OS$	a = 26.494 (5) Å
M_r = 307.79	b = 4.6736 (9) Å
Orthorhombic, $Pca2_1$	c = 11.931 (2) Å

V = 1477.3 (5) Å^3	μ = 0.40 mm^{-1}
Z = 4	T = 113 K
Mo $K\alpha$ radiation	$0.30 \times 0.26 \times 0.20$ mm

Data collection

Rigaku Saturn diffractometer	8870 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2573 independent reflections
T_{min} = 0.890, T_{max} = 0.925	2445 reflections with $I > 2\sigma(I)$
	R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.061	
S = 1.07	
2573 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
187 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
2 restraints	Absolute structure: Flack (1983), 1199 Freidel pairs
	Flack parameter: 0.00 (5)

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1^i	0.873 (11)	2.054 (12)	2.8414 (18)	149.6 (18)
C2—H2⋯O1^{ii}	0.93	2.46	3.213 (2)	138
C8—H8A⋯Cg1^i	0.97	2.92	3.832 (2)	157
C13—H13B⋯Cg1^{iii}	0.96	2.99	3.592 (2)	122

Symmetry codes: (i) $x, y + 1, z$; (ii) $-x + 1, -y + 2, z + \frac{1}{2}$; (iii) $x, y - 1, z$. Cg1 is the centroid of the N2/N3/C9–C12 ring.

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

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supporting information

N-(2-Chlorophenyl)-2-(4,6-dimethylpyrimidin-2-ylsulfanyl)acetamide

Qiang Li, Wei Wang, Hui Wang, Yan Gao and Hong Qiu

S1. Comment

The heteroatom-rich compounds have been intensively studied due to their applications including effective precursors for active molecule (Ye *et al.*, 2006; Huynh *et al.*, 2005). Now, we have synthesized the title compound, (I), from 4,6-di-methylpyrimidin-2-thiol with 2-chlorophenyl carbamic chloride. Here we report the crystal structure determination of the title compound.

   The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The molecule contains a benzene ring and a pyrimidine ring. The dihedral angle between the benzene ring and benzo[*d*]thiazole ring is 80.0 (2)°, which indicate the two rings are close to be perpendicular. Cl atom attached to the benzene ring is coplanar to the benzene ring with an r.m.s deviation of 0.0130 (3) Å. The deviations with the pyrimidine ring plane of C13 and C14 atoms are 0.0544 (3) and 0.0005 (3) Å, respectively. The C6—N1—C7—C8 torsion angle of 177.61 (15)° indicates that the acyl-amide group are nearly coplanar with the benzene ring plane. As a result of π-π conjugation, the Csp^2—S bond [S1—C9 = 1.7646 (17) Å] is significantly shorter than the Csp^3—S bond [S1—C8 = 1.7947 (17) Å]. These values compare with the values of 1.772 (3) and 1.801 (2) Å reported in the literature (Gao *et al.*, 2007).

   The crystal structure is stabilized by inter molecular C—H···O and C—H···N hydrogen bonding, and C—H···π interactions (Table 1).

S2. Experimental

The title compound was synthesized by the reaction of from the 4,6-dimethylpyrimidin-2-thiol with 2-chlorophenyl carbamic chloride in the refluxing ethanol. Crystals of (I) suitable for single-crystal X-ray analysis were grown by slow evaporation of a solution in chloroform/acetone.

S3. Refinement

The H atoms attached to N atom was located in a different density map and the atomic coordinates allowed to refine freely. Other H atoms were positioned geometrically and refined as riding (C—H = 0.93–0.97 Å) and allowed to ride on their parent atoms, with U_{iso}(H) =1.2U_{eq}(parent) or 1.5U_{eq}(parent).
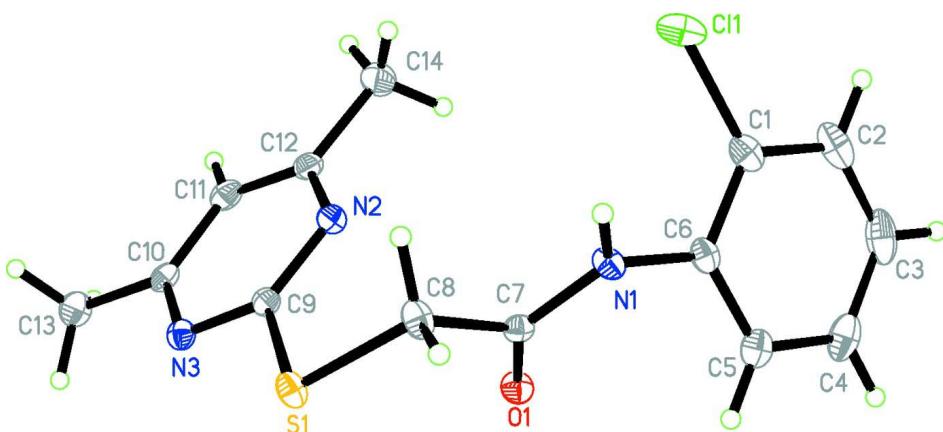
Figure 1

View of the molecule of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level.

N-(2-Chlorophenyl)-2-(4,6-dimethylpyrimidin-2-ylsulfanyl)acetamide

Crystal data

$C_{14}H_{14}ClN_3OS$	$F(000) = 640$
$M_r = 307.79$	$D_x = 1.384$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2c -2ac	Cell parameters from 4682 reflections
$a = 26.494$ (5) Å	$\theta = 1.5–27.9°$
$b = 4.6736$ (9) Å	$\mu = 0.40$ mm^{-1}
$c = 11.931$ (2) Å	$T = 113$ K
$V = 1477.3$ (5) Å^3	Prism, colourless
$Z = 4$	$0.30 \times 0.26 \times 0.20$ mm

Data collection

Rigaku Saturn diffractometer	8870 measured reflections
Radiation source: rotating anode	2573 independent reflections
Confocal monochromator	2445 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.031$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
	$h = -23 \rightarrow 31$
	$k = -5 \rightarrow 5$
$T_{min} = 0.890$, $T_{max} = 0.925$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.024$	
$wR(F^2) = 0.061$	
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2]$
2573 reflections	where $P = (F_o^2 + 2F_c^2)/3$
187 parameters	$(\Delta/\sigma)_{max} = 0.001$
2 restraints	$\Delta\rho_{max} = 0.17$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.20$ e Å^{-3}
	Absolute structure: Flack (1983), 1199 Freidel pairs
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.00 (5)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.689926 (15)	1.07672 (8)	0.28992 (4)	0.02168 (11)
Cl1	0.53054 (2)	1.55461 (10)	0.63460 (4)	0.03848 (14)
O1	0.58637 (4)	0.8871 (2)	0.35668 (11)	0.0216 (3)
N1	0.55577 (5)	1.3204 (3)	0.40980 (12)	0.0193 (3)
N2	0.67886 (5)	1.0007 (3)	0.51049 (13)	0.0216 (3)
N3	0.74415 (5)	0.7412 (3)	0.41469 (12)	0.0198 (3)
C1	0.49282 (7)	1.3208 (4)	0.55761 (16)	0.0250 (4)
C2	0.44695 (7)	1.2326 (4)	0.60204 (17)	0.0353 (5)
H2	0.4361	1.3025	0.6710	0.042*
C3	0.41781 (7)	1.0427 (5)	0.5439 (2)	0.0403 (6)
H3	0.3873	0.9816	0.5740	0.048*
C4	0.43334 (7)	0.9406 (4)	0.44072 (19)	0.0350 (5)
H4	0.4134	0.8106	0.4017	0.042*
C5	0.47894 (6)	1.0334 (4)	0.39558 (18)	0.0254 (4)
H5	0.4891	0.9674	0.3256	0.031*
C6	0.50924 (6)	1.2233 (3)	0.45406 (14)	0.0204 (4)
C7	0.59151 (6)	1.1465 (3)	0.36648 (14)	0.0171 (3)
C8	0.63851 (6)	1.3054 (4)	0.32946 (15)	0.0233 (4)
H8A	0.6495	1.4287	0.3901	0.028*
H8B	0.6300	1.4267	0.2663	0.028*
C9	0.70557 (6)	0.9262 (3)	0.42103 (14)	0.0179 (3)
C10	0.75680 (6)	0.6156 (3)	0.51203 (15)	0.0201 (4)
C11	0.73123 (6)	0.6745 (4)	0.61057 (15)	0.0238 (4)
H11	0.7400	0.5841	0.6772	0.029*
C12	0.69218 (6)	0.8721 (4)	0.60723 (16)	0.0231 (4)
C13	0.80089 (7)	0.4138 (4)	0.50769 (18)	0.0280 (4)
H13A	0.8046	0.3412	0.4329	0.042*
H13B	0.7952	0.2576	0.5584	0.042*
H13C	0.8311	0.5135	0.5291	0.042*
C14	0.66199 (8)	0.9520 (5)	0.70853 (18)	0.0363 (5)
H14A	0.6604	1.1567	0.7146	0.054*
H14B	0.6778	0.8745	0.7743	0.054*
H14C	0.6285	0.8762	0.7016	0.054*
H1	0.5635 (7)	1.500 (2)	0.4204 (17)	0.027 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02112 (19)	0.0217 (2)	0.0222 (2)	0.00522 (16)	0.00482 (17)	0.0057 (2)
Cl1	0.0551 (3)	0.0323 (3)	0.0280 (3)	0.0023 (2)	0.0035 (2)	−0.0090 (2)
O1	0.0241 (6)	0.0138 (6)	0.0270 (7)	0.0011 (5)	0.0005 (5)	−0.0002 (5)
N1	0.0224 (7)	0.0105 (7)	0.0249 (8)	−0.0024 (5)	0.0059 (6)	−0.0004 (6)
N2	0.0201 (7)	0.0226 (7)	0.0221 (8)	−0.0003 (6)	0.0016 (6)	−0.0034 (7)
N3	0.0188 (7)	0.0180 (7)	0.0224 (8)	−0.0013 (5)	0.0005 (6)	0.0008 (6)
C1	0.0303 (9)	0.0197 (9)	0.0251 (9)	0.0060 (7)	0.0049 (7)	0.0054 (8)
C2	0.0357 (11)	0.0317 (10)	0.0386 (12)	0.0123 (8)	0.0153 (9)	0.0113 (9)
C3	0.0226 (9)	0.0422 (13)	0.0560 (15)	0.0027 (9)	0.0127 (9)	0.0221 (11)
C4	0.0235 (10)	0.0342 (11)	0.0474 (14)	−0.0054 (8)	−0.0060 (9)	0.0125 (10)
C5	0.0239 (9)	0.0234 (9)	0.0290 (10)	0.0000 (7)	−0.0017 (8)	0.0070 (8)
C6	0.0206 (9)	0.0163 (9)	0.0243 (9)	0.0042 (6)	0.0028 (7)	0.0052 (7)
C7	0.0207 (8)	0.0157 (8)	0.0150 (8)	0.0016 (6)	−0.0026 (6)	0.0016 (7)
C8	0.0232 (8)	0.0168 (8)	0.0299 (10)	0.0042 (7)	0.0048 (7)	0.0043 (7)
C9	0.0179 (8)	0.0157 (8)	0.0203 (9)	−0.0031 (6)	0.0001 (7)	−0.0007 (7)
C10	0.0203 (9)	0.0171 (8)	0.0230 (10)	−0.0046 (6)	−0.0050 (7)	0.0011 (7)
C11	0.0256 (9)	0.0255 (9)	0.0203 (10)	−0.0056 (7)	−0.0047 (7)	0.0038 (8)
C12	0.0219 (9)	0.0282 (9)	0.0192 (10)	−0.0072 (7)	0.0004 (6)	−0.0042 (8)
C13	0.0269 (9)	0.0276 (10)	0.0294 (11)	0.0031 (7)	−0.0037 (8)	0.0038 (8)
C14	0.0322 (11)	0.0547 (13)	0.0220 (11)	0.0013 (9)	0.0024 (8)	−0.0055 (9)

Geometric parameters (Å, º)

S1—C9	1.7646 (17)	C4—H4	0.9300
S1—C8	1.7947 (17)	C5—C6	1.385 (3)
Cl1—C1	1.742 (2)	C5—H5	0.9300
O1—C7	1.2252 (19)	C7—C8	1.516 (2)
N1—C7	1.351 (2)	C8—H8A	0.9700
N1—C6	1.416 (2)	C8—H8B	0.9700
N1—H1	0.874 (9)	C10—C11	1.385 (3)
N2—C9	1.327 (2)	C10—C13	1.502 (3)
N2—C12	1.348 (2)	C11—C12	1.387 (2)
N3—C9	1.341 (2)	C11—H11	0.9300
N3—C10	1.344 (2)	C12—C14	1.497 (3)
C1—C6	1.387 (2)	C13—H13A	0.9600
C1—C2	1.388 (3)	C13—H13B	0.9600
C2—C3	1.365 (3)	C13—H13C	0.9600
C2—H2	0.9300	C14—H14A	0.9600
C3—C4	1.383 (3)	C14—H14B	0.9600
C3—H3	0.9300	C14—H14C	0.9600
C4—C5	1.392 (3)		
C9—S1—C8	100.53 (8)	S1—C8—H8A	108.7
C7—N1—C6	124.06 (14)	C7—C8—H8B	108.7
C7—N1—H1	117.9 (13)	S1—C8—H8B	108.7

C6—N1—H1	117.4 (13)	H8A—C8—H8B	107.6
C9—N2—C12	115.58 (14)	N2—C9—N3	128.40 (15)
C9—N3—C10	115.01 (14)	N2—C9—S1	118.89 (12)
C6—C1—C2	121.14 (18)	N3—C9—S1	112.71 (12)
C6—C1—Cl1	119.72 (14)	N3—C10—C11	121.67 (15)
C2—C1—Cl1	119.13 (16)	N3—C10—C13	116.00 (15)
C3—C2—C1	119.6 (2)	C11—C10—C13	122.32 (16)
C3—C2—H2	120.2	C10—C11—C12	118.23 (16)
C1—C2—H2	120.2	C10—C11—H11	120.9
C2—C3—C4	120.55 (19)	C12—C11—H11	120.9
C2—C3—H3	119.7	N2—C12—C11	121.10 (16)
C4—C3—H3	119.7	N2—C12—C14	116.10 (16)
C3—C4—C5	119.7 (2)	C11—C12—C14	122.79 (17)
C3—C4—H4	120.2	C10—C13—H13A	109.5
C5—C4—H4	120.2	C10—C13—H13B	109.5
C6—C5—C4	120.50 (19)	H13A—C13—H13B	109.5
C6—C5—H5	119.8	C10—C13—H13C	109.5
C4—C5—H5	119.8	H13A—C13—H13C	109.5
C5—C6—C1	118.53 (16)	H13B—C13—H13C	109.5
C5—C6—N1	121.47 (16)	C12—C14—H14A	109.5
C1—C6—N1	120.00 (16)	C12—C14—H14B	109.5
O1—C7—N1	123.63 (14)	H14A—C14—H14B	109.5
O1—C7—C8	123.26 (14)	C12—C14—H14C	109.5
N1—C7—C8	113.10 (14)	H14A—C14—H14C	109.5
C7—C8—S1	114.09 (12)	H14B—C14—H14C	109.5
C7—C8—H8A	108.7		

C6—C1—C2—C3	1.2 (3)	N1—C7—C8—S1	171.73 (12)
Cl1—C1—C2—C3	−178.03 (15)	C9—S1—C8—C7	−68.05 (14)
C1—C2—C3—C4	−0.8 (3)	C12—N2—C9—N3	−0.4 (3)
C2—C3—C4—C5	−0.3 (3)	C12—N2—C9—S1	178.49 (12)
C3—C4—C5—C6	1.1 (3)	C10—N3—C9—N2	0.4 (2)
C4—C5—C6—C1	−0.7 (3)	C10—N3—C9—S1	−178.57 (11)
C4—C5—C6—N1	179.97 (16)	C8—S1—C9—N2	0.91 (15)
C2—C1—C6—C5	−0.4 (3)	C8—S1—C9—N3	179.98 (11)
Cl1—C1—C6—C5	178.80 (13)	C9—N3—C10—C11	0.4 (2)
C2—C1—C6—N1	178.91 (16)	C9—N3—C10—C13	−178.32 (14)
Cl1—C1—C6—N1	−1.9 (2)	N3—C10—C11—C12	−1.0 (2)
C7—N1—C6—C5	−48.9 (2)	C13—C10—C11—C12	177.59 (16)
C7—N1—C6—C1	131.82 (18)	C9—N2—C12—C11	−0.3 (2)
C6—N1—C7—O1	3.2 (3)	C9—N2—C12—C14	−179.39 (16)
C6—N1—C7—C8	−177.61 (15)	C10—C11—C12—N2	1.0 (2)
O1—C7—C8—S1	−9.1 (2)	C10—C11—C12—C14	179.99 (17)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1[i]	0.87 (1)	2.05 (1)	2.8414 (18)	150 (2)

C2—H2···O1ⁱⁱ	0.93	2.46	3.213 (2)	138
C8—H8*A*···*Cg*1ⁱ	0.97	2.92	3.832 (2)	157
C13—H13*B*···*Cg*1ⁱⁱⁱ	0.96	2.99	3.592 (2)	122

Symmetry codes: (i) x, $y+1$, z; (ii) $-x+1$, $-y+2$, $z+1/2$; (iii) x, $y-1$, z.