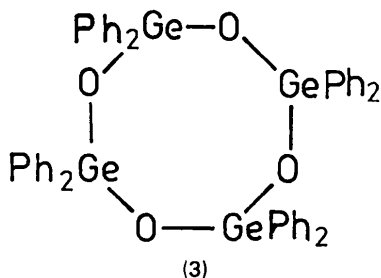and 4.433 (7) Å, respectively] are longer. The bond angles of Ge—O—Ge in (1) [124.9 (2), 126.6 (2)°] are significantly smaller than those in (3) [131.4 (5)–136.9 (5)°].

$$Ph_2Ge—O$$
(3)

Although there is no intramolecular distance shorter than the sum of the van der Waals radii, except for Ge1···Ge1 and Ge2···Ge2, there is an intermolecular distance that is shorter, i.e. H252···H232 [$-0.5 + x$, $0.5 - y$, $0.5 + z$; 2.15 (6) Å], where H252 and H232 are the H atoms bonded to C25 and C23, respectively. That is, short intermolecular contacts only occur between the germacyclohexane rings with Ge1, suggesting that the effect of molecular packing on the structure is less in the germacyclohexane ring with Ge1 than in the other germacyclohexane ring.

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Structure of 1,2;1,3-Di-μ-carbonyl-1,1,2,2,3,3,4-heptacarbonyl-2,3-μ-η³-cyclooctenyl-2,4-μ-hydrido-4-tricyclohexylphosphine-tetrahedro-trirutheniumplatinum

BY LOUIS J. FARRUGIA,* NEIL MACDONALD AND ROBERT D. PEACOCK

Department of Chemistry, The University, Glasgow G12 8QQ, Scotland

Abstract. [Ru$_3$Pt(μ-η^3-C$_8$H$_{13}$)(μ-CO)$_2$(CO)$_7$(μ-H){P(C$_6$H$_{11}$)$_3$}], $M_r = 1141.02$, triclinic, $P\bar{1}$, $a = 10.192$ (1), $b = 21.121$ (7), $c = 21.235$ (6) Å, $\alpha = 118.83$ (3), $\beta = 96.67$ (2), $\gamma = 95.56$ (2)°, $V = 3916$ (2) Å^3, $Z = 4$, $D_x = 1.94$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 48.0$ cm^{-1}, $F(000) = 2216$, $T = 298$ K, $R = 0.035$ for 5663 unique observed reflections. The metal atoms adopt a tetrahedral core, with a 58 cluster valence-electron count. A cyclo-C$_8$H$_{13}$ ligand bridges two Ru atoms in a three-electron donor μ-allyl mode, while the other two Ru—Ru edges are bridged by carbonyl ligands.

Introduction. A considerable number of triosmium-platinum clusters have now been structurally characterized (Farrugia, 1990), and these may have either a tetrahedral Os$_3$Pt core for cluster valence-electron (CVE) counts of 58 or 60, or a 'butterfly' core with 60 CVE's. The tetrahedral 58 CVE clusters, exemplified by Os$_3$Pt(μ-H)$_2$(CO)$_{10}$(PR_3) (1) (Farrugia, Howard, Mitrprachachon, Stone & Woodward, 1981), are chemically unsaturated and highly reac-

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* Author to whom all correspondence should be addressed.

tive. Few ruthenium–platinum clusters have been reported (Farrugia, 1990), and herein we detail the structure of a 58 CVE tetrahedral Ru₃Pt cluster, which is electronically related to (1).

Experimental. The title complex was isolated as a minor product (*ca* 5%) from a 1:1:1 reaction of bis(cyclooctadiene)platinum, tricyclohexylphosphine and the anion $[Ru_3(\mu\text{-H})(CO)_{11}]^-$ in diethyl ether at 273 K under an ethylene atmosphere. After stirring for *ca* 1 min, the dark red–brown mixture was treated with HBF₄.Et₂O, and chromatographed on a florosil column. This gave several uncharacterized yellow, orange and red–orange bands, and a green band for the title complex. Dark-green prisms were obtained from dichloromethane/hexane solution: crystal dimensions *ca* 0.1 × 0.1 × 0.06 mm; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters* refined by least-squares methods from setting angles of 25 independent reflections with $12 < \theta < 13°$; intensities measured to $\theta = 22.5°$ over *hkl* range 0 to 10, −22 to 22, −22 to 22; $\overline{2}59$, $\overline{4}30$ and $56\overline{1}$ measured every 2 h with a 4% decay over 144 h data collection; 10 891 data measured, 10 200 independent data with 5663 having $I > 3.0\sigma(I)$ considered observed and used in structure determination and refinement; $R_{int} = 0.028$ before and 0.024 after absorption correction; corrected for decomposition, Lp and absorption (*DIFABS*; Walker & Stuart, 1983), maximum, minimum values of applied absorption correction 1.16, 0.89; solved by direct methods (*MITHRIL*; Gilmore, 1984) and subsequent least squares; anisotropic thermal parameters for all non-H atoms, except cyclohexyl C atoms, fixed isotropic thermal parameters ($U = 0.05$ Å²) for all H atoms; the positions of the allylic hydrogens H(10*A*)–H(12*B*) were determined from difference Fourier maps, but were not refined; all other H atoms included at calculated positions [C—H = 1.0, Pt—H ≈ 1.85, Ru—H ≈ 1.75 Å (*HYDEX*; Orpen, 1980)]; $\Sigma w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o)]^{-1}$; maximum $\Delta/\sigma = 0.29$, average = 0.05; $\Delta\rho_{max} = 1.25$, $\Delta\rho_{min} = -0.97$ e Å⁻³ in vicinity of Pt atom; final $R = 0.035$, $wR = 0.037$, blocks of 235 and 253 parameters were refined in alternate cycles owing to limitations on matrix size; $S = 1.48$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71–151); calculations carried out on a MicroVAX 3600 computer using the *GX* suite of programs (Mallinson & Muir, 1985).

* The transformation $\overline{1}00/01\overline{1}/0\overline{1}\overline{1}$ yields a pseudo-monoclinic *A*-centred cell with $a = 10.192$, $b = 36.464$, $c = 21.551$ Å, $\alpha = 89.65$, $\beta = 102.08$, $\gamma = 90.67°$; the Laue symmetry and the departures of α and γ from 90° preclude this as a possibility.

Table 1. *Fractional coordinates and isotropic thermal parameters (Å²) (equivalent isotropic parameters U_{eq} for anisotropic 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}
Pt(1*A*)	−0.92872 (5)	0.73192 (3)	0.92914 (3)	0.039
Pt(1*B*)	−0.80466 (5)	0.25854 (3)	0.55654 (3)	0.046
Ru(1*A*)	−0.91503 (10)	0.71142 (5)	1.05368 (5)	0.041
Ru(1*B*)	−0.82341 (11)	0.24756 (5)	0.41539 (5)	0.049
Ru(2*A*)	−1.14426 (10)	0.74285 (5)	1.00611 (6)	0.050
Ru(2*B*)	−1.04939 (11)	0.21217 (7)	0.45404 (6)	0.070
Ru(3*A*)	−1.09274 (10)	0.61066 (5)	0.91650 (5)	0.043
Ru(3*A*)	−0.94085 (11)	0.35447 (6)	0.52793 (6)	0.058
P(1*A*)	−0.7693 (3)	0.8141 (2)	0.9186 (2)	0.036
P(1*B*)	−0.6620 (3)	0.1948 (2)	0.5927 (2)	0.045
O(1*A*)	−0.6857 (11)	0.8058 (5)	1.1810 (5)	0.089
O(1*B*)	−0.6513 (12)	0.1563 (5)	0.3103 (5)	0.108
O(2*A*)	−1.0579 (10)	0.6534 (5)	1.1366 (5)	0.101
O(2*B*)	−0.9839 (11)	0.2759 (6)	0.3077 (5)	0.102
O(3*A*)	−0.9666 (10)	0.8699 (4)	1.1467 (5)	0.074
O(3*B*)	−0.9489 (12)	0.0838 (5)	0.3336 (5)	0.101
O(4*A*)	−1.2423 (9)	0.8660 (5)	0.9892 (5)	0.085
O(4*B*)	−1.1558 (12)	0.1039 (6)	0.4975 (7)	0.146
O(5*A*)	−1.3669 (9)	0.6427 (5)	0.8748 (5)	0.082
O(5*B*)	−1.2091 (11)	0.3108 (7)	0.5611 (7)	0.146
O(6*A*)	−1.3418 (10)	0.7460 (5)	1.1007 (5)	0.097
O(6*B*)	−1.2938 (11)	0.1699 (8)	0.3396 (7)	0.171
O(7*A*)	−1.2651 (9)	0.5086 (5)	0.9456 (5)	0.089
O(7*B*)	−1.0924 (11)	0.4155 (6)	0.4466 (6)	0.121
O(8*A*)	−1.1933 (10)	0.5212 (5)	0.7530 (5)	0.092
O(8*B*)	−0.9802 (16)	0.4796 (8)	0.6690 (6)	0.189
O(9*A*)	−1.1256 (10)	0.6896 (6)	0.7936 (5)	0.105
O(9*B*)	−0.9237 (12)	0.3335 (6)	0.6915 (5)	0.122
C(1*A*)	−0.7710 (13)	0.7697 (6)	1.1316 (6)	0.058
C(1*B*)	−0.7121 (14)	0.1910 (7)	0.3505 (7)	0.066
C(2*A*)	−1.0022 (13)	0.6753 (6)	1.1044 (7)	0.059
C(2*B*)	−0.9174 (15)	0.2655 (7)	0.3488 (7)	0.073
C(3*A*)	−0.9981 (13)	0.8092 (7)	1.1058 (7)	0.064
C(3*B*)	−0.9456 (15)	0.1456 (7)	0.3776 (7)	0.076
C(4*A*)	−1.2090 (12)	0.8181 (7)	0.9957 (7)	0.059
C(4*B*)	−1.1186 (17)	0.1425 (9)	0.4780 (8)	0.105
C(5*A*)	−1.2603 (12)	0.6593 (7)	0.9131 (7)	0.065
C(5*B*)	−1.1196 (14)	0.2996 (8)	0.5322 (7)	0.082
C(6*A*)	−1.2672 (13)	0.7462 (6)	1.0647 (7)	0.059
C(6*B*)	−1.2000 (15)	0.1837 (10)	0.3833 (10)	0.117
C(7*A*)	−1.1982 (12)	0.5450 (6)	0.9335 (6)	0.056
C(7*B*)	−1.0304 (13)	0.3937 (7)	0.4786 (7)	0.069
C(8*A*)	−1.1542 (13)	0.5536 (7)	0.8146 (7)	0.067
C(8*B*)	−0.9639 (17)	0.4322 (9)	0.6184 (8)	0.111
C(9*A*)	−1.0522 (14)	0.7037 (7)	0.8454 (7)	0.076
C(9*B*)	−0.8856 (14)	0.3043 (8)	0.6371 (7)	0.080
C(10*A*)	−0.7794 (13)	0.6296 (6)	1.0230 (6)	0.051
C(10*B*)	−0.6482 (13)	0.3364 (7)	0.4453 (7)	0.061
C(11*A*)	−0.8897 (13)	0.5761 (6)	0.9706 (7)	0.052
C(11*B*)	−0.7368 (12)	0.3879 (7)	0.4819 (7)	0.058
C(12*A*)	−0.9249 (13)	0.5465 (6)	0.8957 (8)	0.059
C(12*B*)	−0.7450 (14)	0.4302 (7)	0.5559 (8)	0.062
C(13*A*)	−0.8294 (13)	0.5605 (7)	0.8508 (7)	0.062
C(13*B*)	−0.6460 (15)	0.4363 (7)	0.6176 (7)	0.068
C(14*A*)	−0.7118 (15)	0.5174 (7)	0.8406 (8)	0.075
C(14*B*)	−0.5082 (19)	0.4819 (9)	0.6305 (13)	0.122
C(15*A*)	−0.6449 (14)	0.5152 (7)	0.9081 (9)	0.081
C(15*B*)	−0.440 (2)	0.467 (2)	0.572 (2)	0.187
C(16*A*)	−0.5708 (13)	0.5863 (7)	0.9714 (7)	0.067
C(16*B*)	−0.4153 (16)	0.3993 (10)	0.5157 (12)	0.115
C(17*A*)	−0.6511 (12)	0.6493 (6)	1.0026 (7)	0.055
C(17*B*)	−0.5223 (14)	0.3329 (8)	0.4857 (8)	0.077
C(111*A*)	−0.6560 (11)	0.8846 (5)	1.0032 (6)	0.033 (3)
C(111*B*)	−0.6214 (12)	0.1120 (6)	0.5158 (6)	0.048 (3)
C(112*A*)	−0.5588 (11)	0.8515 (6)	1.0356 (6)	0.040 (3)
C(112*B*)	−0.5340 (13)	0.1301 (6)	0.4714 (7)	0.053 (3)
C(113*A*)	−0.4633 (12)	0.9111 (6)	1.1039 (6)	0.048 (3)
C(113*B*)	−0.5033 (15)	0.0600 (8)	0.4078 (8)	0.071 (4)
C(114*A*)	−0.5391 (13)	0.9625 (7)	1.1613 (7)	0.058 (4)
C(114*B*)	−0.6310 (15)	0.0064 (8)	0.3611 (8)	0.072 (4)
C(115*A*)	−0.6300 (14)	0.9946 (7)	1.1298 (7)	0.059 (4)
C(115*B*)	−0.7128 (15)	−0.0117 (8)	0.4055 (8)	0.076 (4)
C(116*A*)	−0.7308 (12)	0.9351 (6)	1.0611 (6)	0.046 (3)
C(116*B*)	−0.7484 (14)	0.0584 (7)	0.4679 (7)	0.058 (4)
C(121*A*)	−0.8564 (11)	0.8616 (5)	0.8778 (5)	0.031 (3)
C(121*B*)	−0.7273 (13)	0.1636 (7)	0.6528 (7)	0.054 (3)
C(122*A*)	−0.7785 (12)	0.9264 (6)	0.8784 (6)	0.048 (3)
C(122*B*)	−0.6701 (14)	0.0999 (7)	0.6540 (7)	0.061 (4)
C(123*A*)	−0.8612 (14)	0.9505 (7)	0.8317 (7)	0.060 (4)
C(123*B*)	−0.7102 (17)	0.0902 (9)	0.7167 (9)	0.091 (5)

Table 1 *(cont.)*

	x	y	z	U_{eq}
C(124A)	−0.9929 (13)	0.9682 (7)	0.8551 (7)	0.054 (3)
C(124B)	−0.8525 (17)	0.0799 (8)	0.7126 (8)	0.082 (5)
C(125A)	−1.0699 (13)	0.9066 (7)	0.8587 (7)	0.056 (4)
C(125B)	−0.9143 (16)	0.1414 (8)	0.7100 (8)	0.086 (5)
C(126A)	−0.9857 (13)	0.8841 (6)	0.9063 (6)	0.049 (3)
C(126B)	−0.8743 (16)	0.1490 (8)	0.6452 (8)	0.082 (5)
C(131A)	−0.6553 (11)	0.7617 (6)	0.8579 (6)	0.038 (3)
C(131B)	−0.5001 (12)	0.2543 (6)	0.6448 (6)	0.046 (3)
C(132A)	−0.7339 (13)	0.7044 (7)	0.7816 (7)	0.059 (4)
C(132B)	−0.5165 (14)	0.3270 (7)	0.7130 (7)	0.058 (4)
C(133A)	−0.6431 (15)	0.6546 (7)	0.7352 (7)	0.067 (4)
C(133B)	−0.3843 (15)	0.3801 (7)	0.7481 (7)	0.067 (4)
C(134A)	−0.5249 (15)	0.6989 (8)	0.7290 (8)	0.074 (4)
C(134B)	−0.2767 (17)	0.3451 (8)	0.7682 (9)	0.087 (5)
C(135A)	−0.4496 (14)	0.7575 (7)	0.8021 (7)	0.062 (4)
C(135B)	−0.2576 (16)	0.2736 (8)	0.7040 (8)	0.078 (4)
C(136A)	−0.5424 (13)	0.8079 (7)	0.8480 (7)	0.055 (4)
C(136B)	−0.3926 (14)	0.2216 (7)	0.6680 (7)	0.059 (4)
H(1A)	−0.81650	0.74580	1.01080	0.050
H(1B)	−0.72200	0.23170	0.47690	0.050

Table 2. *Selected bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses*

Pt(1A)—Ru(1A)	2.869 (1)		Pt(1B)—Ru(1B)	2.874 (1)
Pt(1A)—Ru(2A)	2.849 (1)		Pt(1B)—Ru(2B)	2.851 (1)
Pt(1A)—Ru(3A)	2.798 (1)		Pt(1B)—Ru(3B)	2.817 (1)
Ru(1A)—Ru(2A)	2.705 (1)		Ru(1B)—Ru(2B)	2.690 (2)
Ru(1A)—Ru(3A)	2.889 (1)		Ru(1B)—Ru(3B)	2.868 (2)
Ru(2A)—Ru(3A)	2.662 (1)		Ru(2B)—Ru(3B)	2.669 (2)
Pt(1A)—P(1A)	2.370 (3)		Pt(1B)—P(1B)	2.374 (4)
Ru(1A)—C(10A)	2.215 (13)		Ru(1B)—C(10B)	2.247 (13)
Ru(1A)—C(11A)	2.596 (13)		Ru(1B)—C(11B)	2.594 (13)
Ru(3A)—C(11A)	2.586 (13)		Ru(3B)—C(11B)	2.576 (13)
Ru(3A)—C(12A)	2.237 (13)		Ru(3B)—C(12B)	2.255 (13)
Ru(1A)—C(10A)—C(11A)	88.2 (8)		Ru(1B)—C(10B)—C(11B)	86.0 (8)
C(10A)—C(11A)—C(12A)	131.8 (12)		C(10B)—C(11B)—C(12B)	131.9 (12)
Ru(3A)—C(12A)—C(11A)	87.9 (9)		Ru(3B)—C(12B)—C(11B)	86.2 (9)

Discussion. Final positional parameters are given in Table 1, with selected bond distances and angles in Table 2.* The atomic labelling scheme and molecular structure of the two crystallographically independent molecules (*A*) and (*B*) are shown in Figs. 1 and 2. There are no significant differences between these two molecules and the discussion will focus on the metrical parameters of molecule (*A*). A *cyclo*-{μ-η^3-C$_8$H$_{13}$} ligand, formally derived from protonation of cyclooctadiene, is bonded to Ru(1*A*) and Ru(3*A*) and acts as a three-electron donor. Since the Ru(1*A*)—C(10*A*) distance is marginally shorter than the Ru(3*A*)—C(12*A*) separation, the ligand could be regarded as an enyl ligand, σ bonded to Ru(1*A*) and π bonded to Ru(3*A*). However, since the three-carbon-chain bridges the Ru(1*A*)—Ru(3*A*) bond in a nearly symmetric fashion, the ligand may be more profitably viewed as a cyclic μ-η^3-allyl. Similar symmetric μ-allyl bonding modes have been observed in a number of Pd$_2$ and Pt$_2$ complexes (Werner, 1981).

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* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positional parameters, and a complete listing of bond lengths, angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54459 (73 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The formation of this μ-allyl—Ru$_2$ linkage in the title cluster is somewhat unusual. Olefinic bonds normally react with clusters to afford σ-π-vinyl-M_2 moieties through activation of an olefinic C—H bond (see, for example, Bhadhuri, Johnson, Kelland, Lewis, Raithby, Rehani, Sheldrick, Wong & McPartlin, 1979). A five-electron-donor μ_3-allyl bonding mode has also been observed in Ru$_3$(μ_2-H){μ_3-η^3-(Me)C.C(H).C(Et)}(CO)$_9$ (Evans, Hursthouse, Randall, Rosenberg, Milone & Valle, 1972).

The CVE count is 58, implying that the title cluster is electronically unsaturated. The Pt—Ru separations are in the upper range of previously reported distances [2.609 (1)–2.858 (1) Å (Farrugia, 1990)], while the Ru(2*A*)—Ru(3*A*) distance of 2.662 (1) Å is somewhat shorter than the other carbonyl bridged Ru(1*A*)—Ru(2*A*) vector of 2.705 (1) Å. This suggests
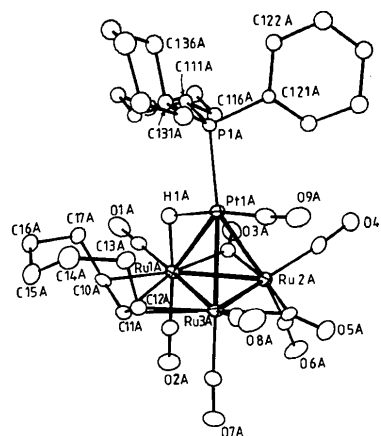

Fig. 1. Molecular structure and atomic labelling scheme for molecule (*A*). Carbonyl C atoms have the same label as attached O atoms.
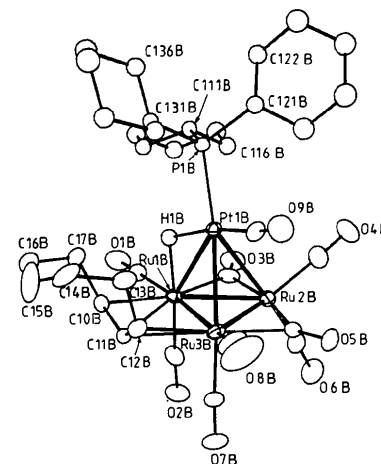

Fig. 2. Molecular structure and atomic labelling scheme for molecule (*B*). Carbonyl C atoms have the same label as attached O atoms.

some possible degree of localization of unsaturation along the former Ru—Ru vector, although the evidence is not strong.

The structure is closely related to that reported for the 58 CVE cluster $Ru_3Pt(\mu\text{-}H)(\mu\text{-}PPh_2)(\mu\text{-}CO)_2\text{-}(CO)_7(PCy_3)$ (2), in which a three-electron donor phosphido group replaces the μ-allyl ligand (Powell, Brewer, Gulia & Sawyer, 1989). The major difference in the metal frameworks is that the M—M distances in the Pt—Ru(1)—Ru(3) face of the title cluster are slightly greater than found for the corresponding face in (2), presumably due to the differing bonding requirements of the PPh_2 versus the allyl ligand. The only other structurally characterized tetrahedral Ru_3Pt species is the 60 CVE cluster $Ru_3Pt\{\mu_3\text{-}\eta^4\text{-}('Bu).C.C.C('Bu).CH_2\}(CO)_8(dppe)$ which has Ru—Pt distances of 2.700 (3)–2.892 (3) Å and Ru—Ru distances of 2.770 (4)–2.904 (4) Å (Farrugia, MacDonald & Peacock, unpublished results).

Note added in proof: A similar $\mu\text{-}\eta^3$ allyl linkage acting as a three-electron donor has recently been reported [Housecroft, Johnson, Lewis, Lunniss, Owen & Raithby (1991). *J. Organomet. Chem.* **409**, 271–284].

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Structure of 2,3-Dicyclohexyl-6,7-dihydro-5H-2aλ^4-thia-2,3,4a,7a-tetraaza-cyclopent[cd]indene-1(2H),4(2H)-diselone

By DAVID G. BILLING, JAN C. A. BOEYENS, LOUIS DENNER, MICHAEL D. HELLYAR, LONG-LI LAI, ANTHONY J. MATTHEE AND DAVID H. REID

Structural Chemistry Group, Department of Chemistry, University of the Witwatersrand, Wits 2050, Johannesburg 2001, South Africa

Abstract. $C_{18}H_{28}N_4SSe_2$, $M_r = 490.435$, monoclinic, $P2_1/c$, $a = 6.455$ (9), $b = 19.990$ (6), $c = 16.43$ (1) Å, $\beta = 101.4$ (10)°, $V = 2078.90$ Å^3, $Z = 4$, $D_x = 1.57$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ(Mo $K\alpha$) = 35.49 cm^{-1}, $F(000) = 992$, $T = 293$ K, $wR = 0.0567$ for 2727 reflections with $F \geq 3\sigma(F)$. The molecule possesses elongated S—N bonds of 1.935 (7) and 1.931 (7) Å, an N—S—N angle of 164.8 (3)° and C=Se bond lengths of 1.844 (9) and 1.826 (9) Å.

Introduction. The determination of the structure of the title compound [(2) R = cyclohexyl] arose from attempts to synthesize new types of 1,6,6aλ^4-triheterapentalene structure (Mitchell & Reid, 1982; Nicol, 1983; Rhodes, 1987; Lai, 1990). 1,6,6aλ^4-Triheterapentalenes contain a sequence of three heteroatoms of Groups V and VI (O, S, Se, Te, NR) in which the two bonds linking adjacent heteroatoms in the sequence are characteristically long, being up

to *ca* 15% longer than the corresponding two-centre two-electron covalent bonds (Mitchell & Reid, 1982; and earlier work and references cited therein). We have found that the 1,2,4-thiadiazolo[4,5-a]-pyrimidine (2), synthesized by the reaction of 5-amino-3-methyl-1,2,4-thiadiazole with 1,3-dibromopropane (Nicol, 1983; Lai, 1990), reacts with isoselenocyanates in boiling toluene with elimination of acetonitrile and concomitant addition of two molecules of the isoselenocyanate to give products which, on the basis of elemental composition, ^{1}H and ^{13}C NMR spectra, and mechanistic considerations, seemed likely to possess structure (2) or structure (3). The same products were also obtained, together with 2,4-dinitrotoluene, by reaction of the tetrahydropyrimidine (4) with the appropriate isoselenocyanates in dichloromethane at room temperature. We now report that the product from the reaction of compound (1) or (4) with cyclohexyl isoselenocyanate