The complex consists of monomeric $PtCl_4(C_6H_{10}N_2)_2$ units. The Pt atom is octahedrally surrounded by four Cl atoms, and by the N atoms from the two 1-propylimidazole ligands in a *cis* geometry. All four Pt—Cl bonds are equal [ranging from 2·310 (3) to 2·322 (3) Å] and are practically identical to those observed in other six-coordinate Pt^{IV} complexes: tetramethylethylenediammonium hexachloroplatinate(IV) [2·304–2·316 (5) Å] (Bisi-Castellani, Manotti-Lanfredi, Tiripicchio, Maresca & Natile, 1984); 2·318 (2) Å in 9-methylguaninium hexachloroplatinate(IV) dihydrate (Terzis & Mentzafos, 1983) and 2·314 (1) Å in K_2PtCl_6 (Ohba & Saito, 1984). The Pt—N(11) and Pt—N(21) bond distances of 2·061 (9) and 2·045 (8) Å are in good agreement with those found for other compounds of Pt^{IV} with nitrogen-donor ligands (Fanizzi, Natile, Maresca, Manotti-Lanfredi & Tiripicchio, 1984).

The structural parameters of the two independent imidazole rings are very similar and are well within the range observed in other metal complexes and derivatives (Navarro-Ranninger, Martinez-Carrera & García-Blanco, 1983; Brouant, Barbe, Marsura & Luu-Duc, 1985). Both imidazole rings are planar; the propyl group is twisted as indicated by the torsion angles C(13)—N(12)—C(14)—C(15) = 80 (1) and C(23)—N(22)—C(24)—C(25) = 67 (2)°. The dihedral angle between the planes of the two imidazole rings is 111 (2)°, while the dihedral angles between the N(11) and N(21) ligand-ring planes and the coordination plane around Pt^{IV} are 124 (1) and 51 (1)° respectively.

There are no abnormally short distances. The crystal structure consists of discrete molecules of $PtCl_4$(1-propylimidazole)$_2$ with adjacent molecules held by van der Waals forces.

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A Neutron Diffraction Study of Bis(cyclopentadienyl)(methyl)(methylene)tantalum(V) at 15 K*

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Abstract. $[Ta(C_5H_5)_2(CH_2)(CH_3)]$, $M_r = 340{\cdot}206$, monoclinic, $P2_1/c$, $a = 6{\cdot}492$ (1), $b = 11{\cdot}539$ (2), $c = 15{\cdot}174$ (2) Å, $\beta = 117{\cdot}33$ (2)°, $V = 1009{\cdot}8$ (3) Å^3, $Z = 4$, $D_x = 2{\cdot}238$ Mg m^{-3}, neutrons, $\lambda = 1{\cdot}1617$ (2) Å, $\mu = 0{\cdot}2473$ mm^{-1}, $F(000) = 122{\cdot}68$ fm. $T = 15(0{\cdot}5)$ K. The structure has been refined based on 2446 reflections to yield an unweighted R value based on F^2 of 0·043. The molecule has approximate C_s point symmetry. All C—H bond distances have been determined with e.s.d.'s ≤ 0·003 Å. The mean C—H distance [1·091 (4) Å] in the methyl group is slightly longer than that in the methylene group [1·081 (5) Å], while the

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respective H—C—H angles are 107·8 (3)° (methyl, mean value) and 112·3 (2)° (methylene). The Ta—C methylene distance is 2·039 (1) Å, corresponding to a full double bond, 0·229 (1) Å shorter than the Ta—C methyl distance of 2·268 (1) Å. The cyclopentadienyl rings have an envelope geometry and exhibit small systematic deviations of the C—C bond distances from the mean value of 1·419 Å; by contrast, the ring C—H distances are equivalent, with a mean value of 1·084 (1) Å.

Introduction. Metal–carbene (or alkylidene) complexes have been postulated as intermediates in catalytic reactions such as reduction of carbon monoxide (Cardin, Cetinkaya, Doyle & Lappert, 1973) and the metathesis of olefins (Ivin, 1983; Dragutan, Balaban & Dimonie, 1985). The simplest of these are methylene complexes. While there are now many examples of complexes that contain bridging methylene (μ-CH_2) ligands (Herrmann, 1982), there are still relatively few stable well characterized complexes that contain terminal methylene ligands (Gilliom & Grubbs, 1986; Holmes, Clark, Turner & Schrock, 1982; Patton, Strouse, Knobler & Gladysz, 1983; Clark, Roper & Wright, 1984; Gallop & Roper, 1986; Guggenberger & Schrock, 1975; Schultz, Williams, Schrock & Holmes, 1984; Hill, Roper, Waters & Wright, 1983; van Asselt, Burger, Gibson & Bercaw, 1986; Fryzuk, MacNeil & Rettig, 1985; Bohle, Clark, Rickard, Roper, Shepard & Wright, 1987). Only two complexes that contain μ-CH_2 ligands have been structurally characterized by neutron diffraction; they are $[Os_3(CO)_{10}(\mu\text{-}H)_2(\mu\text{-}CH_2)]$ (Schultz, Williams, Calvert, Shapley & Stucky, 1979) and $[\{Rh(\eta^5\text{-}C_5H_5)(CO)\}_2(\mu\text{-}CH_2)]$ (Takusagawa, Fumagalli, Koetzle & Herrmann, 1981). To our knowledge none that contains a terminal methylene ligand has been examined by neutron diffraction. Here we report the results of a neutron diffraction study of $[Ta(\eta^5\text{-}C_5H_5)_2(CH_2)(CH_3)]$ that was carried out at Brookhaven.

Experimental. Experimental details and refinement parameters are summarized in Table 1. $[Ta(\eta^5\text{-}C_5H_5)_2(CH_2)(CH_3)]$ was prepared as described in the literature (Schrock, 1975; Schrock & Sharp, 1978). A tabular crystal was mounted on an aluminium pin, oriented approximately parallel to the crystallographic $[10\bar{1}]$ direction. The sample was sealed in quartz under a helium atmosphere and placed in a closed-cycle helium refrigerator (Air Products and Chemicals, Inc. Displex model CS-202) mounted on a four-circle diffractometer at the High Flux Beam Reactor. A Ge (220) crystal monochromator was employed and the neutron-beam wavelength was calibrated based on KBr $a_0 = 6{\cdot}6000$ Å at 298 K.

The cell dimensions are based on $\sin^2\theta$ values of 32 reflections. Intensities were measured in two octants (hkl, $hk\bar{l}$) with $2\theta \leq 106°$ and an additional two octants ($h\bar{k}l$, $h\bar{k}\bar{l}$) with $2\theta \leq 65°$, employing a $\theta/2\theta$ step-scan technique. The scan range was varied according to $\Delta 2\theta = (1{\cdot}69 + 4{\cdot}10\tan\theta)°$ for the high-angle data ($2\theta \geq 65°$) and $\Delta 2\theta = 4{\cdot}0°$ for the low-angle data. The step size was adjusted to give between 60 and 75 steps in each scan. At each point, counts were accumulated for approximately two seconds, the exact time interval being determined by monitoring the incident-beam intensity. Two reflections were monitored every 100 measurements; their intensities did not vary to any significant degree during the entire period of data collection.

Table 1. *Experimental details and refinement parameters*

Crystal weight	6·4 mg
Crystal faces (8)	(100) $(\bar{1}00)$ (010) $(0\bar{1}0)$ (011) $(0\bar{1}\bar{1})$ $(0\bar{1}1)$ $(01\bar{1})$
Range of $\exp(-\mu t)$	0·645–0·805
Number of reflections measured	4009
$R_{\rm int}$	0·044
Number of independent reflections	2446
Range of $(\sin\theta)/\lambda$ (Å^{-1})	0·023–0·687
Range of hkl in asymmetric unit	$0 \leq h \leq 8$, $0 \leq k \leq 15$, $-19 \leq l \leq 17$
Number of parameters	255
$R(F^2)$	0·043
$wR(F^2)$	0·050
$S(F^2)$	1·191

Integrated intensities of reflections were obtained by a conventional peak-minus-background procedure, assuming 15% of the scan at either extremity represented the background intensity. Absorption corrections consisted of numerical integration over a Gaussian grid of points (Busing & Levy, 1957). The error in F^2 ($F^2 = I\sin2\theta$) was estimated as follows: $\sigma^2(F_o^2) = \sigma^2_{\rm count}\sin^2 2\theta + AF_o^4 + B$, where $\sigma_{\rm count}$ is the standard deviation based on counting statistics, and the coefficients A and B were estimated from the discrepancy between symmetry-related reflections. We found that $A = 7{\cdot}2 \times 10^{-4}$ and $B = 2{\cdot}2 \times 10^{-2}$ on an absolute scale. All independent values of F_o^2 were retained in the subsequent refinements.

The X-ray atomic coordinates of non-H atoms (Guggenberger & Schrock, 1975) were used as initial values for the structure refinement. After three least-squares cycles the H atoms were located in a difference map. The resulting model, including positional and anisotropic thermal parameters for all atoms and a type I isotropic-extinction correction (Becker & Coppens, 1975) was refined by a full-matrix least-squares technique, minimizing $\sum w(F_o^2 - k^2F_c^2)^2$, employing a locally modified version of *ORFLS* (Busing, Martin & Levy, 1962). Weights were taken as $w = 1/\sigma^2(F_o^2)$. The most significant extinction-correction value applied to F_c^2 was 0·864 for the reflection 100. Neutron scattering lengths used were $b_{\rm Ta} = 6{\cdot}91$, $b_{\rm O} = 5{\cdot}80$, $b_{\rm C} = 6{\cdot}65$ and $b_{\rm H} = -3{\cdot}74$ fm (Koester, 1977). The neutron scattering length of the Ta atom was varied along with the other parameters, but the value obtained, 6·97 (3) fm,

Table 2. *Positional and equivalent isotropic thermal parameters at* 15 K

	x	y	z	U_{eq}(Å^2)
Ta	0·0847 (1)	0·20999 (5)	0·14729 (4)	0·0100 (2)
C(1*A*)	0·3051 (1)	0·11171 (6)	0·30403 (5)	0·0161 (2)
C(2*A*)	0·1704 (1)	0·02500 (6)	0·23567 (5)	0·0156 (2)
C(3*A*)	−0·0659 (1)	0·04955 (6)	0·20405 (5)	0·0154 (2)
C(4*A*)	−0·0792 (1)	0·15202 (6)	0·25385 (5)	0·0160 (2)
C(5*A*)	0·1505 (1)	0·18960 (6)	0·31635 (5)	0·0166 (3)
C(1*B*)	−0·0280 (1)	0·33966 (6)	0·00779 (5)	0·0165 (3)
C(2*B*)	0·1338 (1)	0·23175 (6)	−0·03227 (5)	0·0150 (2)
C(3*B*)	0·2940 (1)	0·20616 (6)	0·00374 (5)	0·0143 (2)
C(4*B*)	−0·2914 (1)	0·29981 (6)	0·06563 (5)	0·0161 (2)
C(5*B*)	−0·1283 (1)	0·38266 (6)	0·06739 (5)	0·0175 (3)
C(6)	0·2775 (1)	0·10379 (6)	0·08294 (5)	0·0169 (2)
C(7)	0·3393 (1)	0·33089 (7)	0·21297 (5)	0·0186 (3)
H(1*A*)	0·4921 (3)	0·1179 (2)	0·3409 (1)	0·0349 (6)
H(2*A*)	0·2399 (3)	−0·0467 (2)	0·2116 (1)	0·0331 (6)
H(3*A*)	−0·2124 (3)	0·0003 (2)	0·1509 (1)	0·0333 (6)
H(4*A*)	−0·2382 (3)	0·1930 (2)	0·2460 (1)	0·0334 (6)
H(5*A*)	0·1997 (4)	0·2645 (2)	0·3644 (1)	0·0339 (6)
H(1*B*)	0·1062 (4)	0·3809 (2)	−0·0042 (1)	0·0349 (7)
H(2*B*)	−0·0949 (3)	0·1764 (2)	−0·0799 (1)	0·0305 (6)
H(3*B*)	−0·3976 (3)	0·1273 (1)	−0·0113 (1)	0·0304 (6)
H(4*B*)	−0·3972 (3)	0·3068 (2)	0·1037 (1)	0·0319 (7)
H(5*B*)	−0·0859 (4)	0·4633 (1)	0·1087 (1)	0·0346 (7)
H(6*A*)	0·4395 (3)	0·0703 (2)	0·1415 (1)	0·0400 (7)
H(6*B*)	0·3169 (4)	0·1569 (2)	0·0336 (1)	0·0397 (7)
H(6*C*)	0·1754 (4)	0·0292 (2)	0·0398 (1)	0·0384 (7)
H(7*A*)	0·4709 (4)	0·3262 (2)	0·2898 (1)	0·0410 (7)
H(7*B*)	0·3444 (4)	0·4117 (2)	0·1786 (2)	0·0417 (7)
Cp(*A*)*	0·0962	0·1056	0·2628	—
Cp(*B*)*	−0·1751	0·2920	0·0225	—

* Ring centroid.

agreed with the literature value [6·91 (7) fm] (Koester, 1977) within one standard deviation. Final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 2.*

Discussion. A schematic perspective drawing of the molecule is shown in Fig. 1(*a*), and a view that shows atomic nomenclature and essential bond distances can be found in Fig. 1(*b*). A list of complete bond distances and selected bond angles is presented in Table 3. There are no unusual intermolecular contacts: The shortest intermolecular H···H distance is H(3*B*)···H(6*B*) ($x, y, -1+z$) = 2·275 (3) Å. A stereoview showing the crystal packing is given in Fig. 2.

The molecule has approximate C_s point symmetry in which C(6), H(6*c*), Ta and C(7) lie in the mirror plane; the root-mean-square displacement from idealized point symmetry, $\langle \Delta^2 \rangle^{1/2}$, is 0·020 Å. The cyclopentadienyl rings are eclipsed relative to one another with a centroid–Ta–centroid angle of 135·95 (4)°. The cyclopentadienyl rings have an envelope geometry and exhibit small systematic deviations of the C—C bond distances about the mean value of 1·419 Å. This pattern of deviations follows the C_s molecular symmetry, as do the displacements of the ring C atoms from their planes which range in magnitude from 0·001 to 0·008 Å. Similar deviations in ring C—C bond distances have been observed in other η^5-C_5H_5 complexes, *e.g.* in [{Rh(η^5-C_5H_5)(CO)}$_2$(μ-CH_2)] (Takusagawa, Fumagalli, Koetzle & Herrmann, 1981), [Fe(η^5-C_5H_5)$_2$(CO)$_2$]$_2$ (Mitschler, Rees & Lehmann,

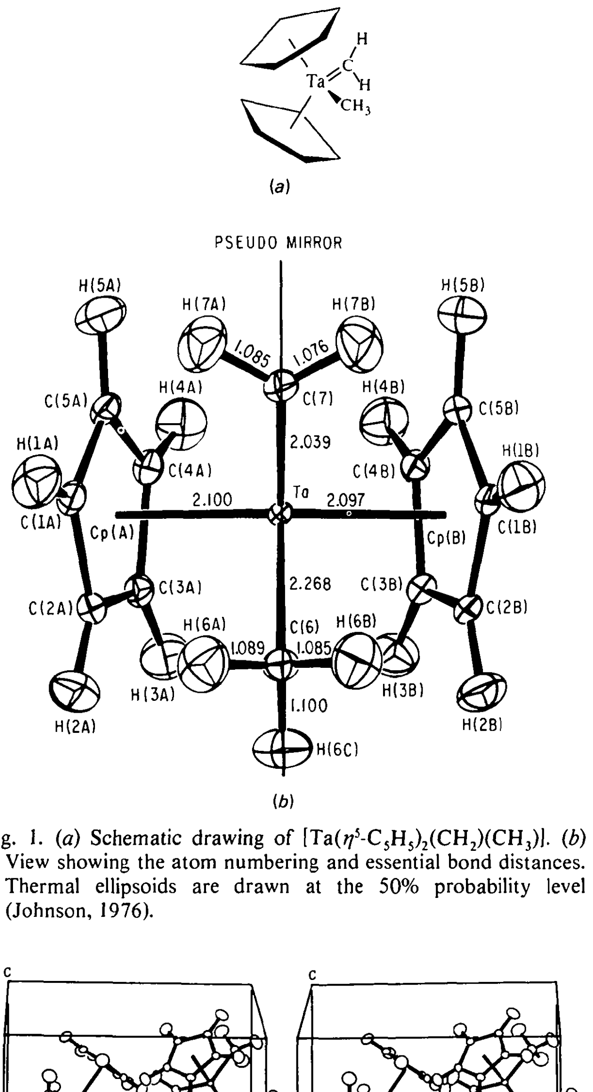

Fig. 1. (*a*) Schematic drawing of [Ta(η^5-C_5H_5)$_2$(CH_2)(CH_3)]. (*b*) View showing the atom numbering and essential bond distances. Thermal ellipsoids are drawn at the 50% probability level (Johnson, 1976).

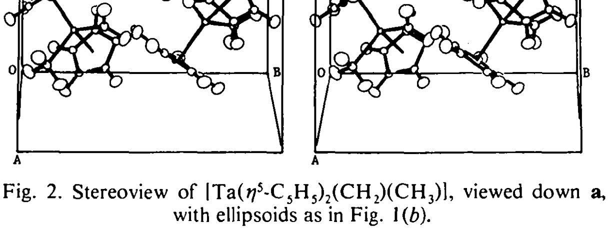

Fig. 2. Stereoview of [Ta(η^5-C_5H_5)$_2$(CH_2)(CH_3)], viewed down **a**, with ellipsoids as in Fig. 1(*b*).

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* Lists of anisotropic thermal parameters, observed and calculated squared structure factors, and complete bond angles, together with a figure showing displacements of atoms in η^5-C_5H_5 rings from least-squares planes, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44469 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond distances* (Å) *and selected bond angles* (°)

Ta—C(6)	2·268 (1)	Ta—C(7)	2·039 (1)
C(6)—H(6*A*)	1·089 (2)	C(7)—H(7*A*)	1·085 (2)
C(6)—H(6*B*)	1·085 (2)	C(7)—H(7*B*)	1·076 (3)
C(6)—H(6*C*)	1·100 (2)		
Ta—Cp(*A*)	2·100 (1)	Ta—Cp(*B*)	2·097 (1)
C(1*A*)—C(2*A*)	1·417 (1)	C(1*B*)—C(2*B*)	1·417 (1)
C(1*A*)—C(5*A*)	1·422 (1)	C(1*B*)—C(5*B*)	1·424 (1)
C(2*A*)—C(3*A*)	1·410 (1)	C(2*B*)—C(3*B*)	1·410 (1)
C(3*A*)—C(4*A*)	1·427 (1)	C(3*B*)—C(4*B*)	1·427 (1)
C(4*A*)—C(5*A*)	1·419 (1)	C(4*B*)—C(5*B*)	1·418 (1)
C(1*A*)—H(1*A*)	1·081 (2)	C(1*B*)—H(1*B*)	1·079 (2)
C(2*A*)—H(2*A*)	1·083 (2)	C(2*B*)—H(2*B*)	1·078 (2)
C(3*A*)—H(3*A*)	1·083 (2)	C(3*B*)—H(3*B*)	1·092 (2)
C(4*A*)—H(4*A*)	1·091 (2)	C(4*B*)—H(4*B*)	1·085 (2)
C(5*A*)—H(5*A*)	1·080 (2)	C(5*B*)—H(5*B*)	1·085 (1)
Mean values*			
C—H_{methyl}	1·091 (4)	C—C_{ring}	1·419
C—$H_{methylene}$	1·081 (5)	C—H_{ring}	1·084 (1)
Ta—Cp	2·099 (2)		
Cp(*A*)—Ta—Cp(*B*)	135·95 (4)	Ta—C(6)—H(6*A*)	110·9 (1)
Cp(*A*)—Ta—C(6)	103·55 (4)	Ta—C(6)—H(6*B*)	110·1 (1)
Cp(*A*)—Ta—C(7)	105·93 (4)	Ta—C(6)—H(6*C*)	112·2 (1)
Cp(*B*)—Ta—C(6)	103·24 (3)	H(6*A*)—C(6)—H(6*B*)	108·5 (2)
Cp(*B*)—Ta—C(7)	105·55 (4)	H(6*A*)—C(6)—H(6*C*)	107·5 (2)
C(6)—Ta—C(7)	95·53 (4)	H(6*B*)—C(6)—H(6*C*)	107·5 (1)
		Ta—C(7)—H(7*A*)	123·6 (1)
		Ta—C(7)—H(7*B*)	123·7 (1)
		H(7*A*)—C(7)—H(7*B*)	112·3 (2)
Mean values			
H—C—H_{methyl}	107·8 (3)		
C—C—C_{ring}	108·00 (8)		
C—C—H_{ring}	126·0 (1)		

* E.s.d.'s of mean values calculated as $\sigma = [\sum \Delta_i^2/n(n-1)]^{1/2}$.

1978), and [$M(\eta^5$-$C_5H_5)(CO)_3$], M = Mn (Fitzpatrick, Le Page, Sedman & Butler, 1981) or M = Re (Fitzpatrick, Le Page & Butler, 1981). The ring C—H distances in [$Ta(\eta^5$-$C_5H_5)_2(CH_2)(CH_3)$] are equivalent, with a mean value of 1·084 (1) Å, essentially identical to that found by neutron diffraction at 15 K in [$Ru(\eta^5$-$C_5H_5)_2$] (Takusagawa & Koetzle, 1980) and [{$Rh(\eta^5$-$C_5H_5)(CO)$}$_2(\mu$-$CH_2)$] (Takusagawa, Fumagalli, Koetzle & Herrmann, 1981).

The mean value for the H—C—H angles in the methyl ligand is 107·8 (3)° and the mean C—H distance is 1·091 (4) Å. The mean C—H value is essentially identical to that found in ethane (Kuchitsu, 1968) [1·096 (2) Å].

The methylene ligand is the main focus of this study. The H—C—H angle [112·3 (2)°] is significantly larger than those found in bridging methylene ligands, *e.g.* 106·0(8)° in [$Os_3(CO)_{10}(\mu$-$H)_2(\mu$-$CH_2)$] (Schultz, Williams, Calvert, Shapley & Stucky, 1979), and 110·4 (1)° in [Rh{$(\eta^5$-$C_5H_5)(CO)$}$_2(\mu$-$CH_2)$] (Takusagawa, Fumagalli, Koetzle & Herrmann, 1981), but significantly smaller than that [117·4 (3)°] found in ethylene in the gas phase (Duncan, 1974). The mean methylene C—H distance in [$Ta(\eta^5$-$C_5H_5)_2(CH_2)$-(CH_3)] is 1·081 (5) Å, not significantly different from the value in ethylene of 1·087 (2) Å (Duncan, 1974). Note that the mean C—H distance in the μ-CH_2 ligand in [$Os_3(CO)_{10}(\mu$-$H)_2(\mu$-$CH_2)$] is 1·091 (1) Å (Schultz, Williams, Calvert, Shapley & Stucky, 1979) and in [{$Rh(\eta^5$-$C_5H_5)(CO)$}$_2(\mu$-$CH_2)$] is 1·095 (1) Å (Takusagawa, Fumagalli, Koetzle & Herrmann, 1981), more consistent with the C—H distance in a methyl group, which is logically what one might expect.

Further evidence that the Ta—C(7) bond is a full double bond consists of the 0·229 (1) Å difference in bond distance between it and Ta—C(6). For comparison the difference between the C—C distances in ethane (Kuchitsu, 1968) and ethylene (Duncan, 1974) is 0·194 (2) Å.

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5-Methyl-2,8-diselena-1,3,7-triphosphatricyclo[3.3.1.$0^{3,7}$]nonane 1-Selenide

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Abstract. $C_5H_9P_3Se_3$, $M_r = 398{\cdot}93$, monoclinic, $P2_1/c$, $a = 6{\cdot}643\ (2)$, $b = 14{\cdot}318\ (5)$, $c = 11{\cdot}636\ (7)$ Å, $\beta = 95{\cdot}40\ (9)°$, $V = 1102\ (2)$ Å^3, $Z = 4$, $D_x = 2{\cdot}405$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}70926$ Å, $\mu = 102{\cdot}89$ cm^{-1}, $F(000) = 744$, $T = 295\ (2)$ K, final $R = 0{\cdot}061$ for 1551 unique reflections. The molecule consists of a tricyclic noradamantane cage. The basis of the cage is a five-membered $P_2Se_2P(Se)$ ring with an exocyclic Se atom. The organic group $CH_3C(CH_2)_3$ bridging the three P atoms forms a heteronoradamantane ring system. It has an enlarged cage structure with an expanded C—C(H$_2$)—P(=Se) bond angle of 121° at the methylene C atom. The mean value of the P—Se distances within the P_2Se_2P ring is 2·246 Å and the exocyclic P=Se length is 2·089 (2) Å. All other distances and angles are normal.

Introduction. Melting of 4-methyl-1,2,6-triphosphatricyclo[2.2.1.$0^{2,6}$]heptane [$CH_3C(CH_2P)_3$, P_3-nortricyclane] with selenium in the molar ratio of 1:3 results in the formation of the title compound $CH_3C(CH_2P)_3Se_3$ (Ellermann, Demuth & Bauer, 1986). From the composition it is comparable to $CH_3C(CH_2P)_3S_3$ (Zimmermann, Gomm, Ellermann & Köck, 1987) and $CH_3C(CH_2As)_3S_3$ (Ellermann, Lietz, Merbach, Thiele & Zoubek, 1979). Whereas the phosphorus–sulfur compound consists of a noradamantane cage with a five-membered $P_2S_2P(=S)$ ring and an exocyclic P=S bond, the arsenic–sulfur derivative consists of an adamantane cage containing a six-membered As_3S_3 ring. In order to establish whether $CH_3C(CH_2P)_3Se_3$ is a noradamantane or an adamantane derivative, a crystal-structure analysis has been undertaken.

Experimental. Recrystallization of $C_5H_9P_3Se_3$ from tetrahydrofuran gave light-yellow crystals; spherical crystal with $r = 0{\cdot}11\ (1)$ mm used for X-ray analysis; Philips PW 1100 diffractometer (graphite monochromator, Mo $K\alpha$ radiation), ω–2θ scans with scan width $(0{\cdot}800 + 0{\cdot}345\tan\theta)°$; profile analysis with a modified Lehmann & Larsen (1974) procedure; lattice parameters refined by least-squares procedure *GIVER* (Krogmann, 1966) on the basis of 34 reflections with $22{\cdot}3 \leq 2\theta \leq 25{\cdot}1°$; spherical absorption-correction factor $A = 4{\cdot}723 \pm 0{\cdot}185$; index range $h \pm 7$, $k\ 0/15$, $l\ 0/12$; six standard reflections (intensity variation 0·7%); $(\sin\theta)_{\max}/\lambda = 0{\cdot}55$ Å^{-1}, 6198 reflections were measured, 266 reflections affected strongly by absorption were eliminated, after averaging symmetrical equivalents, 1551 reflections [40 unobserveds included, $I < 3\sigma(I)$] remained; $R_{\text{int}} = 0{\cdot}032$. Structure solved by direct methods for Se and P, subsequent Fourier map revealed all non-H atoms, all H atoms were located from a difference Fourier map; the residual electron density (0·58 to $-0{\cdot}66$ e Å^{-3}) prevents a precise refinement of the H coordinates. Blocked least-squares refinement based on F and weights $w = 1/\sigma^2(|F_o|)$, final cycles refined scale factor, coordinates, anisotropic temperature factor for non-H atoms; the isotropic temperature factor of some H atoms became negative and a fixed overall $U = 0{\cdot}0396$ Å^2 was employed; $R = 0{\cdot}061$, $wR = 0{\cdot}029$, $S = 2{\cdot}02$, max. $\Delta/\sigma = 0{\cdot}56$; scattering factors taken from *International Tables for X-ray Crystallography* (1974). The *CRYSTAN* system (Burzlaff, Gomm & Zimmermann, 1986) was used for all computations and diagrams were drawn with *ORTEP*II (Johnson, 1977).

Discussion. Atomic coordinates and equivalent isotropic temperature factors are reported in Table 1.

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