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3-(Pyridin-4-ylthio)pentane-2,4-dione

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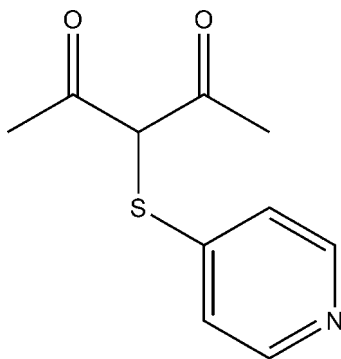
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.038; wR factor = 0.120; data-to-parameter ratio = 14.0.

In the title compound, $\text{C}_{10}\text{H}_{11}\text{NO}_2\text{S}$, the acetylacetone group crystallizes in the keto form with all the non-hydrogen atoms in the acetylacetone group approximately co-planar with a maximum atomic deviation 0.055 (2) Å; the dihedral angle between the acetylacetone group and the pyridine ring is 85.90 (6)°. An intramolecular O—H...O hydrogen bond involving the acetylacetone group forms a six-membered ring.

Related literature

For applications of β -diketones and their derivatives in metallo-supramolecular chemistry, see: Aromí *et al.* (2008); Chen *et al.* (2003; 2004); Domasevitch *et al.* (2006); Massue *et al.* (2005); Soldatov & Ripmeester (2001); Tabellion *et al.* (2001); Vigato *et al.* (2009); Vreshch *et al.* (2003, 2004); Won *et al.* (2007); Zhang *et al.* (2006).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{11}\text{NO}_2\text{S}$
 $M_r = 209.26$
 Monoclinic, $P2_1/c$
 $a = 8.3273$ (7) Å
 $b = 9.5614$ (8) Å
 $c = 13.0681$ (11) Å
 $\beta = 92.698$ (1)°

$V = 1039.34$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.28$ mm⁻¹
 $T = 298$ K
 $0.35 \times 0.30 \times 0.28$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.907$, $T_{\max} = 0.925$

5011 measured reflections
 1822 independent reflections
 1351 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.120$
 $S = 1.06$
 1822 reflections

130 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1...O2	0.88	1.58	2.427 (3)	161

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); 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: ZJ2007).

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supplementary materials

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3-(Pyridin-4-ylthio)pentane-2,4-dione

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Comment

The β -diketones and their derivatives have attracted much attention in recent years, not only for their valuable intrinsic chemical and physical properties but also for their wide applications in metallo-supramolecular chemistry (Aromí & Gamez *et al.*, 2008; Massue & Bellec *et al.*, 2005; Soldatov & Ripmeester, 2001; Tabellion & Seidel *et al.*, 2001; Vigato & Peruzzo *et al.*, 2009; Chen & Fronczek *et al.*, 2003; Chen & Fronczek *et al.*, 2004; Domasevitch & Vreshch, *et al.*, 2006; Vreshch & Chernega *et al.* 2003; Vreshch & Lysenko *et al.* 2004; Won & Clegg *et al.*, 2007; Zhang & Chen *et al.*, 2006). Among them, the N-containing bifunctional derivatives of β -diketones have been successfully applied to construct various metal-organic supramolecular complexes (Chen & Fronczek *et al.*, 2003; Chen & Fronczek *et al.*, 2004; Domasevitch & Vreshch, *et al.*, 2006; Vreshch & Chernega *et al.* 2003; Vreshch & Lysenko *et al.* 2004; Won & Clegg *et al.*, 2007; Zhang & Chen *et al.*, 2006). We report here on the structure of an interesting nonlinear N-containing bifunctional compound, 3-(pyridin-4-ylthio)pentane-2,4-dione, which is prepared according to the previously reported method (Won & Clegg *et al.*, 2007).

As shown in Fig. 1, the acetylacetonone group is with keto-enol tautomerism, where all the non-hydrogen atoms in the acetylacetonone group are approximately co-planar with a maximum atomic deviation 0.0547 (24) Å, and the dihedral angle between the acetylacetonone group and the pyridine ring is about of 85.90 (6)°. The bond angle of C3—S1—C6 is about of 105.16 (10)°, leading to a V-shaped conformation of the whole molecule. In the crystal structure, the intramolecular O—H...O hydrogen bonding interactions have been found in the same acetylacetonone group, forming a six-membered cyclic structure. However, no intermolecular H-bonding interactions have been found in the title compound (Fig. 2).

Experimental

The title compound, 3-(pyridin-4-ylthio)pentane-2,4-dione, was prepared according to the previously reported method (Won & Clegg *et al.*, 2007). The yellow crystals of title compound suitable for X-ray crystallographic analysis were obtained by recrystallization from acetonitrile.

Refinement

All H atoms on C atoms were positioned geometrically and refined as riding atoms with $d(\text{C—H}) = 0.93\text{--}0.97$ Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atom on O atom of enol group was located from difference Fourier map and refined with $d(\text{O—H}) = 0.881$ Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

Figures

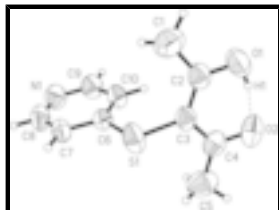


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

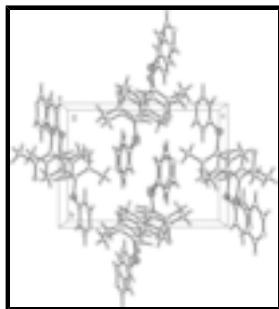


Fig. 2. The packing diagram of the title compound.

3-(Pyridin-4-ylthio)pentane-2,4-dione

Crystal data

$C_{10}H_{11}NO_2S$

$M_r = 209.26$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 8.3273\ (7)\ \text{\AA}$

$b = 9.5614\ (8)\ \text{\AA}$

$c = 13.0681\ (11)\ \text{\AA}$

$\beta = 92.698\ (1)^\circ$

$V = 1039.34\ (15)\ \text{\AA}^3$

$Z = 4$

$F(000) = 440$

$D_x = 1.337\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2174 reflections

$\theta = 2.5\text{--}27.3^\circ$

$\mu = 0.28\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, yellow

$0.35 \times 0.30 \times 0.28\ \text{mm}$

Data collection

Siemens SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

phi and ω scans

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

$T_{\min} = 0.907$, $T_{\max} = 0.925$

5011 measured reflections

1822 independent reflections

1351 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -9 \rightarrow 9$

$k = -5 \rightarrow 11$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.120$	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.3241P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
1822 reflections	$(\Delta/\sigma)_{\max} < 0.001$
130 parameters	$\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL</i> , $F_c^* = kFc[1 + 0.001 \times Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.136 (9)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.87871 (8)	0.22005 (6)	0.95516 (6)	0.0688 (3)
O1	0.7336 (2)	0.60208 (19)	1.01493 (15)	0.0761 (6)
H1	0.7715	0.6213	0.9547	0.091*
O2	0.8608 (2)	0.60632 (19)	0.85162 (14)	0.0769 (6)
N1	0.4443 (3)	-0.0271 (2)	0.83293 (19)	0.0714 (6)
C1	0.6930 (4)	0.3985 (4)	1.1103 (2)	0.0863 (9)
H1A	0.7813	0.3688	1.1550	0.129*
H1B	0.6305	0.3185	1.0886	0.129*
H1C	0.6266	0.4623	1.1461	0.129*
C2	0.7557 (3)	0.4691 (3)	1.01961 (19)	0.0566 (6)
C3	0.8327 (3)	0.3980 (2)	0.94164 (17)	0.0495 (6)
C4	0.8804 (3)	0.4736 (3)	0.85562 (18)	0.0580 (6)
C5	0.9512 (4)	0.4065 (4)	0.7658 (2)	0.0890 (10)
H5A	1.0051	0.4759	0.7269	0.133*
H5B	0.8674	0.3639	0.7235	0.133*
H5C	1.0270	0.3363	0.7889	0.133*

supplementary materials

C6	0.7057 (3)	0.1310 (2)	0.90682 (16)	0.0466 (5)
C7	0.7102 (3)	-0.0134 (2)	0.90713 (19)	0.0584 (6)
H7	0.8011	-0.0607	0.9327	0.070*
C8	0.5786 (3)	-0.0856 (3)	0.8692 (2)	0.0730 (8)
H8	0.5843	-0.1827	0.8690	0.088*
C9	0.4412 (3)	0.1119 (3)	0.8347 (2)	0.0630 (7)
H9	0.3474	0.1561	0.8107	0.076*
C10	0.5674 (3)	0.1949 (2)	0.86962 (18)	0.0542 (6)
H10	0.5591	0.2919	0.8680	0.065*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0565 (4)	0.0432 (4)	0.1043 (6)	0.0034 (3)	-0.0216 (3)	-0.0036 (3)
O1	0.0818 (13)	0.0532 (11)	0.0934 (14)	0.0072 (9)	0.0045 (10)	-0.0142 (9)
O2	0.0901 (14)	0.0563 (11)	0.0827 (13)	-0.0179 (10)	-0.0131 (10)	0.0166 (9)
N1	0.0674 (14)	0.0546 (13)	0.0923 (17)	-0.0155 (11)	0.0029 (12)	-0.0121 (11)
C1	0.090 (2)	0.095 (2)	0.0750 (19)	-0.0214 (18)	0.0168 (15)	-0.0053 (16)
C2	0.0498 (13)	0.0525 (14)	0.0668 (15)	-0.0059 (10)	-0.0058 (11)	-0.0064 (11)
C3	0.0459 (12)	0.0407 (12)	0.0610 (14)	-0.0075 (9)	-0.0053 (10)	-0.0053 (10)
C4	0.0484 (13)	0.0634 (16)	0.0614 (15)	-0.0137 (11)	-0.0080 (11)	-0.0028 (12)
C5	0.080 (2)	0.116 (3)	0.0717 (19)	-0.0176 (18)	0.0129 (15)	-0.0173 (17)
C6	0.0502 (13)	0.0403 (12)	0.0495 (12)	-0.0014 (9)	0.0039 (9)	-0.0023 (9)
C7	0.0605 (15)	0.0398 (12)	0.0751 (16)	0.0029 (10)	0.0060 (12)	0.0046 (11)
C8	0.076 (2)	0.0402 (13)	0.103 (2)	-0.0097 (13)	0.0085 (16)	-0.0015 (13)
C9	0.0570 (14)	0.0594 (16)	0.0720 (16)	-0.0022 (12)	-0.0043 (12)	-0.0048 (12)
C10	0.0577 (14)	0.0413 (12)	0.0630 (14)	-0.0010 (10)	-0.0044 (11)	-0.0041 (10)

Geometric parameters (\AA , $^\circ$)

S1—C3	1.752 (2)	C4—C5	1.484 (4)
S1—C6	1.764 (2)	C5—H5A	0.9600
O1—C2	1.286 (3)	C5—H5B	0.9600
O1—H1	0.8814	C5—H5C	0.9600
O2—C4	1.280 (3)	C6—C10	1.373 (3)
N1—C8	1.318 (3)	C6—C7	1.381 (3)
N1—C9	1.330 (3)	C7—C8	1.368 (4)
C1—C2	1.480 (4)	C7—H7	0.9300
C1—H1A	0.9600	C8—H8	0.9300
C1—H1B	0.9600	C9—C10	1.378 (3)
C1—H1C	0.9600	C9—H9	0.9300
C2—C3	1.404 (3)	C10—H10	0.9300
C3—C4	1.409 (3)		
C3—S1—C6	105.16 (10)	H5A—C5—H5B	109.5
C2—O1—H1	101.1	C4—C5—H5C	109.5
C8—N1—C9	115.8 (2)	H5A—C5—H5C	109.5
C2—C1—H1A	109.5	H5B—C5—H5C	109.5
C2—C1—H1B	109.5	C10—C6—C7	117.9 (2)

H1A—C1—H1B	109.5	C10—C6—S1	124.71 (17)
C2—C1—H1C	109.5	C7—C6—S1	117.38 (17)
H1A—C1—H1C	109.5	C8—C7—C6	118.8 (2)
H1B—C1—H1C	109.5	C8—C7—H7	120.6
O1—C2—C3	120.9 (2)	C6—C7—H7	120.6
O1—C2—C1	115.7 (2)	N1—C8—C7	124.6 (2)
C3—C2—C1	123.4 (2)	N1—C8—H8	117.7
C2—C3—C4	119.1 (2)	C7—C8—H8	117.7
C2—C3—S1	120.14 (18)	N1—C9—C10	124.5 (2)
C4—C3—S1	120.67 (18)	N1—C9—H9	117.7
O2—C4—C3	120.0 (2)	C10—C9—H9	117.7
O2—C4—C5	116.8 (2)	C6—C10—C9	118.4 (2)
C3—C4—C5	123.1 (3)	C6—C10—H10	120.8
C4—C5—H5A	109.5	C9—C10—H10	120.8
C4—C5—H5B	109.5		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H1...O2	0.88	1.58	2.427 (3)	161

Fig. 1

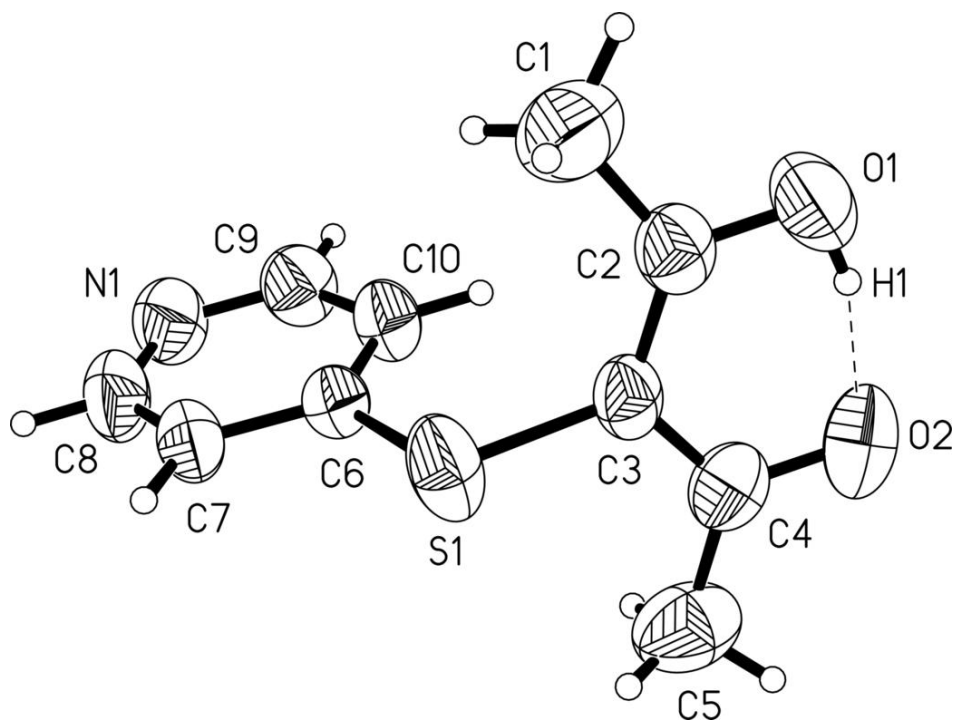


Fig. 2

