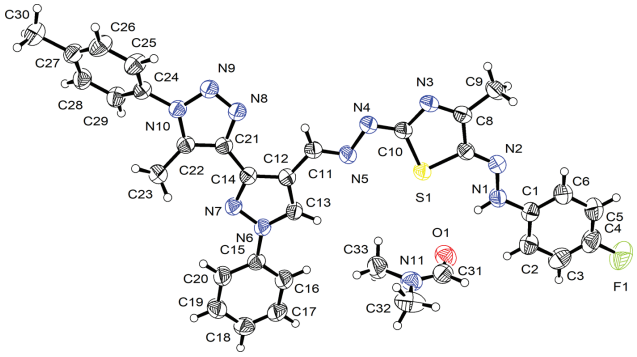
Abdullah A. Alotaibi, Bakr F. Abdel-Wahab, Amany S. Hegazy, Benson M. Kariuki and Gamal A. El-Hiti*

# The crystal structure of 5-(2-(4-fluorophenyl)hydrazono)-4-methyl-2-((3-(5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazol-4-yl)-1-phenyl-1*H*-pyrazol-4-yl)methylene) hydrazono)-2,5-dihydrothiazole dimethylformamide monosolvate, $C_{30}H_{25}FN_{10}S{\cdot}C_3H_7NO$

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## Abstract

$C_{30}H_{25}FN_{10}S{\cdot}C_3H_7NO$, triclinic, $P\bar{1}$ (no. 2), $a = 10.9189(6)$ Å, $b = 12.3898(7)$ Å, $c = 13.9206(7)$ Å, $\alpha = 199.412(4)°$, $\beta = 110.024(5)°$, $\gamma = 105.904(5)°$, $V = 1631.17(17)$ Å$^3$, $Z = 2$, $R_{gt}(F) = 0.0536$, $wR_{ref}(F^2) = 0.1471$, $T = 296$ K.

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The 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.

***Corresponding author: Gamal A. El-Hiti,** Cornea Research Chair, Department of Optometry, College of Applied Medical Sciences, King Saud University, P.O. Box 10219, Riyadh 11433, Saudi Arabia, e-mail: gelhiti@ksu.edu.sa. https://orcid.org/0000-0001-6675-3126

**Abdullah A. Alotaibi:** Department of Chemistry, College of Science and Humanities, Shaqra University, 11911, Al-Dawadmi, Saudi Arabia

**Bakr F. Abdel-Wahab:** Applied Organic Chemistry Department, National Research Centre, Dokki, Giza 12622, Egypt

**Amany S. Hegazy and Benson M. Kariuki:** School of Chemistry, Cardiff University, Main Building, Park Place, Cardiff CF10 3AT, UK

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

| | |
|---|---|
| Crystal: | Yellow block |
| Size: | 0.31 × 0.21 × 0.07 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.15 mm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova, $\omega$ |
| $\theta_{max}$, completeness: | 29.9°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 28922, 8174, 0.039 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 5125 |
| $N(param)_{refined}$: | 427 |
| Programs: | CrysAlis$^{PRO}$ [1], SHELX [2, 3], WinGX/ORTEP [4] |

## Source of material

2-((3-(5-Methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazol-4-yl)-1-phenyl-1*H*-pyrazol-4-yl)methylene)hydrazinecarbothioamide (0.83 g, 2.0 mmol), *N*′-(4-fluorophenyl)-2-oxopropanehydrazonoyl chloride (0.43 g, 2.0 mmol) and triethylamine (0.20 g, 2.0 mmol) in anhydrous ethanol (20 mL) were heated under reflux for 2 h to give the title compound. The solid obtained on cooling was filtered, washed with cold ethanol, dried and recrystallized from dimethylformamide to give yellow crystals in 78% yield.

## Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The electron density indicated disorder in the hydrogen atoms of one methyl group of dimethylformamide and the methyl group linked to the thiazolyl ring. The ordered methyl hydrogens and those bonded to sp$^2$ carbon atoms were idealized during refinement using the options AFIX 33 and AFIX 43, respectively in the SHELXL-2018 program [3]. The SHELX option AFIX 123 was used for disordered methyl groups. The $U_{iso}$ values were set to $1.5U_{eq}$(C) for methyl groups hydrogen atoms and $1.2U_{eq}$(C/N) for the rest.

## Comment

1,2,3-Triazoles are important heterocycles due to their immense pharmacological and biological applications [5–7]. In addition, compounds containing pyrazoles and thiazole

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | −0.0652(2) | 1.03548(17) | 0.20981(18) | 0.0531(5) |
| C2 | 0.0136(2) | 1.0813(2) | 0.31873(19) | 0.0632(6) |
| H2 | 0.0796 | 1.0508 | 0.3533 | 0.076* |
| C3 | −0.0061(3) | 1.1727(2) | 0.3763(2) | 0.0751(7) |
| H3 | 0.0464 | 1.2043 | 0.4494 | 0.090* |
| C4 | −0.1037(3) | 1.2152(2) | 0.3237(3) | 0.0798(8) |
| C5 | −0.1813(3) | 1.1727(2) | 0.2174(3) | 0.0819(8) |
| H5 | −0.2469 | 1.2042 | 0.1840 | 0.098* |
| C6 | −0.1622(3) | 1.0819(2) | 0.1587(2) | 0.0666(6) |
| H6 | −0.2144 | 1.0523 | 0.0854 | 0.080* |
| C7 | −0.0763(2) | 0.82351(17) | −0.00007(16) | 0.0475(5) |
| C8 | −0.1439(2) | 0.77381(18) | −0.11474(16) | 0.0485(5) |
| C9 | −0.2518(2) | 0.8125(2) | −0.18370(18) | 0.0624(6) |
| H9A[a] | −0.2839 | 0.7693 | −0.2569 | 0.094* |
| H9B[a] | −0.2120 | 0.8946 | −0.1755 | 0.094* |
| H9C[a] | −0.3288 | 0.7986 | −0.1631 | 0.094* |
| H9D[a] | −0.2659 | 0.8724 | −0.1401 | 0.094* |
| H9E[a] | −0.3378 | 0.7471 | −0.2215 | 0.094* |
| H9F[a] | −0.2210 | 0.8431 | −0.2339 | 0.094* |
| C10 | −0.0023(2) | 0.67330(17) | −0.07684(15) | 0.0485(5) |
| C11 | 0.2010(2) | 0.51194(17) | −0.03420(16) | 0.0490(5) |
| H11 | 0.1660 | 0.4680 | −0.1049 | 0.059* |
| C12 | 0.3068(2) | 0.48828(17) | 0.04709(15) | 0.0463(4) |
| C13 | 0.3629(2) | 0.53847(17) | 0.15536(16) | 0.0490(5) |
| H13 | 0.3408 | 0.5958 | 0.1906 | 0.059* |
| C14 | 0.37430(19) | 0.40734(16) | 0.03449(16) | 0.0453(4) |
| C15 | 0.5411(2) | 0.51425(17) | 0.31162(15) | 0.0487(5) |
| C16 | 0.5546(3) | 0.6107(2) | 0.38528(18) | 0.0672(6) |
| H16 | 0.5083 | 0.6609 | 0.3635 | 0.081* |
| C17 | 0.6375(3) | 0.6319(2) | 0.49149(19) | 0.0737(7) |
| H17 | 0.6458 | 0.6966 | 0.5412 | 0.088* |
| C18 | 0.7076(3) | 0.5604(2) | 0.52529(19) | 0.0681(6) |
| H18 | 0.7625 | 0.5753 | 0.5973 | 0.082* |
| C19 | 0.6956(3) | 0.4658(2) | 0.45117(19) | 0.0718(7) |
| H19 | 0.7443 | 0.4171 | 0.4731 | 0.086* |
| C20 | 0.6123(2) | 0.4419(2) | 0.34454(18) | 0.0631(6) |
| H20 | 0.6043 | 0.3772 | 0.2951 | 0.076* |
| C21 | 0.36078(19) | 0.33054(17) | −0.06326(15) | 0.0457(4) |
| C22 | 0.4233(2) | 0.24948(17) | −0.07495(15) | 0.0468(4) |
| C23 | 0.5108(3) | 0.2049(2) | 0.00457(18) | 0.0662(6) |
| H23A | 0.4978 | 0.1258 | −0.0282 | 0.099* |
| H23B | 0.4839 | 0.2069 | 0.0635 | 0.099* |
| H23C | 0.6071 | 0.2532 | 0.0298 | 0.099* |
| C24 | 0.4159(2) | 0.12852(17) | −0.24312(15) | 0.0476(5) |
| C25 | 0.3142(2) | 0.0480(2) | −0.33609(18) | 0.0630(6) |
| H25 | 0.2229 | 0.0463 | −0.3592 | 0.076* |
| C26 | 0.3487(3) | −0.0301(2) | −0.39476(19) | 0.0698(7) |
| H26 | 0.2795 | −0.0842 | −0.4577 | 0.084* |
| C27 | 0.4826(2) | −0.03030(19) | −0.36294(18) | 0.0583(5) |
| C28 | 0.5833(2) | 0.0531(2) | −0.27103(18) | 0.0615(6) |
| H28 | 0.6749 | 0.0557 | −0.2487 | 0.074* |
| C29 | 0.5518(2) | 0.13316(18) | −0.21107(17) | 0.0571(5) |
| H29 | 0.6216 | 0.1895 | −0.1498 | 0.069* |
| C30 | 0.5189(3) | −0.1182(2) | −0.4260(2) | 0.0829(8) |
| H30A | 0.5018 | −0.1887 | −0.4045 | 0.124* |
| H30B | 0.6157 | −0.0862 | −0.4130 | 0.124* |
| H30C | 0.4625 | −0.1356 | −0.5007 | 0.124* |
| C31 | 0.0442(3) | 0.7709(3) | 0.33095(18) | 0.0695(7) |
| H31 | −0.0298 | 0.7803 | 0.3457 | 0.083* |
| C32 | −0.0335(3) | 0.5739(3) | 0.3463(2) | 0.0951(10) |
| H32A | −0.1088 | 0.5959 | 0.3525 | 0.143* |
| H32B | 0.0158 | 0.5575 | 0.4106 | 0.143* |
| H32C | −0.0702 | 0.5053 | 0.2870 | 0.143* |
| C33 | 0.1734(3) | 0.6491(2) | 0.3047(2) | 0.0761(7) |
| H33A[a] | 0.1720 | 0.5717 | 0.3068 | 0.114* |
| H33B[a] | 0.2613 | 0.7056 | 0.3561 | 0.114* |
| H33C[a] | 0.1610 | 0.6565 | 0.2347 | 0.114* |
| H33D[a] | 0.2242 | 0.7175 | 0.2916 | 0.114* |
| H33E[a] | 0.1349 | 0.5836 | 0.2423 | 0.114* |
| H33F[a] | 0.2352 | 0.6327 | 0.3637 | 0.114* |
| N1 | −0.04007(18) | 0.94500(15) | 0.15474(14) | 0.0557(4) |
| H1 | 0.0180 | 0.9157 | 0.1892 | 0.067* |
| N2 | −0.10621(17) | 0.90385(14) | 0.04845(14) | 0.0508(4) |
| N3 | −0.10567(18) | 0.69380(15) | −0.15508(13) | 0.0521(4) |
| N4 | 0.05193(19) | 0.59882(15) | −0.10045(13) | 0.0553(4) |
| N5 | 0.15491(18) | 0.59240(15) | −0.01024(13) | 0.0538(4) |
| N6 | 0.45562(17) | 0.48988(14) | 0.20142(13) | 0.0474(4) |
| N7 | 0.46462(17) | 0.40833(14) | 0.12807(13) | 0.0490(4) |
| N8 | 0.28262(18) | 0.33496(16) | −0.16124(13) | 0.0548(4) |
| N9 | 0.29397(18) | 0.26183(16) | −0.23395(14) | 0.0565(4) |
| N10 | 0.38006(16) | 0.20960(14) | −0.18214(13) | 0.0471(4) |
| N11 | 0.06142(19) | 0.66921(19) | 0.32940(15) | 0.0652(5) |
| O1 | 0.11801(19) | 0.85403(16) | 0.31449(14) | 0.0779(5) |
| S1 | 0.04514(5) | 0.75770(5) | 0.05506(4) | 0.05228(16) |
| F1 | −0.1233(2) | 1.30448(15) | 0.38122(16) | 0.1219(7) |

[a]Occupancy: 0.5.

ring systems are versatile scaffolds in organic synthesis and medicinal chemistry due to their biological activities [8–10]. 1,2,3-Triazole connected to other heterocycles can be synthesized from reactions of 1,3-diketones, activated alkenes, and alkynes with nitrogen containing reagents [11]. The synthesis of pyrazoles can be achieved by performing a condensation of hydrazines and unsaturated carbonyl containing compounds [11], whereas thiazoles can be efficiently synthesized through various reactions such Gabriel, Hantzsch and Cook–Heilborn's syntheses [12]. Recently, the X-ray crystal structures for related heterocycles have been reported [13–16].

The asymmetric unit of the structure consists of a molecule of the title compound, $C_{30}H_{25}FN_{10}S$, and a molecule of dimethylformamide solvent (see the figure). The molecule of the title compound comprises fluorophenyl (**A**(C1—C6,F1), methylthiazolyl (**B**(C7—C10,N3,S1), pyrazolyl (**C**(C12—C14,N6,N7), phenyl (**D**(C15—C20), methyltriazolyl (**E**(C21—C23,N8-N10) and methylphenyl (**F**(C24—C30) rings. In the molecule, rings **A**, **B**, **C**, **D** and **E** are almost coplanar with

twist angles **A**/**B** = 4.17(13)°, **B**/**C** = 1.24(14)°, **C**/**D** = 11.59(15)°, **C**/**E** = 7.91(14)°. The plane of the methylphenyl group **F** deviates from the plane of the rest of the molecule with a twist angle **E**/**F** = 43.78(8)°. In the crystal, the planar segments of the molecules are arranged in layers parallel to the (11-1) plane. Hydrogen bonding (N1—H11···N8) occurs between the title molecule and the dimethylformamide solvent. For a pair of adjacent molecules related by inversion symmetry, the N=N bonds of the azo groups are parallel with a separation distance of 3.706 Å and the thiazolyl **B** and pyrazolyl **C** rings of the same molecules are parallel with a centroid-to-centroid separation of 3.613 Å. The phenyl groups **D** of neighbouring molecules are parallel with a centroid-to-centroid separation of 4.126 Å.

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