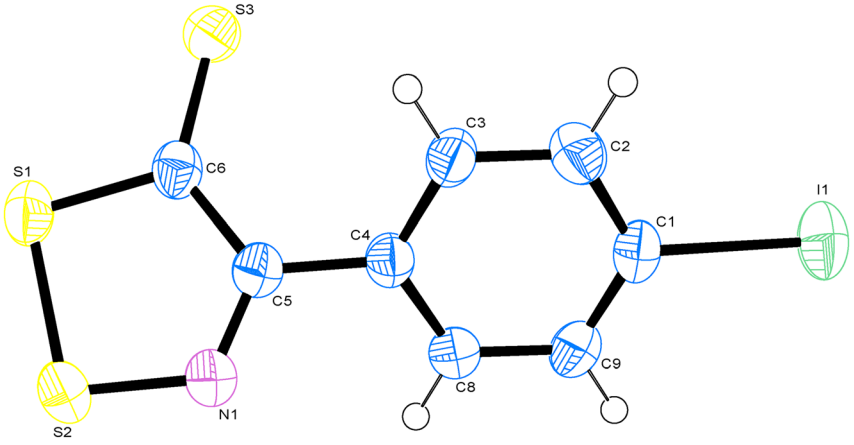
Huiyang Liao and Penghui Ni*

The crystal structure of 4-(4-iodophenyl)-5*H*-1,2,3-dithiazole-5-thione, $C_8H_4INS_3$



https://doi.org/10.1515/ncrs-2023-0084
Received February 22, 2023; accepted March 9, 2023;
published online March 23, 2023

Abstract

$C_8H_4INS_3$, monoclinic, $P2_1/c$ (no. 14), a = 11.8060(19) Å, b = 7.8961(14) Å, c = 11.972(2) Å, β = 110.981 (2)°, V = 1042.1(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0279, $wR_{ref}(F^2)$ = 0.0739, T = 296(2) K.

CCDC no.: 2243717

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.23 × 0.15 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	3.62 mm^{-1}
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
θ_{max}, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6284, 2398, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2070
$N(param)_{refined}$:	118
Programs:	Bruker [1], Shelx [2, 3], WinGX/Ortep [4]

1 Source of material

All chemicals were purchased from commercial sources and used as received without further purification. A mixture of 1-(4-iodophenyl)ethanone *O*-acetyl oxime (15.1 g, 0.05 mol), S_8 (9.6 g, 0.3 mol), CuBr (0.72 g, 0.005 mol), Li_2CO_3 (1.85 g, 0.025 mol), and DMSO (100 mL) were added successfully to a 250 mL oven-dried reaction flask. The sealed reaction flask was stirred at 130° C for 8 h. After cooling to room temperature, the reaction was diluted with ethyl acetate and water. The organic layer was separated, and the aqueous layer was extracted with ethyl acetate (100 mL) for three times. The combined organic layer was brine and dried over magnesium sulfate and the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (PE/EA: 20/1) to yield the desired product (7.2 g, 46%) as a brown solid. Subsequently, dissolve 1 g of the target compound in 30 ml of dichloromethane, heat to reflux until the solid is completely dissolved, filtered. Finally, the title crystal was precipitated by controlling solvent volatilization.

***Corresponding author: Penghui Ni**, School of Chemistry and Materials Science, Key Laboratory of Functional Metal–Organic Compounds of Hunan Province, Hengyang Normal University, Hengyang, Hunan 421008, China, E-mail: penghuini2020@hynu.edu.cn. https://orcid.org/0000-0003-4178-3892
Huiyang Liao, School of Chemistry and Materials Science, Hengyang Normal University, Hengyang, Hunan 421008, China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
I1	0.17125 (2)	0.55094 (3)	0.56857 (2)	0.05658 (11)
S1	0.95419 (7)	0.75629 (11)	0.72613 (7)	0.04593 (19)
S2	0.86658 (9)	0.93158 (12)	0.59960 (9)	0.0596 (2)
S3	0.83166 (8)	0.48844(11)	0.80685 (8)	0.0494 (2)
N1	0.7290 (2)	0.8764 (4)	0.5848 (3)	0.0526 (7)
C1	0.3502 (3)	0.6211 (4)	0.5958 (3)	0.0386 (6)
C2	0.4385 (3)	0.5974 (4)	0.7077 (3)	0.0438 (7)
H2	0.417662	0.554721	0.770198	0.053*
C3	0.5577 (3)	0.6374 (4)	0.7259 (3)	0.0405 (6)
H3	0.617351	0.621527	0.800938	0.049*
C4	0.5890 (2)	0.7016 (3)	0.6325 (2)	0.0360(6)
C5	0.7148 (3)	0.7493 (4)	0.6480 (2)	0.0380 (6)
C6	0.8234 (3)	0.6616 (4)	0.7271 (2)	0.0367 (6)
C8	0.4985 (3)	0.7251 (4)	0.5219 (3)	0.0452 (7)
H8	0.518449	0.768256	0.459050	0.054*
C9	0.3794(3)	0.6856 (4)	0.5033 (3)	0.0467 (7)
H9	0.319408	0.702511	0.428621	0.056*

2 Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5U_{eq} (parent C-atom).

3 Comment

The 1,2,3-dithiazoles are important fused heterocycles because they show to have a broad biological activity profile, including antibacterial [5], anticancer [6], antifungal/herbicidal [7], and anti-melanin activities [8]. The 1,2,3-dithiazole compounds plays an important role in the mechanism of 1,2,3-dithiazoles as Inhibitors of the Feline Immunodeficiency Virus (FIV) Nucleocapsid Protein via a Proposed Zinc Ejection Mechanism [9].

Single-crystal structure analysis revealed that the title compound crystallized in the monoclinic space group $P2_1/c$. The Ortep diagram is presented in Figure. The bond lengths of S1–S2 in the title molecule is 2.0393(13) Å, it is similar to the literature [10–12]. The bond lengths of C6–S3, C6–S1 and N1–S2 in the title molecule are 1.651(3) Å, 1.719(3) Å and 1.628(3) Å, respectively. They are similar to the literature [13, 14]. The bond length of C1–I1 is 2.094(3) Å, which is similar to those reported in the literature [15, 16].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the Scientific & Technological Projects of Hengyang city (No. 202150063426), Scientific Research Fund of Hunan Provincial Education Department of China (No. 21B0634), Science Foundation of Hengyang Normal University of China (No. 2020QD07) and Key Laboratory of Functional Metal–Organic Compounds of Hunan Province (2022HSKFJJ024).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. *Saint, Apex2 and Sadabs*; Bruker AXS Inc.: Madison, WI, USA, 2012.
2. Sheldrick G. M. *Shelxtl* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with Shelxl. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Farrugia L. J. WinGX and Ortep for Windows: an update. *J. Appl. Crystallogr.* 2012, *45*, 849–854.
5. Konstantinova L. S., Bol'shakov O. I., Obruchnikova N. V., Laborie H., Tanga A., Sopena V., Lanneluc I., Picot L., Sable S., Thiery V., Rakitin O. A. One-pot synthesis of 5-phenylimino, 5-thieno or 5-oxo-1,2,3-dithiazoles and evaluation of their antimicrobial and antitumor activity. *Bioorg. Med. Chem. Lett.* 2009, *19*, 136–141.
6. Oppedisano F., Catto M., Koutentis P. A., Nicolotti O., Pochini L., Koyioni M., Introcaso A., Michaelidou S. S., Carotti A., Indiveri C. Inactivation of the glutamine/amino acid transporter ASCT2 by 1,2,3-dithiazoles: proteoliposomes as a tool to gain insights in the molecular mechanism of action and of antitumor activity. *Toxicol. Appl. Pharmacol.* 2012, *265*, 93–102.
7. Konstantinova L. S., Bol'shakov O. A., Obruchnikova N. V., Golova S. P., Nelyubina Y. V., Lyssenko K. A., Rakitin O. A. Synthesis of 1,2,5-thiadiazole-3(2*H*)-thiones and 1,2,5-thiadiazol-3(2*H*)-ones from 1,2,3-dithiazoles. *Mendeleev Commun.* 2009, *19*, 84–86.
8. Charalambous A., Koyioni M., Antoniades I., Pegeioti D., Eleftheriou I., Michaelidou S. S., Amelichev S. A., Konstantinova L. S., Rakitin O. A., Koutentis P. A., Skourides P. A. 1,2,3–dithiazoles – new reversible melanin synthesis inhibitors: a chemical genomics study. *MedChemComm* 2015, *6*, 935–946.
9. Asquith C. R., Konstantinova L. S., Laitinen T., Meli M. L., Poso A., Rakitin O. A., Hofmann–Lehmann R., Hilton S. T. Evaluation of substituted 1,2,3-dithiazoles as inhibitors of the Feline Immunodeficiency Virus (FIV) nucleocapsid protein via a proposed zinc ejection mechanism. *ChemMedChem* 2016, *11*, 2119–2126.
10. Mayo R. A., Morgan I. S., Soldatov D. V., Clérac R., Preuss K. E. Heisenberg spin chains via chalcogen bonding: noncovalent S, O contacts enable long-range magnetic order. *Inorg. Chem.* 2021, *60*, 11338–11346.
11. Konstantinova L. S., Baranovsky I. V., Pritchina E. A., Mikhailov M. S., Bagryanskaya I. Y., Semenov N. A., Irtegova I. G., Salnikov G. E., Lyssenko K. A., Gritsan N. P., Zibarev A. V., Rakitin O. A. Fused 1,2,3-thiaselenazoles synthesized from 1,2,3-dithiazoles through selective chalcogen exchange. *Chem. Eur J.* 2017, *23*, 17037–17047.

12. Konstantinova L. S., Baranovsky I. V., Pritchina E. A., Mikhailov M. S., Bagryanskaya I. Y., Semenov N. A., Irtegova I. G., Salnikov G. E., Lyssenko K. A., Gritsan N. P., Zibarev A. V., Rakitin O. A. Fused 1,2,3-thiaselenazoles synthesized from 1,2,3-dithiazoles through selective chalcogen exchange. *Chem. Eur J.* 2017, *23*, 17037–17047.
13. Koyioni M., Manoli M., Koutentis P. A. The reaction of DABCO with 4-chloro-5*H*-1,2,3-dithiazoles: synthesis and chemistry of 4-[*N*-(2-chloroethyl)piperazin-1-yl]-5*H*-1,2,3-dithiazoles. *J. Org. Chem.* 2016, *81*, 615–631.
14. Bo'shakov O. I., Yushina I. D., Stash A. I., Aysin R. R., Bartashevich E. V., Rakitin O. A. Structure and properties of 4-phenyl-5*H*-1,2,3-dithiazole-5-thione polyiodide with S–I+–S bridged complex. *Struct. Chem.* 2020, *31*, 1729–1737.
15. Ranjan S., Takamizawa S. Two-dimensional organoferroelasticity in a single crystal of 4-iodoaniline. *Cryst. Growth Des.* 2022, *22*, 1831–1836.
16. Dishman S. N., Laconsay C. J., Fettinger J. C., Tantillo D. J., Shaw J. T. Divergent stereochemical outcomes in the insertion of donor/donor carbenes into the C–H bonds of stereogenic centers. *Chem. Sci.* 2022, *13*, 1030–1036.