Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,5-Bis[(1*E*)-3,4-dimethoxybenzylidene]-thiocarbonohydrazide tetrahydrate

B. K. Sarojini,[a] H. S. Yathirajan,[b] B. Narayana,[c] K. Sunil[c] and Michael Bolte[d]*

[a]Department of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalore 574 153, India, [b]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, [c]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India, and [d]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: bolte@chemie.uni-frankfurt.de

Received 12 July 2007; accepted 13 July 2007

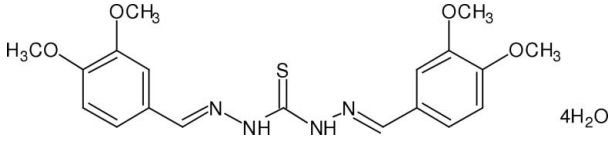
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.006 Å; R factor = 0.042; wR factor = 0.108; data-to-parameter ratio = 13.4.

Geometric parameters of the title compound, $C_{19}H_{22}N_4O_4S\cdot 4H_2O$, are in the usual ranges. Both C=N double bonds are *trans* configured, while only one of the C—N single bonds shows a *trans* configuration (with the other one being *cis* configured). The crystal packing is stabilized by N—H···S, N—H···O, O—H···S and O—H···O hydrogen bonds.

Related literature

For related structures, see: Braibanti *et al.* (1969); Fang *et al.* (2006); Chantrapromma *et al.* (2001); Sarojini *et al.* (2007).

For related literature, see: Hodnett & Dunn (1970); Wiles & Suprunchuk (1970); Misra *et al.* (1981); Agarwal *et al.* (1983); Varma *et al.* (1986); Singh & Dash (1988).



Experimental

Crystal data

$C_{19}H_{22}N_4O_4S\cdot 4H_2O$
M_r = 474.53
Monoclinic, $P2_1/n$
a = 15.9418 (11) Å
b = 8.7714 (7) Å
c = 16.6510 (11) Å
β = 92.815 (5)°
V = 2325.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 173 (2) K
0.33 × 0.32 × 0.28 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.937, T_{max} = 0.951
18188 measured reflections
4349 independent reflections
3712 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.108
S = 1.05
4349 reflections
325 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···S1^{i}	0.87 (6)	2.52 (6)	3.355 (4)	161 (5)
N4—H4···O1W^{ii}	0.86 (6)	2.31 (6)	3.070 (5)	148 (5)
O1W—H1WA···O1	0.85 (2)	2.06 (3)	2.891 (4)	165 (4)
O1W—H1WB···O3^{iii}	0.85 (2)	2.37 (4)	3.021 (4)	133 (4)
O2W—H2WA···S1^{iv}	0.84 (2)	2.52 (4)	3.305 (4)	155 (7)
O2W—H2WB···O1W^{v}	0.84 (2)	2.05 (3)	2.890 (5)	173 (7)
O3W—H3WB···O2W	0.87 (2)	2.02 (3)	2.861 (6)	163 (6)
O3W—H3WA···O4W	0.87 (2)	1.92 (3)	2.749 (6)	158 (6)
O4W—H4WA···S1^{i}	0.85 (2)	2.60 (3)	3.438 (5)	167 (7)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

KS thanks the Department of Studies in Chemistry, Mangalore University, for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2339).

References

Agarwal, R., Chaudhary, K. C. & Misra, V. S. (1983). *Indian J. Chem.* B**22**, 308–310.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Braibanti, A., Tiripicchio, A. & Camellini, M. T. (1969). *Acta Cryst.* B**25**, 2286–2290.
Chantrapromma, S., Razak, I. A., Fun, H.-K., Karalai, C., Zhang, H., Xie, F.-X., Tian, Y.-P., Ma, W., Zhang, Y.-H. & Ni, S.-S. (2001). *Acta Cryst.* C**57**, 289–290.
Fang, X.-N., Xu, Y.-P., Guo, X.-F. & Zeng, X.-R. (2006). *Acta Cryst.* E**62**, o1052–o1054.
Hodnett, E. M. & Dunn, W. J. (1970). *J. Med. Chem.* **13**, 768–770.
Misra, V. S., Singh, S., Agarwal, R. & Chaudhary, K. C. (1981). *J. Chem. Soc. Pak.* **3**, 209–213.
Sarojini, B. K., Narayana, B., Bindya, S., Yathirajan, H. S. & Bolte, M. (2007). *Acta Cryst.* E**63**, o2946.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Singh, W. M. & Dash, B. C. (1988). *Pesticides*, **22**, 33–37.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Varma, R. S., Prakash, R., Khan, M. M. & Ali, A. (1986). *Indian Drugs*, **23**, 345–349.
Wiles, D. M. & Suprunchuk, T. (1970). *J. Med. Chem.* **13**, 323–324.

supplementary materials

Acta Cryst. (2007). E**63**, o3521 [**doi:10.1107/S1600536807034265**]

1,5-Bis[(1*E*)-3,4-dimethoxybenzylidene]thiocarbonohydrazide tetrahydrate

B. K. Sarojini, H. S. Yathirajan, B. Narayana, K. Sunil and M. Bolte

Comment

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. Some Schiff base derivatives were reported to possess antimicrobial, *anti*-inflammatory and central nervous system activities. Moreover, Schiff bases are also known to have biological activities such as antimicrobial, antifungal, antitumor, and as herbicides. A new Schiff base of thiocarbohydrazide with 3,4-dimethoxy benzaldehyde was synthesized and its crystal structure is reported here.

Geometric parameters of the title compound (Fig. 1) are in the usual ranges. Both C═N double bonds are *trans* configured, but only one of the C—N single bond shows a *trans* configuration whereas the other one is *cis* configured. The crystal packing is stabilized by N—H···S, N—H···O, O—H···S and O—H···O hydrogen bonds.

Experimental

A mixture of 3,4-dimethoxybenzaldehyde (1.66 g, 0.01 mol) and thiocarbohydrazide (0.53 g, 0.005 mol) in 15 ml of absolute ethanol containing 2 drops of dilute sulfuric acid was refluxed for about 4 h. On cooling, the solid separated was filtered and recrystallized from (2:8) DMF and ethanol mixture (m.p.: 468–470 K). Analysis for $C_{19}H_{22}N_4O_4S·4H_2O$: Found (Calculated): C 47.96 (48.05); H 6.27 (6.32); N 11.74 (11.80); S 6.68% (6.74%).

Refinement

H atoms were found in a difference map, but those bonded to C were refined using a riding model with C—H = 0.95Å and U_{iso}(H) = 1.2U_{eq}(C) [C—H = 0.98Å and U_{iso}(H) = 1.5U_{eq}(C) for the methyl group, which was allowed to rotate but not to tip]. The water H atoms were refined with the O—H distance restrained to 0.84 (1)Å and the H···H distance restrained to 1.40 (1)Å and U_{iso}(H) = 1.2U_{eq}(O). The amino H atoms were freely refined.

Figures

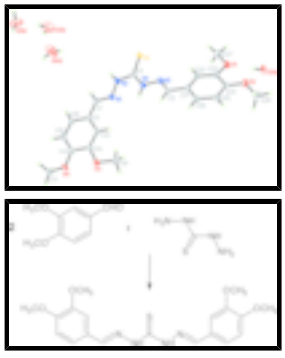


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

1,5-Bis[(1E)-3,4-dimethoxybenzylidene]thiocarbonohydrazide tetrahydrate

Crystal data

$C_{19}H_{22}N_4O_4S·4H_2O$	F_{000} = 1008
M_r = 474.53	D_x = 1.355 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 17322 reflections
a = 15.9418 (11) Å	θ = 3.6–25.7º
b = 8.7714 (7) Å	μ = 0.19 mm^{-1}
c = 16.6510 (11) Å	T = 173 (2) K
β = 92.815 (5)º	Block, red-brown
V = 2325.5 (3) Å^3	0.33 × 0.32 × 0.28 mm
Z = 4	

Data collection

Stoe IPDSII two-circle diffractometer	4349 independent reflections
Radiation source: fine-focus sealed tube	3712 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 173(2) K	θ_{max} = 25.6º
ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −19→19
T_{min} = 0.937, T_{max} = 0.951	k = −10→10
18188 measured reflections	l = −20→19

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.108	$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 1.0873P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
4349 reflections	$\Delta\rho_{max}$ = 0.54 e Å^{-3}
325 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
12 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
S1	0.62197 (6)	0.59999 (11)	0.48744 (7)	0.0229 (3)
N1	0.6007 (2)	0.1935 (4)	0.5838 (2)	0.0194 (7)
N2	0.5838 (2)	0.3291 (4)	0.5438 (2)	0.0206 (7)
H2	0.533 (4)	0.346 (7)	0.524 (4)	0.048 (17)*
N3	0.7878 (2)	0.4980 (4)	0.5639 (2)	0.0212 (7)
N4	0.7202 (2)	0.4006 (4)	0.5727 (2)	0.0204 (7)
H4	0.728 (3)	0.309 (7)	0.590 (3)	0.034 (14)*
O1	1.02971 (18)	0.8313 (3)	0.46858 (19)	0.0284 (7)
O2	1.15681 (17)	0.7740 (3)	0.56386 (19)	0.0264 (7)
O3	0.70084 (17)	−0.3072 (3)	0.72897 (18)	0.0237 (7)
O4	0.56812 (18)	−0.4716 (3)	0.73882 (19)	0.0267 (7)
C1	0.5380 (2)	0.1016 (4)	0.5873 (2)	0.0197 (8)
H1	0.4850	0.1314	0.5637	0.024*
C2	0.8533 (2)	0.4659 (5)	0.6089 (2)	0.0208 (8)
H2A	0.8509	0.3859	0.6472	0.025*
C3	0.6448 (2)	0.4356 (4)	0.5375 (2)	0.0181 (8)
C11	0.5458 (2)	−0.0475 (4)	0.6265 (2)	0.0191 (8)
C12	0.6238 (2)	−0.1012 (4)	0.6591 (2)	0.0184 (8)
H12	0.6727	−0.0401	0.6561	0.022*
C13	0.6288 (2)	−0.2430 (4)	0.6954 (2)	0.0184 (8)
C14	0.5556 (3)	−0.3341 (4)	0.7008 (2)	0.0208 (8)
C15	0.4790 (3)	−0.2810 (5)	0.6688 (3)	0.0240 (9)
H15	0.4299	−0.3418	0.6720	0.029*
C16	0.4742 (3)	−0.1377 (5)	0.6319 (3)	0.0236 (9)
H16	0.4217	−0.1015	0.6103	0.028*
C17	0.4996 (3)	−0.5796 (5)	0.7329 (3)	0.0297 (10)
H17A	0.4509	−0.5372	0.7588	0.045*
H17B	0.5168	−0.6749	0.7596	0.045*
H17C	0.4846	−0.6000	0.6761	0.045*
C18	0.7780 (2)	−0.2235 (5)	0.7209 (3)	0.0258 (9)
H18A	0.7878	−0.2090	0.6638	0.039*
H18B	0.8249	−0.2809	0.7464	0.039*
H18C	0.7737	−0.1239	0.7470	0.039*

C21	0.9316 (2)	0.5514 (4)	0.6017 (2)	0.0198 (8)
C22	0.9387 (2)	0.6590 (5)	0.5394 (3)	0.0211 (8)
H22	0.8913	0.6827	0.5048	0.025*
C23	1.0145 (2)	0.7296 (4)	0.5290 (3)	0.0211 (8)
C24	1.0852 (2)	0.6970 (4)	0.5814 (3)	0.0208 (8)
C25	1.0777 (2)	0.5930 (5)	0.6435 (3)	0.0228 (9)
H25	1.1246	0.5714	0.6791	0.027*
C26	1.0006 (2)	0.5200 (5)	0.6532 (2)	0.0215 (8)
H26	0.9955	0.4483	0.6955	0.026*
C27	0.9587 (3)	0.8724 (6)	0.4161 (3)	0.0325 (10)
H27A	0.9398	0.7830	0.3848	0.049*
H27B	0.9750	0.9533	0.3794	0.049*
H27C	0.9129	0.9087	0.4483	0.049*
C28	1.2329 (3)	0.7296 (6)	0.6074 (3)	0.0301 (10)
H28A	1.2280	0.7515	0.6647	0.045*
H28B	1.2802	0.7870	0.5873	0.045*
H28C	1.2424	0.6202	0.6001	0.045*
O1W	1.18874 (19)	0.9053 (4)	0.4019 (2)	0.0301 (7)
H1WA	1.147 (2)	0.882 (6)	0.430 (2)	0.036*
H1WB	1.171 (3)	0.922 (6)	0.3536 (15)	0.036*
O2W	0.2300 (3)	0.5813 (5)	0.9698 (2)	0.0592 (12)
H2WA	0.214 (4)	0.666 (5)	0.987 (4)	0.071*
H2WB	0.252 (4)	0.535 (7)	1.010 (3)	0.071*
O3W	0.2901 (3)	0.5995 (5)	0.8110 (3)	0.0601 (12)
H3WA	0.274 (4)	0.529 (6)	0.777 (3)	0.072*
H3WB	0.268 (4)	0.576 (7)	0.856 (2)	0.072*
O4W	0.2830 (4)	0.3797 (5)	0.6932 (3)	0.0706 (15)
H4WA	0.300 (5)	0.396 (8)	0.646 (2)	0.085*
H4WB	0.259 (5)	0.293 (5)	0.695 (4)	0.085*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0204 (5)	0.0187 (5)	0.0295 (6)	−0.0011 (4)	0.0002 (4)	0.0063 (4)
N1	0.0198 (16)	0.0167 (16)	0.0216 (18)	0.0005 (13)	−0.0002 (13)	0.0034 (13)
N2	0.0174 (17)	0.0184 (16)	0.0258 (19)	−0.0014 (13)	−0.0022 (14)	0.0059 (14)
N3	0.0178 (16)	0.0210 (16)	0.0248 (19)	−0.0034 (13)	0.0015 (13)	0.0012 (14)
N4	0.0182 (16)	0.0182 (17)	0.0246 (19)	−0.0030 (13)	−0.0008 (13)	0.0037 (14)
O1	0.0248 (15)	0.0308 (16)	0.0296 (17)	−0.0023 (13)	0.0008 (12)	0.0128 (13)
O2	0.0184 (14)	0.0300 (16)	0.0309 (17)	−0.0056 (12)	0.0016 (12)	0.0045 (13)
O3	0.0198 (14)	0.0216 (14)	0.0295 (17)	−0.0001 (11)	−0.0011 (12)	0.0069 (12)
O4	0.0272 (15)	0.0186 (14)	0.0344 (18)	−0.0040 (12)	0.0023 (13)	0.0083 (13)
C1	0.0183 (18)	0.0195 (19)	0.021 (2)	0.0001 (15)	−0.0013 (15)	0.0007 (16)
C2	0.022 (2)	0.0205 (19)	0.020 (2)	−0.0022 (16)	0.0024 (15)	0.0010 (16)
C3	0.0198 (19)	0.0183 (18)	0.0164 (19)	−0.0016 (15)	0.0019 (14)	−0.0009 (15)
C11	0.0208 (19)	0.0191 (19)	0.017 (2)	−0.0017 (15)	0.0007 (15)	−0.0001 (16)
C12	0.0185 (18)	0.0185 (19)	0.018 (2)	−0.0033 (15)	0.0018 (15)	−0.0012 (15)
C13	0.0202 (19)	0.0197 (19)	0.015 (2)	−0.0001 (15)	0.0005 (14)	−0.0017 (15)

C14	0.026 (2)	0.0171 (18)	0.019 (2)	−0.0019 (16)	0.0033 (16)	0.0013 (16)
C15	0.022 (2)	0.023 (2)	0.027 (2)	−0.0072 (16)	0.0016 (16)	0.0008 (17)
C16	0.020 (2)	0.024 (2)	0.026 (2)	−0.0031 (16)	−0.0036 (16)	0.0008 (17)
C17	0.036 (2)	0.021 (2)	0.033 (3)	−0.0096 (18)	0.0076 (19)	0.0022 (18)
C18	0.0183 (19)	0.026 (2)	0.033 (2)	−0.0009 (16)	−0.0003 (16)	0.0051 (18)
C21	0.0193 (19)	0.0185 (18)	0.022 (2)	−0.0006 (15)	0.0028 (15)	−0.0022 (16)
C22	0.0188 (19)	0.0223 (19)	0.022 (2)	0.0008 (15)	−0.0005 (15)	0.0000 (16)
C23	0.023 (2)	0.0187 (19)	0.021 (2)	0.0003 (16)	0.0034 (16)	0.0020 (16)
C24	0.0186 (19)	0.0198 (19)	0.024 (2)	−0.0016 (15)	0.0044 (15)	−0.0025 (16)
C25	0.0199 (19)	0.026 (2)	0.022 (2)	0.0003 (16)	−0.0011 (15)	−0.0002 (17)
C26	0.024 (2)	0.0207 (19)	0.020 (2)	−0.0006 (16)	0.0018 (16)	0.0024 (16)
C27	0.033 (2)	0.035 (2)	0.029 (3)	0.0017 (19)	−0.0029 (19)	0.012 (2)
C28	0.018 (2)	0.039 (3)	0.033 (3)	−0.0034 (18)	−0.0018 (17)	0.001 (2)
O1W	0.0291 (16)	0.0345 (17)	0.0270 (18)	−0.0094 (14)	0.0026 (13)	0.0030 (14)
O2W	0.094 (3)	0.051 (2)	0.033 (2)	0.043 (2)	0.008 (2)	0.0042 (18)
O3W	0.090 (3)	0.050 (2)	0.041 (2)	0.002 (2)	0.006 (2)	0.000 (2)
O4W	0.127 (5)	0.047 (3)	0.039 (2)	−0.019 (3)	0.026 (3)	−0.004 (2)

Geometric parameters (Å, °)

S1—C3	1.696 (4)	C17—H17A	0.9800
N1—C1	1.289 (5)	C17—H17B	0.9800
N1—N2	1.384 (5)	C17—H17C	0.9800
N2—C3	1.355 (5)	C18—H18A	0.9800
N2—H2	0.87 (6)	C18—H18B	0.9800
N3—C2	1.285 (5)	C18—H18C	0.9800
N3—N4	1.388 (5)	C21—C26	1.389 (6)
N4—C3	1.347 (5)	C21—C22	1.411 (6)
N4—H4	0.86 (6)	C22—C23	1.377 (6)
O1—C23	1.375 (5)	C22—H22	0.9500
O1—C27	1.442 (5)	C23—C24	1.420 (6)
O2—C24	1.370 (5)	C24—C25	1.387 (6)
O2—C28	1.436 (5)	C25—C26	1.403 (6)
O3—C13	1.374 (5)	C25—H25	0.9500
O3—C18	1.445 (5)	C26—H26	0.9500
O4—C14	1.372 (5)	C27—H27A	0.9800
O4—C17	1.446 (5)	C27—H27B	0.9800
C1—C11	1.464 (5)	C27—H27C	0.9800
C1—H1	0.9500	C28—H28A	0.9800
C2—C21	1.466 (5)	C28—H28B	0.9800
C2—H2A	0.9500	C28—H28C	0.9800
C11—C16	1.395 (5)	O1W—H1WA	0.85 (2)
C11—C12	1.413 (5)	O1W—H1WB	0.85 (2)
C12—C13	1.383 (5)	O2W—H2WA	0.84 (2)
C12—H12	0.9500	O2W—H2WB	0.84 (2)
C13—C14	1.420 (5)	O3W—H3WA	0.87 (2)
C14—C15	1.389 (6)	O3W—H3WB	0.87 (2)
C15—C16	1.399 (6)	O4W—H4WA	0.85 (2)
C15—H15	0.9500	O4W—H4WB	0.85 (2)

C16—H16	0.9500		
C1—N1—N2	115.2 (3)	H17A—C17—H17C	109.5
C3—N2—N1	120.4 (3)	H17B—C17—H17C	109.5
C3—N2—H2	120 (4)	O3—C18—H18A	109.5
N1—N2—H2	119 (4)	O3—C18—H18B	109.5
C2—N3—N4	114.6 (3)	H18A—C18—H18B	109.5
C3—N4—N3	119.7 (3)	O3—C18—H18C	109.5
C3—N4—H4	118 (3)	H18A—C18—H18C	109.5
N3—N4—H4	121 (3)	H18B—C18—H18C	109.5
C23—O1—C27	116.4 (3)	C26—C21—C22	119.8 (4)
C24—O2—C28	116.8 (3)	C26—C21—C2	120.0 (4)
C13—O3—C18	116.9 (3)	C22—C21—C2	120.1 (4)
C14—O4—C17	116.9 (3)	C23—C22—C21	119.7 (4)
N1—C1—C11	122.0 (3)	C23—C22—H22	120.1
N1—C1—H1	119.0	C21—C22—H22	120.1
C11—C1—H1	119.0	O1—C23—C22	124.8 (4)
N3—C2—C21	120.8 (4)	O1—C23—C24	114.7 (3)
N3—C2—H2A	119.6	C22—C23—C24	120.5 (4)
C21—C2—H2A	119.6	O2—C24—C25	125.8 (4)
N4—C3—N2	116.0 (3)	O2—C24—C23	114.4 (4)
N4—C3—S1	124.9 (3)	C25—C24—C23	119.7 (4)
N2—C3—S1	119.1 (3)	C24—C25—C26	119.6 (4)
C16—C11—C12	119.5 (4)	C24—C25—H25	120.2
C16—C11—C1	118.9 (3)	C26—C25—H25	120.2
C12—C11—C1	121.6 (3)	C21—C26—C25	120.7 (4)
C13—C12—C11	119.9 (3)	C21—C26—H26	119.7
C13—C12—H12	120.1	C25—C26—H26	119.7
C11—C12—H12	120.1	O1—C27—H27A	109.5
O3—C13—C12	125.1 (3)	O1—C27—H27B	109.5
O3—C13—C14	114.6 (3)	H27A—C27—H27B	109.5
C12—C13—C14	120.3 (4)	O1—C27—H27C	109.5
O4—C14—C15	125.2 (4)	H27A—C27—H27C	109.5
O4—C14—C13	115.0 (3)	H27B—C27—H27C	109.5
C15—C14—C13	119.8 (4)	O2—C28—H28A	109.5
C14—C15—C16	119.8 (4)	O2—C28—H28B	109.5
C14—C15—H15	120.1	H28A—C28—H28B	109.5
C16—C15—H15	120.1	O2—C28—H28C	109.5
C11—C16—C15	120.8 (4)	H28A—C28—H28C	109.5
C11—C16—H16	119.6	H28B—C28—H28C	109.5
C15—C16—H16	119.6	H1WA—O1W—H1WB	109 (3)
O4—C17—H17A	109.5	H2WA—O2W—H2WB	106 (7)
O4—C17—H17B	109.5	H3WA—O3W—H3WB	106 (3)
H17A—C17—H17B	109.5	H4WA—O4W—H4WB	110 (4)
O4—C17—H17C	109.5		
C1—N1—N2—C3	178.9 (4)	C13—C14—C15—C16	−0.4 (6)
C2—N3—N4—C3	170.5 (4)	C12—C11—C16—C15	−0.3 (6)
N2—N1—C1—C11	179.2 (3)	C1—C11—C16—C15	179.9 (4)
N4—N3—C2—C21	175.3 (3)	C14—C15—C16—C11	0.3 (6)

N3—N4—C3—N2	175.1 (3)	N3—C2—C21—C26	177.1 (4)
N3—N4—C3—S1	−5.1 (5)	N3—C2—C21—C22	−6.8 (6)
N1—N2—C3—N4	−0.6 (5)	C26—C21—C22—C23	1.5 (6)
N1—N2—C3—S1	179.5 (3)	C2—C21—C22—C23	−174.6 (4)
N1—C1—C11—C16	176.6 (4)	C27—O1—C23—C22	4.1 (6)
N1—C1—C11—C12	−3.2 (6)	C27—O1—C23—C24	−177.5 (4)
C16—C11—C12—C13	0.5 (6)	C21—C22—C23—O1	177.2 (4)
C1—C11—C12—C13	−179.7 (4)	C21—C22—C23—C24	−1.2 (6)
C18—O3—C13—C12	−4.1 (6)	C28—O2—C24—C25	7.8 (6)
C18—O3—C13—C14	176.4 (3)	C28—O2—C24—C23	−171.4 (4)
C11—C12—C13—O3	179.9 (4)	O1—C23—C24—O2	0.9 (5)
C11—C12—C13—C14	−0.7 (6)	C22—C23—C24—O2	179.4 (4)
C17—O4—C14—C15	11.1 (6)	O1—C23—C24—C25	−178.4 (4)
C17—O4—C14—C13	−168.9 (4)	C22—C23—C24—C25	0.1 (6)
O3—C13—C14—O4	0.1 (5)	O2—C24—C25—C26	−178.5 (4)
C12—C13—C14—O4	−179.3 (4)	C23—C24—C25—C26	0.6 (6)
O3—C13—C14—C15	−179.9 (4)	C22—C21—C26—C25	−0.7 (6)
C12—C13—C14—C15	0.6 (6)	C2—C21—C26—C25	175.4 (4)
O4—C14—C15—C16	179.5 (4)	C24—C25—C26—C21	−0.4 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···S1^{i}	0.87 (6)	2.52 (6)	3.355 (4)	161 (5)
N4—H4···O1W^{ii}	0.86 (6)	2.31 (6)	3.070 (5)	148 (5)
O1W—H1WA···O1	0.85 (2)	2.06 (3)	2.891 (4)	165 (4)
O1W—H1WB···O3^{iii}	0.85 (2)	2.37 (4)	3.021 (4)	133 (4)
O2W—H2WA···S1^{iv}	0.84 (2)	2.52 (4)	3.305 (4)	155 (7)
O2W—H2WB···O1W^{v}	0.84 (2)	2.05 (3)	2.890 (5)	173 (7)
O3W—H3WB···O2W	0.87 (2)	2.02 (3)	2.861 (6)	163 (6)
O3W—H3WA···O4W	0.87 (2)	1.92 (3)	2.749 (6)	158 (6)
O4W—H4WA···S1^{i}	0.85 (2)	2.60 (3)	3.438 (5)	167 (7)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) *x*+1/2, −*y*+1/2, *z*−1/2; (iv) *x*−1/2, −*y*+3/2, *z*+1/2; (v) −*x*+3/2, *y*−1/2, −*z*+3/2.

Fig. 1

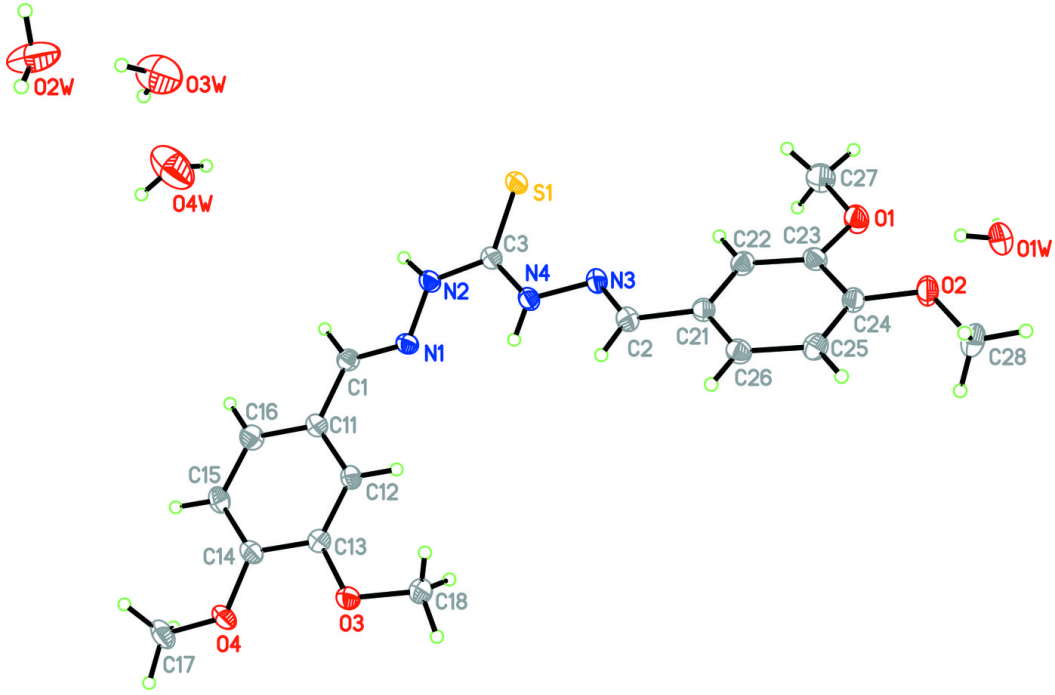

O2W
O3W
O4W
S1
C3
N2
N4
N3
C27
O1
C22
C23
O1W
O2
C24
C2
C21
C25
C28
C26
N1
C1
C16
C11
C15
C12
C14
C13
O3
C18
O4
C17

Fig. 2