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# Condensation of Cyclopentadiene with Bridging Hydroxo Organopalladium and -platinum Dimers: A Novel Simple Entry to $\eta^5$-Cyclopentadienyl Complexes of Palladium and Platinum

Vladimir V. Grushin, Corinne Bensimon, and Howard Alper*

*Department of Chemistry, Ottawa-Carleton Chemistry Institute, University of Ottawa, Ottawa, Ontario K1N 6N5, Canada*

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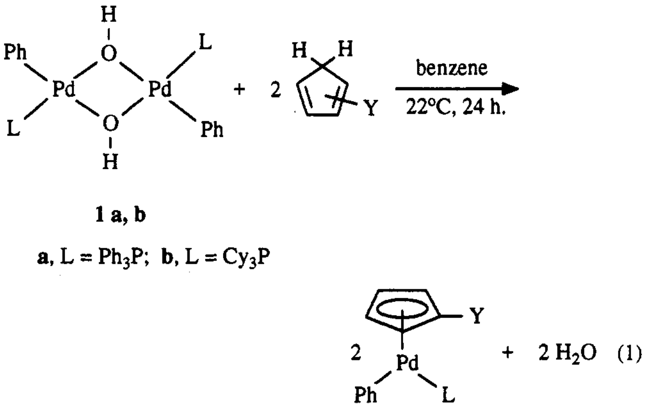
Dinuclear complexes of palladium and platinum, containing two bridging hydroxo ligands, $[L_2M_2Ph_2(\mu\text{-}OH)_2]$, where M = Pd, Pt and L = $Ph_3P$, $Cy_3P$, readily react with cyclopentadiene or methylcyclopentadiene to give the corresponding $\eta^5$-cyclopentadienyl complexes, $[(\eta^5\text{-}C_5H_4Y)M(Ph)L]$ (Y = H, Me), **2a–d** and **3**, in 65–95% yield. This novel method does not require any organometallic reagents for the Cp transfer to palladium and platinum. Complex **2a** (M = Pd, L = $PPh_3$, R = H), was structurally characterized by single crystal diffraction. Crystallographic data for **2a**: space group *C2/c*, $a = 32.934(13)$ Å, $b = 9.736(2)$ Å, $c = 18.355(8)$ Å, $\alpha = 90.00°$, $\beta = 120.16(3)°$, $\gamma = 90.00°$, $V = 5089(3)$ Å$^3$, $Z = 8$, $R = 0.056$, and $R_w = 0.067$.

Organometallic complexes of palladium[1] and platinum,[2] containing cyclopentadienyl (Cp) ligands coordinated to the metal in a $\eta^5$-fashion, are rare. Consequently, the chemistry of $\eta^5$-Cp Pd and Pt compounds is not as developed as that of earlier transition metals, one of the main obstacles being a lack of good methods to introduce the Cp ligand to platinum and palladium. The most common and effective Cp-transfer reagent in the chemistry of Pd and Pt is cyclopentadienylthallium,[1,2] a highly toxic chemical which sometimes is used in a large excess (e.g. 10-fold)[3] in order to obtain the desired compounds in reasonable yield. The development of new, efficient methods to prepare ($\eta^5$-cyclopentadienyl)palladium and -platinum complexes from readily available, relatively inexpensive, and nontoxic materials, is a desirable goal.



**1 a, b**

**a**, L = $Ph_3P$; **b**, L = $Cy_3P$

79-95%

**2 a-d**

**2 a**: L = $Ph_3P$; Y = H
**2 b**: L = $Cy_3P$; Y = H
**2 c**: L = $Ph_3P$; Y = Me
**2 d**: L = $Cy_3P$; Y = Me

We have found that the complexes [CpPd(L)R] (L = tertiary phosphine; R = $\sigma$-organic ligand, e.g. Ph) can be easily prepared in excellent yields by reacting the hydroxo-bridging organopalladium dimers, $[L_2Pd_2R_2(\mu\text{-}OH)_2]$, with cyclopentadiene (eq 1). The reaction (eq 1) occurs in benzene at room temperature, affording **2a–d** in quantitative yields, which can subsequently be isolated, with minor losses, in analytically pure form.

The starting dimers **1a,b** are easily prepared in high yield from readily available $[L_2PdCl_2]$, halobenzenes, and alkali according to eq 2.[4]

$$2[L_2PdCl_2] + 2PhI + 6KOH \xrightarrow[\text{reflux, 3-5 h}]{\text{benzene-H}_2\text{O}} \underset{\mathbf{1}}{[LPd(Ph)(\mu\text{-}OH)]_2} + 2LO + 2KI + 4KCl + 2H_2O \quad (2)$$

Both cyclopentadiene and methylcyclopentadiene exhibited sufficient reactivity toward the hydroxo dimers **1a,b**; however, pentamethylcyclopentadiene remained unreactive under the same conditions. Attempts to enforce the reaction between **1a** and $C_5Me_5H$ appeared unsuccessful. Decomposition of **1a**, accompanied by palladium metal precipitation was observed at an elevated temperature (60 °C), while no reaction took palce at ambient temperature under phase-transfer catalysis conditions (benzene–50% KOH–$Bu_4N^+HSO_4^-$).

The synthesis of neutral complexes **2a–d** requires neither CpTl nor *any other organometallics*, representing a unique example in the chemistry of cyclopentadienylpalladium compounds. The complexes [CpM(L)X] (M = Pd, Pt; L = tertiary phosphine or other $\eta^2$ ligand; R = $\eta^1$-inorganic or -organic ligand) are usually prepared by treatment of the corresponding halogeno (acetato) palladium or platinum dimers with toxic cyclopentadienyl derivatives of

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mercury,[5] thallium,[5–14] or tin.[15,16] There are few examples of the use of cyclopentadiene itself in the synthesis of $\eta^5$-Cp derivatives of Pd. Smidt and Jira[17] reported preparation of [CpPdCl] from cyclopentaidene and wet $PdCl_2$. The product was not reliably characterized but probably is a polymer of nonstoichiometric composition.[1] The cationic complexes $[CpPdL_2]^{2+}$, where L = tertiary phosphine, arsine, and stibine, were obtained by addition of CpH to the species $[L_2Pd(Me_2CO)]^{2+}$, generated from $[L_2PdCl_2]$ and $AgPF_6$ in acetone.[18] One neutral $\eta^5$-methylcyclopentadienyl cyclopalladated complex was obtained in 35% yield by the treatment of the corresponding chloro-bridging dimer with methylcyclopentadiene in the presence of NaOEt.[13]

Platinum complexes of the general formula [CpPt(L)R] are even less common than their palladium analogues.[11] Following the above described methodology, we prepared the complex $[CpPt(PPh_3)Ph]$, **3**, in 65% yield. The only literature method[11] to synthesize this compound (22% yield) requires cyclopentadienylthallium and $[(Ph_3P)_2Pt_2(Ph)_2(\mu\text{-}Cl)_2]$; we failed to obtain the latter in pure form by following the recommended[11] general procedure.[19] Unfortunately, the isostructural and isoelectronic platinum analogues of **1a** cannot be synthesized from $[L_2PtCl_2]$, PhI, and KOH by reaction 2. Nonetheless, organometallic platinum dimers with bridging hydroxo groups can be easily prepared from the corresponding chloro complexes and alkali.[20,21] Equation 3 summarizes the synthesis of **3**

$$[L_2Pt_2Cl_4] \xrightarrow{Ph_2Hg} [L_2Pt_2Ph_2(\mu\text{-}Cl)_2] \xrightarrow{OH^-}$$

$$[L_2Pt_2Ph_2(\mu\text{-}OH)_2] \xrightarrow[-2H_2O]{2CpH} \underset{3,\,65\%}{2[(\eta^5\text{-}Cp)Pt(L)Ph]} \quad (3)$$

$$L = PPh_3$$

from $[(Ph_3P)_2Pt_2Cl_4]$; the intermediate organometallic dimers were not isolated in pure form but were used successfully as crude solids. As the platinum hydroxo dimer is less reactive toward cyclopentadiene than its palladium analogues, **1a,b**, the synthesis of **3** was performed in a sealed tube at elevated (60 °C) temperature.

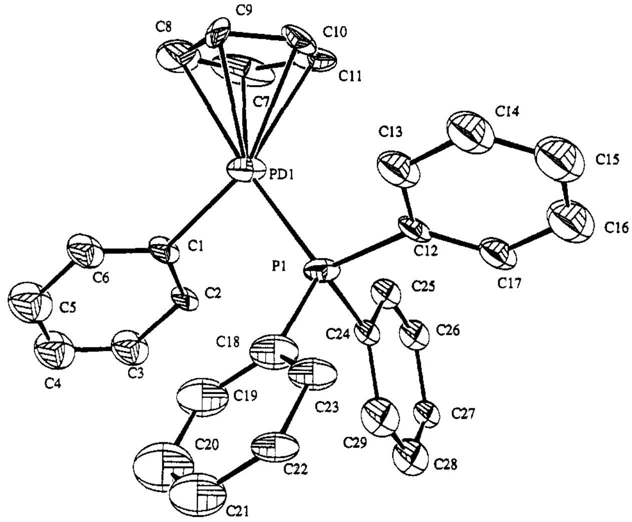

**Figure 1.** Molecular structure of **2a**. Selected bond distances (Å) and bond angles (deg) are as follows: Pd–P, 2.229(4); Pd–C7, 2.332(16); Pd–C8, 2.291(17); Pd–C9, 2.334(15); Pd–C10, 2.314(15); Pd–C11, 2.418(16); Pd–C1, 2.030(12); C7–C8, 1.49(4); C8–C9, 1.36(3); C9–C10, 1.346(25); C10–C11, 1.429(23); C11–C7, 1.37(3); P–Pd–C1, 91.6(3); P–Pd–C7, 146.8(6); P–Pd–C8, 167.3(7); P–Pd–C9, 133.2(5); P–Pd–C10, 112.3(4); P–Pd–C11, 118.1(4).

**Table I. Crystallographic Data for $[CpPd(Ph)PPh_3]\cdot{}^1/_2C_6H_6$ (2a)**

| | |
|---|---|
| formula | $C_{32}H_{28}PPd$ |
| fw | 549.95 |
| *a*, Å | 32.934(13) |
| *b*, Å | 9.736(2) |
| *c*, Å | 18.355(8) |
| α, deg | 90 |
| β, deg | 120.16(3) |
| γ, deg | 90 |
| space group | *C*2/*c* |
| *Z* | 8 |
| *V*, Å³ | 5089(3) |
| $d_{calc}$, g/cm³ | 1.436 |
| *T*, K | 153 |
| λ(Mo Kα radiation), Å | 0.709 30 |
| μ, mm⁻¹ | 0.80 |
| *R* ($R_w$), % | 5.6 (6.7) |

Complexes **2a–d** and **3** are orange or yellow crystalline materials, air-stable in the solid state, but decomposing slowly in solution. It is interesting that the platinum derivative **3** is more air-sensitive in solution than palladium complexes **2a–d**, and **3** is much more soluble in pentane than **2a**. The cyclopentadienylpalladium complexes were characterized by elemental anaysis and $^1H$ and $^{31}P$ NMR spectra,[22] while **3** was identified by comparison of its $^1H$ and $^{31}P$ NMR spectral data with those described in the literature.[11] The structure of **2a** (Figure 1, Table I) was determined by a single crystal X-ray diffraction of its 2:1 benzene solvate. The main structural parameters of **2a** lie in the range of those previously established for other ($\eta^5$-cyclopentadienyl)palladium complexes.[13,14,18,23,24] The

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(21) As anticipated, the metathetic replacement of bridging chloro ligands in $[L_2PtR_2(\mu\text{-}Cl)_2]$ for OH readily occurs in the absence of any phase-transfer catalyst (e.g. crown ethers were used by Fakley and Pidcock[20] for this purpose), as the process is biphasic in nature.[4]

(22) Complex **2a** was described in the literature;[7] however, the yield, elemental analysis, and spectral data (except the $^1H$ NMR of the Cp ligand) were not reported. The compound was originally synthesized[7] by the sequential treatment of $[(Ph_3P)_2Pd_2Cl_4]$ with CpTl and PhMgBr.

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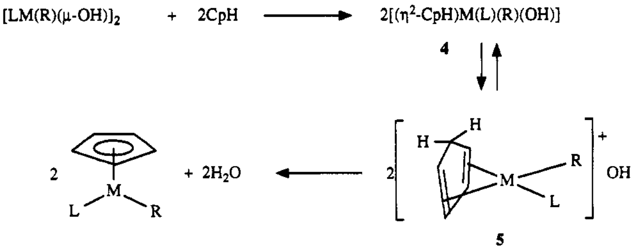
## Scheme I



Pd–C(Cp) bond distances vary from 2.291 to 2.418 Å with Pd–C8 being the shortest. A similar pattern has already been observed in a number of other structures of nickel, palladium, and platinum cyclopentadienyl complexes.[23] The M–C(CP) (M = Ni, Pd, Pt) bond length differences have been rationalized in terms of the trans effect of the $\sigma$-aryl ligand. The Cp ring in **2a** is almost planar with a slight deviation of <0.04 Å. Surprisingly, the C–C bond lengths in the Cp ring of **2a** differ from each other. Such a variation is characteristic of other cyclopentaidenyl complexes of nickel, platinum, and especially palladium.[23] In **2a**, the C–C bond distances of the Cp ring range from 1.34 to 1.49 Å. Again, this could be attributed to the different trans effects of the $\sigma$-Ph and $PPh_3$ ligands. However, some thermal motion in the Cp ring cannot be ruled out although the X-ray experiment was conducted at −120 °C.

Yoshida, Okano, and Otsuka[25] reported the condensation between *monomeric* hydroxo complexes of Pd and Pt with some CH acids, resulting in the corresponding $\sigma$-organometallics. The mechanism of this transformation remains unclear. A possible pathway for the condensation of $[L_2M_2R_2(\mu\text{-}OH)_2]$, where M = Pd or Pt, with cyclopentadiene is shown in Scheme I. First, two molecules of cyclopentadiene coordinate with the dimer in a $\eta^2$-fashion, cleaving the hydroxo bridges. The resulting mononuclear species **4** could rearrange reversibly to the cationic complex **5** that contains a $\eta^4$-coordinated cyclopentadiene ligand along with the hydroxide anion in the *outer* coordination sphere. Subsequent deprotonation of the coordinated cyclopentadiene ligand irreversibly results in the formation of a $\eta^5$-cyclopentadienyl complex. The hydroxide anion of **5** is likely to possess an enhanced basicity due to its negligible solvation by benzene. The mechanism (Scheme I) accounts for the low reactivity of pentamethylcyclopentadiene, which is probably caused by its incapability to coordinate with **1** (steric reasons), rather than by its insufficient acidity.

A number of palladium[4,26] and platinum[20] complexes of the general formula $[L_2M_2R_2(\mu\text{-}OH)_2]$ are known, and even more can be easily synthesized from numerous Pd and Pt dimers with bridging chloro ligands[19,20,27,28] by simply treating them with aqueous alkali.[4] In conclusion, the novel preparative method described above is an exceedingly simple and highly efficient approach to the synthesis of the complexes $[(\eta^5\text{-}C_5H_5)M(PR_3)R']$ from cyclopentadiene.

## Experimental Section

$^1H$ and $^{31}P$ NMR spectra were measured on a Varian XL 300 NMR spectrometer. All chemicals and solvents were purchased from Aldrich, Strem, and Organometallics chemical companies, and used without further purification. Freshly cracked cyclopentadiene and methylcyclopentadiene were immediately used in the synthesis of **2** and **3**. All reactions were run under a nitrogen atmosphere, in $N_2$-saturated solvents, whereas isolation of the complexes was conducted in air. Dimers **1a,b**[4] and $[(Ph_3P)_2Pt_2Cl_4]$[29] were prepared as described in the literature.

**$[(\eta^5\text{-}C_5H_5)Pd(PPh_3)Ph]$, 2a.** A mixture of $[(Ph_3P)_2Pd_2Ph_2(\mu\text{-}OH)_2]$, **1a** (0.11 g; 0.12 mmol), cyclopentadiene (0.08 g; 1.2 mmol), and benzene (3 mL) was stirred at 22 °C for 27 h. The clear, orange-yellow solution was concentrated to ca. 1 mL, diluted with boiling hexane (6 mL), filtered hot, and then stored at −20 °C for 1 day. Nice orange-yellow crystals were separated, washed with cold (−20 °C) pentane (2 mL), and dried under vacuum. The yield of **2a** was 0.115 g (95%). $^1H$ NMR, $\delta$; 5.7 (d, $J$(P–H) = 1.5 Hz, 5H, $\eta^5\text{-}C_5H_5$), 6.65 (m, 3H, 3,4,5-$C_6H_5Pd$), 6.95 (m, 2H, 2,6-$C_6H_5$), 7.1–7.4 (m, 15H, $Ph_3P$). $^{31}P$ NMR, $\delta$: 40.4 (s). Anal. Calcd for $C_{29}H_{25}PPd$: C, 68.18; H, 4.93. Found: C, 68.15; H, 5.22.

**$[(\eta^5\text{-}C_5H_5)Pd(PCy_3)Ph]$, 2b.** A mixture of $[(Cy_3P)_2Pd_2Ph_2(\mu\text{-}OH)_2]$, **1b** (0.12 g, 0.13 mmol), cyclopentadiene (0.10 g; 1.5 mmol), and benzene (5 mL) was stirred at 22 °C for 24 h. After evaporation of benzene, the residue was dissolved in hot hexane, and the solution was filtered and concentrated to ca. 3 mL. Cooling the solution to −78 °C led to precipitation of **2b** that was filtered out cold, washed with cold (−78 °C) pentane (3 mL), and dried under vacuum. The yield was 0.11 g (84%). $^1H$ NMR, $\delta$: 1.0–1.9 (m, 33H, $Cy_3P$), 5.7 (d, $J$(P–H) = 1.3 Hz, 5H, $\eta^5\text{-}C_5H_5$), 6.85 (m, 3H, 3,4,5-$C_6H_5Pd$), 7.35 (m, 2 H, 2,6-$C_6H_5Pd$). $^{31}P$ NMR, $\delta$: 49.7 (s). Anal. Calcd for $C_{29}H_{43}PPd$: C, 65.84; H, 8.19. Found: C, 66.00; H, 8.24.

**$[(\eta^5\text{-}C_5H_4CH_3)Pd(PPh_3)Ph]$, 2c.** A mixture of $[(Ph_3P)_2Pd_2Ph_2(\mu\text{-}OH)_2]$, **1a** (0.12 g; 0.13 mmol), methylcyclopentadiene (0.09 g; 1.4 mmol), and benzene (4 mL) was stirred at 22 °C for 24 h. Benzene was removed under vacuum, and the residue was recrystallized from hexane (+60 to −78 °C). The orange crystals were collected cold, washed with cold (−78 °C) pentane (2 mL), and dired under vacuum. The yield of **2c** was 0.115 g (85%). $^1H$ NMR, $\delta$: 1.7 (d, $J$(P–H) = 3.0 Hz, 3H, $CH_3$), 5.5 (m, 2H, 2,5-$\eta^5\text{-}C_5H_4Me$), 5.65 (m, 2H, 3,4-$\eta^5C_5H_4Me$), 6.65 (m, 3H, 3,4,5-$C_6H_5Pd$), 6.95 (m, 2H, 2,6-$C_6H_5Pd$), 7.15–7.4 (m, 15H, $Ph_3P$). $^{31}P$, $\delta$: 40.3 (s). Anal. Calcd for $C_{30}H_{27}PPd$: C, 68.64; H, 5.19. Found: C, 68.47; H, 5.43.

**$[(\eta^5\text{-}C_5H_4CH_3)Pd(PCy_3)Ph]$, 2d.** A mixutre of $[(Cy_3P)_2Pd_2Ph_2(\mu\text{-}OH)_2]$, **1b** (0.10 g; 0.10 mmol), methylcyclopentadiene (0.08 g, 1.2 mmol), and benzene (4 mL) was stirred at 22 °C for 25 h. Benzene was removed under vacuum, the residue was dissolved in pentane, and the solution was filtered and evaporated. The remaining crystals of **2d** were washed with cold (−78 °C) pentane (2 mL) and dried under vacuum. The yield was 0.089 g (79%). $^1H$ NMR, $\delta$: 1.0–1.8 (m, 33H, $Cy_3P$), 1.85 (d, $J$(P–H) = 2.7 Hz, 3H, $CH_3$), 5.5 (m, 4H, $\eta^5\text{-}C_5H_4Me$), 6.8 (m, 3H, 3,4,5-$C_6H_5Pd$), 7.3 (m, 2H, 2,6-$C_6H_5Pd$). $^{31}P$, $\delta$: 49.8 (s). Anal. Calcd for $C_{30}H_{45}PPd$: C, 66.36; H, 8.35. Found: C, 66.16; H, 8.05.

**$[(\eta^5\text{-}C_5H_5)Pt(PPh_3)Ph]$, 3.** A mixture of $[(Ph_3P)_2Pt_2Cl_4]$ (0.15 g; 0.14 mmol), diphenylmercury (0.115 g; 0.32 mmol), and anhydrous dichloromethane (5 mL) was stirred at 22 °C for 16 h. The solvent was evaporated, and organomercurials were removed from the remaining solid by sublimation (100 °C, 0.2 mmHg, 5 h). The dark residue (crude $[(Ph_3P)_2Pt_2Ph_2(\mu\text{-}Cl)_2]$) was stirred at 22 °C with benzene (5 mL) and 40% KOH (4 mL) for 5 h. The organic layer was filtered, and the clear, yellowish filtrate was evaporated to leave crystalline $[(Ph_3P)_2Pt_2Ph_2(\mu\text{-}OH)_2]$ (0.144 g; 0.13 mmol). A mixture of the crude hydroxo complex (0.13 g; 0.12 mmol) was heated (60 °C, oil bath) with cyclopentadiene (0.33 g; 5 mmol) and benzene (1.5 mL) in a sealed

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5-mL glass tube for 20 h. Benzene was removed under vacuum, the residue was extracted with degassed pentane, filtered, and evaporated. The residual oil was thoroughly dried under vacuum (0.1 mmHg), to give 0.092 g (0.15 mmol; 65%) of spectrally pure[11] **3** as nice yellow crystals.

**X-ray Diffraction.** A crystal of **2a** having approximate dimensions of 0.2 × 0.2 × 0.4 mm was mounted on a glass capillary. All the measurements were made on a Rigaku diffractometer with Mo Kα radiation. Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range $40 \leq 2\theta < 47$, and corresponded to a monoclinic cell with dimensions $a$ = 32.934(13), $b$ = 9.736(2) Å, and $c$ = 18.355(8) Å. For $Z$ = 8 and fw = 549.95, the calculated density is 1.436 g/cm$^3$. On the basis of the systematic absences, the space group was determined to be *C2/c* or *Cc*. There was an inversion center missing in the packing diagram in *Cc*, therefore indicating that the space group is *C2/c*. The MISSYM program confirms the presence of an inversion center at 0.322, 0.000, 0.207. The data were collected at 22 °C using the $\omega$–$2\theta$ scan technique to a maximum $2\theta$ value of 47.

A total of 2507 reflections was collected. The unique set contains only 2123 reflections. The standards were measured after every 150 reflections. No crystal decay was noticed. The data were corrected for Lorentz and polarization effects.[30] No correction was made for absorption. The structure was solved by direct methods. All the atoms were refined anisotropically except the hydrogens. The hydrogen atoms were calculated, with a bonding distance of 1.07 Å. All the phenyl groups were refined as rigid groups to increase the reflections/parameters ratio. The final cycle of full-matrix least-squares refinement was based on 1623 observed reflections ($I > 2.5\sigma(I)$) and 125 variable parameters. Weights based on counting statistics were used. The maximum and minimum peaks on the final difference Fourier map corresponded to +0.700 and −0.790 e/Å$^3$, respectively. All the calculations were performed using the NRCVAX crystallographic software package.[31]

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**Supplementary Material Available:** Textual presentation of the full details of the structure solution, tables of experimental data, atomic coordinates, bond lengths and angles, and thermal parameters, and figures for complex **2a** (13 pages). Ordering information is given on any current masthead page.

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