$h = -22-21$, $k = 0-11$, $l = 1-21$ (room temperature), $h = -21-21$, $k = 0-11$, $l = 1-21$ (110 K). The structure was solved by *MULTAN* (Germain, Main & Woolfson, 1971) and refined with full-matrix least

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *for* Ph₃ClSn

	Room temperature	110 K
Sn(1)–Cl(1)	2·3538 (14)	2·3620 (10)
Sn(2)–Cl(2)	2·3557 (14)	2·3692 (10)
Sn(1)–C(11)	2·113 (4)	2·117 (3)
Sn(1)–C(21)	2·114 (4)	2·124 (3)
Sn(1)–C(31)	2·120 (4)	2·126 (3)
Sn(2)–C(41)	2·109 (4)	2·117 (3)
Sn(2)–C(51)	2·118 (4)	2·123 (3)
Sn(2)–C(61)	2·116 (4)	2·120 (3)
Cl(1)–Sn(1)–C(11)	105·28 (10)	105·85 (12)
Cl(1)–Sn(1)–C(21)	104·81 (10)	105·08 (11)
Cl(1)–Sn(1)–C(31)	105·01 (10)	104·98 (12)
C(11)–Sn(1)–C(21)	118·45 (14)	117·11 (14)
C(11)–Sn(1)–C(31)	109·46 (13)	109·83 (14)
C(21)–Sn(1)–C(31)	112·59 (13)	112·90 (15)
Cl(2)–Sn(2)–C(41)	106·66 (10)	106·74 (12)
Cl(2)–Sn(2)–C(51)	102·41 (10)	103·38 (12)
Cl(2)–Sn(2)–C(61)	104·12 (10)	104·45 (12)
C(41)–Sn(2)–C(51)	114·52 (13)	114·18 (14)
C(41)–Sn(2)–C(61)	110·70 (13)	110·94 (14)
C(51)–Sn(2)–C(61)	116·94 (13)	115·91 (15)
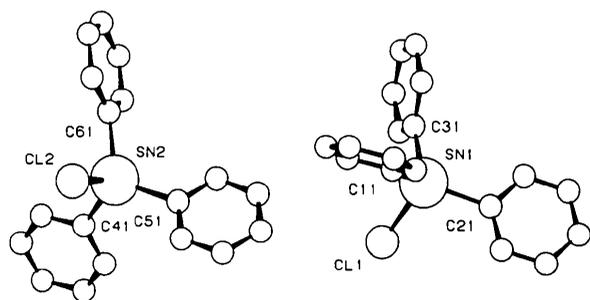
Fig. 1. Numbering scheme and perspective drawing of the two independent molecules of Ph₃SnCl.

squares on F. All H atoms were located in difference Fourier maps. Refinement using anisotropic and isotropic temperature factors for non-H and H atoms respectively yielded $R = 0·033$ (room temperature) and $R = 0·031$ (110 K), number of variables 482, max. $\Delta/\sigma = 0·031$. Highest peak in final difference synthesis $0·69$ e Å⁻³ (room temperature), $1·97$ e Å⁻³ (110 K) near the Sn atoms. Details of the computer programs used and experimental procedure have been presented previously (Grant & Gabe, 1978; Gabe, Lee & Le Page, 1985). The molecules are depicted in Fig. 1. The atomic coordinates and isotropic thermal parameters are given in Table 1, selected bond distances and bond angles are listed in Table 2.*

Related literature. Bancroft, Bulter & Sham (1975); Bokii, Zalkharova & Struchkov (1970); Bokii, Struchkov & Prokofiev (1972); Bondi (1964); Srivistawa (1967).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43162 (86 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(η⁵-methylcyclopentadienyl)divanadium Pentaselenide

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Abstract. μ-Diseleno-*Se,Se'*-μ-(η-diseleno-μ-*Se*,μ-*Se'*)-μ-seleno-bis(methylcyclopentadienylvanadium)(V–V), [V₂(C₆H₇)₂Se₅], $M_r = 654·9$, triclinic, $P\bar{1}$, $a = 7·121$ (3), $b = 10·473$ (4), $c = 11·694$ (4) Å, $\alpha = 96·66$ (3), $\beta = 93·80$ (3), $\gamma = 109·12$ (3)°, $V = 813·4$ (5) Å³, $Z = 2$, $D_x = 2·674$ g cm⁻³, Mo $K\alpha$ (λ

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Table 1. *Atomic coordinates* $(\times 10^4)$ *and isotropic thermal parameters* $(\text{Å}^2 \times 10^4)$

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

	x	y	z	U_{eq}
Se(1)	8471 (2)	6967 (2)	6368 (1)	42 (1)
Se(2)	4543 (2)	5795 (2)	8690 (1)	44 (1)
Se(3)	3762 (2)	5535 (2)	6723 (1)	44 (1)
Se(4)	8990 (3)	8261 (2)	9372 (1)	47 (1)
Se(5)	9369 (2)	6160 (2)	9198 (1)	47 (1)
V(1)	6492 (4)	7722 (2)	7688 (2)	35 (1)
V(2)	6965 (4)	5176 (2)	7466 (2)	34 (1)
Cp(1)	7565 (23)	10084 (13)	7872 (14)	47 (6)
Cp(2)	6088 (26)	9664 (16)	8622 (15)	53 (7)
Cp(3)	4271 (27)	8866 (16)	7958 (16)	62 (8)
Cp(4)	4620 (23)	8801 (15)	6771 (14)	46 (6)
Cp(5)	6655 (25)	9579 (14)	6707 (13)	45 (6)
Cm(5)	7634 (31)	9943 (19)	5659 (15)	75 (10)
Cp(6)	6897 (22)	3110 (14)	8044 (12)	42 (6)
Cp(7)	8645 (23)	3652 (14)	7474 (14)	49 (7)
Cp(8)	8018 (22)	3690 (14)	6308 (14)	47 (6)
Cp(9)	5912 (25)	3214 (14)	6165 (13)	47 (7)
Cp(0)	5199 (23)	2815 (15)	7244 (13)	48 (6)
Cm(0)	3102 (24)	2156 (16)	7468 (16)	63 (8)

$= 0.71073$ Å), $\mu = 132.2 \text{ cm}^{-1}$, $F(000) = 608$, $T = 296$ K, $R = 5.57\%$ for 1647 reflections with $F_o \geq 3\sigma(F_o)$. The compound is isostructural with its S analogue. The cyclopentadienyl rings are inclined toward each other [21·5 (3)°]; the V—V bond, 2·779 (4) Å, is bridged by Se atoms in three distinctly different modes: μ-η-Se$_2$, syn-μ-Se$_2$ and μ-Se. Both of the Se$_2$ groups possess short bond distances: μ-η^2 = 2·295 (2) and syn-μ = 2·290 (2) Å, indicating considerable multiple-bond character.

Experimental. Black, poorly formed crystals of $(CH_3C_5H_4)_2V_2Se_5$, 0·12 × 0·20 × 0·27 mm, were obtained in good yield from the thermal rearrangement of $(CH_3C_5H_4)_2VSe_5$ [*in situ* generation from $(CH_3C_5-H_4)_2VCl_2$ and H_2Se] in refluxing tetrahydrofuran after recrystallization from CH_2Cl_2/toluene. Nicolet $P3$ diffractometer, $\theta/2\theta$ scans, scan rate 5° min^{-1}. Lattice parameters from 25 reflections, $21 \leq 2\theta \leq 26°$, least-squares fit, empirical ellipsoidal absorption correction, seven reflections, 10° increments, 0·038/0·021 (max./min. transmission from *XEMP*), $2\theta_{max} = 45°$ ($h = \pm 8$, $k = \pm 12$, $l = 13$), three standard reflections ($2\bar{8}1$, $\bar{1}35$, $4\bar{1}1$), <1% variation. 2340 reflections collected, 2115 unique, $R_{int} = 3.2\%$, 381 unobserved reflections, $F_o \geq 3\sigma(F_o)$. Structure solved by direct methods ($SOLV$), refinement on F for 172 parameters, all non-H atoms anisotropic, H atoms in calculated positions, methyl-group rotation fixed by location of two H atoms, $d(C-H) = 0.96$ Å, $R_F = 5.57\%$, $wR_F = 6.32\%$, $S = 1.508$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0.001$, $(\Delta/\sigma)_{max} = 0.005$, $\Delta\rho_{max} = 1.4$ e Å^{-3}, $\Delta\rho_{min} = 0.9$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* (Sheldrick, 1984) computer programs. The atomic coordinates are given

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

|			
Se(1)—V(1)	2·394 (2)	V(2)—Cp(6)	2·33 (1)
Se(2)—V(1)	2·510 (3)	V(2)—Cp(7)	2·29 (2)
Se(3)—V(1)	2·549 (2)	V(2)—Cp(8)	2·29 (2)
Se(4)—V(1)	2·447 (3)	V(2)—Cp(9)	2·29 (1)
Se(1)—V(2)	2·396 (3)	V(2)—Cp(0)	2·35 (1)
Se(2)—V(2)	2·507 (3)	Cp(1)—Cp(2)	1·41 (2)
Se(3)—V(2)	2·545 (3)	Cp(2)—Cp(3)	1·41 (2)
Se(5)—V(2)	2·450 (3)	Cp(3)—Cp(4)	1·43 (2)
Se(2)—Se(3)	2·295 (2)	Cp(4)—Cp(5)	1·42 (2)
Se(4)—Se(5)	2·290 (2)	Cp(1)—Cp(5)	1·43 (2)
CNT(1)*—V(1)	1·97 (1)	Cp(6)—Cp(7)	1·43 (2)
CNT(2)*—V(2)	1·97 (1)	Cp(7)—Cp(8)	1·42 (2)
V(1)—Cp(1)	2·32 (1)	Cp(8)—Cp(9)	1·41 (2)
V(1)—Cp(2)	2·31 (1)	Cp(9)—Cp(0)	1·45 (2)
V(1)—Cp(3)	2·29 (2)	Cp(6)—Cp(0)	1·40 (2)
V(1)—Cp(4)	2·30 (2)	Cp(5)—Cm(5)	1·48 (2)
V(1)—Cp(5)	2·34 (2)	Cp(0)—Cm(0)	1·48 (2)
V(1)—Se(1)—V(2)	70·9 (1)	Se(3)—V(2)—V(1)	57·0 (1)
Se(3)—Se(2)—V(1)	63·9 (1)	Se(5)—V(2)—V(1)	84·3 (1)
Se(3)—Se(2)—V(2)	63·8 (1)	Cp(2)—Cp(1)—Cp(5)	108 (1)
V(1)—Se(2)—V(2)	67·3 (1)	Cp(1)—Cp(2)—Cp(3)	109 (1)
Se(2)—Se(3)—V(1)	62·2 (1)	Cp(2)—Cp(3)—Cp(1)	108 (1)
Se(2)—Se(3)—V(2)	62·1 (1)	Cp(3)—Cp(4)—Cp(5)	108 (1)
V(1)—Se(3)—V(2)	66·1 (1)	Cp(4)—Cp(5)—Cp(1)	107 (1)
Se(5)—Se(4)—V(1)	95·8 (1)	Cp(4)—Cp(5)—Cm(5)	128 (1)
Se(4)—Se(5)—V(2)	95·6 (1)	Cp(1)—Cp(5)—Cm(5)	125 (1)
Se(1)—V(1)—Se(2)	110·5 (1)	Cp(7)—Cp(6)—Cp(0)	109 (1)
Se(1)—V(1)—Se(3)	82·9 (1)	Cp(6)—Cp(7)—Cp(8)	108 (1)
Se(2)—V(1)—Se(3)	53·9 (1)	Cp(7)—Cp(8)—Cp(9)	108 (1)
Se(1)—V(1)—Se(4)	94·9 (1)	Cp(8)—Cp(9)—Cp(10)	109 (1)
Se(2)—V(1)—Se(4)	84·8 (1)	Cp(9)—Cp(0)—Cp(6)	107 (1)
Se(3)—V(1)—Se(4)	133·7 (1)	Cp(6)—Cp(0)—Cm(0)	126 (1)
Se(1)—V(1)—V(2)	54·6 (1)	Cp(9)—Cp(0)—Cm(0)	128 (1)
Se(2)—V(1)—V(2)	56·3 (1)	CNT(1)—V(1)—V(2)	169·8 (3)
Se(3)—V(1)—V(2)	56·9 (1)	CNT(1)—V(1)—Se(1)	122·3 (2)
Se(4)—V(1)—V(2)	84·2 (1)	CNT(1)—V(1)—Se(2)	124·3 (2)
Se(1)—V(2)—Se(2)	110·5 (1)	CNT(1)—V(1)—Se(3)	114·2 (2)
Se(1)—V(2)—Se(3)	82·9 (1)	CNT(1)—V(1)—Se(4)	105·9 (2)
Se(2)—V(2)—Se(3)	54·0 (1)	CNT(2)—V(2)—V(1)	169·1 (3)
Se(1)—V(2)—Se(5)	95·2 (1)	CNT(2)—V(2)—Se(1)	122·5 (2)
Se(2)—V(2)—Se(5)	84·6 (1)	CNT(2)—V(2)—Se(2)	123·7 (2)
Se(3)—V(2)—Se(5)	133·6 (1)	CNT(2)—V(2)—Se(3)	113·3 (2)
Se(1)—V(2)—V(1)	54·5 (1)	CNT(2)—V(2)—Se(5)	106·6 (2)
Se(2)—V(2)—V(1)	56·4 (1)		

* CNT(1) and CNT(2) centroids of Cp(1–5) and Cp(6–0) rings, respectively.
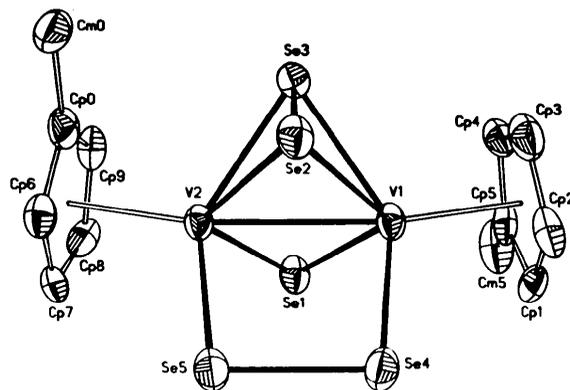
Fig. 1. Structure and labelling scheme for $(\eta^5\text{-}CH_3C_5H_4)_2V_2Se_5$ with 40% thermal-ellipsoid probabilities and H atoms deleted.

in Table 1 and bond lengths and angles in Table 2.* Fig. 1 shows the structure and atom-labelling scheme.

Related literature. Two other structures are known which contain V—Se bonds: [(dppe)V(CO)₃]₂Se (Albrecht, Hübener, Behren & Weiss, 1985), and V₂Se₉ (Furuseth & Klewe, 1984). The isostructural S

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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 43128 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

analogue has been reported (Bolinger, Rauchfuss & Rheingold, 1982).

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Structure of Monoclinic o-Aminobenzoic Acid

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Abstract. C₇H₇NO₂, $M_r = 137 \cdot 1$, monoclinic, $P2_1/c$, $a = 6 \cdot 537$ (3), $b = 15 \cdot 351$ (5), $c = 7 \cdot 086$ (3) Å, $\beta = 112 \cdot 64$ (3)°, $V = 656 \cdot 3$ (5) Å³, $Z = 4$, $D_m = 1 \cdot 34$ (1), $D_x = 1 \cdot 39$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71073$ Å, $\mu = 0 \cdot 097$ mm⁻¹, $F(000) = 288$, $T = 298$ (1) K, final $R = 0 \cdot 039$ for 1187 unique reflections. The molecule is non-zwitterionic. The adjacent molecules form a cyclic dimer by intermolecular hydrogen bonds with their carboxyl groups, the O···O distance being 2·651 (3) Å. One of the amino H atoms takes part in an intramolecular hydrogen bond with N···O distance 2·688 (4) Å and the other in a weak intermolecular hydrogen bond with N···O distance 3·385 (5) Å.

Experimental. Colorless prisms of the title compound occasionally grew from an aqueous solution in which O₂ gas was bubbled by cooling gradually from 343 to 313 K. D_m determined by flotation in an aqueous solution of ZnI₂. Crystal size 0·5 × 0·5 × 0·5 mm, Rigaku AFC-5 four-circle diffractometer, Mo $K\alpha$ radiation, graphite monochromator, θ–2θ scan with scan speed 6° min⁻¹ in θ, scan width $(1 \cdot 3 + 0 \cdot 5\tan\theta)$°. Range of indices, $-6 \leq h \leq 6$, $0 \leq k \leq 14$, $0 \leq l \leq 6$ ($2\theta \leq 55°$). Lattice constants determined based on 20 2θ values ($20 < 2\theta < 31°$). Variation of standards <1%; 1618 reflections measured; 1272 observed reflections with $|F_o| > 3\sigma(|F_o|)$. Systematic absences $h0l$, l odd; $0k0$, k odd. No corrections for absorption or extinction. Structure solved by direct methods with *MULTAN*78

Table 1. *Fractional coordinates* (×10⁴) *and equivalent isotropic temperature factors* (*Hamilton*, 1959)

$$B_{eq} = \tfrac{1}{3} \sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	$B_{eq}(\text{Å}^2 \times 10)$
C(1)	9484 (2)	4408 (1)	7434 (2)	30
C(2)	9057 (2)	3992 (1)	5459 (2)	29
C(3)	6887 (2)	3982 (1)	3976 (2)	35
C(4)	6388 (3)	3577 (1)	2122 (2)	42
C(5)	8073 (3)	3179 (1)	1710 (2)	45
C(6)	10206 (3)	3187 (1)	3114 (2)	42
C(7)	10780 (2)	3597 (1)	5032 (2)	33
N	12921 (2)	3572 (1)	6407 (2)	45
O(1)	7702 (2)	4708 (1)	7665 (1)	42
O(2)	11336 (2)	4476 (1)	8799 (1)	40

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

C(1)—C(2)	1·465 (2)	C(7)—C(2)—C(3)	119·7 (1)
C(2)—C(3)	1·403 (6)	C(2)—C(3)—C(4)	121·3 (1)
C(3)—C(4)	1·375 (3)	C(3)—C(4)—C(5)	119·1 (1)
C(4)—C(5)	1·385 (3)	C(4)—C(5)—C(6)	120·9 (1)
C(5)—C(6)	1·366 (5)	C(5)—C(6)—C(7)	121·6 (2)
C(6)—C(7)	1·410 (3)	C(6)—C(7)—C(2)	117·4 (1)
C(7)—C(2)	1·411 (3)	C(7)—C(2)—C(1)	121·2 (1)
C(7)—N	1·364 (5)	C(3)—C(2)—C(1)	119·1 (1)
C(1)—O(1)	1·320 (2)	C(6)—C(7)—N	119·7 (1)
C(1)—O(2)	1·229 (5)	C(2)—C(7)—N	122·9 (1)
O(1)···O(2ⁱ)	2·651 (3)	C(2)—C(1)—O(1)	114·7 (1)
N···O(2)	2·688 (4)	C(2)—C(1)—O(2)	123·8 (1)
N···O(1ⁱⁱ)	3·385 (5)	O(1)—H(O1)···O(2ⁱ)	173 (2)
		N—H(N)1···O(2)	127 (2)
		N—H(N)2···O(1ⁱⁱ)	104 (1)

Symmetry code: (i) 2−x, 1−y, 2−z; (ii) 1+x, y, z.