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Crystal structure of (3*E*,3'*E*)-3,3'-((1,3,4-thiadiazole-2,5-diyl)bis(sulfanediyl))bis(4-hydroxy-4-phenylbut-3-en-2-one), C₂₂H₁₈N₂O₄S₃

Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.22 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.37 mm ⁻¹
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	16265, 3854, 0.037
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3049
$N(\text{param})_{\text{refined}}$:	290
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Source of material

To the solution of dipotassium 1,3,4-thiadiazole-2,5-dithiolate (2.26 g, 10 mmol) in 50 mL MeOH was added 2-bromo-1-phenylbutane-1,3-dione (4.82 g 20 mmol). The reaction mixture was stirred for 6 h and then filtered. 4.2 g white powder was collected in 90% yield, which was recrystallized in CH₂Cl₂ to get square crystals of the title compound.

Experimental details

The data were scaled and corrected for absorption using SADABS-2016/2 [1–3]. The hydrogen atoms were placed at calculated positions as riding atoms.

Comment

As an important heterocyclic unit, 1,3,4-thiadiazoles were widely used, because these compounds may demonstrated high neuroprotective [4], antifungal [5], antibacterial [6], anti-inflammatory [7], antioxidative [8] and anti-cancer activity [9]. Thus, design and synthesis of new 1,3,4-thiadiazoles have attracted attention in recent years. In this paper, a new b-diketone-based was synthesized by nucleophilic substitution reaction using 2-bromo-1-phenylbutane-1,3-dione as starting material. Such compounds can be used to construct dimetallic complexes [10].

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Abstract

C₂₂H₁₈N₂O₄S₃, monoclinic, $P2_1/c$ (no. 14), $a = 9.632(2)$ Å, $b = 10.000(3)$ Å, $c = 22.760(6)$ Å, $\beta = 93.196(18)^\circ$, $V = 2188.9(10)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0354$, $wR_{\text{ref}}(F^2) = 0.0933$, $T = 293(2)$ K.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
S1	0.00498(5)	−0.30905(5)	0.44517(3)	0.05110(17)
S2	0.19922(5)	−0.07174(5)	0.42572(2)	0.04823(16)
S3	0.19979(5)	0.23198(5)	0.40573(3)	0.05311(17)
O1	0.36111(16)	−0.45450(16)	0.50993(7)	0.0654(4)
H1	0.389(3)	−0.468(3)	0.4567(6)	0.136(12)*
O2	0.36773(15)	−0.45614(16)	0.40259(7)	0.0635(4)
O3	0.57272(16)	0.13017(18)	0.46963(7)	0.0691(5)
H3	0.599(3)	0.111(4)	0.4170(6)	0.148(13)*
O4	0.57157(16)	0.10860(19)	0.36326(7)	0.0745(5)
N1	−0.06491(16)	−0.05440(17)	0.42954(8)	0.0495(4)
N2	−0.01846(17)	0.07460(17)	0.42007(8)	0.0505(4)
C1	0.1768(3)	−0.3690(2)	0.56193(10)	0.0664(6)
H1A	0.1678	−0.2737	0.5651	0.100*
H1B	0.2348	−0.4018	0.5944	0.100*
H1C	0.0866	−0.4096	0.5625	0.100*
C2	0.2404(2)	−0.4027(2)	0.50595(10)	0.0520(5)
C3	0.17387(19)	−0.37773(19)	0.44938(9)	0.0447(5)
C4	0.2428(2)	−0.4071(2)	0.39863(9)	0.0492(5)
C5	0.1879(2)	−0.3796(2)	0.33782(9)	0.0535(5)
C6	0.2694(3)	−0.3046(3)	0.30183(11)	0.0676(7)
H6	0.3555	−0.2732	0.3164	0.081*
C7	0.2244(3)	−0.2765(3)	0.24494(12)	0.0892(9)
H7	0.2790	−0.2244	0.2215	0.107*
C8	0.1000(4)	−0.3244(4)	0.22274(13)	0.0962(10)
H8	0.0695	−0.3042	0.1843	0.115*
C9	0.0197(3)	−0.4022(4)	0.25683(14)	0.0991(11)
H9	−0.0637	−0.4371	0.2410	0.119*
C10	0.0620(3)	−0.4293(3)	0.31499(12)	0.0764(8)
H10	0.0062	−0.4804	0.3384	0.092*
C11	0.03663(18)	−0.1393(2)	0.43351(8)	0.0399(4)
C12	0.11505(19)	0.07977(19)	0.41703(8)	0.0404(4)
C13	0.3834(3)	0.2025(3)	0.52202(10)	0.0666(6)
H13A	0.3490	0.2926	0.5205	0.100*
H13B	0.4516	0.1937	0.5542	0.100*
H13C	0.3078	0.1420	0.5276	0.100*
C14	0.4478(2)	0.1702(2)	0.46604(10)	0.0526(5)
C15	0.3757(2)	0.1820(2)	0.40989(9)	0.0458(5)
C16	0.4453(2)	0.1545(2)	0.35902(9)	0.0528(5)
C17	0.3889(2)	0.1740(2)	0.29767(9)	0.0537(5)
C18	0.4089(3)	0.0742(3)	0.25706(12)	0.0757(8)
H18	0.4517	−0.0055	0.2688	0.091*
C19	0.3645(3)	0.0936(4)	0.19833(14)	0.0992(11)
H19	0.3764	0.0262	0.1709	0.119*
C20	0.3035(3)	0.2120(5)	0.18101(14)	0.1024(12)
H20	0.2733	0.2246	0.1419	0.123*
C21	0.2870(3)	0.3111(4)	0.22091(14)	0.0934(10)
H21	0.2473	0.3920	0.2087	0.112*
C22	0.3283(3)	0.2931(3)	0.27897(11)	0.0700(7)
H22	0.3154	0.3612	0.3059	0.084*

In the crystal structure of the title compound, the bond length of C15—C16 1.397(3) Å is a little bit shorter than C16—C17 1.483(4) Å. The same situation is seen in the other

leg of the molecule (C3—C4 = 1.395(3); C4—C5 = 1.480(3) Å). Hydrogen bonds were observed in molecular structure as following: O2—H2···O1 (*d*(H2···O2) = 2.451 Å, C4—H4···O3 (*d*(H4···O3) = 2.432 Å).

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References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
2. Sheldrick, G. M.: Crystal structure refinement with ShelXL. *Acta Crystallogr. C* **27** (2015) 3–8.
3. 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.
4. Wang, C.; Han, Y.; Wu, A.; Solyom, S.; Niu, L.: Mechanism and site of inhibition of AMPA receptors: pairing a thiadiazole with a 2,3-benzodiazepine scaffold. *ACS Chem. Neurosci.* **5** (2014) 138–147.
5. Li, Q.; Ren, J.; Dong, F.; Feng, Y.; Gu, G.; Guo, Z.: Synthesis and antifungal activity of thiadiazole-functionalized chitosan derivatives. *Carbohydr. Res.* **373** (2013) 103–107.
6. Rezki, N.; Al-Yahyawi, A.; Bardaweel, S.; Al-Blewi, F.; Aouad, M.: Synthesis of novel 2,5-disubstituted-1,3,4-thiadiazoles clubbed 1,2,4-triazole, 1,3,4-thiadiazole, 1,3,4-oxadiazole and/or Schiff base as potential antimicrobial and antiproliferative agents. *Molecules* **20** (2015) 16048–16067.
7. Salgın-Gökşen, U.; Gökhan-Kelekçi, N.; Göktaş, Ö.; Köysal, Y.; Kılıç, E.; Işık, Ş.; Aktay, G.; Özalp, M.: 1-Acylthiosemicarbazides, 1,2,4-triazole-5(4*H*)-thiones, 1,3,4-thiadiazoles and hydrazones containing 5-methyl-2-benzoxazolinones: synthesis, analgesic-anti-inflammatory and antimicrobial activities. *Bioorg. Med. Chem.* **15** (2007) 5738–5751.
8. Cressier, D.; Prouillac, C.; Hernandez, P.; Amourette, C.; Diserbo, M.; Lion, C.; Rima, G.: Synthesis, antioxidant properties and radioprotective effects of new benzothiadiazoles and thiadiazoles. *Bioorg. Med. Chem.* **17** (2009) 5275–5284.
9. Dawood, K. M.; Gomha, S. M.: Synthesis and anti-cancer activity of 1,3,4-thiadiazole and 1,3-thiazole derivatives having 1,3,4-oxadiazole moiety. *J. Heterocycl. Chem.* **52** (2015) 1400–1405.
10. Zhang, X.; Chen, H.; Ma, C.; Chen, C.; Liu, Q.: Syntheses and structural characterizations of 24-membered dimetal (Mn, Ni, Fe) macrocyclic complexes and the C–S bond formation between acetylacetone and a mercapto *N*-heterocycle. *Dalton Trans.* (2006) 4047–4055.