

**Table II. Atomic Coordinates ( $\times 10^4$ ) and Isotropic Thermal Parameters ( $\text{\AA}^2 \times 10^3$ ) for 8**

	x	y	z	$U^a$
Rh	2397.2 (4)	1846.8 (2)	1876.4 (2)	36.9 (1)
P(1)	1967 (2)	443.0 (9)	1797.1 (8)	57.6 (5)
P(2)	2871 (2)	3236.9 (9)	2049.1 (7)	54.8 (5)
O(1)	4519 (3)	1607 (2)	1755 (2)	55 (1)
O(2)	3103 (4)	1720 (2)	2890 (2)	59 (1)
C(1)	1873 (8)	-29 (5)	2568 (3)	99 (3)
C(2)	3254 (7)	-153 (4)	1481 (4)	89 (3)
C(3)	382 (7)	104 (4)	1317 (4)	89 (3)
C(4)	2429 (7)	3996 (4)	1428 (3)	85 (3)
C(5)	2194 (9)	3670 (5)	2727 (4)	108 (4)
C(6)	4676 (7)	3420 (4)	2262 (4)	89 (3)
C(7)	1655 (4)	2021 (3)	935 (2)	36 (2)
C(8)	460 (4)	2395 (3)	690 (2)	38 (2)
C(9)	-448 (4)	2794 (3)	1106 (2)	41 (2)
C(10)	-397 (4)	2621 (3)	1726 (2)	44 (2)
C(11)	458 (5)	1984 (4)	2103 (3)	57 (2)
C(12)	5457 (5)	1362 (3)	2190 (3)	58 (2)
C(13)	5363 (6)	1300 (4)	2846 (3)	67 (2)
C(14)	4281 (7)	1512 (4)	3151 (3)	70 (2)
C(15)	6774 (6)	1142 (4)	1952 (4)	84 (3)
C(16)	4455 (8)	1481 (6)	3870 (3)	113 (4)
C(21)	2037 (3)	1001 (2)	79 (2)	67 (2)
C(22)	2853	666	-342	102 (3)
C(23)	4146	989	-365	115 (4)
C(24)	4625	1645	32	103 (4)
C(25)	3809	1979	453	64 (2)
C(26)	2516	1657	476	46 (2)
C(31)	798 (3)	2822 (3)	-424 (2)	64 (2)
C(32)	308	2913	-1075	84 (3)
C(33)	-993	2635	-1320	88 (3)
C(34)	-1805	2265	-913	74 (2)
C(35)	-1315	2174	-262	58 (2)
C(36)	-14	2452	-17	47 (2)
C(41)	-977 (3)	4075 (2)	451 (2)	66 (2)
C(42)	-1878	4689	194	81 (3)
C(43)	-3232	4670	300	75 (3)
C(44)	-3684	4036	663	84 (3)
C(45)	-2783	3422	920	59 (2)
C(46)	-1430	3442	814	41 (2)

<sup>a</sup> Equivalent isotropic  $U$  defined as one-third of the trace of the orthogonalized  $U_{ij}$  tensor.

selected bond distances and angles is shown in Figure 2, and fractional atomic coordinates appear in Table II. In the solid state the metallacyclic ring is puckered and contains localized single and double bonds between carbon atoms as does its previously characterized 1-platinacyclohexa-2,4-diene relative 1.<sup>2a</sup> In sharp contrast to 1, whose  $\text{CH}_2$  protons are inequivalent at room temperature,<sup>2a</sup> low-temperature NMR studies of 6 indicate that even at  $-96^\circ\text{C}$  the analogous protons are either equivalent or the ring is undergoing rapid inversion. The metallacyclic ring in octahedral 8 is significantly less puckered in the solid state than that in square-planar 1, presumably due to the presence of  $\text{PMe}_3$  above and below the organic ring. An iridacyclohexadiene complex 9 has recently been structurally characterized by Bleeker;<sup>13</sup> in this compound the

(11) Crystal data for 8: monoclinic,  $P2_1/c$ ,  $a = 9.934(2) \text{\AA}$ ,  $b = 16.243(5) \text{\AA}$ ,  $c = 21.070(7) \text{\AA}$ ,  $\beta = 98.42(2)^\circ$ ,  $V = 3361(1) \text{\AA}^3$ ,  $Z = 4$ ,  $D(\text{calcd}) = 1.28 \text{ g cm}^{-3}$ ,  $\mu(\text{Mo K}\alpha) = 6.2 \text{ cm}^{-1}$ ,  $t = 294 \text{ K}$ . A yellow crystal,  $0.20 \times 0.23 \times 0.31 \text{ mm}$ , was obtained by crystallization from hexane. Of 6335 reflections collected ( $4^\circ \leq 2\theta \leq 50^\circ$ ), 5924 were unique ( $R_{\text{int}} = 1.95\%$ ) and 3989 were observed at the  $3\sigma(R_{\text{int}})$  level. No correction for absorption was required. A Patterson synthesis provided the location of the Rh atom. With all non-hydrogen atoms anisotropic and all hydrogen atoms idealized:  $R(F) = 4.37\%$ ,  $R(wF) = 5.51\%$ ,  $\text{GOF} = 1.113$ ,  $\Delta/\sigma = 0.013$ ,  $\Delta(\rho) = 0.49 \text{ e \AA}^{-3}$  ( $1.05 \text{ \AA}$  from Rh),  $N_{\text{obs}}/N_{\text{calc}} = 12.6$ . All software as in ref 7.

(12) While metallabenzenes have been the subject of theoretical studies (Thorn, D. L.; Hoffmann, R. *Nouv. J. Chim.* 1979, 3, 39), only one example of an osmabenzene has been reported to date (Elliot, G. P.; Roper, W. R.; Waters, J. M. *J. Chem. Soc., Chem. Commun.* 1982, 811).

(13) Bleeker, J. R.; Peng, W.-J. *Organometallics*, preceding paper in this issue.

metallacyclic ring is essentially planar. In light of these results we feel that the puckering of the metallacyclohexadiene rings in 1 and 8 probably results from minimization of steric repulsions between the phenyl substituents.

In conclusion, our results clearly demonstrate the stepwise nature of metal-promoted ring opening/ring closure reactions of vinylcyclopropenes. Further studies on the chemistry of these new metallacycles, particularly their potential for transformation to metallabenzenes (or metallacyclohexatrienes), is in progress.

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**Registry No.** 2, 62747-62-0; 3a, 108560-73-2; 3b, 108560-74-3; 5a, 108560-76-5; 5b, 108560-78-7; 6, 108560-77-6; 7, 108560-79-8; *trans*-8, 108646-09-9; *cis*-8, 108560-72-1;  $\text{RhCl}(\text{PMe}_3)_3$ , 36103-64-7;  $[\text{RhCl}(\text{C}_6\text{H}_4)_2]_2$ , 12279-09-3;  $\text{IrCl}(\text{PMe}_3)_2$ , 108560-75-4;  $\text{Rh}(\text{acac})(\text{N-C}_2\text{H}_4)_2$ , 12082-47-2;  $\text{Ti}(\text{acac})$ , 14219-90-0;  $\text{PMe}_3$ , 594-09-2; *P-i-Pr*<sub>3</sub>, 6476-36-4; 1,2,3-triphenylcyclopentadiene, 108535-09-7.

**Supplementary Material Available:** Tables of bond distances and angles, anisotropic thermal parameters, and hydrogen-atom coordinates (8 pages); listings of observed vs. calculated structure factors (54 pages). Ordering information is given on any current masthead page.

### New Tantalum Ylide Complexes: Crystal and Molecular Structure of $(\eta^5\text{-C}_5\text{Me}_5)\text{Cl}_4\text{Ta}(\text{CH}_2=\text{PMePh}_2)$ Containing a Neutral Phosphorus Ylide

Rosa Fandos, Manuel Gómez, and Pascual Royo\*

*Departamento de Química Inorgánica*

*Universidad de Alcalá de Henares, Campus Universitario Alcalá de Henares, Spain*

Severino García-Blanco, Sagrario Martínez-Carrera, and Juliana Sanz-Aparicio

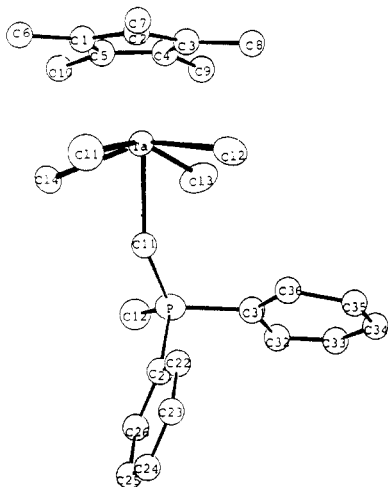
*Departamento de Rayos X, Instituto Rocasolano CSIC Madrid, Spain*

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**Summary:**  $(\eta^5\text{-C}_5\text{Me}_5)\text{TaCl}_4$  reacts with methylene-phosphoranes to give the neutral ylide derivatives  $(\eta^5\text{-C}_5\text{Me}_5)\text{Cl}_4\text{Ta}(\text{CH}_2=\text{PRR}')_2$  where  $\text{R} = \text{Me}$  or  $\text{Ph}$  and  $\text{R}' = \text{Me}$  or  $\text{Ph}$ . The X-ray crystal structure of complex with  $\text{R} = \text{Me}$  and  $\text{R}' = \text{Ph}$  shows a pseudooctahedral geometry with the ylide ligand trans to the pentamethylcyclopentadienyl ring.

Intermediate tantalum phosphorus ylide derivatives have been formulated in alkylidene-transfer reactions from alkylidene-phosphoranes,<sup>1</sup> but as far as we know, only two tantalum complexes containing a phosphoniomethanide fragment have been isolated.<sup>2</sup> Here we report the first

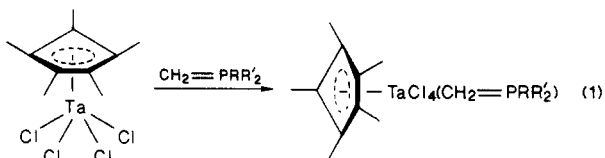
(1) Sharp, P. R.; Schrock, R. R. *J. Organomet. Chem.* 1979, 171, 43.



**Figure 1.** Molecular structure of  $(\eta^5\text{-C}_5\text{Me}_5)\text{Cl}_4\text{Ta}(\text{CH}_2=\text{PMePh}_2)$  with the atomic numbering scheme. Bond lengths (Å): Ta–C<sub>5</sub>Me<sub>5</sub>, 2.252; Ta–Cl1, 2.450 (16); Ta–Cl2, 2.470 (25); Ta–Cl3, 2.295 (26); Ta–Cl4, 2.418 (18); Ta–C11, 2.349 (29); P–C11, 1.812 (30); P–C12, 1.835 (42); P–C21, 1.877; P–C31, 1.840. Bond angles (deg): C<sub>5</sub>Me<sub>5</sub>–Ta–Cl1, 101.6; C<sub>5</sub>Me<sub>5</sub>–Ta–Cl2, 103.4; C<sub>5</sub>Me<sub>5</sub>–Ta–Cl3, 103.2; C<sub>5</sub>Me<sub>5</sub>–Ta–Cl4, 100.2; C<sub>5</sub>Me–Ta–C11, 173.0; C11–Ta–C12, 84.7 (7); C11–Ta–C13, 155.3 (8); C11–Ta–C14, 84.9 (5); C11–Ta–C11, 71.7 (8); C12–Ta–Cl3, 88.0 (8); C12–Ta–Cl4, 155.5 (7); C12–Ta–C11, 75.0 (9); C13–Ta–Cl4, 92.3 (8); C13–Ta–C11, 83.6 (10); C14–Ta–C11, 80.7 (8); C21–P–C31, 99.3; C12–P–C31, 106.2; C12–P–C21, 120.6; C11–P–C31, 113.3; C11–P–C21, 98.3; C11–P–C12, 117.7 (19); Ta–C11–P, 124.9 (15).

tantalum complexes containing neutral phosphorus ylide ligands.

The reaction of  $(\eta^5\text{-C}_5\text{Me}_5)\text{TaCl}_4$  with an equimolar amount of methylenephosphoranes ( $\text{CH}_2=\text{PRR}'_2$ ) gives orange crystals of  $(\eta^5\text{-C}_5\text{Me}_5)\text{Cl}_4\text{Ta}(\text{CH}_2=\text{PRR}'_2)_3$  (R = R' = Me, 1; R = Ph and R' = Me, 2; R = Me and R' = Ph, 3; R = R' = Ph, 4) (eq 1).



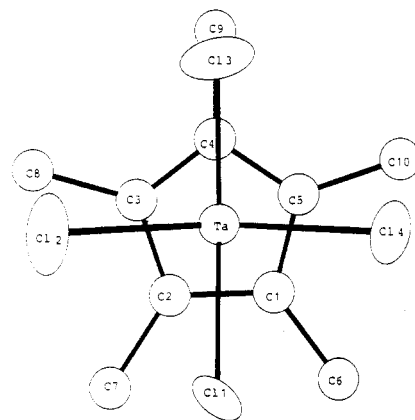
Complexes 1–4 were analytically<sup>4</sup> and spectroscopically characterized. The <sup>1</sup>H NMR spectra<sup>5</sup> show two doublets for the methylene ( $\delta$  2.74–3.97 (<sup>2</sup>J<sub>P–H</sub> = 15.3–16.15 Hz)) and the methyl ( $\delta$  0.94–2.16 (<sup>2</sup>J<sub>P–H</sub> = 13.2–13.7 Hz)) protons of the ylide ligand and one singlet for the methyl ring protons ( $\delta$  2.34–2.41). The <sup>31</sup>P NMR spectra show one

(2) (a) Belmonte, P.; Schrock, R. R.; Churchill, M. R.; Youngs, W. J. *J. Am. Chem. Soc.* **1980**, *102*, 2858. (b) Churchill, M. R.; Youngs, W. J. *Inorg. Chem.* **1981**, *20*, 382. (c) Messerle, L. W.; Jenische, P.; Schrock, R. R.; Stucky, G. J. *Am. Chem. Soc.* **1980**, *102*, 6744.

(3) In a typical experiment,  $(\eta^5\text{-C}_5\text{Me}_5)\text{TaCl}_4$  (0.92 g, 2.00 mmol) was added to a THF (50 mL) solution of phosphorus ylide (2.05 mmol). After the solution was stirred for 20 h at room temperature, the solvent was pumped off and the residue extracted with toluene and crystallized by evaporation after addition of hexane, to give the title compounds in ca. 70% yield.

(4) Anal. Found (Calcd) for 1: C, 31.1 (30.9); H, 4.3 (4.7). Found (Calcd) for 2: C, 37.2 (37.4); H, 4.7 (4.6). Found (Calcd) for 3: C, 43.2 (42.9); H, 4.8 (4.5). Found (Calcd) for 4: C, 47.9 (47.4).

(5) <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>, 80 MHz,  $\delta$ ): 1, 0.94 (9 H, d, <sup>2</sup>J<sub>P–H</sub> = 13.4 Hz, PMe<sub>3</sub>), 2.41 (15 H, s, C<sub>5</sub>Me<sub>5</sub>), 2.74 (2 H, d, <sup>2</sup>J<sub>P–H</sub> = 15.4 Hz, CH<sub>2</sub>P); 2, 1.62 (6 H, d, <sup>2</sup>J<sub>P–H</sub> = 13.24 Hz, PMe<sub>2</sub>Ph), 2.42 (15 H, s, C<sub>5</sub>Me<sub>5</sub>), 2.98 (2 H, d, <sup>2</sup>J<sub>P–H</sub> = 16.15 Hz, CH<sub>2</sub>P), 6.87 (5 H, m, PhP); 3, 2.16 (3 H, d, <sup>2</sup>J<sub>P–H</sub> = 13.7 Hz, PMe), 2.36 (15 H, s, C<sub>5</sub>Me<sub>5</sub>), 3.49 (2 H, d, <sup>2</sup>J<sub>P–H</sub> = 15.3 Hz, CH<sub>2</sub>P), 6.45–7.40 (10 H, m, PhP); 4, 2.34 (15 H, s, C<sub>5</sub>Me<sub>5</sub>), 3.97 (2 H, d, <sup>2</sup>J<sub>P–H</sub> = 16.15 Hz, CH<sub>2</sub>P), 6.90–7.68 (5 H, m, PhP), with respect to Me<sub>4</sub>Si. <sup>31</sup>P NMR (THF, gated decoupled,  $\delta$ ): 1, 27.62 (s, CH<sub>2</sub>PMe<sub>3</sub>); 2, 29.66 (s, CH<sub>2</sub>PMe<sub>2</sub>Ph); 3, 34.99 (s, CH<sub>2</sub>PMePh<sub>2</sub>); 4, 36.43 (s, CH<sub>2</sub>PPh<sub>3</sub>), with respect to H<sub>3</sub>PO<sub>4</sub>.



**Figure 2.** The fragment  $(\eta^5\text{-C}_5\text{Me}_5)\text{Cl}_4\text{Ta}$  projected into the plane of the  $(\eta^5\text{-C}_5\text{Me}_5)$  ring.

singlet for the phosphorus atom between  $\delta$  27.62 and  $\delta$  36.43. This signal is shifted to higher fields in the order expected from the different electronegativity of the R and R' groups.

An X-ray analysis<sup>6–11</sup> of **3** was undertaken to give the structure shown in Figure 1, with the tantalum atom in a pseudooctahedral geometry with the ylide ligand trans to the pentamethylcyclopentadienyl ring. The compound presents a mean value of 102.1° for Cp(centroid)–Ta–Cl angles, a value of 173.0° for the Cp(centroid)–Ta–C(ylide) angle, and average angles between Cl atoms in a cis position of 87.5° and in a trans position of 155.4°. The average value for the Ta–C(Cp) distances is 2.557 Å, and the tantalum atom lies 2.252 Å from the pentamethylcyclopentadienyl plane. Such a value is slightly longer than the distance found in  $\{(\eta^5\text{-C}_5\text{Me}_5\text{Et})\text{TaCl}_2\}_2(\text{H})(\mu\text{-CHPMe}_3)(\mu\text{-O})$ ,<sup>2b</sup> and this could be attributed either to the higher electronic density around the tantalum atom or to steric effects.

(6) Crystal data for **3**: TaCl<sub>4</sub>PC<sub>24</sub>H<sub>30</sub>, *M*, 672.23; orthorhombic; space group *P2<sub>1</sub>c1*; *a* = 13.247 (1), *b* = 20.335 (3), *c* = 9.495 (2) Å; *V* = 2557.0 (6) Å<sup>3</sup>; *Z* = 4, *D*<sub>calcd</sub> = 1.746 Mg m<sup>−3</sup>;  $\lambda$  (Mo K $\alpha$ ) = 0.7107 Å;  $\mu$  = 4.743 mm<sup>−1</sup>; *F*(000) = 1320. X-ray experiments were carried out by using an Enraf-Nonius CAD-4 four circle diffractometer, with graphite-monochromated Mo K $\alpha$  radiation ( $\omega/2\theta$  scan). Intensities of 3837 unique reflections were measured ( $2 < \theta < 30^\circ$ ), 2112 reflections observed with *I* > 4 $\sigma$ (*I*). In reducing the data Lorentz and polarization factors were applied, while absorption correction was made after the isotropic refinement.<sup>8</sup> The structure was solved by a combination of Patterson, direct<sup>9</sup>, and Fourier methods. When trying to refine atom parameters by full-matrix least-squares procedures with isotropic temperature factors, the geometry of the pentamethylcyclopentadienyl and the phenyl (C21–C26) rings were destroyed and thus their positions were optimized and maintained fixed. Later on, the same effect was produced with the other phenyl ring, so it was fixed as well; in the subsequent refinement, anisotropic temperature factors were applied only to non-C atoms. H atoms were located by geometric calculations (C–H = 1.0 Å) and included fixed in the refinement. Empirical weighting scheme<sup>10</sup> was used as to give no trends in  $\langle w\Delta F \rangle$  vs.  $\langle F_o \rangle$  and  $\langle (\sin \theta) / \lambda \rangle$ ; final refinement gave *R* = 0.094 (*R*<sub>w</sub> = 0.108). The calculations were performed with the X-ray System<sup>11</sup> on a Vax 11/750 computer.

(7) (a) Engelter, C.; Moss, J. R.; Niven, N. L.; Nassimbeni, L. R.; Reid, G. J. *Organomet. Chem.* **1982**, *232*, C78. (b) Kermod, N. J.; Lappert, M. F.; Skelton, B. W.; White, A. H.; Holton, J. J. *Organomet. Chem.* **1982**, *228*, C71. (c) Stein, J.; Fackler, J. P.; Pappazios, C.; Chen, H. W. *J. Am. Chem. Soc.* **1981**, *103*, 2192.

(8) Walker, N.; Stuart, D. *Acta Crystallogr., Sect. A: Found. Crystallogr.* **1983**, *A39*, 158.

(9) Beurskens, P. T.; Bosman, W. P.; Doesburg, H. M.; Gould, R. O.; van der Hark, Th. E. M.; Prick, P. A. J.; Noordik, J. H.; Beurskens, G.; Parthasarathi, V. *Dirdif Manual*; Crystallography Laboratory: Toerenoiveld, Netherlands, 1981.

(10) Martínez-Ripoll, M.; Cano, F. H. PESOS, a Computer Program for the Automatic Treatment of Weighting Schemes; Instituto Rocasolano: Madrid, Spain, 1975.

(11) Stewart, J. M.; Kundell, F. A.; Baldwin, J. C. The X-ray System; Computer Science Center, University of Maryland: College Park, MD, 1976.

The mean Ta-Cl bond distance for Cl1, Cl2, and Cl4 is as expected 2.446 Å but a shorter bond length of 2.295 Å is observed for Ta-Cl3 which is an eclipsed conformation with one of the C-Me bonds of the ring (see Figure 2), giving bigger angles with the nearest Cl atoms.

The tantalum-C(ylide) bond length of 2.349 (29) Å corresponds to a single bond and is of the same order than that found for a  $\mu$ -CHPMe<sub>3</sub> bridging group.<sup>2b</sup> The Ta-Cl1-P angle of 125 (1)° and the four equivalent P-C bond lengths averaging 1.841 Å are in the range of those observed for other metal-neutral phosphorus ylide complexes reported.<sup>7</sup>

The reactions of these compounds with an excess of the methylenephosphoranes in order to obtain bidentate anionic ylide (CH<sub>2</sub>)<sub>2</sub>PR<sub>2</sub> or alternatively anionic ylide Ta=CHPR<sub>3</sub> complexes are being studied.

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**Registry No.** 1, 108418-44-6; 2, 108418-45-7; 3, 108418-46-8; 4, 108418-47-9; ( $\eta^5$ -C<sub>5</sub>Me<sub>6</sub>)TaCl<sub>4</sub>, 71414-47-6; CH<sub>2</sub>=PMe<sub>3</sub>, 14580-91-7; CH<sub>2</sub>=PPhMe<sub>2</sub>, 29949-96-0; CH<sub>2</sub>=PMePh<sub>2</sub>, 4554-22-7; CH<sub>2</sub>=PPh<sub>3</sub>, 3487-44-3.

**Supplementary Material Available:** ORTEP drawings and tables of crystal data, fractional coordinates and thermal parameters, least-squares planes, torsion angles, and bond lengths and angles (41 pages); a listing structure factor amplitudes (26 pages). Ordering information is given on any current masthead page.

### X-ray Crystal Structure of the Triple-Decker-Sandwich Complex Bis( $\eta^6$ -mesitylene)( $\mu$ - $\eta^6$ : $\eta^6$ -mesitylene)dichromium

William M. Lamanna\* and William B. Gleason

3M Corporate Research Laboratories  
St. Paul, Minnesota 55144

Doyle Britton

Department of Chemistry, University of Minnesota  
Minneapolis, Minnesota 55455

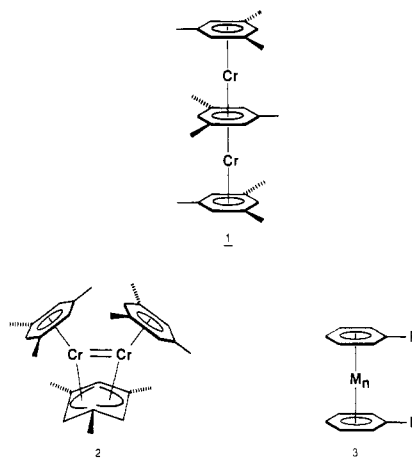
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**Summary:** The X-ray crystal structure of the previously reported tris(mesitylene)dichromium triple-decker-sandwich complex, prepared by co-condensation of Cr vapor with mesitylene at high metal loadings, is described. The material is disordered in the solid with all possible ring methyl positions being half occupied. The three aromatic rings are parallel and nearly perfectly eclipsed when viewed down the threefold Cr/Cr axis which lies normal to the ring planes.

Recently we reported that the reaction of chromium vapor with mesitylene at high metal-to-ligand ratios leads to the formation of an unusual dinuclear complex with the stoichiometry tris(mesitylene)dichromium. On the basis of evidence from variable-temperature <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy, the dichromium species was formulated as the novel triple-decker-sandwich structure 1,<sup>1</sup> the first

homoleptic triple-decker containing bridging and terminal arene ligands.

As previously noted,<sup>1</sup> the NMR data in this instance are not sufficient for a completely unambiguous structural assignment. That is, they do not strictly rule out alternative structures of equivalent stoichiometry but lower symmetry than 1, provided that these alternative structures are rapidly fluxional below -90 °C on the NMR time scale. One such hypothetical structure, for which there exists some literature precedent,<sup>2</sup> is 2. In our previous account we disfavored such structures on the basis that they do not as readily explain the matrix-isolation evidence of Lamanna<sup>1</sup> and Ozin<sup>3</sup> for the stepwise formation of higher arene-metal species with more than two metal atoms. However, it was apparent from the outset that a conclusive resolution of this issue would only be provided by an X-ray crystal structure.



Preliminary attempts to obtain X-ray quality crystals of tris(mesitylene)dichromium were hampered by its tendency to crystallize in the form of thin sheets and by its extreme oxygen sensitivity. These problems were overcome by conducting crystallizations in a N<sub>2</sub>-filled drybox at -40 °C by allowing petroleum ether vapor to diffuse into a concentrated solution of 1 in diethyl ether. The resultant deep purple crystals were loaded into glass capillaries and sealed under N<sub>2</sub>. Crystal data: C<sub>27</sub>H<sub>36</sub>Cr<sub>2</sub>, fw 464.58, rhombohedral, space group *R*3*m*, *a* = 11.063 (3) Å,  $\alpha$  = 43.97 (2)°, *Z* = 1,  $\omega$ -2 $\theta$  scans, 497 data of which 409 with *I* > 1 $\sigma$ (*I*) were used, *R* = 0.029, *R*<sub>w</sub> = 0.039. Additional details of the crystal structure determination are given in the supplementary material.

The structural features in 1 generally resemble those reported for the closely related bis( $\eta^5$ -cyclopentadienyl)( $\mu$ - $\eta^6$ : $\eta^6$ -arene)divanadium<sup>4</sup> and bis( $\eta^5$ -cyclo-

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