Package (B. A. Frenz & Associates, Inc., 1985), *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1976). Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are given in Table 1.*

Discussion. Our X-ray analysis unequivocally establishes the molecular structure and absolute stereochemistry of the title compound. A view of the molecule is given in Fig. 1 with the crystallographic numbering scheme. The molecular dimensions are in accord with accepted values, mean bond lengths being $C(sp^3)$—$C(sp^3)$ 1·537 (12) [range 1·516 (3)–1·573 (3) Å], $C(sp^3)$—$C(sp^2)$ 1·505 (12), $C(sp^2)$—$C(sp^2)$ 1·451 (3), $C(sp^2)$=$C(sp^2)$ 1·328 (7), $C(sp^3)$—O 1·425(12), $C(sp^2)$—O 1·362(3) and C=O 1·208(3)Å. Similar bond lengths have been reported in the structure of (+)-jaborosalactone M (Parvez, Fajardo & Shamma, 1988) which has an identical ring skeleton with different ring conformations of rings *A* and *B* than those observed in (2).

In the molecule of (2), ring *A* has a half-chair conformation; it is a twist boat in (+)-jaborosalactone M. Ring *B* is *cis* fused to ring *A* and has a chair conformation with an 8β,9α orientation while it is a half chair in (+)-jaborosalactone M. Rings *C* and *E* also have regular chair conformations. The five-membered ring *D*, which is *cis* fused to ring *C*, has a C(13) envelope conformation with C(13) 0·707 (3) Å above the plane C(14)–C(17). The lac-

* Lists of anisotropic temperature factors, hydrogen parameters, bond lengths, angles and torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53234 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

tone moiety, ring *F*, is nearly planar, with maximum deviation of 0·021 (4) Å. The methyl groups, C(18) and C(19), the hydroxyl groups, O(2) and O(5) and the methoxyl group [O(4), C(22)] are all β oriented, while Cl and the lactone moiety exhibit α orientation. The crystal structure, Fig. 2, consists of a strong hydrogen bond $O(1)\cdots O(2)^i$ 2·84 Å, where (i) $= 1 - x, \frac{1}{2} + y, \frac{1}{2} - z + 1$.

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Structure of 6,7-Dihydro-2-phenyl-5*H*-2aλ⁴-thia-3-selena-1,2-diazacyclopent[*cd*]indene

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Abstract. $C_{13}H_{12}N_2SSe$, $M_r = 307·27$, triclinic, $P\bar{1}$, $a = 7·698$ (2), $b = 7·818$ (4), $c = 11·685$ (2) Å, $\alpha = 90·24$ (2), $\beta = 97·89$ (2), $\gamma = 117·60$ (3)°, $V = 615·4$ Å³, $Z = 2$, $D_x = 1·66$ g cm⁻³, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 59·7$ cm⁻¹, $F(000) = 376$, $T = 295$ K, final $R = 0·059$ for 1865 unique observed [$F \geq 4·0\sigma(F)$] diffractometer data. The tricyclic portion of the molecule is almost planar and possesses elon-

gated Se—S and S—N bonds of 2·585 (2) and 1·812 (6) Å, respectively, and an Se—S—N bond angle of 172·5 (2)°.

Introduction. The determination of the structure of the title compound (1) described herein is part of a systematic study of the synthesis, reactions and structure of 1,6,6aλ^4-triheterapentalenes and related heterocyclic systems (Czyzewski & Reid, 1983, and earlier references cited therein; Allen, Boeyens, Briggs, Denner, Markwell, Reid & Rose, 1987). 1,6,6aλ^4-Triheterapentalenes contain a sequence of three heteroatoms of Groups V and VI (N,O,S,Se,Te) in which the two bonds linking adjacent heteroatoms are characteristically long. Hitherto the structures of six 1,6,6aλ^4-triheterapentalenes (2)–(7) have been determined in which the hetero-atom sequence contains Se—S bonds. These Se—S bonds are present in Se–Se–S systems (Allen, Boeyens, Briggs, Denner, Markwell, Reid & Rose, 1987), in an Se–S–S system (Van Hende & Klingsberg, 1966) and in S–Se–S systems (Hordvik & Julshamn, 1971; Hordvik, Rimala & Saethre, 1972, 1973). The Se—S bond lengths in these compounds lie in the range 2·33–2·51 Å and are thus 6·2–14·4% greater than the (two-centre two-electron) covalent Se—S single-bond length [2·195 (15) Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987]. We have determined the structure of the title compound, which contains an Se—S bond in an Se–S–N sequence, since compounds of the type (1) have not hitherto been investigated structurally.
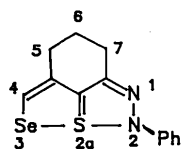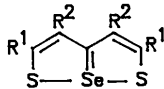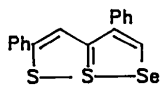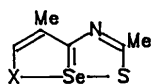

Experimental. The title compound (1) was prepared from 6,7-dihydro-2-phenyl-5H-3-oxa-2aλ^4-thia-1,2-diazacyclopent[cd]indene by the method of Czyzewski & Reid (1983). The compound was recrystallized from hexane and obtained as green crystals, m.p. 368–370 K. A crystal of dimensions $\sim 0.85 \times 0.45 \times 0.45$ mm was used for the X-ray diffraction measurements; $\omega/2\theta$ scans, scan speed 1·2–5·49° min^{-1} in ω was used on a CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The lattice parameters were determined from a least-squares fit of 25 reflections in the range $16 \le 2\theta \le 38°$. Three standard reflections were recorded every hour, only random deviations were observed; 3734 reflections were measured ($2 \le \theta \le 30.0°$, $-10 \le h \le 10$, $-11 \le k \le 11$, $0 \le l \le 16$), averaging ($R_{int} = 0.026$) gave 2975 unique reflections, 1865 of which had $F_o \ge 4.0\sigma(F)$. The data were corrected for Lorentz, polarization and absorption effects, the latter via ψ scans, max./min. transmission 0·99/0·85. The structure was solved via Patterson methods and ΔF syntheses and refined with full-matrix least squares with anisotropic thermal parameters for all non-H atoms and a common isotropic temperature factor for H atoms, the latter being placed in geometrically calculated positions (C—H 1·08 Å). Refinement on F with 1865 reflections, 155 parameters and $w = 3.57/\sigma^2(F)$ gave $R = 0.059$, $wR = 0.054$ and $(\Delta/\sigma)_{max} = 0.004$. No extinction correction was applied. The max. and min. heights in final $\Delta\rho$ map were ± 0.87 (9) e Å^{-3} near the Se atom and ± 0.4 (2) e Å^{-3} in remaining parts of the unit cell. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from International Tables for X-ray Crystallography (1974, Vol. IV). The programs SHELX76, SHELXS86 (Sheldrick, 1976, 1985), NRCVAX (Gabe, Lee & Le Page, 1985) and ORTEP (Johnson, 1965) were used.

Discussion. Final atomic coordinates and bond distances and angles are listed in Tables 1 and 2.* The structure of 6,7-dihydro-2-phenyl-5H-2aλ^4-thia-3-selena-1,2-diazacyclopent[cd]indene (1) is shown in Fig. 1. The tricyclic frame of the molecule is almost planar with maximum deviations from planarity not exceeding 0·033 Å.

The bond lengths and angles may be compared with the relevant ones of the structurally related S–S–N analogues (8) (Hansen & Tomren, 1977) and (9) (Darmo & Hansen, 1977). The S—N bond length in (1) [1·812 (6) Å] is intermediate between those in

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Table 1. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\mathring{A}^2 \times 10^3$)

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

	x	y	z	U_{eq}
Se	1402 (1)	3019 (1)	1174 (1)	75
S	656 (3)	2682 (3)	3276 (2)	51
N1	−195 (8)	2223 (8)	4667 (5)	48 (1)
N2	−2046 (8)	817 (8)	4661 (5)	50 (2)
C1	−1190 (10)	1054 (11)	739 (7)	64 (2)
C2	−2325 (10)	234 (10)	1535 (6)	49 (2)
C3	−1673 (9)	844 (9)	2710 (6)	45 (2)
C4	−2814 (10)	61 (9)	3590 (5)	47 (2)
C5	−4901 (10)	−1542 (10)	3306 (6)	57 (2)
C6	−5753 (10)	−1459 (10)	2046 (7)	60 (2)
C7	−4389 (10)	−1442 (9)	1203 (7)	58 (2)
C8	923 (10)	3293 (10)	5720 (6)	46 (2)
C9	2708 (11)	5002 (10)	5683 (7)	58 (2)
C10	3826 (12)	6083 (11)	6708 (7)	64 (2)
C11	3197 (12)	5551 (12)	7749 (7)	67 (2)
C12	1392 (12)	3812 (12)	7778 (6)	66 (2)
C13	299 (10)	2729 (11)	6770 (6)	54 (2)

Table 2. *Bond lengths* ($\mathring{A}$) *and angles* (°)

|			
Se—S	2·585 (2)	Se—C1	1·864 (7)
S—N1	1·812 (6)	S—C3	1·731 (6)
N1—N2	1·336 (7)	N1—C8	1·410 (8)
N2—C4	1·316 (8)	C1—C2	1·323 (9)
C2—C3	1·396 (9)	C2—C7	1·513 (8)
C3—C4	1·399 (9)	C4—C5	1·498 (8)
C5—C6	1·542 (9)	C6—C7	1·530 (9)
C8—C9	1·411 (9)	C8—C13	1·379 (9)
C9—C10	1·387 (9)	C10—C11	1·366 (10)
C11—C12	1·429 (10)	C12—C13	1·368 (9)
S—Se—C1	86·5 (3)	Se—S—N1	172·5 (2)
Se—S—C3	86·8 (2)	N1—S—C3	85·8 (3)
S—N1—N2	116·3 (4)	S—N1—C8	124·6 (4)
N2—N1—C8	119·1 (6)	N1—N2—C4	108·5 (6)
Se—C1—C2	120·2 (6)	C1—C2—C3	122·0 (7)
C1—C2—C7	120·9 (7)	C3—C2—C7	117·2 (6)
S—C3—C2	124·5 (5)	S—C3—C4	110·8 (5)
C2—C3—C4	124·7 (6)	N2—C4—C3	118·6 (6)
N2—C4—C5	120·9 (6)	C3—C4—C5	120·4 (6)
C4—C5—C6	109·4 (6)	C5—C6—C7	111·3 (6)
C2—C7—C6	110·8 (6)	N1—C8—C9	118·4 (6)
N1—C8—C13	121·8 (6)	C9—C8—C13	119·8 (7)
C8—C9—C10	119·4 (7)	C9—C10—C11	121·1 (7)
C10—C11—C12	119·0 (7)	C11—C12—C13	120·0 (7)
C8—C13—C12	120·6 (7)		
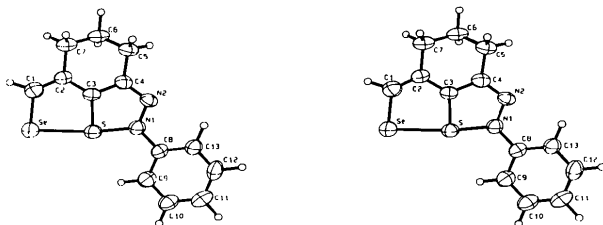
Fig. 1. Stereoview of the title compound showing the numbering of the atoms. The thermal ellipsoids of the non-H atoms enclose 50% probability.

(8) [1·849 (3) Å] and (9) [1·779 (2) Å]. The corresponding S—N bond elongations relative to the typical two-centre two-electron covalent S—N bond length (1·75 Å; Pauling, 1960) are (1) 3·5%; (8) 5·7%; (9) 1·7%.

The Se—S bond in (1) [2·585 (2) Å] is 17·8% longer than that of an accepted two-centre two-electron covalent Se—S bond [2·195 (15) Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987] and is much longer than the longest Se—S bonds in compounds (2)–(7) [(6) 2·507 (2); (7) 2·467 (2) and 2·372 (2) Å]. The sum of the Se—S and S—N bond lengths in (1) is 4·397 Å, approximately 11·5% greater than the sum of two-centre two-electron covalent Se—S and S—N bond lengths (3·945 Å). Note that some of the bond lengths quoted above have been corrected for libration.

Aside from the Se—S and S—N bonds, the remaining bonds in the triheterapentalene unit of (1) show multiple-bond character similar to that shown by the corresponding bonds in the analogues (8) and (9). The C1—C2 bond in (1) [1·323 (8) Å] is, however, markedly shorter than the corresponding bonds in compounds (8) [1·376 (5) Å] and (9) [1·370 (2) Å]. The Se—S—N bond angle in (1) [172·5 (2)°] is close to the corresponding S—S—N angles found in compounds (8) [171·7 (9)°] and (9) [174·4 (1) °].

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