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4-(2-Methoxyphenyl)-3-(3,4,5-trimethoxyphenethyl)-2*H*-1,2,4-triazole-5(4*H*)-thione

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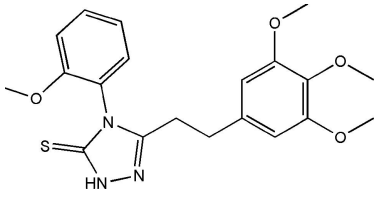
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.099; data-to-parameter ratio = 15.9.

The title compound, $C_{20}H_{23}N_3O_4S$, is an important biologically active heterocyclic compound. The five-membered ring is oriented with respect to the six-membered rings at dihedral angles of 78.60 (3) (trimethoxyphenyl ring) and 71.57 (3)° (methoxyphenyl ring). In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules into infinite chains along the *c* axis.

Related literature

For general background, see: Holla *et al.* (1998); Turan-Zitouni *et al.* (1999); Demirbas *et al.* (2002); Paulvannan *et al.* (2000); Kritsanida *et al.* (2002); Omar *et al.* (1986). For related structures, see: Öztürk *et al.* (2004*a*,*b*); Zhang *et al.* (2004). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{20}H_{23}N_3O_4S$
M_r = 401.47
Triclinic, $P\overline{1}$
a = 8.6368 (6) Å
b = 10.5422 (7) Å
c = 11.6944 (8) Å
α = 91.733 (1)°
β = 92.955 (1)°
γ = 104.075 (1)°
V = 1030.44 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 100 (2) K
0.55 × 0.35 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: integration (*SADABS*; Sheldrick, 1996)
T_{min} = 0.926, T_{max} = 0.946
8266 measured reflections
4149 independent reflections
3689 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.099
S = 1.07
4149 reflections
261 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O2^{i}	0.878 (17)	1.890 (18)	2.7558 (15)	168.4 (15)

Symmetry code: (i) x, y, $z+1$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); 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: HK2411).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Demirbas, N., Ugurluoglu, R. & Demirbas, A. (2002). *Bioorg. Med. Chem.* **10**, 3717–3723.
Holla, B. S., Gonsalves, R. & Shenoy, S. (1998). *Il Farmaco*, **53**, 574–578.
Kritsanida, M., Mouroutsou, A., Marakos, P., Pouli, N., Papakonstantinou-Garoufalias, S., Pannecouque, C., Witvrouw, M. & Clercq, E. D. (2002). *Il Farmaco*, **57**, 253–257.
Omar, A., Mohsen, M. E. & Wafa, O. A. (1986). *J. Heterocycl. Chem.* **23**, 1339–1341.
Öztürk, S., Akkurt, M., Cansız, A., Koparır, M., Şekerci, M. & Heinemann, F. W. (2004*a*). *Acta Cryst.* E**60**, o425–o427.
Öztürk, S., Akkurt, M., Cansız, A., Koparır, M., Şekerci, M. & Heinemann, F. W. (2004*b*). *Acta Cryst.* E**60**, o642–o644.
Paulvannan, K., Chen, T. & Hale, R. (2000). *Tetrahedron*, **56**, 8071–8076.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Turan-Zitouni, G., Kaplancikli, Z. A., Erol, K. & Kilic, F. S. (1999). *Il Farmaco*, **54**, 218–223.
Zhang, L.-X., Zhang, A.-J., Lei, X.-X., Zou, K.-H. & Ng, S. W. (2004). *Acta Cryst.* E**60**, o613–o615.

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supplementary materials

Acta Cryst. (2008). E**64**, o284 [**doi:10.1107/S1600536807066433**]

4-(2-Methoxyphenyl)-3-(3,4,5-trimethoxyphenethyl)-2*H*-1,2,4-triazole-5(4*H*)-thione

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Comment

Substituted triazole derivatives display significant biological activities including antimicrobial (Holla *et al.*, 1998), analgesic (Turan-Zitouni *et al.*, 1999), antitumor (Demirbas *et al.*, 2002), antihypertensive (Paulvannan *et al.*, 2000) and antiviral activities (Kritsanida *et al.*, 2002). The biological activity is closely related to the structure, possibly being due to the presence of the —N—C ?S unit (Omar *et al.*, 1986). We are interested in the synthesis and biological activities of aryloxyacetyl hydrazide derivatives and report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987), and are comparable with those observed in related structures Öztürk *et al.*, 2004*a*,b). The C2?S1 [1.6775 (14) Å] bond is in accordance with the corresponding values of 1.6773 (19) Å in 4-(4-chlorophenyl)-3-(furan-2-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione Öztürk *et al.*, 2004*a*) and 1.668 (5) Å in 4-amino-3-(1,2,3,4,5-pentahydroxypentyl)-1*H*-1,2,4-triazole-5(4*H*)-thione (Zhang *et al.*, 2004). In the triazole ring, the N2 ?C2 [1.3326 (19) Å] bond shows double-bond character.

The rings A (N1–N3/C1/C2), B (C5–C10) and C (C14–C19) are, of course, planar and dihedral angles between them are A/B = 78.60 (3)°, A/C = 71.57 (3)° and B/C = 74.12 (3)°.

In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into infinite chains along the *c* axis (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

The synthesis of the title compound was carried out by refluxing a solution of 1-(3-(3,4,5-trimethoxyphenyl)propanoyl)-4-(2-methoxyphenyl)thiosemicarbazide (4.19 g, 10 mmol) in NaOH (2 *M*) for 5 h. Single crystals suitable for X-ray analysis were obtained by recrystallization from an aqeous ethanol solution at room temperature (yield: 82%; m.p. 491–492 K).

Refinement

H2N (for NH) was located in difference syntheses and refined isotropically [N2—H2N = 0.878 (17) Å and U_{iso}(H) = 0.022 (4) Å^2]. The remaining H atoms were positioned geometrically, with C—H = 0.95, 0.99 and 0.98 Å for aromatic, methylene and methyl H atoms, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H, and x = 1.2 for all other H atoms.

Figures

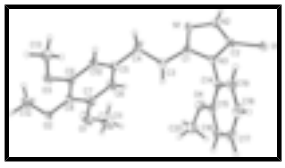

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

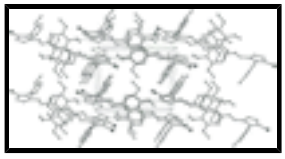

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

4-(2-Methoxyphenyl)-3-(3,4,5-trimethoxyphenethyl)-2*H*-1,2,4-triazole- 5(4*H*)-thione

Crystal data

$C_{20}H_{23}N_3O_4S$	$Z = 2$
$M_r = 401.47$	$F(000) = 424$
Triclinic, $P\overline{1}$	$D_x = 1.294$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 491(1) K
$a = 8.6368$ (6) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 10.5422$ (7) Å	Cell parameters from 5348 reflections
$c = 11.6944$ (8) Å	$\theta = 2.4$–$26.4°$
$\alpha = 91.733$ (1)°	$\mu = 0.19$ mm^{-1}
$\beta = 92.955$ (1)°	$T = 100$ K
$\gamma = 104.075$ (1)°	Rectangular, colourless
$V = 1030.44$ (12) Å^3	0.55 × 0.35 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4149 independent reflections
Radiation source: fine-focus sealed tube	3689 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.061$
ω and φ scans	$\theta_{max} = 26.4°$, $\theta_{min} = 1.8°$
Absorption correction: integration (*SADABS*; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.926$, $T_{max} = 0.946$	$k = -13 \rightarrow 12$
8266 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.099$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 0.3128P]$ where $P = (F_o^2 + 2F_c^2)/3$
4149 reflections	$(\Delta/\sigma)_{max} = 0.001$
261 parameters	$\Delta\rho_{max} = 0.31$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.25$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	1.22156 (4)	0.29397 (4)	1.24108 (3)	0.02234 (11)
O1	0.73558 (18)	0.35378 (12)	0.44984 (10)	0.0440 (3)
O2	0.73876 (11)	0.15824 (11)	0.30066 (8)	0.0224 (2)
O3	0.76675 (13)	−0.07402 (10)	0.37013 (8)	0.0262 (2)
O4	1.16300 (12)	0.44582 (10)	0.89915 (9)	0.0260 (2)
N1	0.87413 (14)	0.11839 (12)	1.02120 (9)	0.0201 (3)
N2	0.94616 (14)	0.15655 (12)	1.12957 (10)	0.0193 (2)
H2N	0.8869 (19)	0.1498 (17)	1.1889 (15)	0.022 (4)*
N3	1.12777 (13)	0.22471 (11)	1.01432 (9)	0.0177 (2)
C1	0.98802 (16)	0.16129 (14)	0.95339 (11)	0.0187 (3)
C2	1.09805 (17)	0.22420 (14)	1.12896 (11)	0.0185 (3)
C3	0.97520 (16)	0.15040 (14)	0.82582 (11)	0.0210 (3)
H3A	1.0517	0.1012	0.7992	0.025*
H3B	1.0058	0.2393	0.7958	0.025*
C4	0.80733 (17)	0.08197 (15)	0.77691 (11)	0.0213 (3)
H4A	0.7863	−0.0128	0.7904	0.026*
H4B	0.7277	0.1174	0.8169	0.026*
C5	0.78916 (16)	0.10143 (14)	0.64979 (11)	0.0198 (3)
C6	0.77190 (19)	0.22128 (15)	0.61242 (12)	0.0268 (3)
H6	0.7712	0.2900	0.6667	0.032*
C7	0.75566 (19)	0.24094 (16)	0.49577 (13)	0.0267 (3)
C8	0.75539 (16)	0.13938 (14)	0.41670 (11)	0.0201 (3)
C9	0.77218 (16)	0.01948 (14)	0.45455 (11)	0.0193 (3)
C10	0.79036 (16)	0.00040 (14)	0.57146 (12)	0.0199 (3)
H10	0.8035	−0.0812	0.5972	0.024*

C11	0.7409 (4)	0.4614 (2)	0.52929 (18)	0.0692 (8)
H11A	0.6571	0.4357	0.5834	0.104*
H11B	0.7236	0.5364	0.4873	0.104*
H11C	0.8457	0.4857	0.5714	0.104*
C12	0.57722 (19)	0.1091 (2)	0.25334 (14)	0.0394 (4)
H12A	0.5360	0.0190	0.2762	0.059*
H12B	0.5757	0.1105	0.1695	0.059*
H12C	0.5100	0.1642	0.2820	0.059*
C13	0.7628 (2)	−0.20286 (16)	0.40713 (14)	0.0354 (4)
H13A	0.8643	−0.2026	0.4488	0.053*
H13B	0.7465	−0.2639	0.3403	0.053*
H13C	0.6748	−0.2303	0.4577	0.053*
C14	1.27708 (16)	0.28093 (14)	0.96606 (11)	0.0185 (3)
C15	1.29400 (17)	0.39505 (14)	0.90645 (11)	0.0207 (3)
C16	1.43893 (18)	0.44820 (15)	0.85927 (12)	0.0258 (3)
H16	1.4522	0.5250	0.8167	0.031*
C17	1.56401 (18)	0.38788 (17)	0.87504 (13)	0.0302 (4)
H17	1.6636	0.4252	0.8440	0.036*
C18	1.54665 (18)	0.27456 (17)	0.93495 (13)	0.0294 (3)
H18	1.6336	0.2347	0.9453	0.035*
C19	1.40069 (17)	0.21988 (15)	0.97969 (12)	0.0237 (3)
H19	1.3861	0.1409	1.0194	0.028*
C20	1.1700 (2)	0.55440 (16)	0.82719 (16)	0.0380 (4)
H20A	1.2536	0.6296	0.8585	0.057*
H20B	1.0666	0.5774	0.8240	0.057*
H20C	1.1947	0.5305	0.7498	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02592 (19)	0.0233 (2)	0.01559 (18)	0.00255 (14)	−0.00210 (13)	0.00064 (13)
O1	0.0882 (10)	0.0284 (7)	0.0253 (6)	0.0323 (7)	0.0050 (6)	0.0065 (5)
O2	0.0211 (5)	0.0317 (6)	0.0138 (5)	0.0047 (4)	0.0009 (4)	0.0054 (4)
O3	0.0389 (6)	0.0217 (6)	0.0171 (5)	0.0057 (5)	0.0037 (4)	−0.0024 (4)
O4	0.0298 (5)	0.0184 (5)	0.0309 (6)	0.0082 (4)	−0.0018 (4)	0.0057 (4)
N1	0.0227 (6)	0.0228 (6)	0.0144 (5)	0.0052 (5)	−0.0009 (4)	0.0003 (5)
N2	0.0222 (6)	0.0225 (6)	0.0127 (5)	0.0041 (5)	0.0015 (5)	0.0018 (5)
N3	0.0208 (6)	0.0175 (6)	0.0142 (5)	0.0037 (5)	0.0006 (4)	0.0023 (4)
C1	0.0221 (7)	0.0161 (7)	0.0176 (7)	0.0044 (5)	−0.0002 (5)	0.0017 (5)
C2	0.0246 (7)	0.0165 (7)	0.0161 (6)	0.0077 (5)	0.0013 (5)	0.0033 (5)
C3	0.0243 (7)	0.0221 (7)	0.0159 (6)	0.0042 (6)	0.0014 (5)	0.0018 (5)
C4	0.0248 (7)	0.0220 (7)	0.0160 (6)	0.0037 (6)	0.0008 (5)	0.0010 (5)
C5	0.0194 (7)	0.0230 (7)	0.0161 (6)	0.0037 (5)	0.0004 (5)	0.0012 (5)
C6	0.0397 (9)	0.0234 (8)	0.0189 (7)	0.0115 (7)	0.0018 (6)	−0.0031 (6)
C7	0.0382 (8)	0.0227 (8)	0.0226 (7)	0.0131 (6)	0.0026 (6)	0.0051 (6)
C8	0.0208 (7)	0.0267 (8)	0.0134 (6)	0.0068 (6)	0.0013 (5)	0.0031 (5)
C9	0.0193 (6)	0.0211 (7)	0.0165 (6)	0.0031 (5)	0.0023 (5)	−0.0021 (5)
C10	0.0225 (7)	0.0186 (7)	0.0183 (7)	0.0044 (5)	0.0008 (5)	0.0023 (5)

C11	0.152 (3)	0.0320 (11)	0.0399 (11)	0.0512 (15)	0.0137 (13)	0.0079 (9)
C12	0.0228 (8)	0.0672 (13)	0.0262 (8)	0.0064 (8)	−0.0028 (6)	0.0151 (8)
C13	0.0555 (11)	0.0198 (8)	0.0284 (8)	0.0029 (7)	0.0116 (7)	−0.0035 (6)
C14	0.0196 (6)	0.0197 (7)	0.0151 (6)	0.0027 (5)	0.0008 (5)	−0.0015 (5)
C15	0.0243 (7)	0.0188 (7)	0.0175 (6)	0.0032 (6)	−0.0008 (5)	−0.0016 (5)
C16	0.0319 (8)	0.0233 (8)	0.0178 (7)	−0.0023 (6)	0.0035 (6)	0.0003 (6)
C17	0.0237 (7)	0.0374 (9)	0.0245 (7)	−0.0022 (6)	0.0072 (6)	−0.0076 (7)
C18	0.0233 (7)	0.0361 (9)	0.0300 (8)	0.0113 (7)	0.0006 (6)	−0.0083 (7)
C19	0.0280 (7)	0.0219 (8)	0.0218 (7)	0.0084 (6)	−0.0006 (6)	−0.0015 (6)
C20	0.0477 (10)	0.0211 (8)	0.0438 (10)	0.0067 (7)	−0.0093 (8)	0.0116 (7)

Geometric parameters (Å, °)

S1—C2	1.6775 (14)	C7—C8	1.393 (2)
O1—C7	1.3650 (18)	C8—C9	1.390 (2)
O1—C11	1.435 (2)	C9—C10	1.3944 (19)
O2—C8	1.3826 (15)	C10—H10	0.9500
O2—C12	1.4397 (18)	C11—H11A	0.9800
O3—C9	1.3647 (17)	C11—H11B	0.9800
O3—C13	1.4309 (19)	C11—H11C	0.9800
O4—C15	1.3642 (17)	C12—H12A	0.9800
O4—C20	1.4324 (18)	C12—H12B	0.9800
N1—C1	1.2987 (19)	C12—H12C	0.9800
N1—N2	1.3861 (15)	C13—H13A	0.9800
N2—C2	1.3326 (19)	C13—H13B	0.9800
N2—H2N	0.878 (17)	C13—H13C	0.9800
N3—C2	1.3778 (17)	C14—C19	1.3792 (19)
N3—C1	1.3789 (18)	C14—C15	1.389 (2)
N3—C14	1.4340 (18)	C15—C16	1.391 (2)
C1—C3	1.4892 (18)	C16—C17	1.388 (2)
C3—C4	1.5266 (19)	C16—H16	0.9500
C3—H3A	0.9900	C17—C18	1.384 (2)
C3—H3B	0.9900	C17—H17	0.9500
C4—C5	1.5122 (18)	C18—C19	1.389 (2)
C4—H4A	0.9900	C18—H18	0.9500
C4—H4B	0.9900	C19—H19	0.9500
C5—C10	1.3859 (19)	C20—H20A	0.9800
C5—C6	1.389 (2)	C20—H20B	0.9800
C6—C7	1.391 (2)	C20—H20C	0.9800
C6—H6	0.9500		
C7—O1—C11	116.44 (13)	C5—C10—H10	120.3
C8—O2—C12	113.01 (11)	C9—C10—H10	120.3
C9—O3—C13	116.17 (11)	O1—C11—H11A	109.5
C15—O4—C20	117.11 (12)	O1—C11—H11B	109.5
C1—N1—N2	103.56 (11)	H11A—C11—H11B	109.5
C2—N2—N1	113.78 (11)	O1—C11—H11C	109.5
C2—N2—H2N	125.4 (11)	H11A—C11—H11C	109.5
N1—N2—H2N	119.5 (11)	H11B—C11—H11C	109.5
C2—N3—C1	107.91 (12)	O2—C12—H12A	109.5

C2—N3—C14	126.41 (11)	O2—C12—H12B	109.5
C1—N3—C14	125.68 (11)	H12A—C12—H12B	109.5
N1—C1—N3	111.33 (12)	O2—C12—H12C	109.5
N1—C1—C3	126.51 (12)	H12A—C12—H12C	109.5
N3—C1—C3	122.13 (12)	H12B—C12—H12C	109.5
N2—C2—N3	103.34 (11)	O3—C13—H13A	109.5
N2—C2—S1	128.07 (11)	O3—C13—H13B	109.5
N3—C2—S1	128.58 (11)	H13A—C13—H13B	109.5
C1—C3—C4	112.98 (12)	O3—C13—H13C	109.5
C1—C3—H3A	109.0	H13A—C13—H13C	109.5
C4—C3—H3A	109.0	H13B—C13—H13C	109.5
C1—C3—H3B	109.0	C19—C14—C15	121.49 (13)
C4—C3—H3B	109.0	C19—C14—N3	119.04 (13)
H3A—C3—H3B	107.8	C15—C14—N3	119.47 (12)
C5—C4—C3	111.07 (12)	O4—C15—C14	115.78 (12)
C5—C4—H4A	109.4	O4—C15—C16	125.22 (13)
C3—C4—H4A	109.4	C14—C15—C16	118.99 (13)
C5—C4—H4B	109.4	C17—C16—C15	119.34 (14)
C3—C4—H4B	109.4	C17—C16—H16	120.3
H4A—C4—H4B	108.0	C15—C16—H16	120.3
C10—C5—C6	120.43 (13)	C18—C17—C16	121.38 (14)
C10—C5—C4	120.11 (13)	C18—C17—H17	119.3
C6—C5—C4	119.45 (12)	C16—C17—H17	119.3
C5—C6—C7	120.17 (13)	C17—C18—C19	119.21 (14)
C5—C6—H6	119.9	C17—C18—H18	120.4
C7—C6—H6	119.9	C19—C18—H18	120.4
O1—C7—C6	125.01 (14)	C14—C19—C18	119.55 (14)
O1—C7—C8	115.33 (13)	C14—C19—H19	120.2
C6—C7—C8	119.65 (14)	C18—C19—H19	120.2
O2—C8—C9	120.15 (12)	O4—C20—H20A	109.5
O2—C8—C7	119.89 (13)	O4—C20—H20B	109.5
C9—C8—C7	119.96 (12)	H20A—C20—H20B	109.5
O3—C9—C8	115.17 (12)	O4—C20—H20C	109.5
O3—C9—C10	124.48 (13)	H20A—C20—H20C	109.5
C8—C9—C10	120.34 (13)	H20B—C20—H20C	109.5
C5—C10—C9	119.45 (13)		
C1—N1—N2—C2	1.85 (15)	C6—C7—C8—C9	−0.4 (2)
N2—N1—C1—N3	−0.12 (15)	C13—O3—C9—C8	−172.13 (13)
N2—N1—C1—C3	−178.15 (13)	C13—O3—C9—C10	6.8 (2)
C2—N3—C1—N1	−1.52 (16)	O2—C8—C9—O3	−1.54 (19)
C14—N3—C1—N1	178.47 (12)	C7—C8—C9—O3	178.61 (13)
C2—N3—C1—C3	176.61 (12)	O2—C8—C9—C10	179.45 (12)
C14—N3—C1—C3	−3.4 (2)	C7—C8—C9—C10	−0.4 (2)
N1—N2—C2—N3	−2.71 (15)	C6—C5—C10—C9	−0.7 (2)
N1—N2—C2—S1	176.30 (10)	C4—C5—C10—C9	179.49 (12)
C1—N3—C2—N2	2.48 (14)	O3—C9—C10—C5	−177.96 (13)
C14—N3—C2—N2	−177.52 (12)	C8—C9—C10—C5	1.0 (2)
C1—N3—C2—S1	−176.53 (11)	C2—N3—C14—C19	72.32 (18)
C14—N3—C2—S1	3.5 (2)	C1—N3—C14—C19	−107.68 (15)

N1—C1—C3—C4	0.6 (2)	C2—N3—C14—C15	−107.91 (15)
N3—C1—C3—C4	−177.23 (12)	C1—N3—C14—C15	72.09 (18)
C1—C3—C4—C5	166.39 (11)	C20—O4—C15—C14	−172.91 (13)
C3—C4—C5—C10	104.08 (15)	C20—O4—C15—C16	6.9 (2)
C3—C4—C5—C6	−75.69 (17)	C19—C14—C15—O4	−179.94 (12)
C10—C5—C6—C7	0.0 (2)	N3—C14—C15—O4	0.29 (18)
C4—C5—C6—C7	179.76 (14)	C19—C14—C15—C16	0.2 (2)
C11—O1—C7—C6	3.6 (3)	N3—C14—C15—C16	−179.52 (12)
C11—O1—C7—C8	−177.92 (19)	O4—C15—C16—C17	178.73 (13)
C5—C6—C7—O1	178.94 (15)	C14—C15—C16—C17	−1.5 (2)
C5—C6—C7—C8	0.6 (2)	C15—C16—C17—C18	1.2 (2)
C12—O2—C8—C9	82.37 (17)	C16—C17—C18—C19	0.3 (2)
C12—O2—C8—C7	−97.78 (17)	C15—C14—C19—C18	1.3 (2)
O1—C7—C8—O2	1.3 (2)	N3—C14—C19—C18	−178.96 (12)
C6—C7—C8—O2	179.79 (13)	C17—C18—C19—C14	−1.5 (2)
O1—C7—C8—C9	−178.88 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···O2^{i}	0.878 (17)	1.890 (18)	2.7558 (15)	168.4 (15)

Symmetry codes: (i) *x*, *y*, *z*+1.

Fig. 1

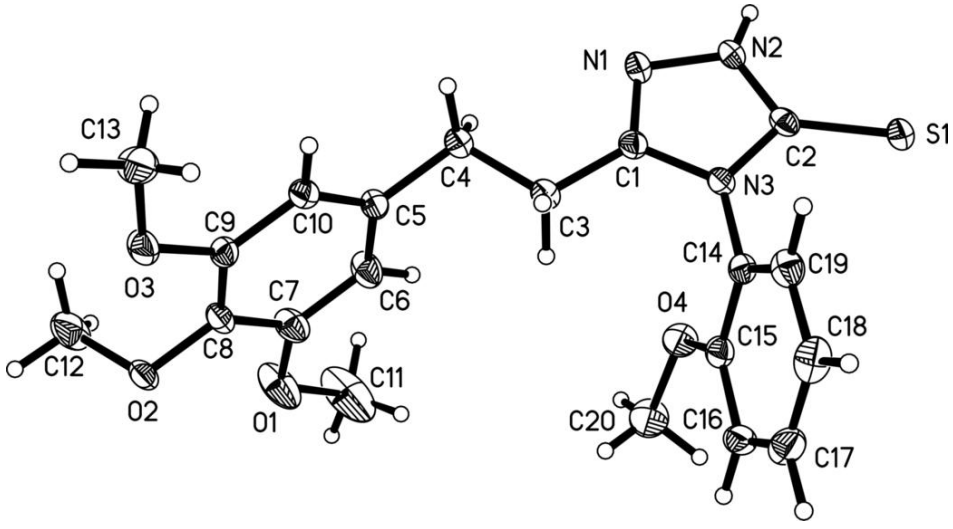
N1
N2
S1
C13
C4
C1
C2
N3
C9
C10
C5
C3
C14
C19
O3
C7
C6
O4
C15
C18
C8
C12
C11
O2
O1
C20
C16
C17

Fig. 2