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S-2-Aminophenyl phenylcarbamothioate

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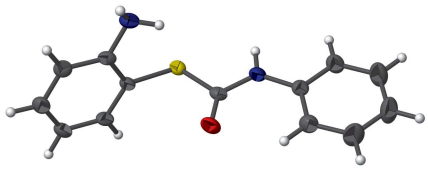
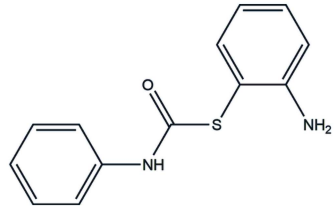
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{12}N_2OS$, which was obtained from the condensation reaction of 2-aminobenzenethiol with isocyanatobenzene, the benzene rings are inclined to one another by 83.5 (1)° and a short intramolecular C—H···O contact is observed. In the crystal, molecules are linked by N—H···O and N—H···N hydrogen bonds, generating (001) sheets.

3D view



Chemical scheme



Structure description

Organic carbamothioates are a class of compounds that play an important role in the synthesis of pharmaceuticals and agricultural chemicals (Torrico-Vallejos *et al.*, 2011; Belkhir *et al.*, 2015). As part of our studies in this area, the title compound (Fig. 1) was obtained from the condensation reaction of 2-aminobenzenethiol with isocyanatobenzene.

The dihedral angle between the aromatic rings is 83.5 (1)° and the major twist occurs about the C6—S1 bond [C1—C6—S1—C7 = 87.6 (2)°]. The C—S bond distances are comparable with those in related structures [average C—S = 1.778 (6) Å in *S*-phenyl 4-methoxybenzothioate (El-Azab *et al.*, 2012) and 1.7733 (2) Å in ethane-1,2-diyl bis(benzenedithioate) (Abe *et al.*, 2011)]. The least-squares plane through the *S*-methyl methylcarbamothioate unit (S1/C7/O1/N2) makes dihedral angles of 88.3 (1) and 6.9 (1)° with the C1–C6 and C8–C13 benzene rings, respectively.

In the crystal, N—H···O and N—H···N hydrogen bonds connect the molecules into (001) sheets (Fig. 2, Table 1).

Synthesis and crystallization

2-Aminobenzenethiol (7.9 mmol, 0.85 ml) was added to an isocyanatobenzene solution (8.7 mmol, 0.9 ml) in 5 ml dimethylformamide (Fig. 3). The mixture was stirred at 0°C until the reaction was complete, and then warmed to room temperature. A precipitate

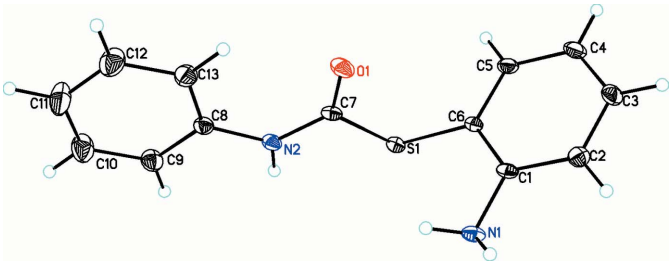


Figure 1
The molecular structure with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as circles of arbitrary size.

was formed after adding 5 ml water. The precipitate was filtered off and washed with toluene. The resulting residue was purified by recrystallization from Et_2O solution to afford *S*-(2-aminophenyl) phenylcarbamothioate (1.2 g, 69% yield) as a white solid. Slow evaporation of the solvent resulted in colourless plates. ^{1}H NMR (400 MHz, DMSO) δ 10.40 (*s*, 1H, NH), 7.49 (*d*, *J* = 7.9 Hz, 2H), 7.29 (*t*, *J* = 7.8 Hz, 2H), 7.21 (*d*, *J* = 7.6 Hz, 1H), 7.14 (*t*, *J* = 7.6 Hz, 1H), 7.08–6.95 (*m*, 1H), 6.77 (*d*, *J* = 8.1 Hz, 1H), 6.55 (*t*, *J* = 7.4 Hz, 1H), 5.36 (*s*, 2H, NH_2).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The title compound was synthesized by Şengül Dilem Doğan, Department of Pharmaceutical Basic Sciences, Faculty of Pharmacy, Erciyes University, Kayseri, 38039, Turkey.

Funding information

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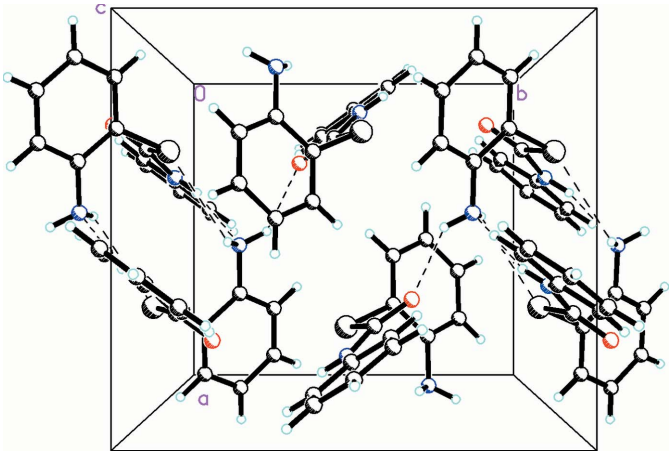

Figure 2
A packing diagram viewed along [001]. Hydrogen-bonding interactions are shown with dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···O1	0.95	2.32	2.928 (4)	121
N1—H1*N*2···O1^{i}	0.87 (4)	2.15 (4)	2.896 (3)	143 (3)
N2—H2*N*···N1^{ii}	0.86 (4)	2.14 (4)	2.993 (3)	173 (4)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{12}N_2OS$
M_r	244.31
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	100
a, *b*, *c* (Å)	9.448 (5), 10.395 (5), 24.337 (5)
V (Å^3)	2390.2 (18)
Z	8
Radiation type	Mo *K*α
μ (mm^{-1})	0.26
Crystal size (mm)	0.35 × 0.19 × 0.08
Data collection	
Diffractometer	Rigaku OD SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.712, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37562, 5122, 2960
R_{int}	0.117
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.812
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.094, 0.217, 1.08
No. of reflections	5122
No. of parameters	166
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.89, −0.49

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2017* (Sheldrick, 2015*b*) and *SHELXTL* (Sheldrick, 2008).

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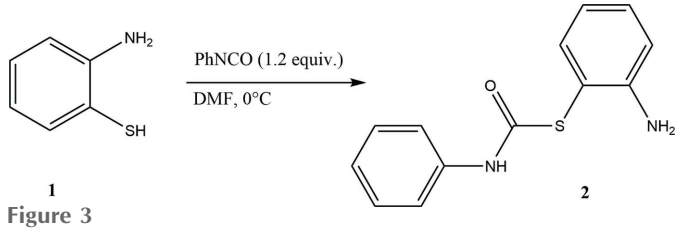

1 2

Figure 3
Reaction scheme.

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full crystallographic data

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Crystal data

$C_{13}H_{12}N_2OS$
M_r = 244.31
Orthorhombic, *Pbca*
a = 9.448 (5) Å
b = 10.395 (5) Å
c = 24.337 (5) Å
V = 2390.2 (18) Å^3
Z = 8
F(000) = 1024
D_x = 1.358 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4641 reflections
θ = 3.8–33.3°
μ = 0.26 mm^{-1}
T = 100 K
Plate, colourless
0.35 × 0.19 × 0.08 mm

Data collection

Rigaku OD SuperNova, Dual, Cu at zero, Atlas diffractometer
Radiation source: micro-focus sealed X-ray tube
Detector resolution: 10.6501 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.712, T_{max} = 1.000
37562 measured reflections
5122 independent reflections
2960 reflections with $I > 2\sigma(I)$
R_{int} = 0.117
θ_{max} = 35.3°, θ_{min} = 3.4°
h = −14→14
k = −16→16
l = −38→38

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.094
$wR(F^2)$ = 0.217
S = 1.08
5122 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 4.6756P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.89 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. H atoms on N atoms were located in difference maps and refined isotropically. The remaining H atoms were positioned geometrically and treated as riding, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.27820 (6)	0.48071 (7)	0.50003 (3)	0.02546 (16)
O1	0.1937 (2)	0.6451 (2)	0.57835 (9)	0.0342 (5)
N1	0.4537 (2)	0.6976 (3)	0.45289 (11)	0.0285 (5)
H1N1	0.463 (3)	0.675 (3)	0.4882 (13)	0.023 (8)*
H1N2	0.499 (4)	0.770 (4)	0.4493 (15)	0.038 (10)*
N2	0.3466 (2)	0.4836 (2)	0.60304 (9)	0.0268 (5)
H2N	0.398 (4)	0.427 (4)	0.5872 (14)	0.043 (10)*
C1	0.3125 (3)	0.6969 (3)	0.43601 (11)	0.0255 (5)
C2	0.2647 (3)	0.7841 (3)	0.39658 (12)	0.0301 (6)
H2A	0.328614	0.845079	0.381456	0.036*
C3	0.1248 (3)	0.7826 (3)	0.37920 (12)	0.0349 (6)
H3A	0.093855	0.843092	0.352511	0.042*
C4	0.0297 (3)	0.6940 (3)	0.40033 (12)	0.0357 (7)
H4A	−0.066487	0.694576	0.388971	0.043*
C5	0.0770 (3)	0.6045 (3)	0.43822 (11)	0.0301 (6)
H5A	0.012911	0.542076	0.452112	0.036*
C6	0.2175 (2)	0.6044 (3)	0.45646 (10)	0.0232 (5)
C7	0.2669 (3)	0.5517 (3)	0.56763 (11)	0.0255 (5)
C8	0.3536 (3)	0.4943 (3)	0.66088 (11)	0.0271 (5)
C9	0.4367 (3)	0.4039 (3)	0.68797 (13)	0.0364 (7)
H9A	0.484475	0.339339	0.667498	0.044*
C10	0.4502 (4)	0.4074 (4)	0.74440 (14)	0.0484 (9)
H10A	0.507490	0.345560	0.762578	0.058*
C11	0.3807 (5)	0.5005 (4)	0.77457 (14)	0.0528 (10)
H11A	0.388073	0.501948	0.813493	0.063*
C12	0.3004 (4)	0.5915 (4)	0.74748 (14)	0.0489 (9)
H12A	0.254610	0.657016	0.768107	0.059*
C13	0.2851 (3)	0.5895 (3)	0.69083 (12)	0.0363 (7)
H13A	0.228691	0.652233	0.672773	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0209 (3)	0.0266 (3)	0.0289 (3)	−0.0001 (2)	−0.0013 (2)	−0.0007 (2)
O1	0.0286 (10)	0.0331 (11)	0.0409 (11)	0.0105 (8)	−0.0030 (8)	−0.0033 (9)
N1	0.0193 (9)	0.0277 (12)	0.0386 (13)	−0.0035 (9)	0.0017 (9)	−0.0009 (10)
N2	0.0212 (10)	0.0296 (12)	0.0296 (11)	0.0047 (9)	0.0000 (8)	−0.0016 (9)
C1	0.0207 (10)	0.0246 (13)	0.0312 (12)	0.0027 (9)	0.0021 (9)	−0.0047 (10)
C2	0.0315 (13)	0.0241 (13)	0.0346 (14)	0.0035 (10)	0.0041 (11)	0.0014 (10)
C3	0.0341 (14)	0.0368 (17)	0.0339 (14)	0.0136 (12)	0.0009 (11)	0.0011 (12)
C4	0.0230 (12)	0.0482 (19)	0.0358 (15)	0.0101 (12)	−0.0024 (10)	−0.0005 (13)
C5	0.0182 (10)	0.0398 (16)	0.0324 (13)	0.0001 (10)	0.0003 (9)	−0.0009 (11)
C6	0.0184 (10)	0.0244 (12)	0.0269 (11)	0.0034 (9)	0.0000 (8)	0.0002 (9)
C7	0.0171 (10)	0.0269 (13)	0.0327 (13)	0.0003 (9)	−0.0003 (9)	−0.0012 (10)
C8	0.0232 (11)	0.0285 (14)	0.0298 (12)	−0.0015 (10)	−0.0003 (9)	0.0002 (10)

C9	0.0386 (15)	0.0344 (16)	0.0363 (15)	0.0087 (13)	−0.0023 (12)	0.0001 (12)
C10	0.063 (2)	0.045 (2)	0.0377 (17)	0.0161 (18)	−0.0070 (15)	0.0025 (14)
C11	0.078 (3)	0.053 (2)	0.0274 (15)	0.015 (2)	−0.0036 (16)	−0.0010 (14)
C12	0.063 (2)	0.047 (2)	0.0371 (16)	0.0152 (18)	0.0056 (15)	−0.0054 (15)
C13	0.0364 (15)	0.0365 (18)	0.0361 (15)	0.0083 (13)	0.0017 (12)	0.0006 (12)

Geometric parameters (Å, º)

S1—C6	1.763 (3)	C4—C5	1.383 (4)
S1—C7	1.806 (3)	C4—H4A	0.9500
O1—C7	1.221 (3)	C5—C6	1.400 (4)
N1—C1	1.397 (3)	C5—H5A	0.9500
N1—H1N1	0.90 (3)	C8—C13	1.389 (4)
N1—H1N2	0.87 (4)	C8—C9	1.391 (4)
N2—C7	1.346 (3)	C9—C10	1.380 (4)
N2—C8	1.414 (3)	C9—H9A	0.9500
N2—H2N	0.86 (4)	C10—C11	1.381 (5)
C1—C2	1.395 (4)	C10—H10A	0.9500
C1—C6	1.406 (4)	C11—C12	1.380 (5)
C2—C3	1.388 (4)	C11—H11A	0.9500
C2—H2A	0.9500	C12—C13	1.387 (5)
C3—C4	1.386 (5)	C12—H12A	0.9500
C3—H3A	0.9500	C13—H13A	0.9500
C6—S1—C7	103.32 (13)	C5—C6—S1	120.0 (2)
C1—N1—H1N1	112 (2)	C1—C6—S1	120.30 (19)
C1—N1—H1N2	116 (2)	O1—C7—N2	126.8 (3)
H1N1—N1—H1N2	106 (3)	O1—C7—S1	123.6 (2)
C7—N2—C8	128.5 (2)	N2—C7—S1	109.58 (19)
C7—N2—H2N	113 (2)	C13—C8—C9	119.7 (3)
C8—N2—H2N	118 (2)	C13—C8—N2	123.8 (3)
C2—C1—N1	120.6 (3)	C9—C8—N2	116.4 (3)
C2—C1—C6	118.8 (2)	C10—C9—C8	120.4 (3)
N1—C1—C6	120.6 (2)	C10—C9—H9A	119.8
C3—C2—C1	120.7 (3)	C8—C9—H9A	119.8
C3—C2—H2A	119.6	C9—C10—C11	120.3 (3)
C1—C2—H2A	119.6	C9—C10—H10A	119.9
C4—C3—C2	120.8 (3)	C11—C10—H10A	119.9
C4—C3—H3A	119.6	C12—C11—C10	119.2 (3)
C2—C3—H3A	119.6	C12—C11—H11A	120.4
C5—C4—C3	119.0 (3)	C10—C11—H11A	120.4
C5—C4—H4A	120.5	C11—C12—C13	121.5 (3)
C3—C4—H4A	120.5	C11—C12—H12A	119.3
C4—C5—C6	121.2 (3)	C13—C12—H12A	119.3
C4—C5—H5A	119.4	C12—C13—C8	119.0 (3)
C6—C5—H5A	119.4	C12—C13—H13A	120.5
C5—C6—C1	119.5 (2)	C8—C13—H13A	120.5

N1—C1—C2—C3	179.2 (3)	C8—N2—C7—S1	−170.7 (2)
C6—C1—C2—C3	2.2 (4)	C6—S1—C7—O1	19.6 (3)
C1—C2—C3—C4	−0.5 (4)	C6—S1—C7—N2	−162.42 (18)
C2—C3—C4—C5	−1.5 (5)	C7—N2—C8—C13	−6.3 (5)
C3—C4—C5—C6	1.7 (4)	C7—N2—C8—C9	174.8 (3)
C4—C5—C6—C1	0.1 (4)	C13—C8—C9—C10	0.7 (5)
C4—C5—C6—S1	−174.4 (2)	N2—C8—C9—C10	179.7 (3)
C2—C1—C6—C5	−2.0 (4)	C8—C9—C10—C11	0.3 (6)
N1—C1—C6—C5	−179.0 (2)	C9—C10—C11—C12	−1.5 (6)
C2—C1—C6—S1	172.4 (2)	C10—C11—C12—C13	1.7 (7)
N1—C1—C6—S1	−4.5 (4)	C11—C12—C13—C8	−0.7 (6)
C7—S1—C6—C5	−98.0 (2)	C9—C8—C13—C12	−0.5 (5)
C7—S1—C6—C1	87.6 (2)	N2—C8—C13—C12	−179.4 (3)
C8—N2—C7—O1	7.2 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···O1	0.95	2.32	2.928 (4)	121
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Symmetry codes: (i) *x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1.