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2-Amino-*N*-(2-methoxyphenyl)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxamide

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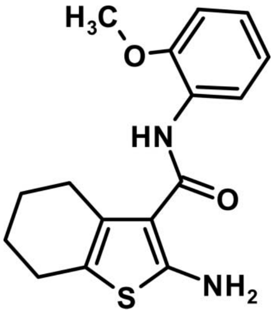
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.052; wR factor = 0.147; data-to-parameter ratio = 14.8.

In the title compound, $C_{16}H_{18}N_2O_2S$, the *o*-methoxyphenyl group is not coplanar with the thiophene ring, making a dihedral angle of 12.9 (1)°. The crystal structure is stabilized by intramolecular N—H···O and C—H···O hydrogen bonds and by intermolecular N—H···O interactions.

Related literature

For related literature, see: Gewald *et al.* (1966); Cremer & Pople (1975); Cohen *et al.* (1977); Csaszar & Morvay (1983); Lakshmi *et al.* (1985); Mohan & Saravanan (2003).



Experimental

Crystal data

$C_{16}H_{18}N_2O_2S$
M_r = 302.38
Monoclinic, $P2_1/n$
a = 8.709 (2) Å
b = 8.576 (2) Å
c = 20.306 (5) Å
β = 90.742 (4)°
V = 1516.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 293 (2) K
0.48 × 0.25 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.937, $T_{\max}$ = 0.998
10964 measured reflections
2831 independent reflections
2266 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.147
S = 1.09
2831 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.86	2.27	2.823 (3)	122
N1—H1B···O1^{i}	0.86	2.28	3.015 (3)	143
N2—H2···O2	0.86	2.19	2.577 (3)	107
C16—H16···O1	0.93	2.39	2.927 (3)	117

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *PARST* (Nardelli, 1995); *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2031).

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supplementary materials

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2-Amino-*N*-(2-methoxyphenyl)-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxamide

K. Chandra Kumar, M. K. Kokila, Puttaraja, S. Mohan and J. Saravanan

Comment

The title compound (I) is one of a series of 3-aryl carboximides. These compounds display different biological activities, such as antitubercular, antibacterial and antifungal activities (Mohan & Saravanan 2003). Schiff bases containing sulfur are the most effective (Csaszar & Morvay 1983; Lakshmi *et al.*, 1985; Cohen *et al.*,1977).

The molecular structure and the packing diagram of (I) are shown in Figures (1) and (2) respectively. The thiophene ring is essentially planar, with a short C8—C9 bond distance [1.352 (3) Å,], indicating the absence of delocalization in the double bonds. This is also reflected in the S1—C2 and S1—C8 distances, which are shorter than the normal C—S distances. The cyclohexene ring adopts a half-chair conformation, with the puckering parameters q_2 = 0.325 (2) Å, Φ = −99.4 (3)° and θ = 130.7 (2)°. (Cremer & Pople, 1975; Spek, 2003). The thiophene ring makes a dihedral angle of 12.9 (1) ° with the *o*-methoxyphenyl ring.

The molecular structure of (I) is stabilized by intramolecular C—H···O and N—H···O hydrogen bonds and by intermolecular N—H···O interactions. (Table 2) The intramolecular C16—H16···O1 and N1—H1···O1 hydrogen bonds form pseudo-six membered rings and N2—H2···O2 forms a pseudo five membered ring, thus locking the molecular conformation and eliminating flexibility. Molecules are linked *via* N—H···O interactions, forming zigzag chains along the *c* axis, (Fig. 2).

Experimental

The title compound (I), was synthesized by mixing cyclohexanone (0.98 g, 0.01 mol) and *o*-methoxycyanoacetanilide (1.94 g, 0.01 mol) and refluxing for 1 h (Gewald *et al.*, 1966) in the presence of 4.0 ml of diethylamine. Sulfur powder (1.28 g, 0.04 mol) and 40 ml e thanol were then added, and the resulting solution was heated for 2 h at 323 K. Crystals of (I) were grown by slow evaporation from a solution in 2-propanol (yield 68%).

Refinement

H atoms were positioned geometrically [N—H = 0.86 Å, and C—H = 0.93 (CH), 0.97 (CH_2) and 0.96 Å (CH_3)] and constrained to ride on their parent atoms with U_{iso}(H) values of 1.2 U_{eq}(C,*N*), or 1.5 U_{eq}(*C*-methyl). A rotating-group model was used for the methyl group.

Figures

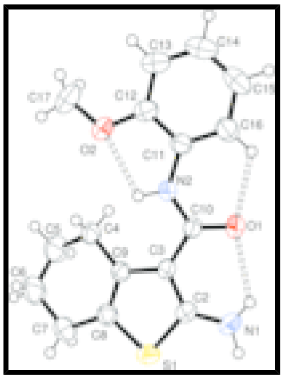

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. Dashed lines indicate intramolecular hydrogen bonds.

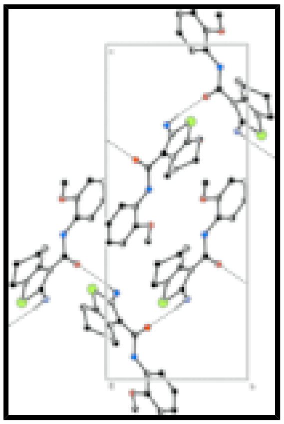

Fig. 2. The packing of (I), viewed down the *a* axis shows molecules connected by N—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

2-Amino-*N*-(2-methoxyphenyl)-4,5,6,7-tetrahydrobenzo-1-thiophene-3-carboxamide

Crystal data

$C_{16}H_{18}N_2O_2S$

$M_r = 302.38$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 8.709\ (2)$ Å

$b = 8.576\ (2)$ Å

$c = 20.306\ (5)$ Å

$\beta = 90.742\ (4)$º

$V = 1516.6\ (6)$ Å^3

$Z = 4$

$F_{000} = 640$

$D_x = 1.324$ Mg m^{-3}

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

Cell parameters from 520 reflections

$\theta = 1.5–28.5$º

$\mu = 0.22$ mm^{-1}

$T = 293\ (2)$ K

Prism, colourless

0.48 × 0.25 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

2831 independent reflections

2266 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$T = 273(2)$ K	$\theta_{max} = 25.5°$
ψ and ω scans	$\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10\rightarrow9$
$T_{min} = 0.937$, $T_{max} = 0.998$	$k = -10\rightarrow10$
10964 measured reflections	$l = -24\rightarrow24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H-atom parameters constrained
$wR(F^2) = 0.147$	$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.3763P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
2831 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
191 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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*	$U_{iso}*/U_{eq}$
N1	0.2332 (3)	0.4280 (3)	0.75854 (10)	0.0537 (6)
H1A	0.2921	0.3616	0.7397	0.064*
H1B	0.2625	0.4724	0.7945	0.064*
N2	0.0765 (2)	0.3030 (2)	0.56763 (9)	0.0431 (5)
H2	0.0161	0.3775	0.5558	0.052*
O1	0.2092 (2)	0.2087 (2)	0.65522 (8)	0.0568 (5)
O2	−0.0842 (2)	0.3124 (2)	0.46026 (9)	0.0602 (6)
S1	−0.02268 (8)	0.59785 (8)	0.76968 (3)	0.0466 (2)
C2	0.0936 (3)	0.4630 (3)	0.73142 (10)	0.0372 (5)
C3	0.0248 (3)	0.4060 (2)	0.67459 (10)	0.0332 (5)
C4	−0.2396 (3)	0.4330 (3)	0.60860 (13)	0.0457 (6)
H4A	−0.2363	0.3217	0.6001	0.055*

H4B	−0.2100	0.4866	0.5687	0.055*
C5	−0.4026 (3)	0.4794 (4)	0.62600 (17)	0.0700 (9)
H5A	−0.4664	0.4747	0.5865	0.084*
H5B	−0.4428	0.4045	0.6572	0.084*
C6	−0.4136 (4)	0.6365 (4)	0.65456 (17)	0.0733 (9)
H6A	−0.5201	0.6572	0.6649	0.088*
H6B	−0.3827	0.7120	0.6217	0.088*
C7	−0.3169 (3)	0.6604 (4)	0.71594 (14)	0.0561 (7)
H7A	−0.2982	0.7709	0.7224	0.067*
H7B	−0.3716	0.6216	0.7539	0.067*
C8	−0.1664 (3)	0.5760 (3)	0.71018 (11)	0.0410 (6)
C9	−0.1266 (3)	0.4723 (2)	0.66323 (10)	0.0348 (5)
C10	0.1099 (3)	0.2970 (2)	0.63284 (10)	0.0358 (5)
C11	0.1258 (3)	0.2057 (3)	0.51622 (11)	0.0393 (5)
C12	0.0353 (3)	0.2094 (3)	0.45871 (11)	0.0440 (6)
C13	0.0714 (4)	0.1160 (3)	0.40563 (12)	0.0556 (7)
H13	0.0112	0.1187	0.3675	0.067*
C14	0.1965 (4)	0.0193 (3)	0.40959 (14)	0.0617 (8)
H14	0.2197	−0.0452	0.3743	0.074*
C15	0.2878 (4)	0.0167 (3)	0.46525 (15)	0.0608 (8)
H15	0.3732	−0.0483	0.4670	0.073*
C16	0.2536 (3)	0.1103 (3)	0.51877 (13)	0.0499 (6)
H16	0.3163	0.1089	0.5562	0.060*
C17	−0.2049 (4)	0.2966 (4)	0.41266 (14)	0.0665 (8)
H17A	−0.1654	0.3157	0.3695	0.100*
H17B	−0.2845	0.3705	0.4219	0.100*
H17C	−0.2460	0.1928	0.4146	0.100*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0486 (14)	0.0669 (15)	0.0452 (11)	0.0091 (11)	−0.0165 (10)	−0.0131 (10)
N2	0.0523 (13)	0.0423 (12)	0.0344 (10)	0.0187 (9)	−0.0073 (9)	−0.0032 (8)
O1	0.0608 (12)	0.0652 (12)	0.0439 (9)	0.0288 (10)	−0.0142 (8)	−0.0020 (8)
O2	0.0615 (13)	0.0734 (13)	0.0450 (10)	0.0203 (10)	−0.0201 (9)	−0.0107 (9)
S1	0.0501 (4)	0.0524 (4)	0.0374 (3)	0.0024 (3)	−0.0013 (3)	−0.0102 (3)
C2	0.0389 (14)	0.0395 (12)	0.0331 (11)	−0.0031 (10)	−0.0017 (9)	0.0011 (9)
C3	0.0340 (13)	0.0343 (11)	0.0314 (10)	0.0010 (9)	−0.0031 (9)	0.0035 (9)
C4	0.0411 (15)	0.0415 (13)	0.0541 (14)	0.0050 (11)	−0.0126 (11)	−0.0061 (11)
C5	0.0432 (18)	0.078 (2)	0.089 (2)	0.0065 (15)	−0.0170 (15)	−0.0128 (17)
C6	0.0479 (19)	0.074 (2)	0.098 (2)	0.0185 (15)	−0.0123 (17)	−0.0151 (18)
C7	0.0481 (17)	0.0589 (17)	0.0614 (16)	0.0145 (13)	0.0041 (13)	−0.0069 (13)
C8	0.0386 (14)	0.0419 (13)	0.0426 (13)	0.0022 (10)	0.0014 (10)	−0.0003 (10)
C9	0.0356 (13)	0.0325 (11)	0.0362 (11)	−0.0010 (9)	−0.0016 (9)	0.0015 (9)
C10	0.0359 (13)	0.0352 (12)	0.0363 (11)	0.0027 (9)	−0.0063 (9)	0.0014 (9)
C11	0.0460 (15)	0.0355 (12)	0.0364 (11)	0.0016 (10)	0.0026 (10)	−0.0015 (9)
C12	0.0514 (16)	0.0437 (14)	0.0369 (12)	−0.0042 (11)	−0.0008 (11)	−0.0003 (10)
C13	0.070 (2)	0.0569 (16)	0.0405 (13)	−0.0074 (15)	0.0029 (12)	−0.0089 (11)

C14	0.082 (2)	0.0519 (17)	0.0519 (15)	−0.0056 (15)	0.0210 (15)	−0.0133 (13)
C15	0.064 (2)	0.0492 (16)	0.0700 (18)	0.0133 (14)	0.0207 (15)	−0.0047 (13)
C16	0.0503 (16)	0.0492 (15)	0.0503 (14)	0.0104 (12)	0.0041 (12)	−0.0007 (11)
C17	0.065 (2)	0.077 (2)	0.0566 (16)	−0.0081 (16)	−0.0254 (14)	0.0132 (15)

Geometric parameters (Å, °)

N1—C2	1.362 (3)	C6—C7	1.509 (4)
N1—H1A	0.8600	C6—H6A	0.9700
N1—H1B	0.8600	C6—H6B	0.9700
N2—C10	1.353 (3)	C7—C8	1.504 (4)
N2—C11	1.408 (3)	C7—H7A	0.9700
N2—H2	0.8600	C7—H7B	0.9700
O1—C10	1.232 (3)	C8—C9	1.352 (3)
O2—C12	1.366 (3)	C11—C16	1.382 (3)
O2—C17	1.425 (3)	C11—C12	1.401 (3)
S1—C2	1.728 (2)	C12—C13	1.383 (3)
S1—C8	1.738 (2)	C13—C14	1.370 (4)
C2—C3	1.383 (3)	C13—H13	0.9300
C3—C9	1.452 (3)	C14—C15	1.374 (4)
C3—C10	1.469 (3)	C14—H14	0.9300
C4—C9	1.512 (3)	C15—C16	1.386 (4)
C4—C5	1.520 (4)	C15—H15	0.9300
C4—H4A	0.9700	C16—H16	0.9300
C4—H4B	0.9700	C17—H17A	0.9600
C5—C6	1.471 (4)	C17—H17B	0.9600
C5—H5A	0.9700	C17—H17C	0.9600
C5—H5B	0.9700		

C2—N1—H1A	120.0	C6—C7—H7B	109.6
C2—N1—H1B	120.0	H7A—C7—H7B	108.1
H1A—N1—H1B	120.0	C9—C8—C7	127.1 (2)
C10—N2—C11	129.7 (2)	C9—C8—S1	111.86 (18)
C10—N2—H2	115.1	C7—C8—S1	120.99 (18)
C11—N2—H2	115.1	C8—C9—C3	112.7 (2)
C12—O2—C17	118.6 (2)	C8—C9—C4	119.6 (2)
C2—S1—C8	92.08 (11)	C3—C9—C4	127.6 (2)
N1—C2—C3	129.4 (2)	O1—C10—N2	121.7 (2)
N1—C2—S1	119.40 (17)	O1—C10—C3	122.4 (2)
C3—C2—S1	111.20 (17)	N2—C10—C3	115.89 (19)
C2—C3—C9	112.2 (2)	C16—C11—C12	119.3 (2)
C2—C3—C10	119.3 (2)	C16—C11—N2	125.2 (2)
C9—C3—C10	128.45 (19)	C12—C11—N2	115.5 (2)
C9—C4—C5	111.8 (2)	O2—C12—C13	125.0 (2)
C9—C4—H4A	109.2	O2—C12—C11	114.6 (2)
C5—C4—H4A	109.2	C13—C12—C11	120.4 (2)
C9—C4—H4B	109.2	C14—C13—C12	119.6 (3)
C5—C4—H4B	109.2	C14—C13—H13	120.2
H4A—C4—H4B	107.9	C12—C13—H13	120.2
C6—C5—C4	113.4 (3)	C15—C14—C13	120.6 (2)

C6—C5—H5A	108.9	C15—C14—H14	119.7
C4—C5—H5A	108.9	C13—C14—H14	119.7
C6—C5—H5B	108.9	C14—C15—C16	120.5 (3)
C4—C5—H5B	108.9	C14—C15—H15	119.7
H5A—C5—H5B	107.7	C16—C15—H15	119.7
C5—C6—C7	114.3 (3)	C11—C16—C15	119.6 (3)
C5—C6—H6A	108.7	C11—C16—H16	120.2
C7—C6—H6A	108.7	C15—C16—H16	120.2
C5—C6—H6B	108.7	O2—C17—H17A	109.5
C7—C6—H6B	108.7	O2—C17—H17B	109.5
H6A—C6—H6B	107.6	H17A—C17—H17B	109.5
C8—C7—C6	110.4 (2)	O2—C17—H17C	109.5
C8—C7—H7A	109.6	H17A—C17—H17C	109.5
C6—C7—H7A	109.6	H17B—C17—H17C	109.5
C8—C7—H7B	109.6		

C8—S1—C2—N1	−179.4 (2)	C5—C4—C9—C3	159.3 (2)
C8—S1—C2—C3	0.96 (18)	C11—N2—C10—O1	9.1 (4)
N1—C2—C3—C9	179.3 (2)	C11—N2—C10—C3	−173.1 (2)
S1—C2—C3—C9	−1.1 (2)	C2—C3—C10—O1	28.5 (3)
N1—C2—C3—C10	−3.4 (4)	C9—C3—C10—O1	−154.7 (2)
S1—C2—C3—C10	176.22 (16)	C2—C3—C10—N2	−149.3 (2)
C9—C4—C5—C6	45.6 (4)	C9—C3—C10—N2	27.4 (3)
C4—C5—C6—C7	−57.6 (4)	C10—N2—C11—C16	−20.0 (4)
C5—C6—C7—C8	37.7 (4)	C10—N2—C11—C12	160.2 (2)
C6—C7—C8—C9	−10.1 (4)	C17—O2—C12—C13	17.7 (4)
C6—C7—C8—S1	173.4 (2)	C17—O2—C12—C11	−163.6 (2)
C2—S1—C8—C9	−0.60 (19)	C16—C11—C12—O2	−177.3 (2)
C2—S1—C8—C7	176.4 (2)	N2—C11—C12—O2	2.6 (3)
C7—C8—C9—C3	−176.7 (2)	C16—C11—C12—C13	1.5 (4)
S1—C8—C9—C3	0.1 (3)	N2—C11—C12—C13	−178.7 (2)
C7—C8—C9—C4	0.9 (4)	O2—C12—C13—C14	178.7 (2)
S1—C8—C9—C4	177.63 (17)	C11—C12—C13—C14	0.0 (4)
C2—C3—C9—C8	0.6 (3)	C12—C13—C14—C15	−1.3 (4)
C10—C3—C9—C8	−176.3 (2)	C13—C14—C15—C16	1.0 (4)
C2—C3—C9—C4	−176.7 (2)	C12—C11—C16—C15	−1.8 (4)
C10—C3—C9—C4	6.4 (4)	N2—C11—C16—C15	178.4 (2)
C5—C4—C9—C8	−17.9 (3)	C14—C15—C16—C11	0.6 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1	0.86	2.27	2.823 (3)	122
N1—H1B···O1^{i}	0.86	2.28	3.015 (3)	143
N2—H2···O2	0.86	2.19	2.577 (3)	107
C16—H16···O1	0.93	2.39	2.927 (3)	117

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

Fig. 1

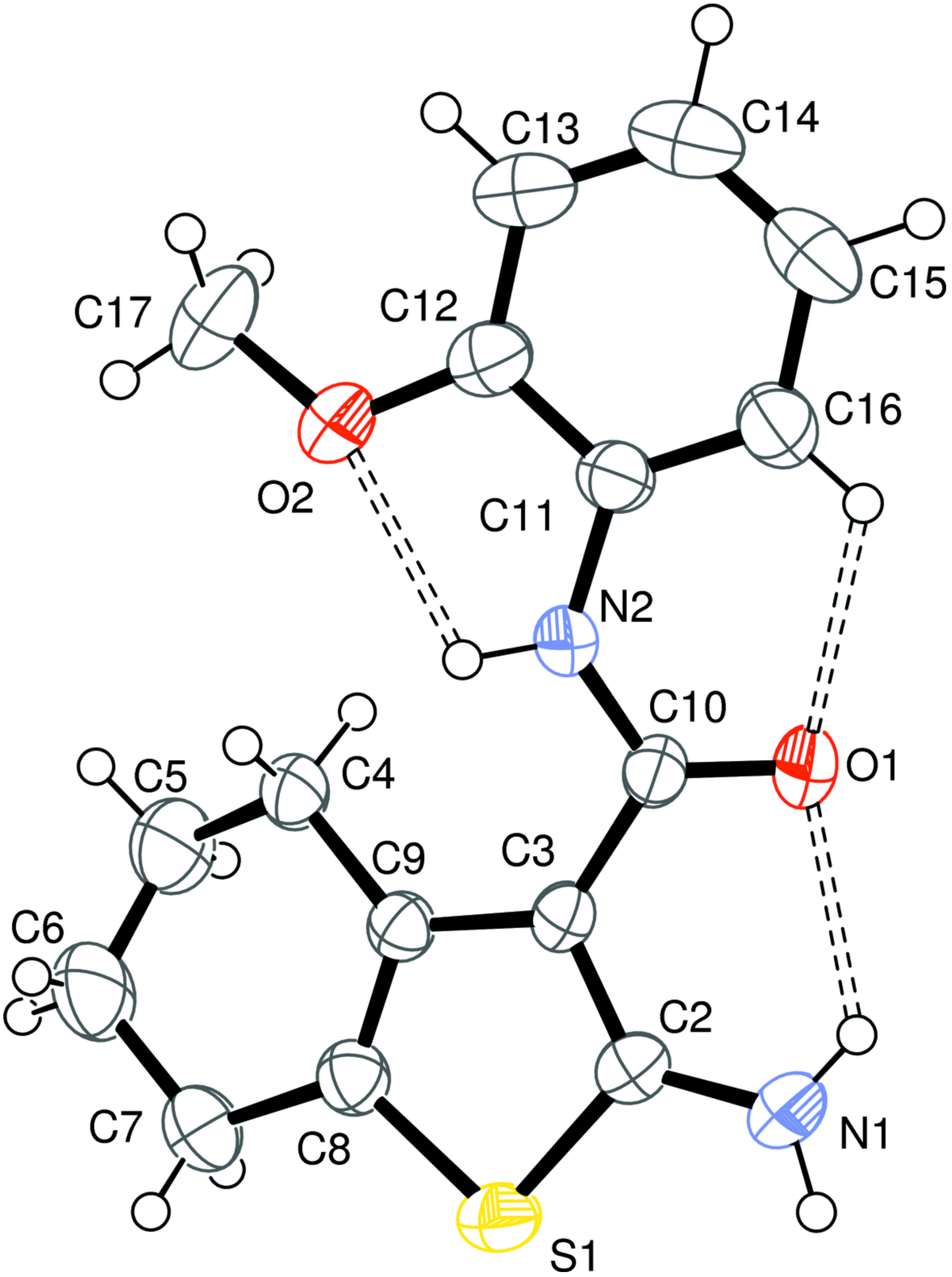
C14
C13
C15
C17
C12
C16
O2
C11
N2
C10
C4
C5
O1
C9
C3
C6
C2
C7
C8
N1
S1

Fig. 2