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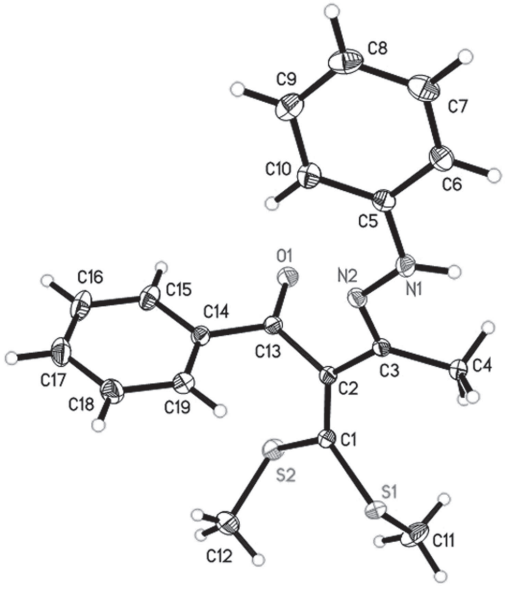
# Crystal structure of (*E*)-2-(bis(methylthio) methylene)-1-phenyl-3-(2-phenylhydrazono) butan-1-one, $C_{19}H_{20}N_2OS_2$

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## Abstract

$C_{19}H_{20}N_2OS_2$, monoclinic, $P2_1/c$ (no. 14), $a = 10.6164(9)$ Å, $b = 8.4131(6)$ Å, $c = 20.2448(16)$ Å, $\beta = 100.021(4)°$, $V = 1780.6(2)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.040$, $wR(F^2) = 0.081$, $T = 100$ K.

**CCDC no.:** 1030324

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

| | |
|---|---|
| Crystal: | Yellow, plates, size 0.15×0.33×0.43 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 3.07 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker Kappa APEXII duo, $\omega + \varphi$-Scans |
| $2\theta_{max}$: | 61.24° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 46960, 5464 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\,\sigma(I_{obs})$, 4781 |
| $N(param)_{refined}$: | 224 |
| Programs: | SHELX [4] |

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

| Atom | Site | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 4*e* | 0.156(2) | 0.387(2) | −0.0211(8) | 0.029(4) |
| H(4A) | 4*e* | −0.0235 | 0.2933 | −0.0321 | 0.025 |
| H(4B) | 4*e* | −0.0925 | 0.1819 | 0.0149 | 0.025 |
| H(4C) | 4*e* | 0.0514 | 0.1452 | 0.0058 | 0.025 |
| H(6) | 4*e* | 0.3054 | 0.4900 | −0.0831 | 0.023 |
| H(7) | 4*e* | 0.4592 | 0.6800 | −0.0950 | 0.028 |
| H(8) | 4*e* | 0.5366 | 0.8526 | −0.0066 | 0.030 |
| H(9) | 4*e* | 0.4567 | 0.8342 | 0.0937 | 0.028 |
| H(10) | 4*e* | 0.3034 | 0.6423 | 0.1071 | 0.024 |
| H(11A) | 4*e* | −0.2819 | −0.0130 | 0.1496 | 0.042 |
| H(11B) | 4*e* | −0.2423 | −0.1517 | 0.1031 | 0.042 |
| H(11C) | 4*e* | −0.2785 | 0.0208 | 0.0721 | 0.042 |
| H(12A) | 4*e* | −0.1271 | 0.0315 | 0.2654 | 0.050 |
| H(12B) | 4*e* | −0.1747 | 0.1512 | 0.3174 | 0.050 |
| H(12C) | 4*e* | −0.0332 | 0.1671 | 0.3009 | 0.050 |
| H(15) | 4*e* | −0.0042 | 0.7447 | 0.2470 | 0.025 |
| H(16) | 4*e* | 0.1289 | 0.7816 | 0.3509 | 0.031 |
| H(17) | 4*e* | 0.2785 | 0.5874 | 0.3931 | 0.031 |
| H(18) | 4*e* | 0.2995 | 0.3574 | 0.3314 | 0.029 |
| H(19) | 4*e* | 0.1633 | 0.3158 | 0.2286 | 0.023 |

**Table 3:** Atomic displacement parameters (Å$^2$).

| Atom | Site | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| S(1) | 4*e* | −0.07224(2) | 0.02230(3) | 0.13364(1) | 0.0195(1) | 0.0113(1) | 0.0180(1) | −0.00014(8) | 0.00302(9) | 0.00020(8) |
| O(1) | 4*e* | −0.07605(7) | 0.61738(9) | 0.13255(4) | 0.0251(4) | 0.0141(3) | 0.0176(3) | 0.0027(3) | 0.0031(3) | 0.0000(3) |
| C(1) | 4*e* | −0.08549(9) | 0.2242(1) | 0.15450(5) | 0.0172(4) | 0.0130(4) | 0.0137(4) | 0.0004(3) | 0.0032(3) | −0.0009(3) |
| N(1) | 4*e* | 0.19657(8) | 0.4306(1) | 0.01713(4) | 0.0195(4) | 0.0177(4) | 0.0135(4) | −0.0026(3) | 0.0054(3) | −0.0022(3) |
| S(2) | 4*e* | −0.18064(3) | 0.27524(3) | 0.21456(1) | 0.0243(1) | 0.0185(1) | 0.0208(1) | −0.00243(9) | 0.0111(1) | −0.00211(9) |
| N(2) | 4*e* | 0.13419(8) | 0.4307(1) | 0.07033(4) | 0.0176(4) | 0.0143(3) | 0.0121(3) | 0.0016(3) | 0.0037(3) | 0.0004(3) |
| C(2) | 4*e* | −0.02341(9) | 0.3428(1) | 0.12802(4) | 0.0164(4) | 0.0121(4) | 0.0123(4) | 0.0010(3) | 0.0019(3) | −0.0007(3) |
| C(3) | 4*e* | 0.03806(9) | 0.3340(1) | 0.06819(4) | 0.0175(4) | 0.0112(4) | 0.0115(4) | 0.0021(3) | 0.0022(3) | 0.0003(3) |
| C(4) | 4*e* | −0.0109(1) | 0.2295(1) | 0.00903(5) | 0.0227(5) | 0.0143(4) | 0.0128(4) | −0.0009(3) | 0.0023(3) | −0.0015(3) |
| C(5) | 4*e* | 0.28873(9) | 0.5478(1) | 0.01314(5) | 0.0150(4) | 0.0157(4) | 0.0167(4) | 0.0012(3) | 0.0029(3) | 0.0013(3) |
| C(6) | 4*e* | 0.3361(1) | 0.5597(1) | −0.04694(5) | 0.0184(5) | 0.0221(5) | 0.0175(4) | 0.0011(4) | 0.0048(4) | 0.0011(4) |
| C(7) | 4*e* | 0.4276(1) | 0.6727(1) | −0.05395(6) | 0.0191(5) | 0.0269(5) | 0.0257(5) | 0.0014(4) | 0.0082(4) | 0.0063(4) |
| C(8) | 4*e* | 0.4736(1) | 0.7754(1) | −0.00169(6) | 0.0175(5) | 0.0208(5) | 0.0355(6) | −0.0015(4) | 0.0042(4) | 0.0054(4) |
| C(9) | 4*e* | 0.4262(1) | 0.7636(1) | 0.05788(6) | 0.0206(5) | 0.0193(5) | 0.0290(5) | −0.0015(4) | 0.0010(4) | −0.0025(4) |
| C(10) | 4*e* | 0.3344(1) | 0.6500(1) | 0.06595(5) | 0.0202(5) | 0.0200(5) | 0.0191(4) | −0.0002(4) | 0.0034(4) | −0.0018(4) |
| C(11) | 4*e* | −0.2376(1) | −0.0372(1) | 0.11216(7) | 0.0216(5) | 0.0182(5) | 0.0412(7) | −0.0033(4) | −0.0025(5) | −0.0040(4) |
| C(12) | 4*e* | −0.1223(2) | 0.1410(2) | 0.28232(6) | 0.0570(8) | 0.0276(6) | 0.0178(5) | −0.0071(6) | 0.0124(5) | 0.0024(4) |
| C(13) | 4*e* | −0.01677(9) | 0.5052(1) | 0.16126(5) | 0.0165(4) | 0.0135(4) | 0.0139(4) | −0.0010(3) | 0.0054(3) | −0.0007(3) |
| C(14) | 4*e* | 0.06615(9) | 0.5254(1) | 0.22809(5) | 0.0175(4) | 0.0160(4) | 0.0139(4) | −0.0028(3) | 0.0047(3) | −0.0015(3) |
| C(15) | 4*e* | 0.0563(1) | 0.6654(1) | 0.26433(5) | 0.0208(5) | 0.0212(5) | 0.0219(5) | −0.0024(4) | 0.0060(4) | −0.0075(4) |
| C(16) | 4*e* | 0.1358(1) | 0.6874(2) | 0.32585(6) | 0.0238(5) | 0.0322(6) | 0.0229(5) | −0.0086(4) | 0.0081(4) | −0.0135(4) |
| C(17) | 4*e* | 0.2251(1) | 0.5721(2) | 0.35083(5) | 0.0240(5) | 0.0363(6) | 0.0162(4) | −0.0121(5) | 0.0019(4) | −0.0035(4) |
| C(18) | 4*e* | 0.2369(1) | 0.4347(1) | 0.31452(5) | 0.0233(5) | 0.0263(5) | 0.0206(5) | −0.0053(4) | −0.0019(4) | 0.0040(4) |
| C(19) | 4*e* | 0.1565(1) | 0.4106(1) | 0.25330(5) | 0.0213(5) | 0.0173(4) | 0.0180(4) | −0.0021(4) | 0.0020(4) | 0.0003(3) |

The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

## Source of material

The reagents and solvents used in this study are commercially available. The synthesis of (*E*)-2-(*bis*(methylthio)methylene)-1-phenyl-3-(2-phenylhydrazono)butan-1-one was carried out according to following procedure: A mixture of 2.80 g (0.01 mol) (*E*)-2-(*bis*(methylthio)methylene)-3-hydrazono-1-phenylbutan-1-one and 0.02 mol of phenylhydrazine in 35 mL of ethanol was refluxed for 3 h. The reaction mixture was cooled filtered. The precipitated product was filtered and washed with ethanol and dried; the precipitated was recrystallized from ethanol. Yield: 79%; m.p. 175 °C; IR ($\nu_{max}$): 3292, 3060, 2916, 1770, 1645, 1512, 1228, 1156 cm$^{-1}$; $^1$H—NMR (400 MHz, $CDCl_3$): δ 2.17 (s, 3H, $CH_3$), 2.23 (s, 3H, $CH_3$), 2.60 (s, 3H, $CH_3$), 7.28–7.73 (m, 10H, Ph); $^{13}$C—NMR (100 MHz, DMSO-$d_6$): δ 13.3, 17.8, 113.0, 12;.5, 125.3, 128.7, 129.6, 129.9, 134.8, 137.9, 143.2, 155.8, 181.6, 195.0; MS *m/z* (%): 356.50 [M+, 50°]; Anal. calcd. for $C_{19}H_{20}N_2OS_2$: C, 64.01; H, 5.65; N, 7.86; O, 4.49; S, 17.99%. Found: C, 64.03; H, 5.66; N, 7.88; S, 18.02%.

## Discussion

Ketene *S*,*S* dithioacetals are very useful intermediates for the construction of heterocyclic compounds such as polysubstituted pyrazoles, pyrimidines and quinolones [1–3]. The structure of the title compound was elucidated by spectroscopic methods and X-ray diffraction. The molecules packing in the crystal structure is stabilized by intermolecular hydrogen bonds (N1—H1···O1).

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