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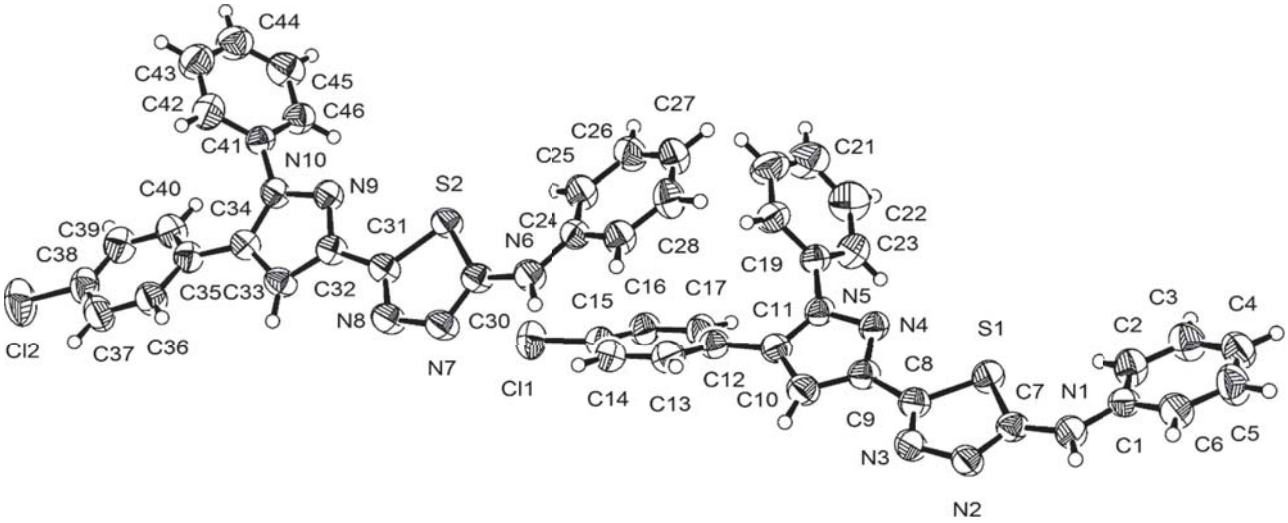
# Crystal structure of 5-(5-(4-chlorophenyl)-1-phenyl-1*H*-pyrazol-3-yl)-*N*-phenyl-1,3,4-thiadiazol-2-amine, $C_{23}H_{16}ClN_5S$

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## Abstract

$C_{23}H_{16}ClN_5S$, monoclinic, $P2_1/n$ (no. 14), $a = 19.8348(9)$ Å, $b = 8.8376(3)$ Å, $c = 23.8486(10)$ Å, $\beta = 97.646(4)°$, $V = 4143.3(3)$ Å$^3$, $Z = 8$, $R_{gt}(F) = 0.0492$, $wR_{ref}(F^2) = 0.1276$, $T = 293(2)$ K.

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The two crystallographically independent molecules of the title crystal structure are shown in the figure. 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 needle |
| Size: | 0.26 × 0.20 × 0.14 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 3.1 cm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova, $\omega$-scans |
| $2\theta_{max}$, completeness: | 59.8°, >84% (>99 up to 50°) |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 27827, 10183, 0.029 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 661 |
| $N(param)_{refined}$: | 523 |
| Programs: | CrysAlis$^{PRO}$ [9], SHELX [10], ORTEP/WinGX [11] |

## Source of material

The title compound was synthesized from treatment of 2-(5-(4-chlorophenyl)-1-phenyl-1*H*-pyrazole-3-carbonyl)-*N*-phenylhydrazinecarbothioamide with concentrated sulfuric acid in an ice bath for 1 h. This reaction mixture was left to stir at room temperature overnight, poured into cooled water and then neutralized with an aqueous sodium hydroxide solution.

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

| Atom | x | y | z | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.24211(11) | 1.5782(2) | 0.12747(8) | 0.0460(5) |
| C2 | 0.22457(13) | 1.5093(3) | 0.17563(10) | 0.0603(6) |
| H2 | 0.2315 | 1.4060 | 0.1811 | 0.072* |
| C3 | 0.19674(14) | 1.5939(3) | 0.21555(10) | 0.0669(7) |
| H3 | 0.1856 | 1.5469 | 0.2480 | 0.080* |
| C4 | 0.18533(13) | 1.7451(3) | 0.20824(10) | 0.0649(6) |
| H4 | 0.1659 | 1.8008 | 0.2351 | 0.078* |
| C5 | 0.20298(15) | 1.8143(3) | 0.16048(11) | 0.0717(7) |
| H5 | 0.1956 | 1.9175 | 0.1552 | 0.086* |
| C6 | 0.23132(13) | 1.7325(2) | 0.12074(10) | 0.0599(6) |
| H6 | 0.2434 | 1.7810 | 0.0889 | 0.072* |
| C7 | 0.29496(11) | 1.3636(2) | 0.08005(8) | 0.0467(5) |
| C8 | 0.34536(11) | 1.1181(2) | 0.08771(9) | 0.0489(5) |
| C9 | 0.37712(11) | 0.9760(2) | 0.10773(9) | 0.0490(5) |
| C10 | 0.40845(12) | 0.8662(2) | 0.07793(10) | 0.0554(5) |
| H10 | 0.4124 | 0.8657 | 0.0395 | 0.066* |
| C11 | 0.43206(11) | 0.7591(2) | 0.11730(9) | 0.0489(5) |
| C12 | 0.46579(11) | 0.6136(2) | 0.10954(9) | 0.0506(5) |
| C13 | 0.51383(12) | 0.6040(3) | 0.07242(10) | 0.0598(6) |
| H13 | 0.5260 | 0.6904 | 0.0539 | 0.072* |
| C14 | 0.54393(13) | 0.4663(3) | 0.06270(11) | 0.0647(6) |
| H14 | 0.5756 | 0.4601 | 0.0374 | 0.078* |
| C15 | 0.52673(13) | 0.3407(3) | 0.09049(10) | 0.0617(6) |
| C16 | 0.47931(12) | 0.3463(2) | 0.12778(10) | 0.0594(6) |
| H16 | 0.4680 | 0.2594 | 0.1464 | 0.071* |
| C17 | 0.44878(12) | 0.4832(2) | 0.13702(10) | 0.0551(5) |
| H17 | 0.4165 | 0.4880 | 0.1619 | 0.066* |
| C18 | 0.43127(11) | 0.7451(2) | 0.22323(9) | 0.0466(5) |
| C19 | 0.49753(12) | 0.7012(3) | 0.24166(10) | 0.0587(6) |
| H19 | 0.5312 | 0.7125 | 0.2184 | 0.070* |
| C20 | 0.51278(16) | 0.6402(3) | 0.29527(12) | 0.0760(8) |
| H20 | 0.5569 | 0.6087 | 0.3079 | 0.091* |
| C21 | 0.46357(18) | 0.6261(3) | 0.32967(13) | 0.0869(9) |
| H21 | 0.4744 | 0.5856 | 0.3657 | 0.104* |
| C22 | 0.39786(17) | 0.6715(3) | 0.31134(12) | 0.0820(8) |
| H22 | 0.3646 | 0.6630 | 0.3352 | 0.098* |
| C23 | 0.38130(13) | 0.7297(3) | 0.25744(11) | 0.0600(6) |
| H23 | 0.3368 | 0.7581 | 0.2445 | 0.072* |
| C24 | 0.69815(10) | 0.5923(2) | 0.12635(8) | 0.0453(5) |
| C25 | 0.69579(12) | 0.5232(3) | 0.17819(9) | 0.0560(6) |
| H25 | 0.7009 | 0.4189 | 0.1817 | 0.067* |
| C26 | 0.68579(13) | 0.6095(3) | 0.22481(10) | 0.0621(6) |
| H26 | 0.6850 | 0.5627 | 0.2597 | 0.075* |
| C27 | 0.67701(13) | 0.7630(3) | 0.22018(11) | 0.0626(6) |
| H27 | 0.6704 | 0.8205 | 0.2517 | 0.075* |
| C28 | 0.67809(13) | 0.8310(3) | 0.16841(11) | 0.0624(6) |
| H28 | 0.6715 | 0.9350 | 0.1649 | 0.075* |
| C29 | 0.68875(12) | 0.7476(2) | 0.12188(10) | 0.0534(5) |
| H29 | 0.6897 | 0.7954 | 0.0872 | 0.064* |
| C30 | 0.72772(11) | 0.3721(2) | 0.06818(8) | 0.0460(5) |
| C31 | 0.77591(11) | 0.1258(2) | 0.06346(8) | 0.0464(5) |
| C32 | 0.81204(11) | −0.0165(2) | 0.07216(8) | 0.0455(5) |
| C33 | 0.82315(11) | −0.1264(2) | 0.03206(9) | 0.0503(5) |
| H33 | 0.8083 | −0.1243 | −0.0066 | 0.060* |
| C34 | 0.86055(11) | −0.2379(2) | 0.06173(9) | 0.0461(5) |
| C35 | 0.88346(11) | −0.3838(2) | 0.04055(9) | 0.0490(5) |
| C36 | 0.89623(12) | −0.3936(3) | −0.01508(10) | 0.0598(6) |
| H36 | 0.8922 | −0.3074 | −0.0376 | 0.072* |
| C37 | 0.91496(13) | −0.5293(3) | −0.03778(11) | 0.0665(7) |
| H37 | 0.9229 | −0.5345 | −0.0753 | 0.080* |
| C38 | 0.92168(12) | −0.6556(3) | −0.00414(12) | 0.0615(6) |
| C39 | 0.90950(12) | −0.6500(3) | 0.05095(11) | 0.0616(6) |
| H39 | 0.9145 | −0.7364 | 0.0734 | 0.074* |
| C40 | 0.88962(5) | −0.51452(10) | 0.07312(4) | 0.0558(6) |
| H40 | 0.8803 | −0.5112 | 0.1103 | 0.067* |
| C41 | 0.90500(5) | −0.25945(10) | 0.16613(4) | 0.0451(5) |
| C42 | 0.97155(5) | −0.31118(10) | 0.16891(4) | 0.0659(6) |
| H42 | 0.9955 | −0.2972 | 0.1383 | 0.079* |
| C43 | 1.00226(5) | −0.38380(10) | 0.21739(4) | 0.0781(8) |
| H43 | 1.0468 | −0.4184 | 0.2193 | 0.094* |
| C44 | 0.96644(5) | −0.40469(10) | 0.26307(4) | 0.0771(8) |
| H44 | 0.9870 | −0.4533 | 0.2955 | 0.093* |
| C45 | 0.89990(5) | −0.35295(10) | 0.26029(4) | 0.0706(7) |
| H45 | 0.8759 | −0.3669 | 0.2909 | 0.085* |
| C46 | 0.86918(5) | −0.28033(10) | 0.21181(4) | 0.0555(5) |
| H46 | 0.8247 | −0.2457 | 0.2099 | 0.067* |
| N1 | 0.26926(10) | 1.50409(18) | 0.08357(7) | 0.0521(4) |
| H1 | 0.2696 | 1.5578 | 0.0535 | 0.062* |
| N2 | 0.31817(10) | 1.31961(19) | 0.03348(7) | 0.0535(5) |
| N3 | 0.34769(10) | 1.17836(19) | 0.03870(7) | 0.0553(5) |
| N4 | 0.38073(9) | 0.94219(18) | 0.16249(7) | 0.0477(4) |
| N5 | 0.41448(9) | 0.80814(18) | 0.16768(7) | 0.0461(4) |
| N6 | 0.70767(9) | 0.51543(19) | 0.07662(7) | 0.0510(4) |
| H6A | 0.6993 | 0.5686 | 0.0463 | 0.061* |
| N7 | 0.72575(10) | 0.32082(19) | 0.01643(7) | 0.0545(5) |
| N8 | 0.75392(10) | 0.17976(19) | 0.01409(7) | 0.0548(5) |
| N9 | 0.84089(9) | −0.05410(18) | 0.12387(7) | 0.0461(4) |
| N10 | 0.87069(9) | −0.18991(18) | 0.11698(7) | 0.0463(4) |
| S1 | 0.30460(3) | 1.22919(6) | 0.13349(2) | 0.05229(15) |
| S2 | 0.76268(3) | 0.24453(6) | 0.11947(2) | 0.04918(14) |
| Cl1 | 0.56410(4) | 0.16672(9) | 0.07866(3) | 0.0902(3) |
| Cl2 | 0.94636(4) | −0.82648(8) | −0.03139(4) | 0.0934(3) |

The solid formed was filtered, dried and recrystallized from dimethylformamide to give needle-shaped colourless crystals of the title compound (Mp. 212–213 °C) [1].

## Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. N—H bonds were fixed at 0.86 Å, with displacement parameters 1.2$U_{eq}$(N). Aromatic C—H distances were set to 0.93 Å and their U(iso) set to 1.2$U_{eq}$ for the atoms to which they are bonded.

## Discussion

1,3,4-Thiadiazole ring systems [2, 3] are present in a number of natural products and have a wide range of pharmacological

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activities such as antibacterial, antifungal, anticancer, antioxidant and anticonvulsant [4–8].

The asymmetric unit comprises two molecules (I and II) of $C_{23}H_{16}ClN_5S$ with slightly different conformations. The twist angles about the bonds between the (phenylamino)-(thiadiazole)-(pyrazole)-(chlorophenyl) groups are 15.13(10)°, 10.49(13)° and 39.56(10)° respectively for one molecule and 22.11(7)°, 8.06(12)° and 30.04(8)° for the other molecule. The angle between the (pyrazole)-(phenyl) groups are 49.07(10)° for I and 52.32(9)° for II. Bond lengths and angles are all in the expected ranges. The two crystallographically independent molecules of the title crystal structure are pairwise connected by two N—H···N hydrogen bonds.

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