Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

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

Se(1)	−647 (3)	1501 (2)	1286 (1)	47 (1)
Se(2)	1925 (3)	2056 (1)	2218 (1)	44 (1)
Br(1)	5289 (3)	2319 (1)	3485 (1)	49 (1)
Br(2)	7611 (3)	1017 (1)	9353 (1)	47 (1)
C(1)	1471 (29)	775 (15)	720 (12)	49 (6)
N(1)	3053 (23)	1358 (12)	369 (10)	52 (5)
N(2)	1386 (23)	−215 (11)	571 (10)	48 (5)
C(2)	−409 (35)	−871 (15)	871 (15)	63 (7)
C(3)	2830 (39)	−741 (14)	21 (12)	57 (6)
C(4)	1475 (24)	808 (14)	2790 (11)	42 (5)
N(3)	−236 (23)	811 (11)	3238 (9)	47 (5)
N(4)	2783 (23)	8 (10)	2703 (10)	46 (5)
C(5)	2389 (28)	−939 (13)	3135 (10)	45 (5)
C(6)	4830 (31)	83 (17)	2278 (12)	59 (6)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Se(1)—Se(2)	2·384 (03)	Se(1)—C(1)	1·885 (19)
Se(2)—C(4)	1·929 (18)	C(1)—N(1)	1·373 (24)
C(1)—N(2)	1·311 (24)	N(2)—C(2)	1·482 (26)
N(2)—C(3)	1·479 (27)	C(4)—N(3)	1·309 (22)
C(4)—N(4)	1·316 (22)	N(4)—C(5)	1·467 (23)
N(4)—C(6)	1·458 (24)		
Se(2)—Se(1)—C(1)	94·1 (06)	Se(1)—Se(2)—C(4)	91·0 (05)
Se(1)—C(1)—N(1)	116·4 (13)	Se(1)—C(1)—N(2)	124·7 (14)
N(1)—C(1)—N(2)	118·5 (17)	C(1)—N(2)—C(2)	121·3 (16)
C(1)—N(2)—C(3)	124·1 (16)	C(2)—N(2)—C(3)	114·1 (15)
Se(2)—C(4)—N(3)	115·4 (13)	Se(2)—C(4)—N(4)	120·9 (13)
N(3)—C(4)—N(4)	123·6 (17)	C(4)—N(4)—C(5)	120·0 (15)
C(4)—N(4)—C(6)	121·6 (15)	C(5)—N(4)—C(6)	117·7 (14)

made with *SHELXTL-Plus* (Sheldrick, 1987). Scattering factors, f and f' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).* The refined coordinates and equivalent isotropic temperature factors for the structure are listed in Table 1, bond distances and bond angles are presented in Table 2 and Fig. 1 presents a stereoview of the ions present in the structure.

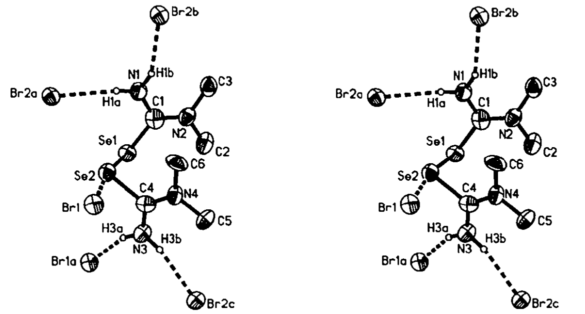

Fig. 1. Stereoview of $(DMSeU)_2$ with Br atoms which make close contacts with H or Se atoms included (methyl H atoms deleted; thermal ellipsoids at 0·50 probability).

Related literature. Husebye (1983, 1988) has thoroughly reviewed the structures of many selenium-containing complexes, and in particular has discussed the variation of bond distances with ligand configuration including possible three-center bond formation. The structure of a closely related compound, $(SU)_2Cl_2$ with SU = selenourea (Villa, Nardelli & Tani, 1970), has been reported in which short halogen to selenium contacts have been observed in a nearly linear Cl···Se—Se configuration.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53535 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (*E*,*E*)-Bis(2-acetoxy-1-iodo-2-phenylvinyl) Disulfide

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Abstract. $C_{20}H_{16}I_2O_4S_2$, $M_r = 638·3$, triclinic, $P\bar{1}$, $a = 14·3123$ (11), $b = 7·5965$ (6), $c = 11·5344$ (9) Å, $\alpha = 109·289$ (5), $\beta = 97·592$ (5), $\gamma = 92·150$ (5)°, $V = 1168·8$ Å^3, $Z = 2$, $D_x = 1·814$ g cm^{-3}, Nb-filtered Mo $K\alpha$ radiation, $\lambda = 0·71074$ Å, $\mu = 29·1$ cm^{-1}, $F(000) = 612$, $T = 295$ K. Final $R = 0·037$ for 2453

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observed unique reflections with $\sin\theta/\lambda \leq 0{\cdot}60$ Å^{-1}. The investigation was necessary to determine the nature of the product of the reaction of *S*-(2-phenylvinyl) thioacetate with iodine; this could not be established by spectroscopic means. The name given in the title describes the structure and agrees with all distances and angles.

Experimental. The product from the reaction of *S*-(2-phenylvinyl) thioacetate with iodine (Pedersen & Senning, 1990) was recrystallized from ethanol giving pale yellow transparent brittle crystals. A prismatic crystal bounded by eight faces and of dimensions $0{\cdot}23 \times 0{\cdot}35 \times 0{\cdot}18$ mm was used. The space group was determined from precession photographs to be triclinic, $P1$ or $P\bar{1}$. Unit-cell dimensions were obtained by least-squares refinement of centred angle settings of 78 reflections $22 < 2\theta < 28°$. 4088 unique reflections, $-16 \leq h \leq 16$, $-9 \leq k \leq 0$, $-13 \leq l \leq 13$ with $2\theta_{\max} = 50°$, were collected in ω–2θ scan mode, scan range: $1{\cdot}20 + 0{\cdot}346 \tan\theta$ in θ, at room temperature using Nb-filtered Mo $K\alpha$ radiation on a Huber four-circle diffractometer. Two test reflections, $0\bar{2}0$ and 005, measured every 50 reflections, diminished to a different degree, $0\bar{2}0$ by 4% and 005 by 20%. Intensities were corrected by a linear function of the intensity-weighted decrease of the standard reflections which corresponds to a total decrease of 9%. The crystal changed colour from pale yellow to reddish brown which might be interpreted as formation of free iodine in the crystal. Corrections for coincidence loss, Lorentz and polarization effects were applied. Absorption correction gave transmission factors in the range from 0·52 to 0·64. The I atoms were located from a Patterson map, subsequent difference maps showed the lighter atoms. H atoms were included at calculated positions. A full-matrix least-squares refinement of atomic position and displacement parameters – anisotropic for non-H atoms and isotropic for H atoms – based on 2453 reflections with $\sin\theta/\lambda \leq 0{\cdot}60$ Å^{-1} and $I > 3\sigma(I)$ and 269 variables gave discrepancy factors $R(F) = 0{\cdot}037$, $wR(F) = 0{\cdot}038$ and goodness of fit $S = 1{\cdot}37$, where the weighting factor w is determined by $w = 1/[\sigma(F)]^2$; $\sigma(F) = [\sigma(F^2) + 1{\cdot}02F^2]^{1/2} - |F|$. Maximum $\Delta/\sigma = 0{\cdot}010$, $\Delta\rho_{\max} = 0{\cdot}9$ (1) and $\Delta\rho_{\min} = -0{\cdot}6$ (1) e Å^{-3}. Computations were carried out on a VAX 6210 computer with the following programs: *INTEG* – based on the Nelmes (1975) algorithm for integration of intensites; *DATAP* and *DSORT* (Coppens, Leiserowitz & Rabinovich, 1965) – data processing; modified *ORFLS* (Busing, Martin & Levy, 1962 – least-squares refinement; *ORFFE* (Busing, Martin & Levy, 1964) – geometry; *ORTEP*II (Johnson, 1976) – drawings. Atomic scattering factors for non-H atoms were taken from *International Tables for X-ray Crystallography* (1974,

Table 1. *Fractional coordinates* ($\times 10^5$ *for* I *and* S *atoms;* $\times 10^4$ *for the rest) and equivalent isotropic thermal parameters* ($\times 10^4$ *for* I *and* S *atoms,* $\times 10^3$ *for the rest) for all non-*H *atoms*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
I(1)	968 (4)	21085 (7)	−10342 (5)	628 (4)
I(11)	35468 (4)	−22710 (7)	−29508 (5)	728 (4)
S(1)	12320 (15)	−17063 (25)	−16125 (19)	571 (12)
S(11)	13162 (13)	−13726 (26)	−32800 (18)	557 (12)
C(3)	1919 (4)	3172 (9)	1396 (7)	43 (4)
C(4)	1912 (6)	3277 (11)	2602 (7)	62 (5)
C(5)	1982 (7)	4975 (14)	3536 (9)	85 (7)
C(6)	2049 (6)	6592 (13)	3260 (10)	78 (7)
C(7)	2072 (5)	6538 (10)	2073 (10)	64 (6)
C(8)	2012 (5)	4827 (10)	1137 (7)	52 (5)
C(2)	1851 (4)	1319 (9)	409 (6)	42 (4)
C(1)	1225 (5)	617 (9)	−626 (7)	46 (4)
O(1)	2480 (3)	86 (6)	687 (4)	49 (3)
C(9)	3406 (5)	416 (11)	586 (7)	53 (5)
C(10)	3992 (7)	−989 (17)	924 (12)	87 (8)
O(2)	3667 (4)	1618 (8)	236 (5)	72 (4)
C(13)	3646 (5)	2163 (10)	−3092 (9)	61 (5)
C(14)	4069 (6)	3486 (12)	−1996 (10)	74 (6)
C(15)	4919 (7)	4454 (14)	−1953 (13)	94 (8)
C(16)	5314 (8)	4103 (18)	−2970 (17)	110 (11)
C(17)	4921 (11)	2832 (23)	−4057 (16)	160 (14)
C(18)	4039 (9)	1828 (18)	−4105 (12)	134 (10)
C(12)	2705 (5)	1219 (11)	−3130 (7)	55 (5)
C(11)	2495 (5)	−492 (10)	−3106 (6)	48 (4)
O(11)	1963 (3)	2396 (6)	−3097 (5)	58 (3)
C(19)	1426 (7)	2205 (11)	−4183 (9)	71 (6)
O(12)	1591 (6)	1171 (10)	−5141 (6)	110 (6)
C(20)	627 (7)	3421 (14)	−4028 (12)	87 (7)

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non-*H *atoms*

S(1)—S(11)	2·039 (3)	C(11)—S(11)	1·752 (7)
C(1)—S(1)	1·757 (7)	C(11)—I(11)	2·090 (7)
C(1)—I(1)	2·098 (7)	C(11)—C(12)	1·333 (10)
C(1)—C(2)	1·332 (9)	C(12)—C(13)	1·490 (10)
C(2)—C(3)	1·478 (9)	C(13)—C(14)	1·374 (11)
C(3)—C(4)	1·368 (10)	C(14)—C(15)	1·384 (12)
C(4)—C(5)	1·372 (11)	C(15)—C(16)	1·321 (16)
C(5)—C(6)	1·372 (13)	C(16)—C(17)	1·339 (17)
C(6)—C(7)	1·360 (12)	C(17)—C(18)	1·435 (17)
C(7)—C(8)	1·378 (10)	C(13)—C(18)	1·318 (12)
C(3)—C(8)	1·389 (10)	O(11)—C(12)	1·410 (8)
O(1)—C(2)	1·405 (7)	O(11)—C(19)	1·342 (10)
O(1)—C(9)	1·367 (8)	C(19)—C(20)	1·487 (12)
C(9)—C(10)	1·500 (11)	O(12)—C(19)	1·184 (9)
O(2)—C(9)	1·180 (8)		

S(11)—S(1)—C(1)	102·0 (2)	S(1)—S(11)—C(11)	103·0 (2)
I(1)—C(1)—S(1)	117·1 (4)	I(11)—C(11)—S(11)	117·5 (4)
I(1)—C(1)—C(2)	121·6 (5)	I(11)—C(11)—C(12)	121·5 (5)
S(1)—C(1)—C(2)	120·9 (5)	S(11)—C(11)—C(12)	120·9 (6)
C(1)—C(2)—C(3)	129·0 (6)	C(11)—C(12)—C(13)	129·1 (7)
O(1)—C(2)—C(3)	114·1 (6)	O(11)—C(12)—C(13)	113·1 (6)
C(1)—C(2)—O(1)	116·5 (6)	C(11)—C(12)—O(11)	117·6 (6)
C(2)—C(3)—C(4)	119·6 (6)	C(12)—C(13)—C(14)	118·7 (8)
C(2)—C(3)—C(8)	121·9 (7)	C(12)—C(13)—C(18)	120·9 (9)
C(4)—C(3)—C(8)	118·5 (7)	C(14)—C(13)—C(18)	120·3 (9)
C(3)—C(4)—C(5)	121·0 (8)	C(13)—C(14)—C(15)	119·8 (10)
C(4)—C(5)—C(6)	119·5 (9)	C(14)—C(15)—C(16)	119·7 (12)
C(5)—C(6)—C(7)	121·0 (8)	C(15)—C(16)—C(17)	122·2 (12)
C(6)—C(7)—C(8)	119·1 (8)	C(16)—C(17)—C(18)	118·3 (12)
C(3)—C(8)—C(7)	120·8 (7)	C(13)—C(18)—C(17)	119·7 (12)
C(2)—O(1)—C(9)	116·4 (5)	C(12)—O(11)—C(19)	117·1 (6)
O(1)—C(9)—O(2)	122·5 (7)	O(11)—C(19)—O(12)	121·9 (9)
O(1)—C(9)—C(10)	110·4 (7)	O(11)—C(19)—C(20)	112·5 (9)
O(2)—C(9)—C(10)	127·0 (8)	O(12)—C(19)—C(20)	125·5 (10)

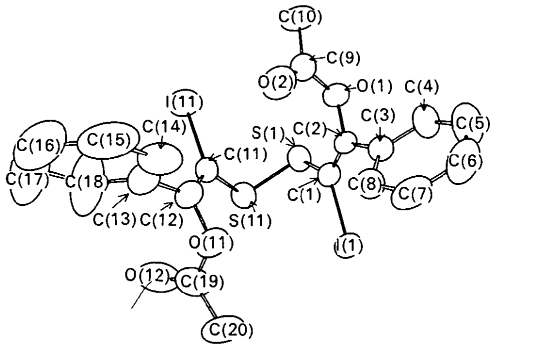

Fig. 1. *ORTEP*II (Johnson, 1976) drawing showing the conformation of a molecule of (*E*,*E*)-bis(2-acetoxy-1-iodo-2-phenylvinyl) disulfide and the atom-numbering scheme.

Vol. IV). The scattering factor for H atoms is that of Stewart, Davidson & Simpson (1965). Atomic coordinates and the equivalent isotropic thermal parameters are given in Table 1.* Bond lengths and angles are in Table 2. The structure is shown in Fig. 1. No hydrogen bonding or close approaches to S atoms are found.

* Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53588 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The present compound is quite different from that resulting from an analogous reaction of a chlorinating agent with *S*-(2-phenylvinyl) thioacetate (Pedersen, Hazell & Senning, 1989). Hordvik (1966) gives a correlation between the S—S distance and the dihedral angle around it; the present values of 2·039 (3) Å and 74·1 (3)° agree with this. Distances and angles in the central part of the molecule agree with values found by Jones & Power (1976) in a similar compound.

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Structure of Calliterpenone Hemihydrate

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Abstract. 16α,17-Dihydroxy-13β-kauran-3-one, $C_{20}H_{32}O_3.\frac{1}{2}H_2O$, $M_r = 329{\cdot}48$, monoclinic, $C2$, $a = 13{\cdot}475$ (6), $b = 6{\cdot}338$ (1), $c = 22{\cdot}904$ (7) Å, $\beta = 114{\cdot}75$ (2)°, $V = 1776$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}232$ g cm^{-3}, graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å), $\mu = 0{\cdot}77$ cm^{-1}, $F(000) = 723{\cdot}92$, $T = 295$ K, $R = 0{\cdot}055$ for 1353 observed reflections. The CH_2OH side chain is found to be in the β-position rather than in the sterically more favourable α-position of the diterpenoid molecule. The hydroxyl groups are involved in intermolecular hydrogen bonding with the water molecule of crystallization and also with the neighbouring diterpenoid molecules. No intramolecular hydrogen bonding was observed between O(2) and O(3) of the two hydroxy groups which could form a sterically favoured five-membered ring.

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Experimental. Calliterpenone was isolated from petroleum ether (60–80° C) extract of *Callicarpa cana*

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