

## 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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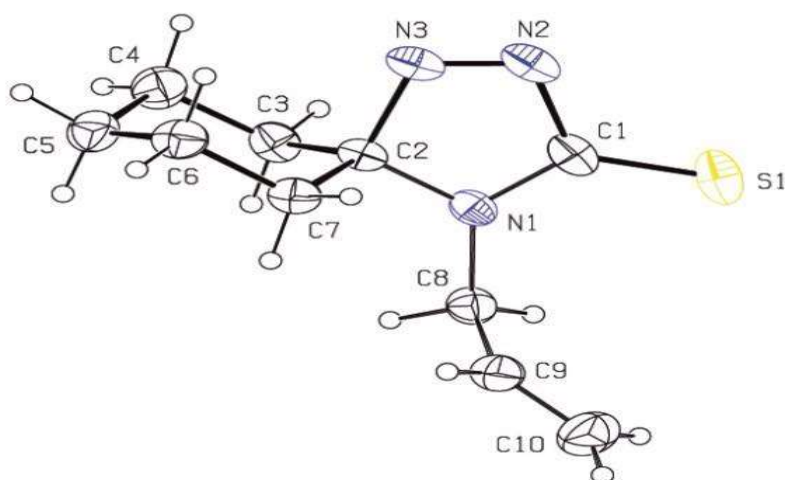
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#### Crystal Structure Report for 7h

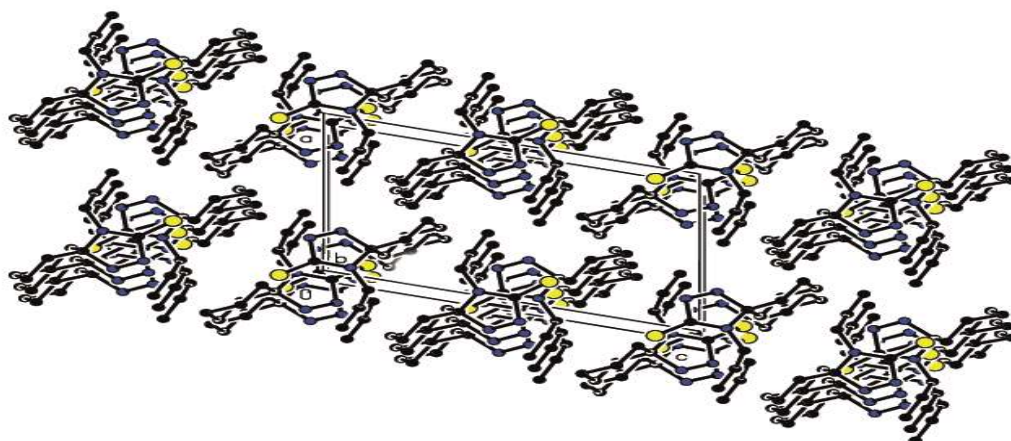
A yellow plate-like specimen of C<sub>13</sub>H<sub>21</sub>N<sub>3</sub>S, approximate dimensions 0.074 mm x 0.197 mm x 0.281 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker D8 VENTURE PHOTON 100 CMOS system equipped with a mirror monochromator and a Cu-K $\alpha$  INCOATEC I $\mu$ S micro--focus source ( $\lambda = 1.54178 \text{ \AA}$ ).

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  $\theta$  angle of 72.37° (0.81  $\text{\AA}$  resolution), of which 2192 were independent (average redundancy 3.868, completeness = 97.5%,  $R_{\text{int}} = 2.52\%$ ,  $R_{\text{sig}} = 2.33\%$ ) and 1945 (88.73%) were greater than  $2\sigma(F^2)$ . The final cell constants of  $a = 9.1362(3) \text{ \AA}$ ,  $b = 9.6025(3) \text{ \AA}$ ,  $c = 13.3880(4) \text{ \AA}$ ,  $\beta = 105.3260(10)^\circ$ , volume = 1132.76(6)  $\text{\AA}^3$ , are based upon the refinement of the XYZ-centroids of 6352 reflections above  $20 \sigma(I)$  with  $9.209^\circ < 2\theta < 144.7^\circ$ . 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  $0.590 \text{ e}/\text{\AA}^3$  and the largest hole was  $-0.536 \text{ e}/\text{\AA}^3$  with an RMS deviation of  $0.063 \text{ e}/\text{\AA}^3$ . On the basis of the final model, the calculated density was  $1.474 \text{ g}/\text{cm}^3$  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.**

<b>Identification code</b>	<b>7h</b>	
<b>Chemical formula</b>	C <sub>13</sub> H <sub>21</sub> N <sub>3</sub> S	
<b>Formula weight</b>	251.39 g/mol	
<b>Temperature</b>	225(2) K	
<b>Wavelength</b>	1.54178 Å	
<b>Crystal size</b>	0.074 x 0.197 x 0.281 mm	
<b>Crystal habit</b>	yellow plate	
<b>Crystal system</b>	monoclinic	
<b>Space group</b>	P 1 21/c 1	
<b>Unit cell dimensions</b>	a = 9.1362(3) Å	α = 90°
	b = 9.6025(3) Å	β = 105.3260(10)°
	c = 13.3880(4) Å	γ = 90°
<b>Volume</b>	1132.76(6) Å <sup>3</sup>	
<b>Z</b>	4	
<b>Density (calculated)</b>	1.474 g/cm <sup>3</sup>	
<b>Absorption coefficient</b>	2.356 mm <sup>-1</sup>	
<b>F(000)</b>	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	2 $\theta$ / $^{\circ}$	$\omega$ / $^{\circ}$	$\varphi$ / $^{\circ}$	$\chi$ / $^{\circ}$	Width/ $^{\circ}$	Frames	Time/s	Wavelength/ $\text{\AA}$	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.**

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

<b>Goodness-of-fit on F<sup>2</sup></b>	1.097	
<b>Final R indices</b>	1945 data;	R1 = 0.0605, wR2 =
	I > 2σ(I)	0.1968
	all data	R1 = 0.0651, wR2 =
		0.2035
<b>Weighting scheme</b>	w = 1/[σ <sup>2</sup> (F <sub>o</sub> <sup>2</sup> ) + (0.1234P) <sup>2</sup> + 0.5208P] where P = (F <sub>o</sub> <sup>2</sup> + 2F <sub>c</sub> <sup>2</sup> )/3	
<b>Largest diff. peak and hole</b>	0.590 and -0.536 eÅ <sup>-3</sup>	
<b>R.M.S. deviation from mean</b>	0.063 eÅ <sup>-3</sup>	

**Table 7. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å<sup>2</sup>) for 7h.**

U(eq) is defined as one third of the trace of the orthogonalized U<sub>ij</sub> 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(4)	C2-N1-C10A-C11A	-117.3(3)
N1-C10A-C11A-C12A	121.3(3)		

**Table 9. Anisotropic atomic displacement parameters ( $\text{\AA}^2$ ) for 7h.**

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

	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
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 ( $\text{\AA}^2$ ) 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 I $\mu$ S micro-focus source' ( $\lambda = 1.54178 \text{ \AA}$ ).

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  $\theta$  angle of  $72.45^\circ$  (0.81  $\text{\AA}$  resolution), of which 3235 were independent (average redundancy 2.070, completeness = 93.9%,  $R_{\text{int}} = 2.19\%$ ,  $R_{\text{sig}} = 2.90\%$ ) and 2787 (86.15%) were greater than  $2\sigma(F^2)$ . The final cell constants of  $a = 7.2036(2) \text{ \AA}$ ,  $b = 8.2321(2) \text{ \AA}$ ,  $c = 16.1168(4) \text{ \AA}$ ,  $\alpha = 89.755(2)^\circ$ ,  $\beta = 82.4190(10)^\circ$ ,  $\gamma = 66.6960(10)^\circ$ , volume =  $868.89(4) \text{ \AA}^3$ , are based upon the refinement of the XYZ-centroids of 5161 reflections above  $20 \sigma(I)$  with  $5.539^\circ < 2\theta < 144.9^\circ$ . 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  $P\bar{1}$ , 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  $0.311 \text{ e}^-/\text{\AA}^3$  and the largest hole was  $-0.283 \text{ e}^-/\text{\AA}^3$  with an RMS deviation of  $0.086 \text{ e}^-/\text{\AA}^3$ . On the basis of the final model, the calculated density was  $1.684 \text{ g/cm}^3$  and  $F(000)$ , 448  $e^-$ .

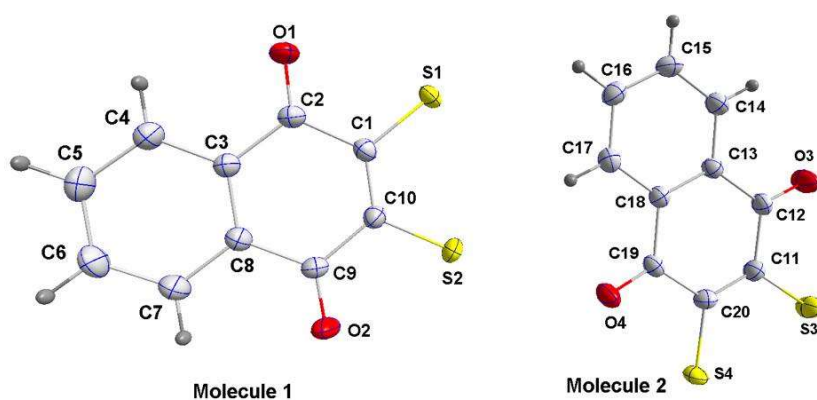


Fig. 3. The asymmetric unit of **12** with labeling scheme and 50% probability ellipsoids

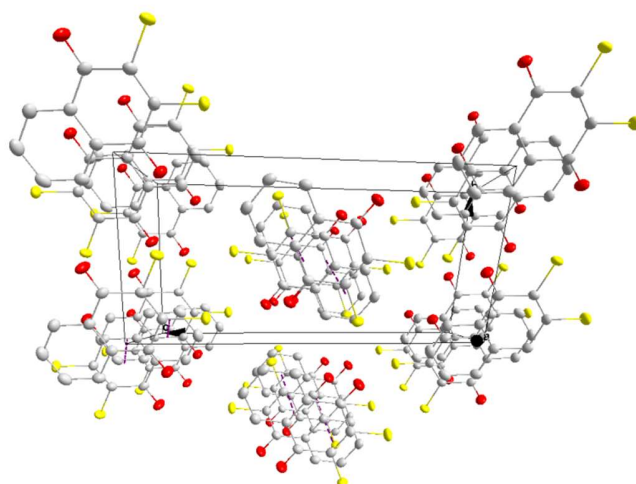
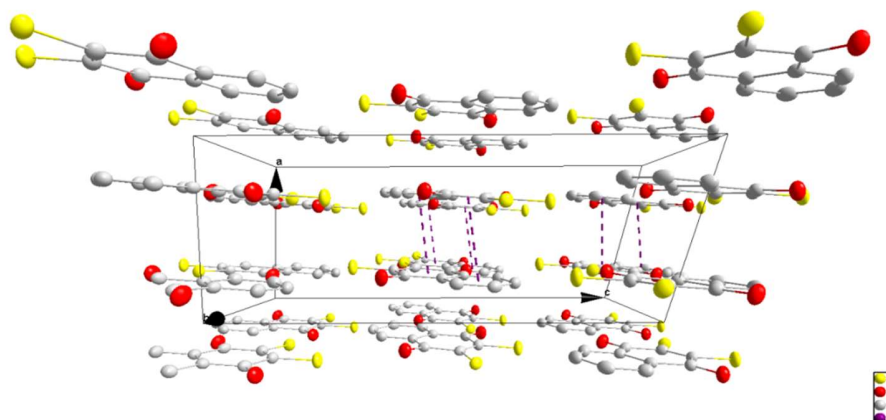


Fig 4. Packing viewed down the  $a$  axis for **12**.





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

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

<b>Identification code</b>	12	
<b>Chemical formula</b>	C <sub>10</sub> H <sub>4</sub> O <sub>2</sub> S <sub>2</sub>	
<b>Formula weight</b>	220.25 g/mol	
<b>Temperature</b>	150(2) K	
<b>Wavelength</b>	1.54178 Å	
<b>Crystal size</b>	0.114 x 0.156 x 0.268 mm	
<b>Crystal habit</b>	colorless block	
<b>Crystal system</b>	Triclinic	
<b>Space group</b>	P -1	
<b>Unit cell dimensions</b>	a = 7.2036(2) Å	$\alpha$ = 89.755(2)°
	b = 8.2321(2) Å	$\beta$ = 82.4190(10)°
	c = 16.1168(4) Å	$\gamma$ = 66.6960(10)°
<b>Volume</b>	868.89(4) Å <sup>3</sup>	
<b>Z</b>	4	
<b>Density (calculated)</b>	1.684 g/cm <sup>3</sup>	
<b>Absorption coefficient</b>	5.271 mm <sup>-1</sup>	
<b>F(000)</b>	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	2 $\theta$ / $^{\circ}$	$\omega$ / $^{\circ}$	$\phi$ / $^{\circ}$	$\chi$ / $^{\circ}$	Width/ $^{\circ}$	Frames	Time/s	Wavelength/ $\text{\AA}$	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.**

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

**Table 13. Atomic coordinates and equivalent isotropic atomic displacement parameters ( $\text{\AA}^2$ ) 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 ( $^\circ$ ) 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 ( $\text{\AA}^2$ ) for 12.**

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

	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
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 ( $\text{\AA}^2$ ) for 12.**

	<b>x/a</b>	<b>y/b</b>	<b>z/c</b>	<b>U(eq)</b>
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