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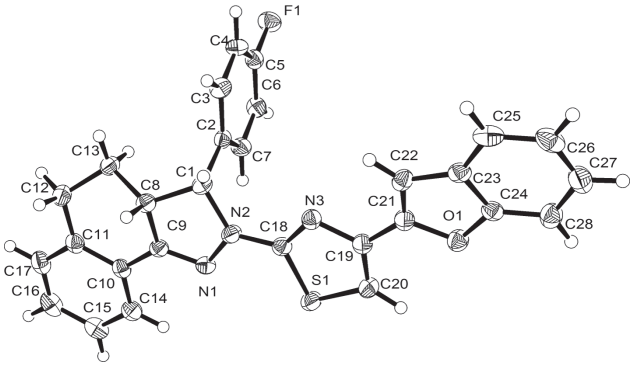
# Crystal structure of 4-(benzofuran-2-yl)-2-(3-(4-fluorophenyl)-3,3*a*,4,5-tetrahydro-2*H*-benzo[*g*]indazol-2-yl)thiazole, $C_{28}H_{20}FN_3OS$

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## Abstract

$C_{28}H_{20}FN_3OS$, triclinic, $P\bar{1}$ (no. 2), $a = 9.5719(5)$ Å, $b = 10.7499(6)$ Å, $c = 10.9238(5)$ Å, $\alpha = 95.470(4)°$, $\beta = 102.133(4)°$, $\gamma = 97.962(4)°$, $V = 1079.30(10)$ Å$^3$, $R_{gt}(F) = 0.0482$, $wR_{ref}(F^2) = 0.1143$, $T = 150(2)$ K.

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The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless plate |
| | Size 0.20 × 0.14 × 0.10 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 1.9 cm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova, $\omega$-scans |
| $2\theta_{max}$, completeness: | 59.6°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 9220, 5066, 0.025 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3916 |
| $N(param)_{refined}$: | 307 |
| Programs: | SHELX [11], CrysAlis$^{PRO}$ [12], WinGX [13] |

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | −0.14281(18) | 0.11637(17) | 0.67571(17) | 0.0246(4) |
| H1 | −0.1328 | 0.1399 | 0.5912 | 0.030* |
| C2 | −0.08131(17) | −0.00434(17) | 0.69912(16) | 0.0227(4) |
| C3 | −0.07582(19) | −0.09076(18) | 0.59862(17) | 0.0278(4) |
| H3 | −0.1074 | −0.0721 | 0.5150 | 0.033* |
| C4 | −0.0253(2) | −0.20383(18) | 0.61704(17) | 0.0285(4) |
| H4 | −0.0213 | −0.2624 | 0.5476 | 0.034* |
| C5 | 0.01878(18) | −0.22837(17) | 0.73945(18) | 0.0264(4) |
| C6 | 0.01447(19) | −0.14668(18) | 0.84189(17) | 0.0282(4) |
| H6 | 0.0450 | −0.1668 | 0.9251 | 0.034* |
| C7 | −0.03553(19) | −0.03385(18) | 0.82147(17) | 0.0275(4) |
| H7 | −0.0387 | 0.0243 | 0.8916 | 0.033* |
| C8 | −0.30185(18) | 0.11105(17) | 0.68765(17) | 0.0249(4) |
| H8 | −0.3495 | 0.1645 | 0.6267 | 0.030* |
| C9 | −0.28178(18) | 0.17815(16) | 0.81924(17) | 0.0232(4) |
| C10 | −0.39902(18) | 0.16903(17) | 0.88609(17) | 0.0245(4) |
| C11 | −0.52773(19) | 0.08579(18) | 0.82889(18) | 0.0277(4) |
| C12 | −0.54500(19) | 0.00729(19) | 0.70234(19) | 0.0323(4) |
| H12A | −0.5969 | 0.0509 | 0.6353 | 0.039* |
| H12B | −0.6055 | −0.0754 | 0.7015 | 0.039* |
| C13 | −0.40109(19) | −0.01597(18) | 0.67175(18) | 0.0280(4) |
| H13A | −0.3554 | −0.0720 | 0.7293 | 0.034* |
| H13B | −0.4181 | −0.0578 | 0.5840 | 0.034* |
| C14 | −0.3859(2) | 0.24056(18) | 1.00242(18) | 0.0296(4) |
| H14 | −0.2976 | 0.2950 | 1.0414 | 0.036* |
| C15 | −0.5004(2) | 0.2326(2) | 1.06119(19) | 0.0350(5) |

**Table 2** (continued)

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| H15 | −0.4920 | 0.2828 | 1.1396 | 0.042* |
| C16 | −0.6285(2) | 0.1507(2) | 1.0049(2) | 0.0382(5) |
| H16 | −0.7076 | 0.1447 | 1.0451 | 0.046* |
| C17 | −0.6407(2) | 0.0783(2) | 0.8910(2) | 0.0343(5) |
| H17 | −0.7283 | 0.0220 | 0.8540 | 0.041* |
| C18 | 0.06315(18) | 0.28820(17) | 0.79507(17) | 0.0243(4) |
| C19 | 0.27786(18) | 0.35947(17) | 0.76643(17) | 0.0254(4) |
| C20 | 0.2901(2) | 0.42708(18) | 0.88014(18) | 0.0297(4) |
| H20 | 0.3735 | 0.4849 | 0.9247 | 0.036* |
| C21 | 0.38786(19) | 0.36524(17) | 0.69299(18) | 0.0273(4) |
| C22 | 0.38299(19) | 0.32063(17) | 0.57326(17) | 0.0266(4) |
| H22 | 0.3011 | 0.2734 | 0.5142 | 0.032* |
| C23 | 0.52349(19) | 0.35703(17) | 0.55059(18) | 0.0260(4) |
| C24 | 0.60774(19) | 0.42322(17) | 0.66407(17) | 0.0248(4) |
| C25 | 0.5896(2) | 0.33990(19) | 0.44821(19) | 0.0346(5) |
| H25 | 0.5358 | 0.2972 | 0.3686 | 0.042* |
| C26 | 0.7360(2) | 0.3875(2) | 0.4673(2) | 0.0379(5) |
| H26 | 0.7826 | 0.3767 | 0.3993 | 0.045* |
| C27 | 0.8156(2) | 0.4498(2) | 0.5820(2) | 0.0388(5) |
| H27 | 0.9160 | 0.4792 | 0.5917 | 0.047* |
| C28 | 0.7527(2) | 0.47028(19) | 0.6832(2) | 0.0336(5) |
| H28 | 0.8068 | 0.5147 | 0.7620 | 0.040* |
| N1 | −0.15439(15) | 0.24229(14) | 0.86502(14) | 0.0241(3) |
| N2 | −0.07305(16) | 0.22275(14) | 0.77533(14) | 0.0266(3) |
| N3 | 0.14703(15) | 0.27865(14) | 0.71629(14) | 0.0253(3) |
| O1 | 0.52411(13) | 0.43076(12) | 0.75277(12) | 0.0288(3) |
| S1 | 0.13323(5) | 0.39370(5) | 0.93305(5) | 0.03009(14) |
| F1 | 0.06926(12) | −0.33894(11) | 0.75986(10) | 0.0352(3) |

## Source of material

4-(Benzofuran-2-yl)-2-(3-(4-fluorophenyl)-3,3*a*,4,5-tetrahydro-2*H*-benzo[*g*]indazol-2-yl)thiazole was prepared from reaction of 3-(4-fluorophenyl)-3,3*a*,4,5-tetrahydro-2*H*-benzo[*g*]indazole-2-carbothioamide with 2-bromoacetylbenzofuran in ethanol under reflux for 4 h. The solid obtained on cooling was recrystallized from dimethylformamide to give colourless crystals of the title compound (Mp 218–219°C) [1].

## Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. Methine C—H bonds were fixed at 1.00 Å and methylene C—H bonds at 0.99 Å. Aromatic C—H distances were set to 0.95 Å. $U_{iso}$ for all hydrogens were set to 1.2 times the $U_{eq}$ for the atoms they are bonded to.

## Discussion

Thiazole ring system has been found in vitamin $B_1$. Many thiazole derivatives show various biological activities such as antibacterial, antifungal, antiallergic and anti-HIV properties [2–4]. Convenient syntheses of thiazole derivatives involve reactions of aryl ketones with a *N,N*-diformylaminomethyl substituent in chloroform in the presence of excess triethylamine and phosphorus pentasulfide at 60°C [5], of isocyanides containing active methylene with carbodithioates in dimethylformamide in the presence of excess sodium hydride at room temperature [6], of thioureas with propargyl bromides in dimethylformamide in the presence of potassium carbonate under microwave condition [7], of *N*-bromosuccinimide with β-keto esters in the presence of thiourea in aqueous acetone at 50°C [8], of aldehydes with 2-amino(thio)phenols and aldehydes in aqueous ethanol in the presence of using samarium trifluoromethanesulfonate 55°C [9], and of β-diketones with 2-aminothiophenols in the presence of a Brønsted acid as a catalyst [10].

The asymmetric unit of the title crystal structure comprises one molecule of $C_{28}H_{20}FN_3OS$ (*cf*. the Figure). All lengths and angles are in the expected ranges. In the molecule, the benzofuran and thiozole groups are planar with a twist angle of 11.93(7)° about the bond between them. The tetrahydrobenzoindazole group is also almost planer with C(1), C(8) and C(13) being only 0.22–0.53 Å apart from the plane calculated for the rest of the group. In the crystal structure, inversion related pairs of molecules are linked by two weak C—H···N interactions with C6···N1 = 3.508(2) Å, C—H···N = 170.8°. The benzofuran groups of neighbouring molecules are involved in π-π interaction with a centroid-to-centroid distance of 3.69 Å.

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