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2-(2,4,5-Trimethoxyphenyl)-1,3-benzothiazole

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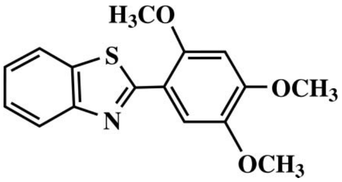
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.089; data-to-parameter ratio = 15.5.

In the title compound, $C_{16}H_{15}NO_3S$, the benzothiazole ring system is essentially planar and makes a dihedral angle of 4.5 (2)° with the plane of the attached benzene ring. The 4- and 5-methoxy groups are coplanar with the attached benzene ring, while the 2-methoxy group is rotated slightly out of the plane [4.8 (3)°].

Related literature

For related literature, see: Kalle Co. (1962); Barbara *et al.* (1980); Leng *et al.* (2001); Kannan *et al.* (2001); Mori *et al.* (2003); Oren *et al.* (1999).



Experimental

Crystal data

$C_{16}H_{15}NO_3S$
M_r = 301.35
Orthorhombic, $P2_12_12_1$
a = 6.9897 (6) Å
b = 12.6809 (11) Å
c = 16.4228 (14) Å
V = 1455.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 273 (2) K
0.18 × 0.12 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.960, $T_{\max}$ = 0.982
9473 measured reflections
3309 independent reflections
2843 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.089
S = 1.03
3309 reflections
213 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.20 e Å^{-3}
$\Delta\rho_{\min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 1374 Friedel pairs
Flack parameter: −0.06 (7)

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2131).

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supplementary materials

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2-(2,4,5-Trimethoxyphenyl)-1,3-benzothiazole

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Comment

2-arylbenzothiazole derivatives have been well known for their biological and pharmaceutical activities, such as antitumor, antiviral, antimicrobial activities and potent inhibitory activity (Altanla *et al.*, 1999). Recently, 2-arylbenzothiazole derivatives have also attracted increasing attention due to their application in the area of organic optoelectronic materials, such as second-order nonlinear optical (NLO) materials (Leng *et al.*, 2001), two-photo absorption (TPA) chromophores (Prasad *et al.*, 2001), photoconducting materials (Kalle & Br, 1962), liquid crystals (Kawamoto *et al.*, 2003) and efficient fluorophores (Barbara *et al.*, 1980). The title compound, (I), plays a very important role in the electroactive and photoative materials, which emits blue luminescence in both solid state and organic solution upon by UV-light at ambient temperature.

In the crystal structure of the title molecule, the five-member thiazole ring is almost coplanar with its fused benzene ring, and the dihedral angle between the mean planes of the rings is only 0.3 (1) ° (Fig. 1). The benzothiazole ring is twisted slightly away from the plane of C13—C8 phenyl ring due to the steric hindrance. The dihedral angle between them is 4.5 (2) °. 2-Methoxy group is twisted out of the C13—C8 benzene plane due to the steric interference from the adjacent benzothiazole moiety. The C10—C9—O1—C16 torsion angle is 4.8 (3) °. the other two methoxy groups almost lies in the C8—C13 benzene plane. The bond distances of C7—N1 (1.301 (2) Å) and C1—N1 (1.387 (2) Å), have an average trend, which is a result of π-electron delocalization. Similarly, the bond lengths of C6—S1 (1.7292 (19) Å) and C7—S1 (1.7588 (17) Å) have small difference (Table 1).

In the stacking structure of compound (I), there exists an effective overlap of the phenyl rings between two adjacent molecules (Fig. 2). The shortest separation distance is about 3.502 Å. There are also interactions between C15—H and the benzene ring of adjacent molecules, whose shortest distance is 3.494 Å.

Experimental

2-aminothiophenol and 2, 4, 5-trimethoxybenzaldehyde were purchased from Acros and used without further purification. 2-aminothiophenol (1.25 g, 10 mmol) and 2, 4, 5-trimethoxybenzaldehyde (1.96 g, 10 mmol) were dissolved in 20 ml of DMSO under argon atmosphere. The mixture was heated at 473 K for 0.5 h. After cooling, the solution was poured into ice water, and then adjusted the solution to pH 8–9 with 1 N $NaHCO_3$ solution. The precipitate was filtered, washed with a great deal of water several times. After dried under vacuum, the crude product was recrystallized with ethanol. Light green crystal, yield: 90%. ^{1}H NMR ($CDCl_3$, p.p.m.) 8.09 (s, 1H), 8.03 (d, J = 7.2 Hz, 1H), 7.88 (d, J = 7.8 Hz, 1H), 7.45 (t, J = 7.5 Hz, 1H), 7.33 (t, J = 7.2 Hz, 1H), 6.61 (s, 1H), 4.04 (s, 3H), 4.01 (s, 3H), 3.96 (s, 3H). mp = 471–472 K. Anal·Calcd for $C_{16}H_{15}O_3NS$: C, 63.77; H, 5.01; N, 4.65. Found: C, 63.69; H, 4.96; N, 4.62%. MS (FAB): m/e, 301 (M^+).

Refinement

H atoms were placed in idealized positions and constrained to ride on their parent atoms with C—H = 0.93 Å, and with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2\ U_{\mathrm{eq}}$(carrier atom).

Figures

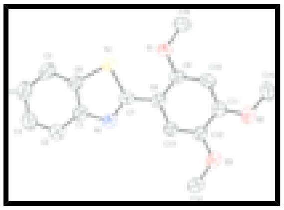

Fig. 1. The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

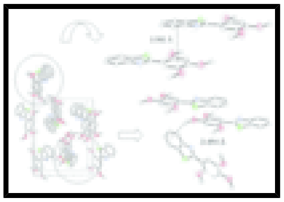

Fig. 2. Part of the crystal packing of (I), showing the intermolecular interactions. All hydrogen atoms have been omitted for clarity.

2-(2,4,5-Trimethoxyphenyl)-1,3-benzothiazole

Crystal data

$C_{16}H_{15}NO_3S$

$M_r = 301.35$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.9897\ (6)$ Å

$b = 12.6809\ (11)$ Å

$c = 16.4228\ (14)$ Å

$V = 1455.6\ (2)$ Å^3

$Z = 4$

$F_{000} = 632$

$D_x = 1.375$ Mg m^{-3}

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

$\mu = 0.23$ mm^{-1}

$T = 273\ (2)$ K

Block, green

0.18 × 0.12 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 273(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\mathrm{min}} = 0.960$, $T_{\mathrm{max}} = 0.982$

9473 measured reflections

3309 independent reflections

2843 reflections with $I > 2\sigma(I)$

$R_{\mathrm{int}} = 0.022$

$\theta_{\mathrm{max}} = 27.5°$

$\theta_{\mathrm{min}} = 2.0°$

$h = -9 \rightarrow 9$

$k = -16 \rightarrow 15$

$l = -21 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.089$

$S = 1.03$

3309 reflections

213 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.0899P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.20$ e Å^{-3}

$\Delta\rho_{min} = -0.18$ e Å^{-3}

Extinction correction: none

Absolute structure: Flack (1983)

Flack parameter: −0.06 (7)

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}
C1	−0.0446 (3)	0.79063 (14)	0.78838 (11)	0.0448 (4)
C2	−0.2035 (3)	0.72585 (17)	0.77923 (14)	0.0592 (5)
C3	−0.3326 (3)	0.71829 (18)	0.84219 (15)	0.0657 (6)
C4	−0.3062 (3)	0.77359 (18)	0.91380 (15)	0.0638 (6)
C5	−0.1498 (3)	0.83768 (17)	0.92492 (14)	0.0590 (5)
C6	−0.0190 (3)	0.84597 (14)	0.86102 (12)	0.0463 (4)
C7	0.2260 (3)	0.87307 (13)	0.75684 (10)	0.0410 (4)
C8	0.3898 (2)	0.90527 (13)	0.70673 (10)	0.0399 (4)
C9	0.5369 (3)	0.96957 (14)	0.73449 (11)	0.0432 (4)
C10	0.6914 (3)	0.99462 (14)	0.68413 (11)	0.0452 (4)
C11	0.6967 (3)	0.95721 (13)	0.60518 (11)	0.0423 (4)
C12	0.5458 (3)	0.89504 (14)	0.57504 (11)	0.0436 (4)
C13	0.3979 (3)	0.86906 (13)	0.62575 (10)	0.0424 (4)
H13	0.3000	0.8263	0.6062	0.051*
C14	0.4136 (3)	0.80441 (18)	0.46223 (13)	0.0669 (6)
H14A	0.4039	0.7396	0.4921	0.100*

H14B	0.4411	0.7893	0.4062	0.100*
H14C	0.2949	0.8422	0.4660	0.100*
C15	0.9938 (3)	1.04209 (18)	0.57777 (14)	0.0623 (6)
H15A	0.9456	1.1112	0.5900	0.093*
H15C	1.0878	1.0470	0.5353	0.093*
H15B	1.0514	1.0124	0.6256	0.093*
C16	0.6571 (3)	1.07802 (17)	0.84273 (12)	0.0641 (6)
H16A	0.7822	1.0469	0.8404	0.096*
H16B	0.6264	1.0949	0.8982	0.096*
H16C	0.6552	1.1412	0.8106	0.096*
H2	−0.222 (3)	0.6833 (15)	0.7316 (14)	0.067 (7)*
H3	−0.450 (4)	0.6731 (19)	0.8373 (15)	0.082 (8)*
H4	−0.398 (3)	0.7704 (18)	0.9581 (14)	0.077 (7)*
H5	−0.128 (3)	0.8792 (16)	0.9739 (14)	0.067 (7)*
H10	0.788 (3)	1.0332 (13)	0.7063 (11)	0.043 (5)*
N1	0.0967 (2)	0.80764 (12)	0.73050 (9)	0.0461 (4)
O1	0.5212 (2)	1.00604 (12)	0.81193 (8)	0.0604 (4)
O2	0.84061 (18)	0.97636 (10)	0.55154 (8)	0.0537 (3)
O3	0.5617 (2)	0.86625 (11)	0.49516 (8)	0.0574 (4)
S1	0.18854 (8)	0.91984 (4)	0.85636 (3)	0.05518 (15)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0448 (10)	0.0467 (9)	0.0429 (10)	0.0012 (8)	0.0007 (8)	0.0006 (8)
C2	0.0579 (12)	0.0641 (12)	0.0556 (12)	−0.0135 (11)	0.0050 (11)	−0.0069 (10)
C3	0.0525 (12)	0.0691 (13)	0.0755 (15)	−0.0110 (11)	0.0099 (11)	−0.0008 (12)
C4	0.0585 (13)	0.0661 (13)	0.0669 (14)	−0.0005 (11)	0.0216 (12)	−0.0010 (11)
C5	0.0655 (13)	0.0604 (12)	0.0510 (11)	0.0045 (11)	0.0141 (10)	−0.0073 (10)
C6	0.0493 (10)	0.0450 (9)	0.0445 (10)	0.0021 (8)	0.0055 (8)	−0.0031 (8)
C7	0.0478 (10)	0.0398 (8)	0.0353 (9)	0.0033 (8)	−0.0023 (7)	−0.0032 (7)
C8	0.0438 (9)	0.0393 (8)	0.0366 (9)	0.0019 (7)	0.0001 (7)	−0.0001 (7)
C9	0.0497 (10)	0.0433 (9)	0.0366 (9)	0.0008 (8)	−0.0044 (8)	−0.0010 (7)
C10	0.0459 (10)	0.0431 (9)	0.0466 (10)	−0.0052 (9)	−0.0057 (9)	0.0004 (8)
C11	0.0422 (9)	0.0396 (8)	0.0451 (9)	0.0021 (8)	0.0023 (8)	0.0061 (7)
C12	0.0505 (10)	0.0434 (10)	0.0369 (9)	−0.0003 (8)	0.0026 (8)	0.0006 (7)
C13	0.0468 (10)	0.0397 (9)	0.0406 (9)	−0.0023 (7)	0.0013 (8)	−0.0024 (7)
C14	0.0804 (15)	0.0773 (14)	0.0432 (11)	−0.0204 (12)	0.0057 (11)	−0.0146 (11)
C15	0.0460 (11)	0.0729 (13)	0.0679 (13)	−0.0114 (10)	0.0046 (10)	0.0100 (11)
C16	0.0781 (14)	0.0650 (12)	0.0492 (11)	−0.0179 (12)	−0.0098 (11)	−0.0125 (10)
N1	0.0498 (9)	0.0514 (8)	0.0371 (8)	−0.0064 (7)	0.0025 (7)	−0.0051 (7)
O1	0.0670 (9)	0.0732 (9)	0.0412 (7)	−0.0227 (8)	0.0023 (6)	−0.0153 (7)
O2	0.0494 (7)	0.0600 (8)	0.0518 (7)	−0.0081 (6)	0.0091 (6)	0.0007 (6)
O3	0.0603 (8)	0.0719 (9)	0.0400 (7)	−0.0122 (7)	0.0081 (7)	−0.0087 (7)
S1	0.0594 (3)	0.0634 (3)	0.0428 (2)	−0.0111 (3)	0.0081 (2)	−0.0165 (2)

Geometric parameters (Å, °)

C1—N1	1.388 (2)	C10—C11	1.381 (3)

C1—C2	1.389 (3)	C10—H10	0.909 (19)
C1—C6	1.396 (3)	C11—O2	1.359 (2)
C2—C3	1.376 (3)	C11—C12	1.407 (3)
C2—H2	0.96 (2)	C12—O3	1.366 (2)
C3—C4	1.382 (3)	C12—C13	1.368 (2)
C3—H3	1.00 (3)	C13—H13	0.9300
C4—C5	1.374 (3)	C14—O3	1.407 (2)
C4—H4	0.97 (2)	C14—H14A	0.9600
C5—C6	1.396 (3)	C14—H14B	0.9600
C5—H5	0.97 (2)	C14—H14C	0.9600
C6—S1	1.7283 (19)	C15—O2	1.424 (2)
C7—N1	1.301 (2)	C15—H15A	0.9600
C7—C8	1.468 (2)	C15—H15C	0.9600
C7—S1	1.7583 (17)	C15—H15B	0.9600
C8—C9	1.389 (2)	C16—O1	1.411 (2)
C8—C13	1.408 (2)	C16—H16A	0.9600
C9—O1	1.358 (2)	C16—H16B	0.9600
C9—C10	1.397 (3)	C16—H16C	0.9600

N1—C1—C2	125.93 (18)	O2—C11—C12	115.28 (15)
N1—C1—C6	114.58 (16)	C10—C11—C12	120.20 (17)
C2—C1—C6	119.49 (18)	O3—C12—C13	125.58 (17)
C3—C2—C1	119.0 (2)	O3—C12—C11	115.25 (16)
C3—C2—H2	119.0 (14)	C13—C12—C11	119.17 (17)
C1—C2—H2	122.0 (14)	C12—C13—C8	121.80 (17)
C2—C3—C4	121.1 (2)	C12—C13—H13	119.1
C2—C3—H3	121.0 (15)	C8—C13—H13	119.1
C4—C3—H3	117.9 (14)	O3—C14—H14A	109.5
C5—C4—C3	121.3 (2)	O3—C14—H14B	109.5
C5—C4—H4	116.7 (14)	H14A—C14—H14B	109.5
C3—C4—H4	122.0 (14)	O3—C14—H14C	109.5
C4—C5—C6	117.8 (2)	H14A—C14—H14C	109.5
C4—C5—H5	123.6 (13)	H14B—C14—H14C	109.5
C6—C5—H5	118.6 (13)	O2—C15—H15A	109.5
C1—C6—C5	121.38 (18)	O2—C15—H15C	109.5
C1—C6—S1	110.02 (14)	H15A—C15—H15C	109.5
C5—C6—S1	128.60 (16)	O2—C15—H15B	109.5
N1—C7—C8	122.20 (15)	H15A—C15—H15B	109.5
N1—C7—S1	114.88 (14)	H15C—C15—H15B	109.5
C8—C7—S1	122.92 (13)	O1—C16—H16A	109.5
C9—C8—C13	118.13 (15)	O1—C16—H16B	109.5
C9—C8—C7	123.81 (15)	H16A—C16—H16B	109.5
C13—C8—C7	118.05 (15)	O1—C16—H16C	109.5
O1—C9—C8	116.58 (16)	H16A—C16—H16C	109.5
O1—C9—C10	122.66 (16)	H16B—C16—H16C	109.5
C8—C9—C10	120.76 (16)	C7—N1—C1	111.47 (15)
C11—C10—C9	119.88 (17)	C9—O1—C16	120.09 (15)
C11—C10—H10	122.8 (11)	C11—O2—C15	117.73 (15)
C9—C10—H10	117.2 (11)	C12—O3—C14	117.27 (15)
O2—C11—C10	124.53 (17)	C6—S1—C7	89.04 (9)

N1—C1—C2—C3	−179.2 (2)
C6—C1—C2—C3	0.3 (3)
C1—C2—C3—C4	−0.2 (4)
C2—C3—C4—C5	−0.3 (4)
C3—C4—C5—C6	0.6 (3)
N1—C1—C6—C5	179.53 (18)
C2—C1—C6—C5	0.0 (3)
N1—C1—C6—S1	−0.5 (2)
C2—C1—C6—S1	179.92 (15)
C4—C5—C6—C1	−0.5 (3)
C4—C5—C6—S1	179.64 (17)
N1—C7—C8—C9	−175.75 (17)
S1—C7—C8—C9	4.5 (2)
N1—C7—C8—C13	4.4 (2)
S1—C7—C8—C13	−175.33 (13)
C13—C8—C9—O1	177.79 (16)
C7—C8—C9—O1	−2.1 (3)
C13—C8—C9—C10	−2.0 (3)
C7—C8—C9—C10	178.11 (16)
O1—C9—C10—C11	−178.34 (16)
C8—C9—C10—C11	1.5 (3)
C9—C10—C11—O2	−179.56 (16)
C9—C10—C11—C12	0.8 (3)
O2—C11—C12—O3	−2.5 (2)
C10—C11—C12—O3	177.22 (16)
O2—C11—C12—C13	177.91 (15)
C10—C11—C12—C13	−2.4 (3)
O3—C12—C13—C8	−177.75 (17)
C11—C12—C13—C8	1.8 (3)
C9—C8—C13—C12	0.4 (3)
C7—C8—C13—C12	−179.76 (16)
C8—C7—N1—C1	−179.19 (15)
S1—C7—N1—C1	0.6 (2)
C2—C1—N1—C7	179.49 (18)
C6—C1—N1—C7	0.0 (2)
C8—C9—O1—C16	−175.31 (17)
C10—C9—O1—C16	4.5 (3)
C10—C11—O2—C15	−1.9 (3)
C12—C11—O2—C15	177.74 (15)
C13—C12—O3—C14	−0.1 (3)
C11—C12—O3—C14	−179.71 (17)
C1—C6—S1—C7	0.69 (14)
C5—C6—S1—C7	−179.4 (2)
N1—C7—S1—C6	−0.75 (15)
C8—C7—S1—C6	179.01 (15)

Fig. 1

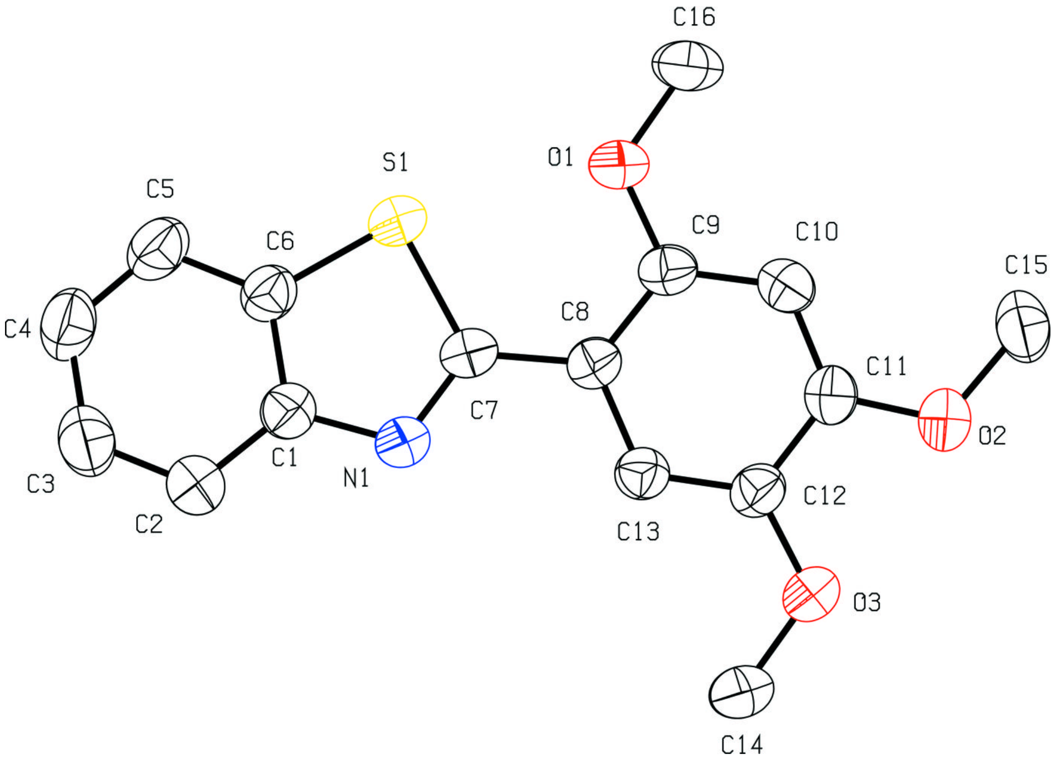

Fig. 2