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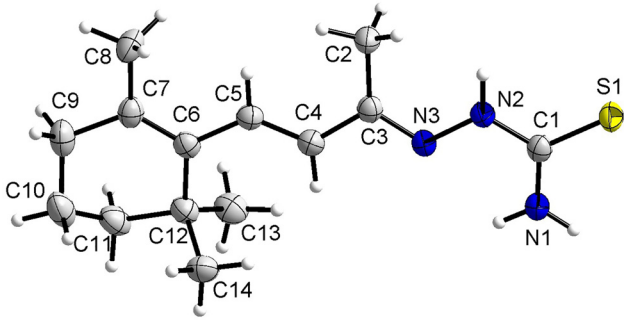
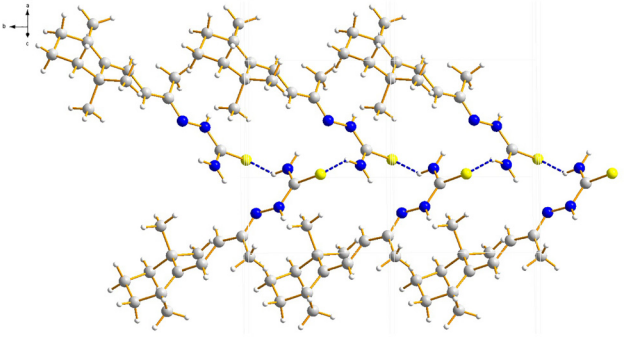
# (*E*)-2-((*E*)-4-(2,6,6-trimethylcyclohex-1-en-1-yl)but-3-en-2-ylidene)hydrazine-1-carbothioamide $C_{14}H_{23}N_3S_1$



https://doi.org/10.1515/ncrs-2023-0238
Received May 16, 2023; accepted July 4, 2023;
published online July 17, 2023

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**Abstract**

$[C_{14}H_{23}N_3S_1]$, monoclinic, $P2_1/c$ (no. 14), $a$ = 15.1642(8) Å, $b$ = 6.9215(3) Å, $c$ = 15.2182(7) Å, $\beta$ = 112.675(2)°, $V$ = 1473.83(12) Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0453, w$R_{ref}(F^2)$ = 0.1240, T = 296(2) K.

**CCDC no.:** 2279145

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

## 1 Source of materials

*β*–Ionone (20 mmol, 3.85 g) and thiosemicarbazide (24 mmol, 2.19 g) were introduced in a flask and dissolved in ethanol, then diluted hydrochloric acid (2.87 mol, 3 mL) was added and refluxed at 80 °C for 1 h. The reaction was monitored by TLC analysis until the starting material disappears completely. It was recrystallized from 70 % ethanol. After filtration and drying, compound (*E*)-2-((*E*)-4-(2,6,6-trimethylcyclohex-1-en-1-yl)but-3-en-2-ylidene)hydrazine-1-carbothioamide was obtained.

## 2 Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with

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

| | |
|---|---|
| Crystal: | Colorless block |
| Size: | 0.20 × 0.15 × 0.10 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 0.5 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II, $\varphi$ and $\omega$-scans |
| $\theta_{max}$, completeness: | 25.5°, >99 % |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 20,046, 2727, 0.048 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\sigma(I_{obs})$, 2291 |
| $N(param)_{refined}$: | 167 |
| Programs: | Bruker programs [1], SHELX [2, 3], DIAMOND [4] |

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.58360 (3) | 1.02285 (7) | 0.42188 (3) | 0.03531 (18) |
| N1 | 0.50633 (11) | 0.8022 (2) | 0.26928 (11) | 0.0342 (4) |
| H1A | 0.4696 | 0.7069 | 0.2372 | 0.041* |
| H1B | 0.5397 | 0.8694 | 0.2437 | 0.041* |
| N2 | 0.45936 (11) | 0.7384 (2) | 0.39255 (11) | 0.0322 (4) |
| H2 | 0.4584 | 0.7677 | 0.4484 | 0.039* |
| N3 | 0.40750 (11) | 0.5831 (2) | 0.34135 (11) | 0.0316 (4) |
| C5 | 0.24128 (13) | 0.2112 (3) | 0.33660 (13) | 0.0312 (4) |
| H5 | 0.2358 | 0.2378 | 0.3955 | 0.037* |
| C3 | 0.35447 (13) | 0.4865 (3) | 0.37571 (13) | 0.0305 (4) |
| C4 | 0.30079 (13) | 0.3293 (3) | 0.31560 (13) | 0.0316 (4) |
| H4 | 0.3083 | 0.3083 | 0.2572 | 0.038* |
| C1 | 0.51149 (13) | 0.8445 (3) | 0.35580 (13) | 0.0299 (4) |
| C6 | 0.18361 (13) | 0.0484 (3) | 0.28366 (13) | 0.0308 (4) |
| C12 | 0.17125 (14) | 0.0089 (3) | 0.17978 (14) | 0.0341 (4) |
| C7 | 0.14312 (13) | −0.0674 (3) | 0.32955 (14) | 0.0346 (4) |
| C2 | 0.34619 (16) | 0.5341 (3) | 0.46823 (14) | 0.0399 (5) |
| H2A | 0.4093 | 0.5688 | 0.5156 | 0.060* |
| H2B | 0.3216 | 0.4215 | 0.4907 | 0.060* |
| H2C | 0.3023 | 0.6430 | 0.4590 | 0.060* |
| C14 | 0.26476 (16) | −0.0666 (3) | 0.17541 (16) | 0.0433 (5) |
| H14A | 0.2549 | −0.0941 | 0.1091 | 0.065* |
| H14B | 0.2842 | −0.1852 | 0.2132 | 0.065* |
| H14C | 0.3149 | 0.0313 | 0.2012 | 0.065* |
| C13 | 0.13847 (16) | 0.1900 (3) | 0.11738 (14) | 0.0436 (5) |
| H13A | 0.1910 | 0.2835 | 0.1351 | 0.065* |
| H13B | 0.0838 | 0.2480 | 0.1269 | 0.065* |
| H13C | 0.1196 | 0.1543 | 0.0503 | 0.065* |
| C8 | 0.14789 (16) | −0.0286 (3) | 0.42942 (15) | 0.0437 (5) |
| H8A | 0.2129 | −0.0535 | 0.4756 | 0.065* |
| H8B | 0.1029 | −0.1138 | 0.4428 | 0.065* |
| H8C | 0.1309 | 0.1064 | 0.4344 | 0.065* |
| C11 | 0.09132 (15) | −0.1407 (3) | 0.13491 (15) | 0.0439 (5) |
| H11A | 0.0287 | −0.0784 | 0.1218 | 0.053* |
| H11B | 0.0924 | −0.1841 | 0.0734 | 0.053* |
| C9 | 0.08943 (16) | −0.2500 (3) | 0.28763 (17) | 0.0456 (5) |
| H9A | 0.0207 | −0.2294 | 0.2735 | 0.055* |
| H9B | 0.1122 | −0.3545 | 0.3355 | 0.055* |
| C10 | 0.10086 (16) | −0.3140 (3) | 0.19764 (18) | 0.0499 (6) |
| H10A | 0.1643 | −0.3745 | 0.2137 | 0.060* |
| H10B | 0.0512 | −0.4106 | 0.1638 | 0.060* |

$U_{iso}$(H) = 1.5 $U_{eq}$(C) for methyl H atoms and 1.2 $U_{eq}$(C) for all other H atoms.

# 3 Comment

Thiosemicarbazone compounds have good fungicide, insecticidal, antiviral and other biological activities, and occupy an important position in the pesticide market [5–7]. For instance, Tabatabaee M. *et al*. have reported the structure of 4–Amino-6-methyl-3-thio-3,4-dihydro-1,2,4-triazin-5(2H)-one [8]. Jouad E. M. *et al*. have reported the structure and antifungal activity of nickel(II) complexes with 5-methyl-2-furfural thiosemicarbazone [9]. However, most of the compounds in these studies are heteroatom containing five membered ring thiosemicarbazones structures [10, 11], while there are relatively few studies on six membered ring thiosemicarbazones structures. Hashimoto H. *et al*. have reported the structure of *β*-ionylidene-2,4-dinitrophenylhydrazone [12].

In the molecules of the title structure bond lengths and angles are very similar to those given in the literature for thiosemicarbazones derivative [13]. In the title structure, the cyclohexene portion is in a half-chair configuration. The dihedral angle formed by the C8–C7–C6–C12 plane and the C10–C11–C12 plane is 45.33(17)°. Due to $\pi$–$\pi$ conjugation, the non hydrogen atoms of thiosemicarbazone moiety are approximately planar. The dihedral angle formed by the carbon–carbon double bond group C4–H4–C5 plane, the carbon-nitrogen double bond group C2–C3–N3 plane and the carbon-sulfur double bond group N2–C1–S1 plane are 1.3(2)°, 6.1(2)° and 7.4(2)°, respectively. The torsion angles of C7–C6–C5–C4, C6–C5–C4–C3, C5–C4–C3–C2, C5–C4–C3–N3, C4–C3–N3–N2, C3–N3–N2–C1, N3–N2–C1–N1 and N3–N2–C1–S1 are 168.3(2)°, –179.7(2)°, –1.3(3)°, –179.7(2)°, 178.4(2)°, –177.3(2)°, 3.9(3)° and –174.4(1)°, respectively. In the title structure, there are abundant intramolecular hydrogen bonds (N2–H2···S1, N1–H1B···N3 and C2–H2A···S1) and intermolecular hydrogen bond (N1–H1A···S1). The intermolecular hydrogen bond (N1–H1A···S1) link adjacent molecules into one-dimensional hydrogen bond chain structure.

**Acknowledgment:** X-ray data were collected at Instrumental Analysis Center Nanchang Hangkong University, Nanchang, 330063, People's Republic of China

**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 National Natural Science Foundation of China (No. 31760295), Major science and technology R & D special project (20203ABC28W016), Jiangxi Province Training Program Leading Talents Project (20204BCJ22022) and Central Finance Forestry Science and Technology Promotion Demonstration Fund Project(JXTG(2021)01).

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

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