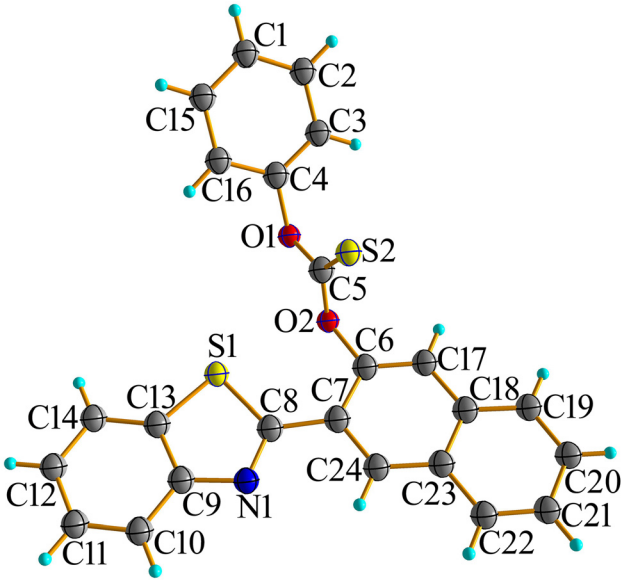
Qian Wang, Yong-Lang Liu, Jiang-Hai Ye, Juan Zou, Chong Wu* and Wu-De Yang*

Crystal structure of *O*-(3-(benzo[d]thiazol-2-yl) naphthalen-2-yl) *O*-phenyl carbonothioate, $C_{24}H_{15}NO_2S_2$



https://doi.org/10.1515/ncrs-2023-0403
Received September 11, 2023; accepted October 13, 2023;
published online October 31, 2023

Abstract

$C_{24}H_{15}NO_2S_2$, monoclinic, $P2_1/c$ (no. 14), a = 20.8250(11) Å, b = 5.3649(3) Å, c = 18.8787(11) Å, β = 106.278(2)°, V = 2024.7(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0439, $wR_{ref}(F^2)$ = 0.1015, T = 273 K.

CCDC no.: 2301075

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

***Corresponding authors: Chong Wu and Wu-De Yang**, College of Pharmacy, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, People's Republic of China, E-mail: wuchong064@gzy.edu.cn (C. Wu), yangwude476@gzy.edu.cn (W.-D. Yang). https://orcid.org/0009-0009-5135-7812 (C. Wu)

Qian Wang, Yong-Lang Liu, Jiang-Hai Ye and Juan Zou, College of Pharmacy, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, People's Republic of China

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.18 × 0.16 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.28 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,411, 3962, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3094
$N(param)_{refined}$:	262
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

1 Source of materials

The synthesis consists of two steps. In the first step, PCl_3 (3 ml) was added dropwise to a solution of 3-hydroxynaphthalene-2-carboxylic acid (6 g) and 2-aminothiophenol (4 ml) in toluene (50 ml). Reflux the mixture for 2 h. Toluene was removed by vacuum distillation and the solid remained was collected and purified by recrystallization from methanol. The product was obtained as yellow crystals. In the second step, the mixture of 3-(benzo[*d*]thiazol-2-yl)naphthalen-2-ol (1 g), phenyl chlorothionocarbonate (1 g), DIPEA (1 ml) in 20 ml DCM were stirred at room temperature for 24 h. Then, the solvent was evaporated and the crude was purified via column chromatography using hexane/ethyl acetate (10:1, v/v) to gain the final product as a white solid. Colourless crystals were grown by slow evaporation of the hexane solution.

2 Experimental details

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(C–H) = 0.97–0.99 Å, U_{iso}(H) = 1.2 times U_{eq}(C) and U_{iso}(H) = 1.5 times U_{eq}(O).

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.72952 (3)	0.39603 (12)	0.55527 (3)	0.05553 (17)
S2	0.66977 (3)	0.53092 (14)	0.74311 (3)	0.0655 (2)
O1	0.60388 (7)	0.7204 (3)	0.61382 (9)	0.0659 (5)
O2	0.70763 (7)	0.7949 (3)	0.64322 (8)	0.0523 (4)
N1	0.85437 (8)	0.2923 (3)	0.61284 (9)	0.0475 (4)
C1	0.42010 (19)	0.4931 (13)	0.6311 (3)	0.140 (2)
H1	0.3775	0.4471	0.6330	0.168*
C2	0.44948 (18)	0.6971 (10)	0.6679 (2)	0.1252 (16)
H2	0.4277	0.7875	0.6963	0.150*
C3	0.51185 (13)	0.7732 (7)	0.66372 (18)	0.0891 (9)
H3	0.5317	0.9168	0.6877	0.107*
C4	0.54291 (11)	0.6335 (5)	0.62405 (13)	0.0613 (6)
C5	0.66030 (10)	0.6834 (4)	0.66718 (12)	0.0488 (5)
C6	0.77354 (10)	0.8014 (4)	0.69139 (11)	0.0453 (5)
C7	0.82069 (9)	0.6284 (4)	0.67908 (11)	0.0427 (5)
C8	0.80731 (9)	0.4400 (4)	0.62040 (11)	0.0430 (5)
C9	0.83140 (10)	0.1343 (4)	0.55365 (11)	0.0457 (5)
C10	0.86972 (12)	−0.0444 (4)	0.53115 (13)	0.0589 (6)
H10	0.9145	−0.0658	0.5568	0.071*
C11	0.84110 (13)	−0.1888 (5)	0.47096 (14)	0.0652 (6)
H11	0.8665	−0.3092	0.4558	0.078*
C12	0.77430 (14)	−0.1570 (5)	0.43216 (14)	0.0683 (7)
H12	0.7558	−0.2563	0.3911	0.082*
C13	0.76405 (10)	0.1621 (4)	0.51484 (11)	0.0493 (5)
C14	0.73529 (13)	0.0162 (5)	0.45283 (13)	0.0656 (7)
H14	0.6907	0.0367	0.4264	0.079*
C15	0.4519 (2)	0.3553 (9)	0.5917 (3)	0.1287 (16)
H15	0.4314	0.2143	0.5667	0.154*
C16	0.51537 (17)	0.4229 (7)	0.58808 (18)	0.0928 (9)
H16	0.5382	0.3272	0.5620	0.111*
C17	0.78918 (11)	0.9792 (4)	0.74349 (13)	0.0517 (5)
H17	0.7565	1.0911	0.7482	0.062*
C18	0.85467 (11)	0.9983 (4)	0.79121 (12)	0.0491 (5)
C19	0.87377(13)	1.1793 (4)	0.84736 (14)	0.0619 (6)
H19	0.8427	1.2958	0.8534	0.074*
C20	0.93741(14)	1.1848 (5)	0.89274 (15)	0.0693 (7)
H20	0.9494	1.3048	0.9296	0.083*
C21	0.98494 (12)	1.0116 (5)	0.88432 (15)	0.0670 (7)
H21	1.0281	1.0165	0.9160	0.080*
C22	0.96860 (11)	0.8369 (4)	0.83053 (13)	0.0576 (6)
H22	1.0008	0.7240	0.8253	0.069*
C23	0.90292 (10)	0.8240 (4)	0.78208 (12)	0.0467 (5)
C24	0.88446 (10)	0.6464 (4)	0.72581 (11)	0.0468 (5)
H24	0.9168	0.5352	0.7197	0.056*

3 Comment

Benzothiazole (BTA) and its derivatives are the most important heterocyclic compounds, which are widespread and essential components of a variety of natural products and pharmaceutical agents [5–7]. Benzothiazoles are the appropriate source for fragments and core scaffolds for the design and synthesis of targeted molecules due to their innate affinity for a variety of biological receptors [8, 9]. Benzothiazole derivatives have been extensively used in various fields, such as pharmaceutical industry, and dye industry [10]. The bond length and bond angle of the target compound are within the normal range [11]. The benzothiazole ring is nearly parallel to the benzene ring, with a dihedral angle of 3.76°. The dihedral angle between the benzene ring and the naphthalene ring is 39.68°.

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

Research funding: Guizhou Provincial Key Technology R&D Program [2020]4Y107, the Natural Science Research Project of Education Department of Guizhou Province (No. QJH-KY-[2021]068) and Doctor Startup Funds of Guizhou University of Traditional Chinese Medicine [2018]04.

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

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