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**Pankaj Bandhoria**  
 Government Gandhi Memorial  
 Science College Jammu,  
 Jammu and Kashmir, India

**Priyanka Kanwal**  
 University of Jammu, Jammu  
 and Kashmir, India

**Satinder Kumari Juneja**  
 Government College for  
 Women, Parade Ground  
 Jammu, Jammu and Kashmir,  
 India

**Aran Kumar**  
 Government Degree College  
 Boys Udhampur Jammu,  
 Jammu and Kashmir, India

**Renu Rajput**  
 Government College for  
 Women, Gandhi Nagar  
 Jammu, Jammu and Kashmir,  
 India

**Sanjay Kumar**  
 Government Degree College,  
 Bhaderwah, Jammu and  
 Kashmir, India

**Corresponding Author:**  
**Priyanka Kanwal**  
 University of Jammu, Jammu  
 and Kashmir, India

## Single Crystal X-Ray crystallographic study of 2, 2-Diiodo-1-phenylvinyl (methyl) sulfane

**Pankaj Bandhoria, Priyanka Kanwal, Satinder Kumari Juneja, Aran Kumar, Renu Rajput and Sanjay Kumar**

### Abstract

The title compound, 2,2-diiodo-1-phenylvinyl(methyl)sulfane (C<sub>9</sub>H<sub>8</sub>I<sub>2</sub>S) crystallized in the monoclinic crystal system with space group P2<sub>1</sub>/n having unit cell parameters: a = 8.6397(3), b = 13.1631(5), c = 20.8706(7) Å, β = 97.617(3)° and Z = 8. A well-defined single crystal was chosen for data collection. X-ray intensity data of 7796 reflections (of which 4141 were unique) were collected using CuKα radiation. The crystal structure was solved by direct methods and refined by full-matrix least-squares procedures to a final R- value of 0.0533 for 3441 observed reflections. There are two independent molecules per asymmetric unit. The experimental crystal F(000) value is 1472. The goodness of fit is 1.029. The phenyl ring is perfectly planar. Maximum shift to e.s.d ratio is 0.001. The crystal structure is stabilized by van der waals interactions.

**Keywords:** Crystal structure, direct methods, van der waals interactions, monoclinic crystal system.

### 1. Introduction

Organosulfur compounds contain C–S–C linkage in them and are essential for plants and animals [1]. Currently, most medicinal garlic products are manufactured from the extracts (e.g., garlic oil) which contain organosulfur compounds [2, 4]. These organosulfur compounds contain one or more sulfur atoms bonded with carbon, which is the basis for their biological activities, including antimicrobial, antioxidant, antitumor and antiasthmatic activities [5]. Fossil fuels, coal, petroleum, and natural gas, which are derived from ancient organisms, necessarily contain organosulfur compounds, the removal of which is a major focus of oil refineries. Sulfides, formerly known as thioethers, are characterized by C–S–C bonds [6, 7]. Relative to C–C bonds, C–S bond are both longer, because S is larger than carbon, and about 10% weaker. Representative bond lengths in sulfur compounds are 183 pm for the S–C single bond in methanethiol and 173 pm in thiophene. The C–S bond dissociation energy for thiomethane is 89 kcal/mol (370 kJ/mol) compared to methane's 100 kcal/mol (420 kJ/mol) and when hydrogen is replaced by a methyl group the energy decreases to 73 kcal/mol (305 kJ/mol)[8]. The single carbon to oxygen bond is shorter than that of the C–C bond. The bond dissociation energies for dimethyl sulfide and dimethyl ether are respectively 73 and 77 kcal/mol (305 and 322 kJ/mol).

### Experimental

White transparent rectangular shaped single crystals of the title compound (Figure 1) were grown at room temperature from a 2:1 mixture of ethyl acetate and methanol by slow evaporation technique. The crystals were found to be monoclinic. The space group was determined to be P2<sub>1</sub>/n from the systematic absences 0k0: k = 2n + 1. X-ray intensity data were collected on an XCALIBUR CCD area-detector diffractometer equipped with graphite monochromated CuKα radiation (λ = 1.5418 Å). The crystal used for data collection was of dimensions 0.3 X 0.2 X 0.2 mm. The intensities were measured by ω scan mode for q ranges 3.98 to 66.92° with h k l values -7 ≤ h ≤ 10, -14 ≤ k ≤ 15, -23 ≤ l ≤ 24. 3441 reflections were treated as observed using (I > 2σ(I)) as criterion with R<sub>int</sub> = 0.0491. Data were corrected for Lorentz, polarization and absorption corrections.

The structure solution was obtained by employing direct methods using SHELX97 software [9] with the best E map revealing positions for all non-hydrogen atoms.

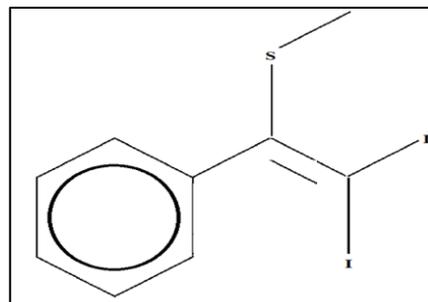
The statistical analysis of ABS ( $E^2-1$ ) indicated overall distribution of intensities to be centric [0.785 (0 k l); 1.012 (h 0 l) 0.759 (h k 0)] ( $E$  represents the normalized structure factor). Reliability index for the normalized  $E$ -values ( $R_E$ ) was 0.179.

Full-matrix least-squares refinement was carried out using SHELXL97 software [10]. During anisotropic refinement of all the non-hydrogen atoms it was observed that there are two independent molecules per asymmetric unit. All H atoms were geometrically fixed and allowed to ride on the corresponding non-H atoms with  $C-H=0.93-0.96\text{\AA}$ , and  $U_{iso}(H)=1.5U_{eq}(C)$  of the attached C atom for methyl H atoms and  $1.2 U_{eq}$  for other H atoms. The final refinement cycles converged  $R = 0.0533$  and  $wR(F^2) = 0.1491$ . The final weighting scheme was  $w=[1/[s^2(F_o)^2+(0.0853P)^2+4.2229P]]$  where  $P=[F_o^2+2F_c^2]/3$ . Maximum shift to e.s.d. ratio for all atoms in the final cycle was 0.001. Atomic scattering factors were taken from International Tables for X-ray Crystallography (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4). The crystallographic data are summarized in Table 1.

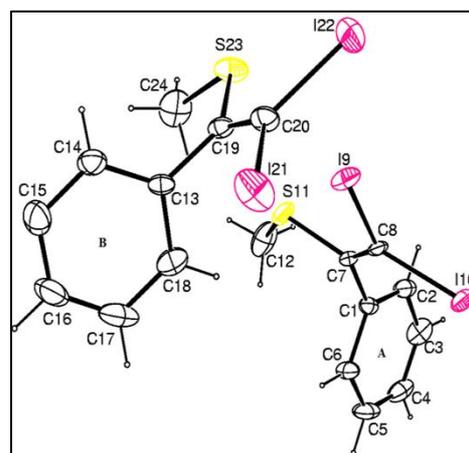
### Results and discussion

The final atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms are listed in Table 2. Bond distances and bond angles, and torsion angles for non-hydrogen atoms are presented in Tables 3 and 4, respectively. A general view of the molecule indicating atom numbering scheme (thermal ellipsoids drawn at 40% probability level) is shown in Figure 2. ORTEP-3 for Windows [11, 12] software was used for making the thermal ellipsoids. Geometrical calculations were performed using PLATON [13, 14] and PARST [15] software.

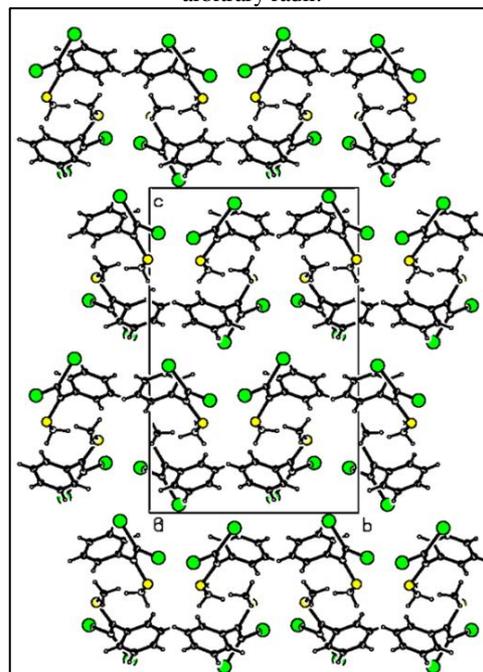
The asymmetric unit contains two independent molecules, A and B, with slightly different geometries (Table 2). The  $C7=C8$  bond length is found to be  $1.310(11)\text{\AA}$  in molecule A and  $1.312(12)\text{\AA}$  ( $C19=C20$ ) in molecule B. These values confirm the localization of double bond at this position. The C-C bond lengths in the phenyl ring range from  $1.336(18)$  to  $1.414(15)\text{\AA}$  in molecule A and from  $1.339(18)$  to  $1.397(14)\text{\AA}$  in molecule B. The modest differences between the corresponding values for the two independent molecules are sufficient to preclude the possibility of additional symmetry. In both the asymmetric molecules, the phenyl ring is perfectly planar with maximum deviation from their mean plane of  $-0.009(11)\text{\AA}$  for  $C3$  (molecule A) and  $0.011(12)\text{\AA}$  for  $C15$  (molecule B). The dihedral angle between the phenyl rings of molecule A and molecule B is  $34.7(3)^\circ$ . The  $S11-C12$  bond length is found to be  $1.788(14)\text{\AA}$  in molecule A and  $1.774(14)\text{\AA}$  ( $S23-C24$ ) in molecule B. Packing of the molecules in the unit cell down a-axis is shown in Figure 3. van der Waals interactions link molecules into dimers. The dimers are arranged in a manner to form layers. Within the layers, the dimers are arranged antiparallel to each other.



**Fig 1:** Chemical structure of (2, 2-diiodo-1 phenylvinyl) (methyl) sulfane



**Fig 2:** View of the molecule with displacement ellipsoids drawn at 40% Probability level. H atoms are shown as small spheres of arbitrary radii.



**Fig 3:** The packing arrangement of molecules viewed down the a-axis.

**Table 1:** Crystal and experimental data

Crystal description	White Transparent
Crystal size	0.3 X 0.2 X 0.2 mm
Formula weight	402.01
Radiation, Wavelength	Cu $\alpha$ , 1.5418 $\text{\AA}$
Unit cell dimensions	a=8.6397(3) b=13.1631(5) c=20.8706(7) $\text{\AA}$ $\beta=97.617(3)^\circ$
Crystal system	Monoclinic
Space group	P2 <sub>1</sub> /n

Unit cell volume	2352.57(14)Å <sup>3</sup>
Density (calculated)	1.201 Mg m <sup>-3</sup>
No. of molecules per unit cell, Z	8
Temperature	293(2) K
F(000)	1472
Reflections collected / unique	7796/ 4141
Reflections observed (I > 2σ(I))	3441
Rint	0.0434
Rsigma	0.0570
Structure determination	Direct methods
No. of parameters refined	219
Final R-factor	0.0533
wR(F <sup>2</sup> )	0.01491
Weight	[1/[s <sup>2</sup> (F <sub>o</sub> <sup>2</sup> )+(0.0853P) <sup>2</sup> +4.2229P] where P=[F <sub>o</sub> <sup>2</sup> +2F <sub>c</sub> <sup>2</sup> ]/3
Goodness-of-fit	1.029
(D / s) max	0.001

**Table 2:** Atomic coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>) for non-hydrogen atoms (e.s.d.'s are given in parenthesis)

Atom	x	y	z	Ueq*
I9	0.73091(7)	0.28917(5)	0.34630(3)	0.0443(2)
I10	0.65275(6)	0.09269(4)	0.45320(2)	0.0386(2)
S11	0.3683(3)	0.2546(2)	0.27606(12)	0.0537(8)
C1	0.3090(8)	0.1053(6)	0.3651(4)	0.0288(19)
C2	0.2902(12)	0.0069(8)	0.3428(4)	0.045(3)
C3	0.1806(13)	-0.0545(9)	0.3647(5)	0.053(3)
C4	0.0861(11)	-0.0211(10)	0.4071(5)	0.054(3)
C5	0.1025(11)	0.0737(10)	0.4298(5)	0.053(4)
C6	0.2141(10)	0.1415(8)	0.4102(4)	0.043(3)
C7	0.4265(9)	0.1764(6)	0.3437(3)	0.030(2)
C8	0.5695(9)	0.1844(6)	0.3733(3)	0.0282(19)
C12	0.1714(16)	0.2182(11)	0.2483(6)	0.073(4)
I21	0.37735(9)	0.14063(7)	0.02537(4)	0.0672(3)
I22	0.28396(9)	-0.04285(6)	0.13863(4)	0.0598(2)
S23	0.6303(3)	0.0083(2)	0.21972(11)	0.0488(7)
C20	0.4470(10)	0.0600(7)	0.1111(4)	0.038(3)
C13	0.6994(9)	0.1483(7)	0.1274(3)	0.032(2)
C14	0.7062(12)	0.2477(8)		0.1504(4)
C15	0.8153(15)	0.3146(9)	0.1316(5)	0.058(4)
C16	0.9124(15)	0.2863(11)	0.0899(6)	0.068(4)
C17	0.9109(15)	0.1892(12)	0.0658(6)	0.073(5)
C18	0.8046(14)	0.1209(10)	0.0850(6)	0.060(4)
C19	0.5828(10)	0.0747(6)	0.1461(4)	0.032(2)
C24	0.8295(16)	0.0381(10)	0.2445(5)	0.067(4)

**Table 3:** Bond distances (Å) and bond angles (°) for non-hydrogen atoms (e.s.d.'s are given in parentheses)

I9-C8	2.091(8)	C1-C2	1.379(13)
I10-C8	2.107(7)	C2-C3	1.369(15)
I21-C20	2.099(9)	C3-C4	1.354(15)
I22-C20	2.088(9)	C4-C5	1.336(18)
S11-C7	1.766(7)	C5-C6	1.414(15)
C20-C19	1.312(12)	C1-C6	1.411(12)
S11-C12	1.788(14)	C1-C7	1.492(11)
C13-C14	1.392(14)	C17-C18	1.382(19)
S23-C19	1.768(9)	C14-C15	1.384(16)
C13-C18	1.397(14)	C15-C16	1.339(18)
S23-C24	1.774(14)	C16-C17	1.370(16)
C13-C19	1.487(12)	C7-C8	1.310(11)
C7-S11-C12	105.1(5)	C19-S23-C24	104.4(5)
C2-C1-C7	122.9(7)	C6-C1-C7	117.8(7)
C2-C1-C6	119.3(8)	I21-C20-I22	115.8(4)
C1-C2-C3	119.9(9)	I21-C20-C19	122.3(7)
C2-C3-C4	122.0(11)	I22-C20-C19	121.9(6)
C3-C4-C5	119.4(10)	C14-C13-C18	117.4(9)
C4-C5-C6	122.1(9)	C14-C13-C19	121.4(7)
C1-C6-C5	117.3(9)	C18-C13-C19	121.2(9)
S11-C7-C8	118.7(6)	C13-C14-C15	120.0(9)

C1-C7-C8	123.1(7)	C14-C15-C16	121.1(11)
S11-C7-C1	118.2(5)	C15-C16-C17	121.4(13)
I9-C8-I10	114.9(3)	C16-C17-C18	118.3(12)
I10-C8-C7	121.9(6)	C13-C18-C17	121.9(12)
I9-C8-C7	123.1(5)	S23-C19-C20	119.9(7)

**Table 4:** Torsion angles ( $\circ$ ) for non-hydrogen atoms (e.s.d.'s are given in parentheses)

C12-S11-C7-C1	-2.9(8)	I21-C20-C19-C13	-1.0(12)
C12-S11-C7-C8	178.1(7)	I22-C20-C19-S23	-1.3(11)
C24-S23-C19-C20	172.6(8)	C19-C13-C14-C15	-179.4(9)
C24-S23-C19-C13	-9.2(8)	C14-C13-C19-S23	-84.6(9)
C6-C1-C2-C3	-0.2(13)	C14-C13-C18-C17	-0.6(16)
C6-C1-C7-S11	-89.1(8)	C19-C13-C18-C17	178.2(10)
C2-C1-C6-C5	-0.5(12)	C2-C3-C4-C5	-1.9(16)
C7-C1-C6-C5	179.6(8)	C2-C1-C7-S11	91.0(9)
C6-C1-C7-C8	89.9(10)	C7-C1-C2-C3	179.7(8)
C2-C1-C7-C8	-90.0(10)	C4-C5-C6-C1	0.1(14)
C1-C2-C3-C4	1.4(16)	C1-C7-C8-I10	2.4(10)
C3-C4-C5-C6	1.1(16)	S11-C7-C8-I10	-178.7(4)
S11-C7-C8-I9	2.2(9)	I21-C20-C19-S23	177.1(4)
C1-C7-C8-I9	-176.8(5)	I22-C20-C19-C13	-179.5(6)
C18-C13-C14-C15	-0.6(13)	C18-C13-C19-C20	-85.2(12)
C14-C13-C19-C20	93.5(10)	C18-C13-C19-S23	96.6(9)
C13-C14-C15-C16	2.0(16)	C14-C15-C16-C17	-2.1(19)
C15-C16-C17-C18	0.8(19)	C16-C17-C18-C13	0.5(19)

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