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N-(4-Chlorobutanoyl)-*N*′-(2,5-dimethoxyphenyl)thiourea

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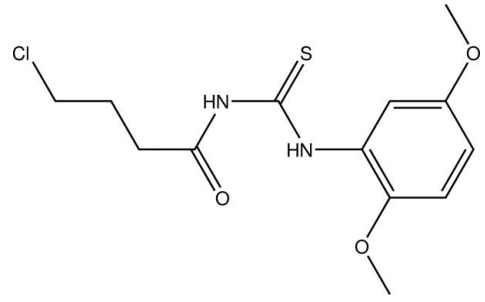
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.105; data-to-parameter ratio = 18.5.

The title molecule, $C_{13}H_{17}ClN_2O_3S$, shows an *anti* and *syn* disposition of the butanoyl and 2,5-dimethoxyphenyl groups with respect to the thione and is stabilized by intramolecular N—H···O and weak C—H···S hydrogen bonds. In the crystal, intermolecular N—H···S hydrogen bonds link the molecules into centrosymmetric dimers. The crystal structure is stabilized by weak C—H···O and C—H···S contacts.

Related literature

For the structures of related thioureas, see: Yamin *et al.* (2011); Yusof *et al.* (2011).



Experimental

Crystal data

$C_{13}H_{17}ClN_2O_3S$
M_r = 316.80
Triclinic, $P\overline{1}$
a = 7.6882 (18) Å
b = 9.151 (2) Å
c = 10.939 (3) Å
α = 98.536 (5)°
β = 97.787 (5)°
γ = 101.489 (5)°
V = 734.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 298 K
0.29 × 0.25 × 0.19 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.890, T_{max} = 0.926
9303 measured reflections
3351 independent reflections
2928 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.06
3351 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86	1.93	2.663 (2)	141
C7—H7A···S1	0.93	2.51	3.1853 (18)	129
N1—H1A···S1^{i}	0.86	2.58	3.4058 (16)	161
C3—H3A···S1^{i}	0.97	2.83	3.5633 (19)	133
C12—H12A···O2^{ii}	0.96	2.50	3.259 (3)	136

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x, -y, -z+3$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2378).

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Yusof, M. S. M., Embong, N. F., Othman, E. A. & Yamin, B. M. (2011). *Acta Cryst.* E**67**, o1849.

supporting information

Acta Cryst. (2011). E**67**, o2609 [https://doi.org/10.1107/S1600536811036002]

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S1. Comment

The title compound (Fig. 1) is analogous to the previously reported *N*-(4-chlorobutanoyl)-*N*′-(2-fluorophenyl)thiourea (Yusof *et al.*, 2011) except that the methoxy groups are attached at the 2 and 5 positions of the phenyl ring. The carbonyl-thiourea fragment C4/O1/N1/C5/S1/N2 and the benzene ring, C6···C11, are each planar with the maximum deviation from the least-squares planes of 0.024 (2) Å for atom C4. The benzene ring and carbonylthiourea moiety form a dihedral angle of 5.67 (6)°, much smaller than angles observed in the previously reported thioureas *N*-(4-chlorobutanoyl)-*N*′-(2-fluorophenyl)thiourea [74.78 (19)° and 82.3 (2)° for two independent molecules] and *N*-(4-chlorobutanoyl)-*N*′-phenyl-thiourea [72.98 (12)° and 81.47 (14)° for two independent molecules] (Yusof *et al.*, 2011; Yamin *et al.*, 2011). The bond lengths and angles in the title thiourea are in normal ranges and comparable to those in the analogous compounds. The molecule maintains the *trans-cis* configuration with respect to the position of the butanoyl and 2,5-dimethoxyphenyl groups against the thiono C=S group bond across their C—N bonds.

The molecule is stabilized by three intramolecular contacts, N—H···O and C—H···S. In the crystal packing, the molecules are linked by N—H···S, C—H···S and C—H···O intermolecular hydrogen bonds (symmetry codes as in Table 1) and form dimers (Fig. 2).

S2. Experimental

A solution of 4-chlorobutanoylisothiocyanate (1.25 g, 6.33 mmol) in 30 ml of acetone was added into a flask containing 30 ml acetone solution of 2,5-dimethoxyaniline (0.82 g, 6.33 mmol). The mixture was refluxed for 1 h. Then, the solution was filtered-off and left to evaporate at room temperature. The colourless solid was obtained after one day of evaporation (yield 74%).

S3. Refinement

H atoms bonded to C atoms were positioned geometrically with C—H = 0.93–0.97 Å and constrained to ride on their parent atoms with U_{iso}(H)= xU_{eq}(parent atom) where x=1.5 for CH_3 group and 1.2 for CH and CH_2 groups. Amine H atoms were also placed in idealized positions and refined with N—H bond lengths restrained to 0.86 Å and U_{iso}(H)= 1.2U_{eq}(parent N atom).

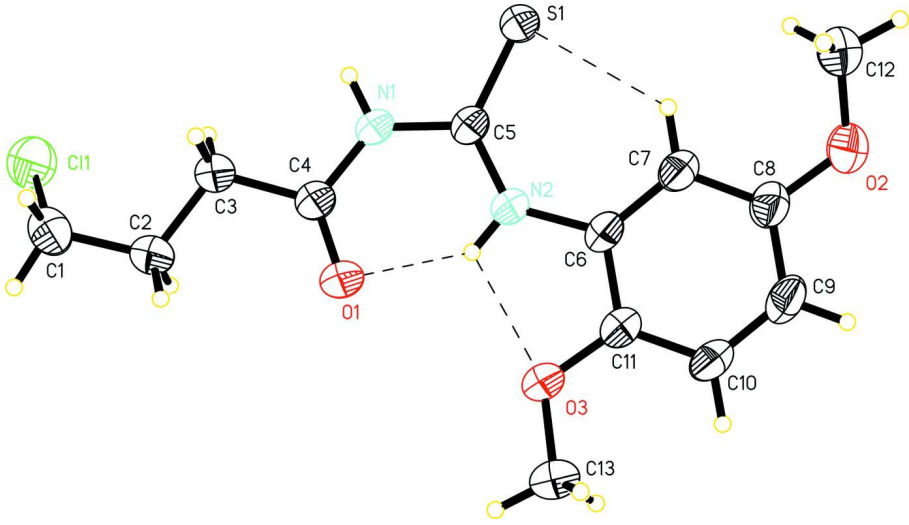

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

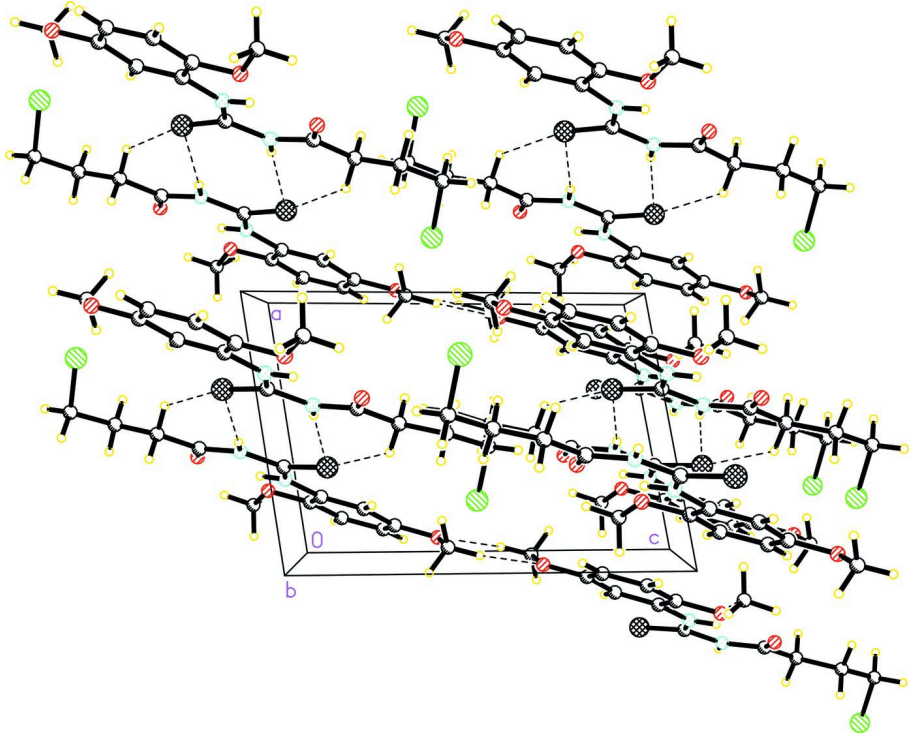

Figure 2

A packing diagram of the title compound viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{13}H_{17}ClN_2O_3S$
$M_r = 316.80$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.6882\ (18)$ Å
$b = 9.151\ (2)$ Å

c = 10.939 (3) Å
α = 98.536 (5)°
β = 97.787 (5)°
γ = 101.489 (5)°
V = 734.9 (3) Å^3
Z = 2
F(000) = 332
D_x = 1.432 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 934 reflections
θ = 1.9–27.5°
μ = 0.41 mm^{-1}
T = 298 K
Slab, colourless
0.29 × 0.25 × 0.19 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 83.66 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.890, T_{max} = 0.926

9303 measured reflections
3351 independent reflections
2928 reflections with $I > 2/s(I)$
R_{int} = 0.018
θ_{max} = 27.5°, θ_{min} = 1.9°
h = −9→9
k = −11→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.06
3351 reflections
181 parameters
0 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.1878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.76739 (8)	0.42437 (6)	0.51080 (5)	0.06766 (17)
S1	0.35046 (6)	0.36496 (4)	1.10956 (4)	0.04799 (14)
O1	0.38199 (19)	0.05467 (12)	0.74925 (12)	0.0523 (3)
O2	0.0551 (2)	−0.00809 (15)	1.36092 (13)	0.0633 (4)
O3	0.23484 (18)	−0.21076 (12)	0.90512 (12)	0.0521 (3)
N1	0.40933 (18)	0.27749 (13)	0.88324 (12)	0.0393 (3)
H1A	0.4464	0.3739	0.8901	0.047*
N2	0.29315 (17)	0.07994 (13)	0.97717 (12)	0.0379 (3)
H2A	0.3046	0.0287	0.9073	0.045*
C1	0.5613 (3)	0.2829 (2)	0.45811 (17)	0.0549 (4)
H1B	0.4647	0.3319	0.4332	0.066*
H1C	0.5743	0.2142	0.3851	0.066*
C2	0.5121 (3)	0.19342 (19)	0.55845 (15)	0.0466 (4)
H2B	0.6092	0.1445	0.5826	0.056*
H2C	0.4052	0.1144	0.5235	0.056*
C3	0.4770 (3)	0.28733 (18)	0.67463 (15)	0.0457 (4)
H3A	0.5857	0.3630	0.7127	0.055*

H3B	0.3837	0.3401	0.6506	0.055*
C4	0.4193 (2)	0.19249 (17)	0.76973 (15)	0.0388 (3)
C5	0.3481 (2)	0.22955 (16)	0.98775 (14)	0.0353 (3)
C6	0.2192 (2)	−0.00946 (16)	1.06058 (14)	0.0356 (3)
C7	0.1739 (2)	0.04605 (17)	1.17392 (15)	0.0414 (3)
H7A	0.1919	0.1500	1.2009	0.050*
C8	0.1013 (2)	−0.05441 (19)	1.24712 (15)	0.0435 (4)
C9	0.0757 (2)	−0.20879 (19)	1.20822 (17)	0.0475 (4)
H9A	0.0292	−0.2752	1.2584	0.057*
C10	0.1191 (2)	−0.26447 (18)	1.09489 (17)	0.0463 (4)
H10A	0.1010	−0.3686	1.0688	0.056*
C11	0.1893 (2)	−0.16674 (17)	1.01961 (15)	0.0394 (3)
C12	0.0271 (3)	0.1408 (2)	1.38725 (18)	0.0584 (5)
H12A	−0.0043	0.1587	1.4693	0.088*
H12B	−0.0689	0.1522	1.3260	0.088*
H12C	0.1354	0.2125	1.3843	0.088*
C13	0.1689 (3)	−0.36576 (19)	0.84650 (18)	0.0540 (4)
H13A	0.2096	−0.3825	0.7675	0.081*
H13B	0.0396	−0.3895	0.8329	0.081*
H13C	0.2130	−0.4296	0.8997	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0734 (3)	0.0600 (3)	0.0700 (3)	0.0030 (2)	0.0293 (3)	0.0139 (2)
S1	0.0672 (3)	0.0281 (2)	0.0468 (2)	−0.00080 (17)	0.0255 (2)	0.00220 (16)
O1	0.0760 (8)	0.0283 (6)	0.0488 (7)	−0.0001 (5)	0.0206 (6)	0.0022 (5)
O2	0.1019 (11)	0.0522 (7)	0.0481 (7)	0.0223 (7)	0.0333 (7)	0.0227 (6)
O3	0.0739 (8)	0.0282 (5)	0.0537 (7)	0.0013 (5)	0.0262 (6)	0.0057 (5)
N1	0.0517 (8)	0.0240 (6)	0.0410 (7)	0.0006 (5)	0.0160 (6)	0.0058 (5)
N2	0.0488 (7)	0.0259 (6)	0.0370 (6)	0.0008 (5)	0.0123 (5)	0.0050 (5)
C1	0.0685 (12)	0.0546 (11)	0.0401 (9)	0.0094 (9)	0.0141 (8)	0.0050 (8)
C2	0.0597 (10)	0.0363 (8)	0.0413 (9)	0.0074 (7)	0.0120 (7)	0.0007 (6)
C3	0.0657 (11)	0.0325 (8)	0.0403 (8)	0.0090 (7)	0.0179 (7)	0.0059 (6)
C4	0.0438 (8)	0.0312 (7)	0.0396 (8)	0.0029 (6)	0.0103 (6)	0.0055 (6)
C5	0.0372 (7)	0.0290 (7)	0.0388 (7)	0.0027 (6)	0.0092 (6)	0.0076 (5)
C6	0.0370 (7)	0.0299 (7)	0.0395 (8)	0.0027 (6)	0.0060 (6)	0.0117 (6)
C7	0.0515 (9)	0.0320 (7)	0.0409 (8)	0.0056 (6)	0.0094 (7)	0.0113 (6)
C8	0.0517 (9)	0.0422 (8)	0.0388 (8)	0.0086 (7)	0.0097 (7)	0.0152 (7)
C9	0.0557 (10)	0.0405 (8)	0.0491 (9)	0.0047 (7)	0.0120 (8)	0.0226 (7)
C10	0.0565 (10)	0.0293 (7)	0.0527 (9)	0.0031 (7)	0.0114 (8)	0.0134 (7)
C11	0.0426 (8)	0.0313 (7)	0.0437 (8)	0.0035 (6)	0.0089 (6)	0.0095 (6)
C12	0.0805 (13)	0.0503 (10)	0.0482 (10)	0.0129 (9)	0.0235 (9)	0.0116 (8)
C13	0.0685 (12)	0.0324 (8)	0.0579 (11)	0.0048 (8)	0.0158 (9)	0.0026 (7)

Geometric parameters (Å, º)

Cl1—C1	1.798 (2)	C3—C4	1.507 (2)
S1—C5	1.6750 (16)	C3—H3A	0.9700
O1—C4	1.2156 (19)	C3—H3B	0.9700
O2—C8	1.370 (2)	C6—C7	1.385 (2)
O2—C12	1.415 (2)	C6—C11	1.405 (2)
O3—C11	1.370 (2)	C7—C8	1.390 (2)
O3—C13	1.4267 (19)	C7—H7A	0.9300
N1—C4	1.3839 (19)	C8—C9	1.380 (2)
N1—C5	1.3905 (19)	C9—C10	1.378 (3)
N1—H1A	0.8600	C9—H9A	0.9300
N2—C5	1.3324 (18)	C10—C11	1.384 (2)
N2—C6	1.4149 (18)	C10—H10A	0.9300
N2—H2A	0.8600	C12—H12A	0.9600
C1—C2	1.508 (2)	C12—H12B	0.9600
C1—H1B	0.9700	C12—H12C	0.9600
C1—H1C	0.9700	C13—H13A	0.9600
C2—C3	1.513 (2)	C13—H13B	0.9600
C2—H2B	0.9700	C13—H13C	0.9600
C2—H2C	0.9700		
C8—O2—C12	117.89 (13)	N1—C5—S1	116.73 (10)
C11—O3—C13	117.32 (13)	C7—C6—C11	119.81 (13)
C4—N1—C5	129.36 (12)	C7—C6—N2	125.40 (13)
C4—N1—H1A	115.3	C11—C6—N2	114.78 (13)
C5—N1—H1A	115.3	C6—C7—C8	119.69 (14)
C5—N2—C6	131.35 (13)	C6—C7—H7A	120.2
C5—N2—H2A	114.3	C8—C7—H7A	120.2
C6—N2—H2A	114.3	O2—C8—C9	116.42 (14)
C2—C1—Cl1	112.05 (13)	O2—C8—C7	123.06 (15)
C2—C1—H1B	109.2	C9—C8—C7	120.50 (15)
Cl1—C1—H1B	109.2	C10—C9—C8	119.96 (15)
C2—C1—H1C	109.2	C10—C9—H9A	120.0
Cl1—C1—H1C	109.2	C8—C9—H9A	120.0
H1B—C1—H1C	107.9	C9—C10—C11	120.59 (15)
C1—C2—C3	114.17 (14)	C9—C10—H10A	119.7
C1—C2—H2B	108.7	C11—C10—H10A	119.7
C3—C2—H2B	108.7	O3—C11—C10	125.02 (14)
C1—C2—H2C	108.7	O3—C11—C6	115.55 (13)
C3—C2—H2C	108.7	C10—C11—C6	119.43 (15)
H2B—C2—H2C	107.6	O2—C12—H12A	109.5
C4—C3—C2	112.46 (13)	O2—C12—H12B	109.5
C4—C3—H3A	109.1	H12A—C12—H12B	109.5
C2—C3—H3A	109.1	O2—C12—H12C	109.5
C4—C3—H3B	109.1	H12A—C12—H12C	109.5
C2—C3—H3B	109.1	H12B—C12—H12C	109.5
H3A—C3—H3B	107.8	O3—C13—H13A	109.5

O1—C4—N1	122.66 (14)	O3—C13—H13B	109.5
O1—C4—C3	123.84 (14)	H13A—C13—H13B	109.5
N1—C4—C3	113.49 (13)	O3—C13—H13C	109.5
N2—C5—N1	115.06 (13)	H13A—C13—H13C	109.5
N2—C5—S1	128.21 (12)	H13B—C13—H13C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	1.93	2.663 (2)	141
N2—H2*A*···O3	0.86	2.15	2.5895 (18)	112
C7—H7*A*···S1	0.93	2.51	3.1853 (18)	129
N1—H1*A*···S1^{i}	0.86	2.58	3.4058 (16)	161
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