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5-[3-(2,5-Dimethoxyphenyl)prop-2-enylidene]-1,3-diethyl-2-thioxohexahydropyrimidine-4,6-dione

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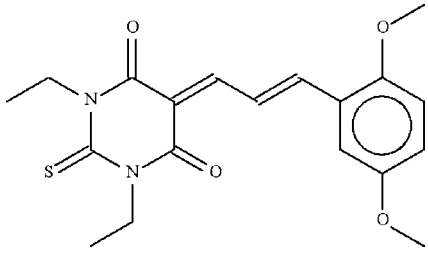
Key indicators: single-crystal X-ray study; T = 140 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.119; data-to-parameter ratio = 17.3.

1,3-Diethyl-2-thiobarbituric acid reacts with 2,5-dimethoxybenzaldehyde to form the title Knoevenagel product, $C_{19}H_{22}N_2O_4S$. In the compound, the two six-membered rings at either end of the three-membered –CHCHCH– chain are nearly coplanar with this fragment (r.m.s. deviation of the two six-membered rings and the three chain atoms = 0.08 Å).

Related literature

For the reaction of 1,3-diethyl-2-thiobarbituric acid with aromatic aldehydes to form the Knoevenagel and Michael products, see: Adamson *et al.* (1999).



Experimental

Crystal data

$C_{19}H_{22}N_2O_4S$
M_r = 374.45
Monoclinic, $P2_1/n$
a = 10.0519 (2) Å
b = 15.5942 (3) Å
c = 11.5920 (2) Å
β = 90.813 (1)°
V = 1816.88 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 140 K
0.35 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.932, T_{max} = 0.970
12384 measured reflections
4124 independent reflections
3351 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.119
S = 1.02
4124 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2548).

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supporting information

Acta Cryst. (2009). E**65**, o1820 [doi:10.1107/S1600536809026099]

5-[3-(2,5-Dimethoxyphenyl)prop-2-enylidene]-1,3-diethyl-2-thioxohexahydro-pyrimidine-4,6-dione

Abdullah Mohamed Asiri, Salman A. Khan and Seik Weng Ng

S1. Experimental

1,3-Diethyl-2-thiobarbituric acid (1 g, 0.005 mol) and 2,5-dimethoxybenzaldehyde (0.83 g, 0.005 mol) were heated in ethanol (15 ml) for 3 h; several drops of pyridine were added. The progress of reaction was monitored by TLC. The solid that seperated from the cool mixture was collected and recrystallized from a methanol-chloroform mixture in 50% yield; m.p. 454 K.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) fixed at 1.2–1.5U_{eq}(C).

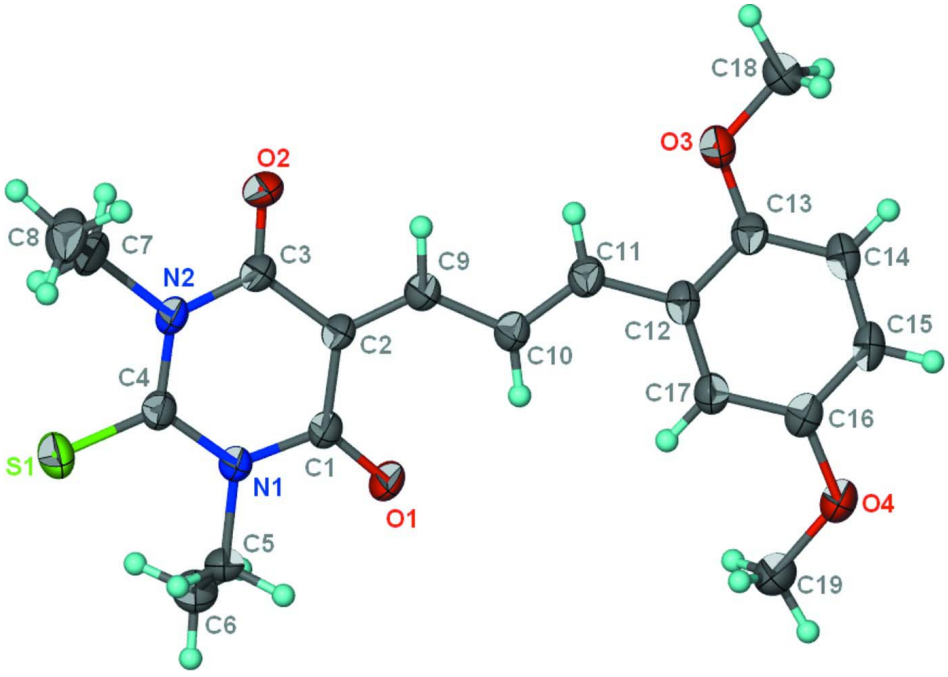

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{19}H_{22}N_2O_4S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

5-[3-(2,5-Dimethoxyphenyl)prop-2-enylidene]-1,3-diethyl-2- thioxohexahydropyrimidine-4,6-dione

Crystal data

$C_{19}H_{22}N_2O_4S$
$M_r = 374.45$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.0519$ (2) Å
$b = 15.5942$ (3) Å
$c = 11.5920$ (2) Å
$\beta = 90.813$ (1)°
$V = 1816.88$ (6) Å^3
$Z = 4$
$F(000) = 792$
$D_x = 1.369$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4985 reflections
$\theta = 2.2$–$28.3°$
$\mu = 0.21$ mm^{-1}
$T = 140$ K
Irregular, gold–green
0.35 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.932$, $T_{max} = 0.970$
12384 measured reflections
4124 independent reflections
3351 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
$h = -12 \rightarrow 13$
$k = -20 \rightarrow 19$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.119$
$S = 1.02$
4124 reflections
239 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.8319P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.45514 (5)	0.17006 (3)	0.48043 (5)	0.04028 (15)
O1	0.65557 (12)	0.44794 (7)	0.58487 (12)	0.0369 (3)
O2	0.88016 (13)	0.19441 (7)	0.68934 (12)	0.0368 (3)
O3	1.30863 (11)	0.51465 (7)	0.76778 (11)	0.0323 (3)
O4	1.02427 (13)	0.79694 (8)	0.61792 (13)	0.0412 (3)
N1	0.57177 (12)	0.31723 (8)	0.53660 (11)	0.0229 (3)
N2	0.67570 (13)	0.19089 (8)	0.60462 (11)	0.0238 (3)
C1	0.67099 (15)	0.37060 (9)	0.58438 (13)	0.0242 (3)
C2	0.78972 (15)	0.32772 (9)	0.63022 (13)	0.0229 (3)
C3	0.78870 (16)	0.23434 (9)	0.64445 (13)	0.0250 (3)
C4	0.57229 (15)	0.22878 (10)	0.54306 (14)	0.0248 (3)
C5	0.45950 (15)	0.36105 (10)	0.47751 (14)	0.0265 (3)
H5A	0.4319	0.3274	0.4088	0.032*

H5B	0.4890	0.4182	0.4508	0.032*
C6	0.34165 (17)	0.37168 (11)	0.55619 (16)	0.0341 (4)
H6A	0.2705	0.4026	0.5152	0.051*
H6B	0.3690	0.4042	0.6249	0.051*
H6C	0.3092	0.3151	0.5794	0.051*
C7	0.67507 (17)	0.09673 (10)	0.62395 (15)	0.0303 (4)
H7A	0.5820	0.0766	0.6301	0.036*
H7B	0.7216	0.0837	0.6977	0.036*
C8	0.7422 (2)	0.04915 (11)	0.52713 (19)	0.0415 (4)
H8A	0.7466	−0.0121	0.5458	0.062*
H8B	0.8325	0.0715	0.5175	0.062*
H8C	0.6911	0.0572	0.4553	0.062*
C9	0.90368 (16)	0.36954 (10)	0.66047 (13)	0.0250 (3)
H9	0.9718	0.3342	0.6923	0.030*
C10	0.93689 (16)	0.45823 (10)	0.65184 (13)	0.0254 (3)
H10	0.8741	0.4981	0.6216	0.031*
C11	1.05873 (16)	0.48485 (10)	0.68724 (14)	0.0258 (3)
H11	1.1170	0.4415	0.7155	0.031*
C12	1.11166 (16)	0.57152 (10)	0.68749 (13)	0.0246 (3)
C13	1.24183 (16)	0.58518 (10)	0.72991 (13)	0.0252 (3)
C14	1.29543 (16)	0.66804 (10)	0.73119 (14)	0.0275 (3)
H14	1.3836	0.6774	0.7589	0.033*
C15	1.21964 (17)	0.73610 (10)	0.69203 (15)	0.0294 (4)
H15	1.2566	0.7922	0.6926	0.035*
C16	1.08972 (17)	0.72399 (10)	0.65159 (14)	0.0288 (3)
C17	1.03685 (16)	0.64216 (10)	0.64830 (14)	0.0262 (3)
H17	0.9490	0.6336	0.6192	0.031*
C18	1.44150 (17)	0.52479 (11)	0.81125 (16)	0.0328 (4)
H18A	1.4763	0.4690	0.8361	0.049*
H18B	1.4415	0.5643	0.8770	0.049*
H18C	1.4978	0.5481	0.7504	0.049*
C19	0.89413 (19)	0.78713 (12)	0.56997 (19)	0.0415 (4)
H19A	0.8576	0.8436	0.5503	0.062*
H19B	0.8368	0.7592	0.6264	0.062*
H19C	0.8983	0.7518	0.5002	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0323 (2)	0.0271 (2)	0.0610 (3)	−0.00414 (17)	−0.0128 (2)	−0.0060 (2)
O1	0.0306 (6)	0.0158 (5)	0.0640 (8)	0.0014 (5)	−0.0106 (6)	0.0012 (5)
O2	0.0364 (7)	0.0202 (5)	0.0534 (8)	0.0041 (5)	−0.0167 (6)	0.0012 (5)
O3	0.0260 (6)	0.0242 (6)	0.0465 (7)	−0.0029 (5)	−0.0060 (5)	−0.0024 (5)
O4	0.0380 (7)	0.0223 (6)	0.0629 (9)	−0.0045 (5)	−0.0123 (6)	0.0066 (6)
N1	0.0214 (6)	0.0185 (6)	0.0287 (6)	−0.0005 (5)	−0.0028 (5)	0.0002 (5)
N2	0.0250 (7)	0.0158 (6)	0.0307 (7)	−0.0010 (5)	−0.0004 (5)	0.0005 (5)
C1	0.0230 (8)	0.0185 (7)	0.0312 (8)	−0.0009 (6)	−0.0002 (6)	0.0016 (6)
C2	0.0249 (8)	0.0167 (7)	0.0270 (7)	0.0005 (6)	−0.0025 (6)	−0.0014 (5)

C3	0.0274 (8)	0.0186 (7)	0.0288 (8)	0.0006 (6)	−0.0017 (6)	−0.0006 (6)
C4	0.0244 (8)	0.0200 (7)	0.0300 (8)	−0.0015 (6)	0.0008 (6)	−0.0011 (6)
C5	0.0242 (8)	0.0241 (7)	0.0309 (8)	0.0020 (6)	−0.0053 (6)	0.0034 (6)
C6	0.0285 (9)	0.0318 (9)	0.0419 (10)	0.0039 (7)	−0.0002 (7)	−0.0008 (7)
C7	0.0322 (9)	0.0160 (7)	0.0427 (9)	−0.0018 (6)	0.0037 (7)	0.0029 (6)
C8	0.0402 (10)	0.0252 (8)	0.0594 (12)	0.0003 (7)	0.0068 (9)	−0.0097 (8)
C9	0.0255 (8)	0.0202 (7)	0.0293 (8)	0.0011 (6)	−0.0020 (6)	−0.0024 (6)
C10	0.0262 (8)	0.0202 (7)	0.0299 (8)	−0.0005 (6)	−0.0010 (6)	−0.0021 (6)
C11	0.0270 (8)	0.0207 (7)	0.0298 (8)	−0.0001 (6)	0.0009 (6)	−0.0033 (6)
C12	0.0260 (8)	0.0220 (7)	0.0260 (7)	−0.0039 (6)	0.0029 (6)	−0.0045 (6)
C13	0.0254 (8)	0.0228 (7)	0.0275 (8)	−0.0009 (6)	0.0030 (6)	−0.0036 (6)
C14	0.0254 (8)	0.0271 (8)	0.0300 (8)	−0.0066 (6)	0.0018 (6)	−0.0054 (6)
C15	0.0313 (9)	0.0226 (7)	0.0345 (8)	−0.0091 (6)	0.0034 (7)	−0.0039 (6)
C16	0.0328 (9)	0.0224 (7)	0.0314 (8)	−0.0021 (6)	0.0013 (7)	0.0000 (6)
C17	0.0251 (8)	0.0230 (7)	0.0305 (8)	−0.0038 (6)	−0.0003 (6)	−0.0008 (6)
C18	0.0264 (8)	0.0320 (9)	0.0398 (9)	−0.0031 (7)	−0.0051 (7)	0.0009 (7)
C19	0.0365 (10)	0.0292 (9)	0.0587 (12)	−0.0017 (7)	−0.0097 (9)	0.0094 (8)

Geometric parameters (Å, º)

S1—C4	1.6516 (16)	C8—H8A	0.9800
O1—C1	1.2160 (19)	C8—H8B	0.9800
O2—C3	1.2207 (19)	C8—H8C	0.9800
O3—C13	1.3582 (19)	C9—C10	1.427 (2)
O3—C18	1.430 (2)	C9—H9	0.9500
O4—C16	1.368 (2)	C10—C11	1.351 (2)
O4—C19	1.422 (2)	C10—H10	0.9500
N1—C4	1.3815 (19)	C11—C12	1.453 (2)
N1—C1	1.4065 (19)	C11—H11	0.9500
N1—C5	1.4791 (19)	C12—C17	1.405 (2)
N2—C4	1.385 (2)	C12—C13	1.407 (2)
N2—C3	1.396 (2)	C13—C14	1.400 (2)
N2—C7	1.4852 (19)	C14—C15	1.379 (2)
C1—C2	1.461 (2)	C14—H14	0.9500
C2—C9	1.360 (2)	C15—C16	1.394 (2)
C2—C3	1.465 (2)	C15—H15	0.9500
C5—C6	1.514 (2)	C16—C17	1.383 (2)
C5—H5A	0.9900	C17—H17	0.9500
C5—H5B	0.9900	C18—H18A	0.9800
C6—H6A	0.9800	C18—H18B	0.9800
C6—H6B	0.9800	C18—H18C	0.9800
C6—H6C	0.9800	C19—H19A	0.9800
C7—C8	1.512 (2)	C19—H19B	0.9800
C7—H7A	0.9900	C19—H19C	0.9800
C7—H7B	0.9900		
C13—O3—C18	118.65 (12)	H8A—C8—H8C	109.5
C16—O4—C19	117.25 (14)	H8B—C8—H8C	109.5

C4—N1—C1	124.56 (13)	C2—C9—C10	130.09 (15)
C4—N1—C5	119.26 (13)	C2—C9—H9	115.0
C1—N1—C5	116.18 (12)	C10—C9—H9	115.0
C4—N2—C3	124.38 (13)	C11—C10—C9	119.24 (15)
C4—N2—C7	119.65 (13)	C11—C10—H10	120.4
C3—N2—C7	115.81 (13)	C9—C10—H10	120.4
O1—C1—N1	119.92 (14)	C10—C11—C12	128.08 (15)
O1—C1—C2	123.75 (14)	C10—C11—H11	116.0
N1—C1—C2	116.32 (13)	C12—C11—H11	116.0
C9—C2—C1	123.71 (14)	C17—C12—C13	119.00 (14)
C9—C2—C3	117.04 (14)	C17—C12—C11	122.30 (15)
C1—C2—C3	119.24 (13)	C13—C12—C11	118.70 (14)
O2—C3—N2	119.86 (14)	O3—C13—C14	123.75 (15)
O2—C3—C2	123.26 (14)	O3—C13—C12	116.31 (13)
N2—C3—C2	116.89 (13)	C14—C13—C12	119.94 (15)
N1—C4—N2	117.14 (13)	C15—C14—C13	119.73 (15)
N1—C4—S1	121.84 (12)	C15—C14—H14	120.1
N2—C4—S1	121.01 (11)	C13—C14—H14	120.1
N1—C5—C6	111.71 (13)	C14—C15—C16	121.12 (14)
N1—C5—H5A	109.3	C14—C15—H15	119.4
C6—C5—H5A	109.3	C16—C15—H15	119.4
N1—C5—H5B	109.3	O4—C16—C17	125.20 (16)
C6—C5—H5B	109.3	O4—C16—C15	115.32 (14)
H5A—C5—H5B	107.9	C17—C16—C15	119.48 (15)
C5—C6—H6A	109.5	C16—C17—C12	120.71 (15)
C5—C6—H6B	109.5	C16—C17—H17	119.6
H6A—C6—H6B	109.5	C12—C17—H17	119.6
C5—C6—H6C	109.5	O3—C18—H18A	109.5
H6A—C6—H6C	109.5	O3—C18—H18B	109.5
H6B—C6—H6C	109.5	H18A—C18—H18B	109.5
N2—C7—C8	111.72 (14)	O3—C18—H18C	109.5
N2—C7—H7A	109.3	H18A—C18—H18C	109.5
C8—C7—H7A	109.3	H18B—C18—H18C	109.5
N2—C7—H7B	109.3	O4—C19—H19A	109.5
C8—C7—H7B	109.3	O4—C19—H19B	109.5
H7A—C7—H7B	107.9	H19A—C19—H19B	109.5
C7—C8—H8A	109.5	O4—C19—H19C	109.5
C7—C8—H8B	109.5	H19A—C19—H19C	109.5
H8A—C8—H8B	109.5	H19B—C19—H19C	109.5
C7—C8—H8C	109.5		

C4—N1—C1—O1	−172.42 (15)	C4—N2—C7—C8	88.82 (18)
C5—N1—C1—O1	6.6 (2)	C3—N2—C7—C8	−86.77 (18)
C4—N1—C1—C2	8.2 (2)	C1—C2—C9—C10	−2.7 (3)
C5—N1—C1—C2	−172.73 (13)	C3—C2—C9—C10	176.85 (15)
O1—C1—C2—C9	−11.3 (3)	C2—C9—C10—C11	−179.44 (16)
N1—C1—C2—C9	167.98 (14)	C9—C10—C11—C12	−179.07 (15)
O1—C1—C2—C3	169.12 (15)	C10—C11—C12—C17	0.0 (3)

N1—C1—C2—C3	−11.6 (2)	C10—C11—C12—C13	179.19 (15)
C4—N2—C3—O2	−173.69 (15)	C18—O3—C13—C14	−0.2 (2)
C7—N2—C3—O2	1.7 (2)	C18—O3—C13—C12	179.70 (14)
C4—N2—C3—C2	6.8 (2)	C17—C12—C13—O3	179.26 (13)
C7—N2—C3—C2	−177.84 (13)	C11—C12—C13—O3	0.1 (2)
C9—C2—C3—O2	5.6 (2)	C17—C12—C13—C14	−0.8 (2)
C1—C2—C3—O2	−174.83 (15)	C11—C12—C13—C14	−179.99 (14)
C9—C2—C3—N2	−174.93 (14)	O3—C13—C14—C15	−179.33 (14)
C1—C2—C3—N2	4.7 (2)	C12—C13—C14—C15	0.7 (2)
C1—N1—C4—N2	2.4 (2)	C13—C14—C15—C16	0.4 (2)
C5—N1—C4—N2	−176.56 (13)	C19—O4—C16—C17	−3.6 (3)
C1—N1—C4—S1	−177.59 (12)	C19—O4—C16—C15	176.54 (16)
C5—N1—C4—S1	3.42 (19)	C14—C15—C16—O4	178.46 (15)
C3—N2—C4—N1	−10.5 (2)	C14—C15—C16—C17	−1.4 (2)
C7—N2—C4—N1	174.31 (13)	O4—C16—C17—C12	−178.52 (15)
C3—N2—C4—S1	169.52 (12)	C15—C16—C17—C12	1.3 (2)
C7—N2—C4—S1	−5.67 (19)	C13—C12—C17—C16	−0.2 (2)
C4—N1—C5—C6	82.80 (17)	C11—C12—C17—C16	178.91 (15)
C1—N1—C5—C6	−96.28 (16)		