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Crystal structure of (*E*)-4-hydroxy-3-((5-phenyl-1,3,4-oxadiazol-2-yl)thio)pent-3-en-2-one, $C_{13}H_{12}N_2O_3S$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.24 × 0.23 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.25 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	12274, 2521, 0.044
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1887
$N(\text{param})_{\text{refined}}$:	175
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

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Abstract

$C_{13}H_{12}N_2O_3S$, monoclinic, $P2_1/c$ (no. 14), $a = 12.3541(8)$ Å, $b = 14.3818(10)$ Å, $c = 7.3757(5)$ Å, $\beta = 96.471(2)^\circ$, $V = 1302.12(15)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0417$, $wR_{\text{ref}}(F^2) = 0.1080$, $T = 293(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Potassium 5-phenyl-1,3,4-oxadiazole-2-thiolate (2.16 g, 10 mmol, 1 eq) was dissolved in 50 mL of MeOH. To the solution at room temperature was added 3-bromoacetylacetone

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3918(2)	−0.08509(19)	1.0954(3)	0.0704(7)
H1A	0.3697	−0.0217	1.1084	0.106*
H1B	0.4684	−0.0907	1.1341	0.106*
H1C	0.3519	−0.1245	1.1694	0.106*
C2	0.36925(18)	−0.11378(14)	0.8997(3)	0.0491(5)
C3	0.26526(16)	−0.11992(13)	0.8045(3)	0.0412(5)
C4	0.25264(18)	−0.14976(13)	0.6204(3)	0.0464(5)
C6	0.14369(18)	−0.16024(16)	0.5128(3)	0.0591(6)
H6A	0.1047	−0.2101	0.5628	0.089*
H6B	0.1529	−0.1740	0.3881	0.089*
H6C	0.1034	−0.1035	0.5183	0.089*
C7	0.12568(15)	0.01862(14)	0.8714(3)	0.0428(5)
C8	0.15430(15)	0.16475(13)	0.8755(2)	0.0393(4)
C9	0.21460(15)	0.25120(14)	0.9068(2)	0.0402(5)
C10	0.32163(17)	0.25073(15)	0.9884(3)	0.0473(5)
H10	0.3558	0.1949	1.0233	0.057*
C11	0.37697(19)	0.33394(17)	1.0175(3)	0.0581(6)
H11	0.4486	0.3338	1.0721	0.070*
C13	0.3270(2)	0.41696(17)	0.9663(3)	0.0666(7)
H13	0.3648	0.4726	0.9867	0.080*
C15	0.16440(19)	0.33558(15)	0.8552(3)	0.0522(5)
H15	0.0928	0.3363	0.8007	0.063*
C14	0.2205(2)	0.41743(16)	0.8847(3)	0.0647(7)
H14	0.1869	0.4735	0.8496	0.078*
N1	0.05322(14)	0.15344(12)	0.8140(3)	0.0522(5)
N2	0.03459(14)	0.05654(13)	0.8093(3)	0.0540(5)
O1	0.45440(12)	−0.13309(13)	0.8209(2)	0.0667(5)
H1	0.4357	−0.1517	0.7170	0.100*
O2	0.33617(13)	−0.16925(11)	0.5404(2)	0.0612(4)
O3	0.20634(10)	0.08245(9)	0.91541(18)	0.0424(3)
S1	0.14909(4)	−0.09857(4)	0.91795(8)	0.0510(2)

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(1.78 g, 10 mmol, 1 eq) slowly in several portions. The reaction mixture was stirred for 4 h. After filtration 2.2 g of a white powder was collected in 80% yield, which was then recrystallized in ethanol to get square block crystals of the title compound.

Experimental details

Crystal data, data collection and structure refinement details are summarized in Table 1. Using Olex2 [1], the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, with the $d(\text{C}-\text{H}) = 0.97\text{--}0.99 \text{ \AA}$, $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H}) = 1.5$ times $U_{\text{eq}}(\text{O})$.

Comment

Oxazoles are an important class of compounds. Especially 1,3,4-oxazoles are of interest [5]. The β -diketonate ligands having played a significant role in coordination chemistry for over a century [6].

The title compound is constructed from the 5-phenyl-1,3,4-oxadiazole-2-thiol and the β -diketone methylene moiety (see the figure). The bond angle of C3—S1—C7 is 101.6°, the dihedral angle of phenyl ring and 1,3,4-oxadiazole ring is 175.34°. The formal β -diketone forms enol tautomeric structure, and the stable six membered ring structure is formed between O1 and O2 of β -diketone through O1—H1...O2

intramolecular hydrogen bond. All geometric parameters are in the expected ranges [7].

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References

1. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
2. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
5. Aksenov, A. V.; Khamraev, V.; Aksenov, N. A.; Kirilov, N. K.; Domenyuk, D. A.; Zelensky, V. A.; Rubin, M.: Electrophilic activation of nitroalkanes in efficient synthesis of 1,3,4-oxadiazoles. *RSC Adv.* **9** (2019) 6636–6642.
6. Li, B.; Li, H. F.; Chen, P.; Sun, W. B.; Wang, C.; Gao, T.; Yan, P. F.: Enhancement of near-infrared luminescence of ytterbium in triple-stranded binuclear helicates. *Phys. Chem. Chem. Phys.* **17** (2015) 30510–30517.
7. Wu, Q.; Zhang, H.-Q.; Ramanaskanda, B.; Chai, K.-Y.: Crystal structure of 2,5-bis(4-(10*H*-phenothiazin-10-yl)phenyl)-1,3,4-oxadiazole, C₃₈H₂₄N₄O₅S₂. *Z. Kristallogr. NCS* **233** (2018) 869–871.