# Supplementary Material
# Synthesis of spiro-1,2,4-triazole-3-thiones from cycloalkanone thiosemicarbazones, and formation of a cyclic 1,2-dithione

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**Crystal Structure Report for 7h**

A yellow plate-like specimen of $C_{13}H_{21}N_3S$, approximate dimensions 0.074 mm x 0.197 mm x 0.281 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker D8 VENTURE PHOTON 100 CMOS system equipped with a mirror monochromator and a Cu-Kα INCOATEC IμS micro--focus source (λ = 1.54178 Å).

A total of 1960 frames were collected. The total exposure time was 36.27 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 8479 reflections to a maximum θ angle of 72.37° (0.81 Å resolution), of which 2192 were independent (average redundancy 3.868, completeness = 97.5%, $R_{int}$ = 2.52%, $R_{sig}$ = 2.33%) and 1945 (88.73%) were greater than $2\sigma(F^2)$. The final cell constants of a = 9.1362(3) Å, b = 9.6025(3) Å, c = 13.3880(4) Å, β = 105.3260(10)°, volume = 1132.76(6) $Å^3$, are based upon the refinement of the XYZ-centroids of 6352 reflections above 20 σ(I) with 9.209° < 2θ < 144.7°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.859. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.5570 and 0.8450.

 

The final anisotropic full-matrix least-squares refinement on $F^2$ with 137 variables converged at R1 = 6.05%, for the observed data and wR2 = 20.35% for all data. The goodness-of-fit was 1.097. The largest peak in the final difference electron density synthesis was 0.590 $e^-/Å^3$ and the largest hole was -0.536 $e^-/Å^3$ with an RMS deviation of 0.063 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.474 $g/cm^3$ and F(000), 544 $e^-$.

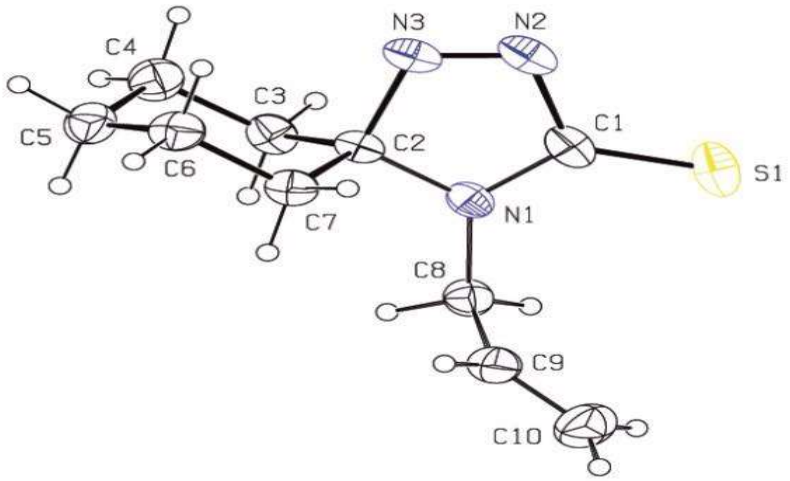


**Fig**. **1** Molecular structure of **7h** with labeling scheme and 50% probability ellipsoids.

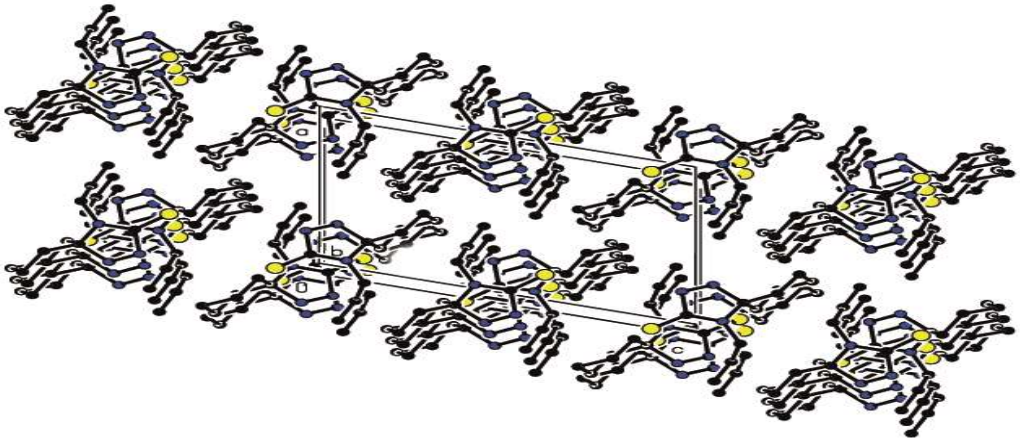

**Fig 2.** Packing viewed of **7h** down the *b* axis with hydrogen atoms omitted for clarity.

 

**Table 1. Sample and crystal data for 7h.**

| | | |
|---|---|---|
| **Identification code** | **7h** | |
| **Chemical formula** | $C_{13}H_{21}N_3S$ | |
| **Formula weight** | 251.39 g/mol | |
| **Temperature** | 225(2) K | |
| **Wavelength** | 1.54178 Å | |
| **Crystal size** | 0.074 x 0.197 x 0.281 mm | |
| **Crystal habit** | yellow plate | |
| **Crystal system** | monoclinic | |
| **Space group** | P 1 21/c 1 | |
| **Unit cell dimensions** | a = 9.1362(3) Å | α = 90° |
| | b = 9.6025(3) Å | β = 105.3260(10)° |
| | c = 13.3880(4) Å | γ = 90° |
| **Volume** | 1132.76(6) $Å^3$ | |
| **Z** | 4 | |
| **Density (calculated)** | 1.474 $g/cm^3$ | |
| **Absorption coefficient** | 2.356 $mm^{-1}$ | |
| **F(000)** | 544 | |

**Table 2. Bond lengths (Å) for 7h.**

| | | | |
|---|---|---|---|
| S1-C1 | 1.643(3) | N1-C1 | 1.330(3) |
| N1-C10 | 1.456(3) | N1-C10A | 1.456(4) |
| N1-C2 | 1.460(3) | C1-C4 | 1.454(4) |
| C2-C3 | 1.467(3) | C2-C5 | 1.528(3) |
| C2-C9 | 1.531(3) | C3-C4 | 1.235(4) |
| C3-H3 | 0.94 | C4-H4 | 0.94 |
| C5-C6 | 1.523(4) | C5-H5A | 0.98 |
| C5-H5B | 0.98 | C6-C7 | 1.519(4) |
| C6-H6A | 0.98 | C6-H6B | 0.98 |
| C7-C8 | 1.522(4) | C7-H7A | 0.98 |
| C7-H7B | 0.98 | C8-C9 | 1.517(4) |
| C8-H8A | 0.98 | C8-H8B | 0.98 |
| C9-H9A | 0.98 | C9-H9B | 0.98 |
| C10-C11 | 1.497(6) | C10-H10A | 0.98 |

| | | | |
|---|---|---|---|
| C10-H10B | 0.98 | C11-C12 | 1.322(6) |
| C11-H11 | 0.94 | C12-H12A | 0.94 |
| C12-H12B | 0.94 | C10A-C11A | 1.496(8) |
| C10A-H10C | 0.98 | C10A-H10D | 0.98 |
| C11A-C12A | 1.321(7) | C11A-H11A | 0.94 |
| C12A-H12C | 0.94 | C12A-H12D | 0.94 |

**Table 5: Data collection details for 7h.**

| Axis | dx/mm | 2θ/° | ω/° | φ/° | χ/° | Width/° | Frames | Time/s | Wavelength/Å | Voltage/kV | Current/mA | Temperature/K |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Omega | 39.980 | 85.20 | -87.30 | 71.17 | 54.74 | 0.50 | 330 | 40.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.980 | 104.95 | -66.55 | -160.00 | 54.74 | 0.50 | 326 | 80.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.980 | 104.95 | -66.55 | -40.00 | 54.74 | 0.50 | 326 | 80.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.980 | 4.97 | -166.53 | 0.00 | 54.74 | 0.50 | 326 | 40.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.980 | 104.95 | -66.55 | -80.00 | 54.74 | 0.50 | 326 | 80.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.980 | 104.95 | -66.55 | 160.00 | 54.74 | 0.50 | 326 | 80.00 | 1.54184 | 50 | 1.0 | n/a |

**Table 6. Data collection and structure refinement for 7h.**

| | |
|---|---|
| **Diffractometer** | Bruker D8 VENTURE PHOTON 100 CMOS |
| **Radiation source** | INCOATEC IμS micro--focus source, Cu-Kα |
| **Theta range for data collection** | 5.02 to 72.37° |
| **Index ranges** | -10<=h<=10, -11<=k<=11, -16<=l<=16 |
| **Reflections collected** | 8479 |
| **Independent reflections** | 2192 [R(int) = 0.0252] |
| **Coverage of independent reflections** | 97.5% |
| **Absorption correction** | multi-scan |
| **Max. and min. transmission** | 0.8450 and 0.5570 |
| **Refinement method** | Full-matrix least-squares on $F^2$ |
| **Refinement program** | SHELXL-2014 (Sheldrick, 2014) |
| **Function minimized** | $\Sigma\, w(F_o^2 - F_c^2)^2$ |
| **Data / restraints / parameters** | 2192 / 4 / 137 |

 

| | | |
|---|---|---|
| **Goodness-of-fit on $F^2$** | 1.097 | |
| **Final R indices** | 1945 data; I>2σ(I) | R1 = 0.0605, wR2 = 0.1968 |
| | all data | R1 = 0.0651, wR2 = 0.2035 |
| **Weighting scheme** | $w=1/[\sigma^2(F_o^2)+(0.1234P)^2+0.5208P]$ where $P=(F_o^2+2F_c^2)/3$ | |
| **Largest diff. peak and hole** | 0.590 and -0.536 eÅ$^{-3}$ | |
| **R.M.S. deviation from mean** | 0.063 eÅ$^{-3}$ | |

**Table 7. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å$^2$) for 7h.**

U(eq) is defined as one third of the trace of the orthogonalized$U_{ij}$ tensor.

| | x/a | y/b | z/c | U(eq) |
|---|---|---|---|---|
| S1 | 0.60011(11) | 0.86628(8) | 0.61196(5) | 0.0686(4) |
| N1 | 0.4722(2) | 0.7544(2) | 0.42361(15) | 0.0439(5) |
| C1 | 0.4709(3) | 0.7895(2) | 0.51946(18) | 0.0512(6) |
| C2 | 0.3276(3) | 0.6915(2) | 0.36941(17) | 0.0425(5) |
| C3 | 0.2476(3) | 0.6927(3) | 0.45106(19) | 0.0471(6) |
| C4 | 0.3242(3) | 0.7461(3) | 0.53191(19) | 0.0516(6) |
| C5 | 0.3453(3) | 0.5412(2) | 0.3371(2) | 0.0480(6) |
| C6 | 0.1937(3) | 0.4766(3) | 0.2809(2) | 0.0581(7) |
| C7 | 0.1130(3) | 0.5637(3) | 0.1879(2) | 0.0620(7) |
| C8 | 0.0918(3) | 0.7127(3) | 0.2203(2) | 0.0650(8) |
| C9 | 0.2405(3) | 0.7797(3) | 0.2777(2) | 0.0522(6) |
| C10 | 0.5981(10) | 0.7788(13) | 0.3781(12) | 0.0499(7) |
| C11 | 0.7144(5) | 0.6652(6) | 0.4022(4) | 0.0609(11) |
| C12 | 0.8591(5) | 0.6920(7) | 0.4458(4) | 0.0870(15) |
| C10A | 0.601(3) | 0.778(4) | 0.381(4) | 0.0499(7) |
| C11A | 0.7362(19) | 0.6964(18) | 0.4394(12) | 0.0609(11) |
| C12A | 0.8030(17) | 0.6037(18) | 0.3934(11) | 0.0870(15) |

**Table 8. Torsion angles (°) for 7h.**

| | | | |
|---|---|---|---|
| C10-N1-C1-C4 | 180.0(4) | C10A-N1-C1-C4 | -178.9(10) |
| C2-N1-C1-C4 | 1.6(3) | C10-N1-C1-S1 | 0.1(5) |
| C10A-N1-C1-S1 | 1.2(10) | C2-N1-C1-S1 | -178.30(19) |
| C1-N1-C2-C3 | -1.8(2) | C10-N1-C2-C3 | 179.8(4) |
| C10A-N1-C2-C3 | 178.7(10) | C1-N1-C2-C5 | -118.1(2) |
| C10-N1-C2-C5 | 63.5(4) | C10A-N1-C2-C5 | 62.4(10) |

 

| | | | |
|---|---|---|---|
| C1-N1-C2-C9 | 115.0(2) | C10-N1-C2-C9 | -63.4(4) |
| C10A-N1-C2-C9 | -64.5(10) | N1-C2-C3-C4 | 1.4(3) |
| C5-C2-C3-C4 | 120.1(2) | C9-C2-C3-C4 | -117.3(3) |
| C2-C3-C4-C1 | -0.6(3) | N1-C1-C4-C3 | -0.6(3) |
| S1-C1-C4-C3 | 179.2(2) | N1-C2-C5-C6 | 179.9(2) |
| C3-C2-C5-C6 | 68.6(3) | C9-C2-C5-C6 | -52.9(3) |
| C2-C5-C6-C7 | 55.6(3) | C5-C6-C7-C8 | -56.2(3) |
| C6-C7-C8-C9 | 55.3(3) | C7-C8-C9-C2 | -53.2(3) |
| N1-C2-C9-C8 | 178.9(2) | C3-C2-C9-C8 | -69.7(3) |
| C5-C2-C9-C8 | 51.6(3) | C1-N1-C10-C11 | 85.6(14) |
| C2-N1-C10-C11 | -96.2(11) | N1-C10-C11-C12 | -125.2(10) |
| C1-N1-C10A-C11A | 63.(4) | C2-N1-C10A-C11A | -117.(3) |
| N1-C10A-C11A-C12A | 121.(3) | | |

**Table 9. Anisotropic atomic displacement parameters (Å$^2$) for 7h.**

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12} ]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| S1 | 0.1045(7) | 0.0510(5) | 0.0431(4) | -0.0010(3) | 0.0064(4) | 0.0001(3) |
| N1 | 0.0566(11) | 0.0374(10) | 0.0411(10) | 0.0012(7) | 0.0188(8) | 0.0012(8) |
| C1 | 0.0789(17) | 0.0372(12) | 0.0388(12) | 0.0049(9) | 0.0179(11) | 0.0101(11) |
| C2 | 0.0498(12) | 0.0400(12) | 0.0430(11) | 0.0006(9) | 0.0219(9) | 0.0029(9) |
| C3 | 0.0487(12) | 0.0520(14) | 0.0500(13) | 0.0030(10) | 0.0298(10) | 0.0058(10) |
| C4 | 0.0696(16) | 0.0529(14) | 0.0427(12) | 0.0018(10) | 0.0331(12) | 0.0091(11) |
| C5 | 0.0527(13) | 0.0366(12) | 0.0581(14) | 0.0028(10) | 0.0206(11) | 0.0029(9) |
| C6 | 0.0599(15) | 0.0465(14) | 0.0726(17) | -0.0027(12) | 0.0260(13) | -0.0078(11) |
| C7 | 0.0551(14) | 0.0628(18) | 0.0670(17) | -0.0095(13) | 0.0142(12) | -0.0056(12) |
| C8 | 0.0579(16) | 0.0664(18) | 0.0662(17) | -0.0002(14) | 0.0082(13) | 0.0142(13) |
| C9 | 0.0655(15) | 0.0397(13) | 0.0516(13) | 0.0015(10) | 0.0159(11) | 0.0098(11) |
| C10 | 0.0543(14) | 0.0502(14) | 0.0481(15) | 0.0050(11) | 0.0184(12) | -0.0053(11) |
| C11 | 0.057(2) | 0.072(3) | 0.059(3) | 0.001(2) | 0.025(2) | 0.0058(19) |
| C12 | 0.064(3) | 0.126(5) | 0.067(3) | 0.019(3) | 0.011(2) | 0.011(2) |
| C10A | 0.0543(14) | 0.0502(14) | 0.0481(15) | 0.0050(11) | 0.0184(12) | -0.0053(11) |
| C11A | 0.057(2) | 0.072(3) | 0.059(3) | 0.001(2) | 0.025(2) | 0.0058(19) |
| C12A | 0.064(3) | 0.126(5) | 0.067(3) | 0.019(3) | 0.011(2) | 0.011(2) |

**Table 10. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å$^2$) for 7h.**

| | x/a | y/b | z/c | U(eq) |
|---|---|---|---|---|
| H3 | 0.1494 | 0.6569 | 0.4428 | 0.056 |
| H4 | 0.2924 | 0.7572 | 0.5927 | 0.062 |
| H5A | 0.4136 | 0.5389 | 0.2916 | 0.058 |
| H5B | 0.3917 | 0.4857 | 0.3988 | 0.058 |
| H6A | 0.2107 | 0.3826 | 0.2578 | 0.07 |
| H6B | 0.1294 | 0.4690 | 0.3288 | 0.07 |
| H7A | 0.0137 | 0.5225 | 0.1554 | 0.074 |
| H7B | 0.1725 | 0.5638 | 0.1369 | 0.074 |
| H8A | 0.0453 | 0.7680 | 0.1585 | 0.078 |

| | | | | |
|---|---|---|---|---|
| H8B | 0.0223 | 0.7131 | 0.2649 | 0.078 |
| H9A | 0.2199 | 0.8716 | 0.3029 | 0.063 |
| H9B | 0.3038 | 0.7932 | 0.2296 | 0.063 |
| H10A | 0.6469 | 0.8674 | 0.4041 | 0.06 |
| H10B | 0.5586 | 0.7869 | 0.3028 | 0.06 |
| H11 | 0.6837 | 0.5727 | 0.3858 | 0.073 |
| H12A | 0.8911 | 0.7841 | 0.4624 | 0.104 |
| H12B | 0.9300 | 0.6189 | 0.4601 | 0.104 |
| H10C | 0.6263 | 0.8775 | 0.3848 | 0.06 |
| H10D | 0.5743 | 0.7504 | 0.3079 | 0.06 |
| H11A | 0.7746 | 0.7115 | 0.5109 | 0.073 |
| H12C | 0.7659 | 0.5874 | 0.3219 | 0.104 |
| H12D | 0.8877 | 0.5539 | 0.4321 | 0.104 |

**Crystal Structure Report for 12**

A colorless block-like specimen of $C_{10}H_4O_2S_2$, approximate dimensions 0.114 mm x 0.156 mm x 0.268 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker D8 VENTURE PHOTON 100 CMOS system equipped with a mirror monochromator and a Cu 'INCOATEC IμS micro-focus source' (λ = 1.54178 Å).

A total of 1960 frames were collected. The total exposure time was 5.44 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a triclinic unit cell yielded a total of 6695 reflections to a maximum θ angle of 72.45° (0.81 Å resolution), of which 3235 were independent (average redundancy 2.070, completeness = 93.9%, $R_{int}$ = 2.19%, $R_{sig}$ = 2.90%) and 2787 (86.15%) were greater than $2\sigma(F^2)$. The final cell constants of a = 7.2036(2) Å, b = 8.2321(2) Å, c = 16.1168(4) Å, α = 89.755(2)°, β = 82.4190(10)°, γ = 66.6960(10)°, volume = 868.89(4) $Å^3$, are based upon the refinement of the XYZ-centroids of 5161 reflections above 20 σ(I) with 5.539° < 2θ < 144.9°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.766. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.3320 and 0.5850.

 

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P -1, with Z = 4 for the formula unit, $C_{10}H_4O_2S_2$. The final anisotropic full-matrix least-squares refinement on $F^2$ with 253 variables converged at R1 = 3.05%, for the observed data and wR2 = 8.07% for all data. The goodness-of-fit was 1.070. The largest peak in the final difference electron density synthesis was 0.311 $e^-/Å^3$ and the largest hole was -0.283 $e^-/Å^3$ with an RMS deviation of 0.086 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.684 $g/cm^3$ and F(000), 448 $e^-$.

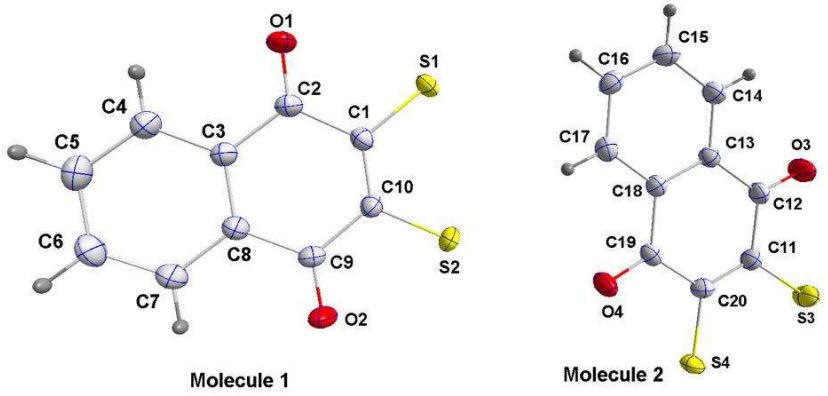


**Fig. 3.**The asymmetric unit of **12** with labeling scheme and 50% probability ellipsoids

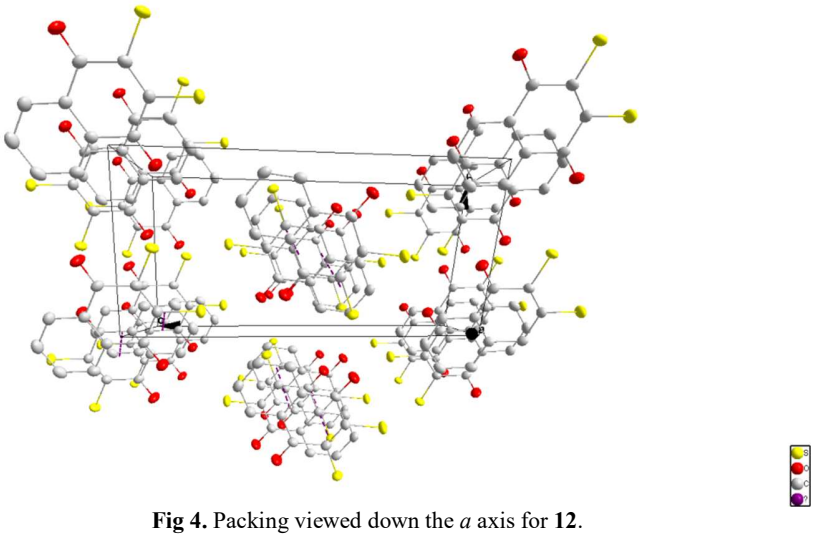

**Fig 4.** Packing viewed down the *a* axis for **12**.

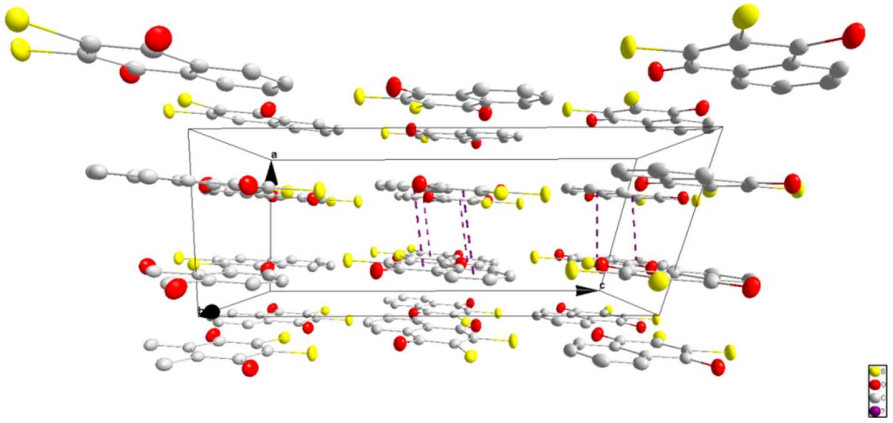

**Fig 5.** Packing viewed down the *b* axis. Selected examples of the π-stacking interactions are shown by dotted lines.

**Table 3. Sample and crystal data for 12.**

| | | |
|---|---|---|
| **Identification code** | 12 | |
| **Chemical formula** | $C_{10}H_4O_2S_2$ | |
| **Formula weight** | 220.25 g/mol | |
| **Temperature** | 150(2) K | |
| **Wavelength** | 1.54178 Å | |
| **Crystal size** | 0.114 x 0.156 x 0.268 mm | |
| **Crystal habit** | colorless block | |
| **Crystal system** | Triclinic | |
| **Space group** | P -1 | |
| **Unit cell dimensions** | a = 7.2036(2) Å | α = 89.755(2)° |
| | b = 8.2321(2) Å | β = 82.4190(10)° |
| | c = 16.1168(4) Å | γ = 66.6960(10)° |
| **Volume** | 868.89(4) $Å^3$ | |
| **Z** | 4 | |
| **Density (calculated)** | 1.684 $g/cm^3$ | |
| **Absorption coefficient** | 5.271 $mm^{-1}$ | |
| **F(000)** | 448 | |

**Table 4. Bond lengths (Å) for 12.**

| | | | |
|---|---|---|---|
| S1-C1 | 1.7111(18) | S2-C10 | 1.7115(18) |
| O1-C2 | 1.218(2) | O2-C9 | 1.212(2) |
| C1-C10 | 1.339(3) | C1-C2 | 1.503(3) |
| C2-C3 | 1.486(3) | C3-C4 | 1.395(3) |
| C3-C8 | 1.395(3) | C4-C5 | 1.387(3) |
| C4-H4 | 0.95 | C5-C6 | 1.388(3) |
| C5-H5 | 0.95 | C6-C7 | 1.387(3) |
| C6-H6 | 0.95 | C7-C8 | 1.392(3) |
| C7-H7 | 0.95 | C8-C9 | 1.488(3) |

| | | | |
|---|---|---|---|
| C9-C10 | 1.492(3) | S3-C11 | 1.7102(18) |
| S4-C20 | 1.7096(19) | O3-C12 | 1.213(2) |
| O4-C19 | 1.218(2) | C11-C20 | 1.337(3) |
| C11-C12 | 1.493(3) | C12-C13 | 1.486(3) |
| C13-C14 | 1.395(3) | C13-C18 | 1.400(3) |
| C14-C15 | 1.384(3) | C14-H14 | 0.95 |
| C15-C16 | 1.386(3) | C15-H15 | 0.95 |
| C16-C17 | 1.392(3) | C16-H16 | 0.95 |
| C17-C18 | 1.391(3) | C17-H17 | 0.95 |
| C18-C19 | 1.484(3) | C19-C20 | 1.500(3) |

## Table 11: Data collection details for 12.

| Axis | dx/mm | 2θ/° | ω/° | φ/° | χ/° | Width/° | Frames | Time/s | Wavelength/Å | Voltage/kV | Current/mA | Temperature/K |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Omega | 39.983 | 85.20 | -87.30 | 71.17 | 54.74 | 0.50 | 330 | 10.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.983 | 104.95 | -66.55 | -160.00 | 54.74 | 0.50 | 326 | 10.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.983 | 104.95 | -66.55 | -40.00 | 54.74 | 0.50 | 326 | 10.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.983 | 4.97 | -166.53 | 0.00 | 54.74 | 0.50 | 326 | 10.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.983 | 104.95 | -66.55 | -80.00 | 54.74 | 0.50 | 326 | 10.00 | 1.54184 | 50 | 1.0 | n/a |
| Omega | 39.983 | 104.95 | -66.55 | 160.00 | 54.74 | 0.50 | 326 | 10.00 | 1.54184 | 50 | 1.0 | n/a |

**Table 12. Data collection and structure refinement for 12.**

| | |
|---|---|
| **Diffractometer** | Bruker D8 VENTURE PHOTON 100 CMOS |
| **Radiation source** | 'INCOATEC IμS micro-focus source', Cu |
| **Theta range for data collection** | 2.77 to 72.45° |
| **Index ranges** | -8<=h<=8, -10<=k<=9, -19<=l<=19 |
| **Reflections collected** | 6695 |
| **Independent reflections** | 3235 [R(int) = 0.0219] |
| **Coverage of independent reflections** | 93.9% |
| **Absorption correction** | multi-scan |
| **Max. and min. transmission** | 0.5850 and 0.3320 |
| **Structure solution technique** | direct methods |
| **Structure solution program** | *SHELXT* (Bruker, 2014) |
| **Refinement method** | Full-matrix least-squares on $F^2$ |
| **Refinement program** | SHELXL-2014/7 (Sheldrick, 2014) |
| **Function minimized** | $\Sigma\, w(F_o^2 - F_c^2)^2$ |
| **Data / restraints / parameters** | 3235 / 0 / 253 |
| **Goodness-of-fit on $F^2$** | 1.070 |
| **$\Delta/\sigma_{max}$** | 0.001 |
| **Final R indices** | 2787 data; I>2σ(I) R1 = 0.0305, wR2 = 0.0770 |
| | all data R1 = 0.0371, wR2 = 0.0807 |
| **Weighting scheme** | $w=1/[\sigma^2(F_o^2)+(0.0422P)^2+0.2615P]$ where $P=(F_o^2+2F_c^2)/3$ |
| **Largest diff. peak and hole** | 0.311 and -0.283 eÅ$^{-3}$ |
| **R.M.S. deviation from mean** | 0.086 eÅ$^{-3}$ |

 

**Table 13. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å$^2$) for 12.**

U(eq) is defined as one third of the trace of the orthogonalized$U_{ij}$ tensor.

| | x/a | y/b | z/c | U(eq) |
|---|---|---|---|---|
| S1 | 0.73394(7) | 0.51591(6) | 0.11937(3) | 0.02659(13) |
| S2 | 0.71335(8) | 0.83698(7) | 0.23298(3) | 0.03018(13) |
| O1 | 0.7713(2) | 0.58215(18) | 0.94256(9) | 0.0320(3) |
| O2 | 0.7145(2) | 0.13325(18) | 0.13554(9) | 0.0313(3) |
| C1 | 0.7429(3) | 0.7051(2) | 0.07825(12) | 0.0226(4) |
| C2 | 0.7617(3) | 0.7089(2) | 0.98431(12) | 0.0226(4) |
| C3 | 0.7678(2) | 0.8724(2) | 0.94701(11) | 0.0208(4) |
| C4 | 0.7882(3) | 0.8819(3) | 0.86007(12) | 0.0263(4) |
| C5 | 0.7919(3) | 0.0342(3) | 0.82418(13) | 0.0297(4) |
| C6 | 0.7738(3) | 0.1776(3) | 0.87448(13) | 0.0283(4) |
| C7 | 0.7527(3) | 0.1695(2) | 0.96103(13) | 0.0255(4) |
| C8 | 0.7508(2) | 0.0164(2) | 0.99741(12) | 0.0208(4) |
| C9 | 0.7313(3) | 0.0096(2) | 0.09029(12) | 0.0221(4) |
| C10 | 0.7317(3) | 0.8422(2) | 0.12612(11) | 0.0227(4) |
| S3 | 0.71122(8) | 0.54038(7) | 0.73049(3) | 0.03102(14) |
| S4 | 0.69562(7) | 0.85409(6) | 0.61434(3) | 0.02472(12) |
| O3 | 0.7529(2) | 0.22621(18) | 0.63572(9) | 0.0342(3) |
| O4 | 0.7547(2) | 0.75039(18) | 0.43776(9) | 0.0324(3) |
| C11 | 0.7296(3) | 0.5190(2) | 0.62388(11) | 0.0230(4) |
| C12 | 0.7510(3) | 0.3433(2) | 0.58950(12) | 0.0228(4) |
| C13 | 0.7663(2) | 0.3219(2) | 0.49700(11) | 0.0207(4) |
| C14 | 0.7852(3) | 0.1609(2) | 0.46148(12) | 0.0250(4) |
| C15 | 0.7996(3) | 0.1381(3) | 0.37549(13) | 0.0271(4) |
| C16 | 0.7938(3) | 0.2752(3) | 0.32429(12) | 0.0272(4) |
| C17 | 0.7767(3) | 0.4359(3) | 0.35895(12) | 0.0259(4) |
| C18 | 0.7631(2) | 0.4597(2) | 0.44526(11) | 0.0205(4) |
| C19 | 0.7463(2) | 0.6314(2) | 0.48124(12) | 0.0215(4) |
| C20 | 0.7239(3) | 0.6519(2) | 0.57490(12) | 0.0218(4) |

**Table 14. Bond angles (°) for 12.**

| | | | |
|---|---|---|---|
| C10-C1-C2 | 121.81(17) | C10-C1-S1 | 122.62(15) |
| C2-C1-S1 | 115.57(13) | O1-C2-C3 | 123.12(18) |
| O1-C2-C1 | 120.20(17) | C3-C2-C1 | 116.68(16) |
| C4-C3-C8 | 119.78(18) | C4-C3-C2 | 119.05(17) |
| C8-C3-C2 | 121.17(17) | C5-C4-C3 | 119.85(19) |
| C5-C4-H4 | 120.1 | C3-C4-H4 | 120.1 |
| C4-C5-C6 | 120.26(19) | C4-C5-H5 | 119.9 |
| C6-C5-H5 | 119.9 | C7-C6-C5 | 120.26(18) |

 

| | | | |
|---|---|---|---|
| C7-C6-H6 | 119.9 | C5-C6-H6 | 119.9 |
| C6-C7-C8 | 119.76(18) | C6-C7-H7 | 120.1 |
| C8-C7-H7 | 120.1 | C7-C8-C3 | 120.09(18) |
| C7-C8-C9 | 118.81(17) | C3-C8-C9 | 121.10(17) |
| O2-C9-C8 | 122.45(17) | O2-C9-C10 | 120.81(17) |
| C8-C9-C10 | 116.73(16) | C1-C10-C9 | 122.47(17) |
| C1-C10-S2 | 121.86(15) | C9-C10-S2 | 115.66(14) |
| C20-C11-C12 | 122.46(17) | C20-C11-S3 | 121.85(15) |
| C12-C11-S3 | 115.69(14) | O3-C12-C13 | 122.47(17) |
| O3-C12-C11 | 120.86(17) | C13-C12-C11 | 116.66(16) |
| C14-C13-C18 | 119.71(17) | C14-C13-C12 | 119.12(17) |
| C18-C13-C12 | 121.17(16) | C15-C14-C13 | 120.05(18) |
| C15-C14-H14 | 120.0 | C13-C14-H14 | 120.0 |
| C14-C15-C16 | 120.30(18) | C14-C15-H15 | 119.9 |
| C16-C15-H15 | 119.9 | C15-C16-C17 | 120.15(18) |
| C15-C16-H16 | 119.9 | C17-C16-H16 | 119.9 |
| C18-C17-C16 | 119.94(18) | C18-C17-H17 | 120.0 |
| C16-C17-H17 | 120.0 | C17-C18-C13 | 119.83(17) |
| C17-C18-C19 | 119.31(17) | C13-C18-C19 | 120.86(16) |
| O4-C19-C18 | 122.52(17) | O4-C19-C20 | 120.55(17) |
| C18-C19-C20 | 116.91(15) | C11-C20-C19 | 121.87(17) |
| C11-C20-S4 | 122.48(15) | C19-C20-S4 | 115.64(13) |

**Table 15. Torsion angles (°) for 12.**

| | | | |
|---|---|---|---|
| C10-C1-C2-O1 | -179.87(17) | S1-C1-C2-O1 | -0.6(2) |
| C10-C1-C2-C3 | 0.2(2) | S1-C1-C2-C3 | 179.42(12) |
| O1-C2-C3-C4 | -0.9(3) | C1-C2-C3-C4 | 179.02(15) |
| O1-C2-C3-C8 | 178.23(17) | C1-C2-C3-C8 | -1.8(2) |
| C8-C3-C4-C5 | 0.1(3) | C2-C3-C4-C5 | 179.27(16) |
| C3-C4-C5-C6 | -0.4(3) | C4-C5-C6-C7 | 0.2(3) |
| C5-C6-C7-C8 | 0.3(3) | C6-C7-C8-C3 | -0.7(3) |
| C6-C7-C8-C9 | 179.06(16) | C4-C3-C8-C7 | 0.5(3) |
| C2-C3-C8-C7 | -178.69(15) | C4-C3-C8-C9 | -179.26(15) |
| C2-C3-C8-C9 | 1.6(3) | C7-C8-C9-O2 | 0.9(3) |
| C3-C8-C9-O2 | -179.34(16) | C7-C8-C9-C10 | -179.51(15) |
| C3-C8-C9-C10 | 0.2(2) | C2-C1-C10-C9 | 1.7(3) |
| S1-C1-C10-C9 | -177.51(13) | C2-C1-C10-S2 | -179.22(13) |
| S1-C1-C10-S2 | 1.6(2) | O2-C9-C10-C1 | 177.68(17) |
| C8-C9-C10-C1 | -1.9(3) | O2-C9-C10-S2 | -1.5(2) |
| C8-C9-C10-S2 | 178.95(12) | C20-C11-C12-O3 | -178.99(17) |
| S3-C11-C12-O3 | 0.6(2) | C20-C11-C12-C13 | 0.1(3) |
| S3-C11-C12-C13 | 179.73(12) | O3-C12-C13-C14 | -0.5(3) |
| C11-C12-C13-C14 | -179.58(15) | O3-C12-C13-C18 | 179.80(17) |
| C11-C12-C13-C18 | 0.7(2) | C18-C13-C14-C15 | -0.3(3) |
| C12-C13-C14-C15 | 179.98(16) | C13-C14-C15-C16 | -0.5(3) |
| C14-C15-C16-C17 | 1.0(3) | C15-C16-C17-C18 | -0.7(3) |
| C16-C17-C18-C13 | -0.1(3) | C16-C17-C18-C19 | 179.57(16) |

 

| | | | |
|---|---|---|---|
| C14-C13-C18-C17 | 0.6(2) | C12-C13-C18-C17 | -179.69(16) |
| C14-C13-C18-C19 | -179.05(15) | C12-C13-C18-C19 | 0.6(2) |
| C17-C18-C19-O4 | -3.9(3) | C13-C18-C19-O4 | 175.72(16) |
| C17-C18-C19-C20 | 177.69(15) | C13-C18-C19-C20 | -2.6(2) |
| C12-C11-C20-C19 | -2.2(3) | S3-C11-C20-C19 | 178.14(13) |
| C12-C11-C20-S4 | 179.00(13) | S3-C11-C20-S4 | -0.6(2) |
| O4-C19-C20-C11 | -174.91(17) | C18-C19-C20-C11 | 3.5(2) |
| O4-C19-C20-S4 | 3.9(2) | C18-C19-C20-S4 | -177.68(12) |

**Table 16. Anisotropic atomic displacement parameters (Å$^2$) for 12.**

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12} ]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0344(2) | 0.0179(2) | 0.0281(3) | 0.00452(19) | -0.00212(19) | -0.01187(19) |
| S2 | 0.0458(3) | 0.0305(3) | 0.0171(2) | 0.0021(2) | -0.00650(19) | -0.0177(2) |
| O1 | 0.0441(8) | 0.0252(7) | 0.0293(7) | -0.0071(6) | 0.0014(6) | -0.0185(6) |
| O2 | 0.0435(8) | 0.0253(7) | 0.0281(7) | -0.0041(6) | -0.0072(6) | -0.0161(6) |
| C1 | 0.0224(8) | 0.0192(9) | 0.0252(10) | 0.0017(8) | -0.0017(7) | -0.0077(7) |
| C2 | 0.0208(8) | 0.0217(9) | 0.0251(10) | -0.0037(8) | -0.0015(7) | -0.0086(7) |
| C3 | 0.0184(8) | 0.0212(9) | 0.0225(9) | -0.0009(8) | -0.0020(7) | -0.0077(7) |
| C4 | 0.0262(9) | 0.0285(10) | 0.0232(9) | -0.0015(8) | -0.0021(7) | -0.0102(8) |
| C5 | 0.0267(9) | 0.0371(11) | 0.0234(10) | 0.0058(9) | -0.0025(7) | -0.0111(8) |
| C6 | 0.0251(9) | 0.0263(10) | 0.0335(11) | 0.0097(9) | -0.0049(8) | -0.0100(8) |
| C7 | 0.0246(8) | 0.0214(9) | 0.0313(10) | 0.0014(8) | -0.0047(8) | -0.0096(7) |
| C8 | 0.0185(7) | 0.0203(9) | 0.0238(9) | 0.0012(8) | -0.0038(7) | -0.0076(7) |
| C9 | 0.0218(8) | 0.0198(9) | 0.0244(9) | -0.0027(8) | -0.0042(7) | -0.0078(7) |
| C10 | 0.0241(8) | 0.0231(9) | 0.0199(9) | 0.0000(8) | -0.0027(7) | -0.0085(7) |
| S3 | 0.0493(3) | 0.0315(3) | 0.0169(2) | 0.0024(2) | -0.0067(2) | -0.0203(2) |
| S4 | 0.0307(2) | 0.0184(2) | 0.0264(2) | -0.00299(19) | -0.00234(18) | -0.01175(18) |
| O3 | 0.0514(9) | 0.0261(7) | 0.0270(7) | 0.0083(6) | -0.0058(6) | -0.0177(7) |
| O4 | 0.0464(8) | 0.0245(7) | 0.0290(7) | 0.0068(6) | -0.0041(6) | -0.0174(6) |
| C11 | 0.0250(8) | 0.0242(9) | 0.0204(9) | 0.0009(8) | -0.0042(7) | -0.0101(7) |
| C12 | 0.0237(8) | 0.0207(9) | 0.0243(9) | 0.0057(8) | -0.0049(7) | -0.0086(7) |
| C13 | 0.0193(8) | 0.0197(9) | 0.0233(9) | 0.0021(8) | -0.0033(7) | -0.0077(7) |
| C14 | 0.0237(8) | 0.0211(9) | 0.0304(10) | 0.0025(8) | -0.0041(8) | -0.0092(7) |
| C15 | 0.0250(8) | 0.0243(9) | 0.0316(10) | -0.0054(8) | -0.0034(8) | -0.0097(8) |
| C16 | 0.0257(9) | 0.0303(10) | 0.0230(10) | -0.0035(8) | -0.0031(7) | -0.0086(8) |
| C17 | 0.0257(9) | 0.0253(10) | 0.0253(10) | 0.0041(8) | -0.0035(7) | -0.0088(8) |
| C18 | 0.0185(8) | 0.0206(9) | 0.0219(9) | 0.0016(8) | -0.0025(7) | -0.0073(7) |
| C19 | 0.0203(8) | 0.0193(9) | 0.0253(9) | 0.0043(8) | -0.0024(7) | -0.0087(7) |
| C20 | 0.0198(8) | 0.0212(9) | 0.0243(9) | -0.0022(8) | -0.0014(7) | -0.0086(7) |

**Table 17. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å$^2$) for 12.**

 

| | x/a | y/b | z/c | U(eq) |
|---|---|---|---|---|
| H4 | 0.7997 | 0.7843 | -0.1745 | 0.032 |
| H5 | 0.8068 | 1.0404 | -0.2351 | 0.036 |
| H6 | 0.7759 | 1.2818 | -0.1505 | 0.034 |
| H7 | 0.7397 | 1.2680 | -0.0047 | 0.031 |
| H14 | 0.7881 | 0.0668 | 0.4964 | 0.03 |
| H15 | 0.8135 | 0.0280 | 0.3515 | 0.033 |
| H16 | 0.8015 | 0.2594 | 0.2654 | 0.033 |
| H17 | 0.7743 | 0.5293 | 0.3237 | 0.031 |