Synthesis of tetrahydrofurfurylcyclopentadiene and its thallium, manganese, and rhodium complexes and the low temperature matrix photolysis of the manganese complex

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Abstract

Reaction of tetrahydrofurfuryl chloride and sodium cyclopentadienide has been found to yield tetrahydrofurfurylcyclopentadiene, I, which is converted to its thallium salt, II, by thallium ethoxide. II reacts with $BrMn(CO)_5$ and $[ClRh(CO)_2]_2$ to give the corresponding tetrahydrofurfurylcymantrene, III, and both tetrahydrofurfurylcyclopentadienyl rhodium dichloride, IV, and its dinuclear species, V. 2-D COSY analysis of the ^1H NMR spectrum of V has permitted assignment of all resonances. Photolysis of the manganese complex as a Nujol solution at 77 K afforded CO ejection to give both tetrahydrofurfurylcyclopentadienylmanganesedicarbonyl, and a second isomer of this dicarbonyl species in which the pendant tetrahydrofurfuryl group was bound to the metal. Photochemical and thermal reversal of the former process gave III and more of the tetrahydrofurfuryl bound complex, while reversal of the latter process was only observed upon warming to higher temperatures. This is the first time that such an intramolecular reaction with such a remote ligand has been observed in low-temperature matrix experiments.

Introduction

Recent work in our laboratory and others has greatly expanded the number of ring-substituted cyclopentadienyl reagents which are available for synthesis of cyclopentadienyl metal complexes. For example, we have recently reported improved routes to RCpTl compounds, where R = CHO, $COCH_3$, CO_2CH_3 [1], and 1,4-bis(cyclopentadienylthallium)-1,4-butadione [2], and the use of these materials in the synthesis of a range of metal complexes. Several papers have appeared describing the synthesis of phosphine substituted cyclopentadienyl compounds in which the phosphine can serve as a ligand to bind a second metal center [3]. Rauchfuss et al. described the preparation of thienylmethylcyclopentadiene which is quite similar to the compounds described herein, and its conversion to a ruthenium complex [4].

In this paper we report the preparation of tetrahydrofurfurylcyclopentadiene, its subsequent conversion to tetrahydrofurfurylcyclopentadienyl metal complexes, and

the low temperature matrix photochemistry [5] of the manganese complex in order to investigate the ability of the complex to eject a CO ligand and to bind to the pendant tetrahydrofurfuryl group. The closely related (2-furyl)methylcyclopentadiene has been reported, but its application to organometallic chemistry remains unexplored [6].

Results

(a) Preparation and physical properties of the compounds

Reaction of tetrahydrofurfuryl chloride with sodium cyclopentadienide in THF yielded, after workup and distillation, tetrahydrofurfuryl cyclopentadiene, I, as a water-white oil which was shown by NMR to be a nearly 1:1 mixture of the two isomers, IA and IB, with a small ether or hydrocarbon contamination. Reaction of
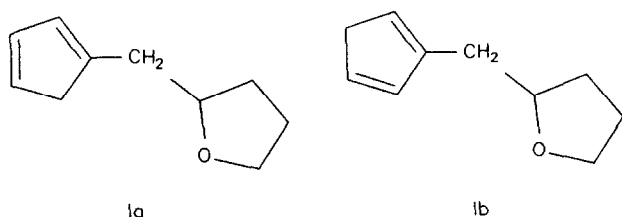
this mixture with thallium ethoxide in ethanol gave tetrahydrofurfurylcyclopentadienyl thallium, II, as a white, air-stable powder. Subsequent reactions of II with BrMn(CO)$_5$ and [ClRh(CO)$_2$]$_2$ in refluxing benzene yielded compounds III–V. All compounds were fully characterized by IR, ^{1}H and ^{13}C NMR spectroscopy and mass spectroscopy. The tar-like consistency of the rhodium compounds made them difficult to manipulate for elemental analyses. However, a high resolution mass measurement of IV which was consistent with the calculated mass was successfully obtained. The small parent ion peak of V even under chemical ionization conditions precluded exact mass measurements on that compound.

The proton NMR spectra of III was characteristically broadened by coupling with ^{55}Mn. The tetrahydrofurfuryl regions of compounds I–V were found to be very complex so that only a limited analysis could be made. A 2-D COSY spectrum of V, which had the best resolved spectrum in this region, was performed making it
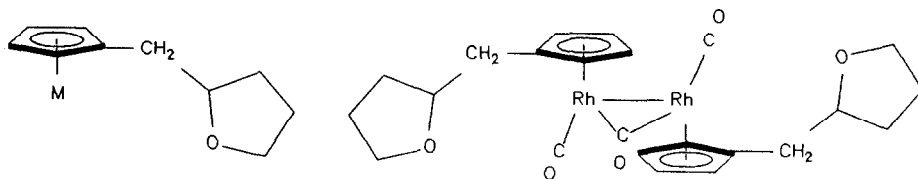
II: M = Tl

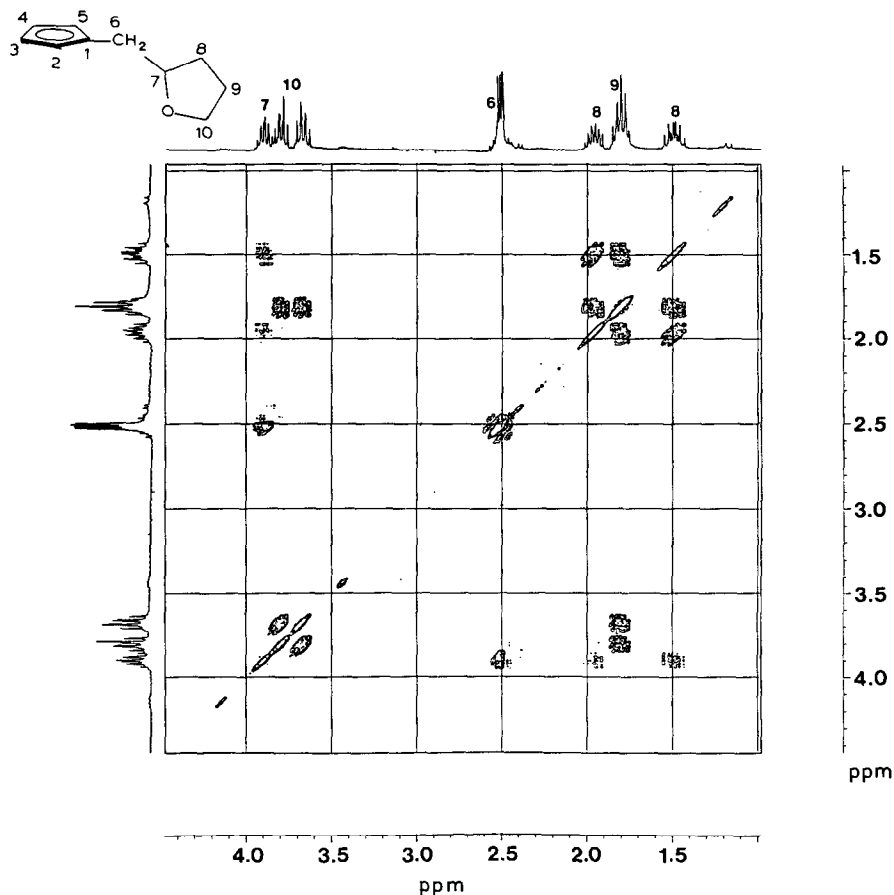III: M = Mn(CO)$_3$

IV: M = Rh(CO)$_2$

V

Fig. 1. Tetrahydrofurfuryl region of 2-D COSY ^{1}H NMR spectrum of V in CDCl$_3$.

possible to assign the proton resonances for this compound. The COSY spectrum of the tetrahydrofurfuryl portion of this compound is presented in Fig. 1. Spectral assignments were made for other compounds by analogy.

(b) Photolysis of III in Nujol solutions at 77–298 K

The infrared spectrum of III as a Nujol solution at 77 K showed two carbonyl bands, an "A_1" band * at 2021 cm^{-1} and a broader "E" band * at 1939 cm^{-1} (Fig. 2a). The compound was found to have an electronic transition at 315 nm at 77 K. Photolysis of the matrix (filter: 250 nm < λ < 350 nm) produced four apparent new peaks in the carbonyl region at 2132, 1951, 1882, and 1866 cm^{-1} (Fig. 2b). The peak at 2132 cm^{-1} is known to be characteristic of free CO in the matrix [5]. By using spectral subtraction to ratio out the peaks of III, a fourth peak was found at 1937 cm^{-1} (Fig. 2c). Warming of the solution caused the peaks at 1951 and 1882

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* The designations "A_1" and "E" refer to the local C_{3v} symmetry of the Mn(CO)$_3$ fragment. The broadness of the "E" band indicates symmetry lowering by the asymmetric cyclopentadienyl ligand corresponding to "E" band splitting of analogous complexes in gas matrices [7].
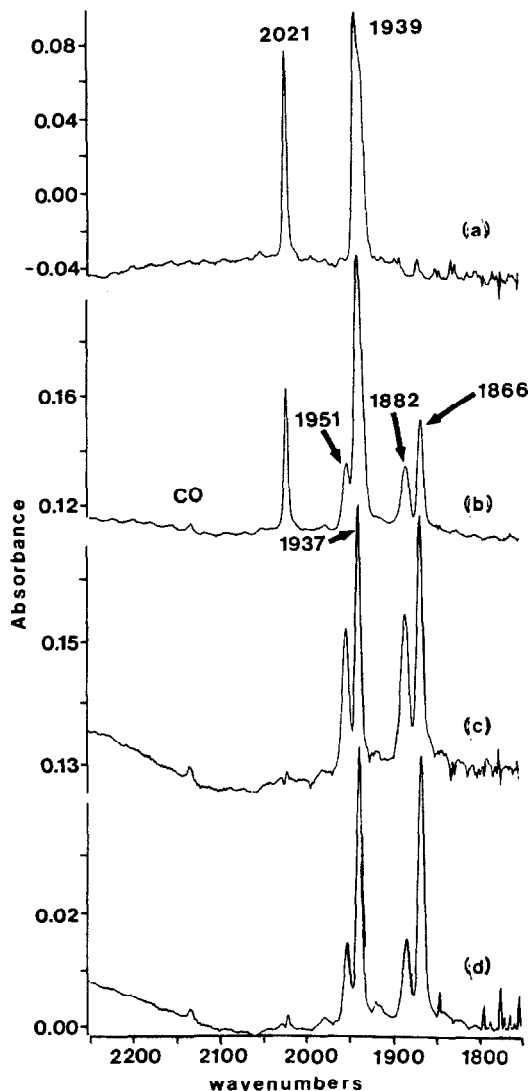
Fig. 2. Infrared spectra from an experiment with III in a Nujol matrix: (a) starting spectrum at 77 K, (b) after 15 min photolysis (filter 250 nm $< \lambda < _3 50$ nm), (c) subtraction spectrum [(b)−(a)] to remove parent peaks only, (d) after 10 min of warming the mull then refreezing to 77 K with parent peaks (a) removed by subtraction.

cm^{-1} to decrease in intensity first while the other two photoproduct peaks did not disappear until the solution had reached room temperature (Fig. 2d). IR spectral data and assignments are summarized in Table 1.

Photolysis of III as before (Fig. 3a–3c) followed by longer wavelength photolysis (filter $\lambda > 350$ nm) resulted in the growth in carbonyl peaks of III as well as the peaks at 1937 and 1866 cm^{-1} accompanied by the decrease in intensity of the peaks at 1951 and 1882 cm^{-1} (Fig. 3d).

Comparison of the band positions for the new peaks in Nujol with those for $(\eta^5-C_5H_5)Mn(CO)_2$ in CH_4 gas matrices at 12 K [7], and for $(\eta^5-C_5H_5)Mn(CO)_2$-

Table 1

Infrared band positions (cm^{-1}) observed in the carbonyl stretching region for tetrahydrofurfurylcyclopentadienyl manganese tricarbonyl, III, its photoproducts and related species in various media at ca. 12–298 K

Complex	Nujol (77 K)	CH$_4$ (12 K)	Glass (77 K) [a]	Solution
III	2021	–	–	2019 [b]
	1939	–	–	1930
VI	1951	–	–	–
	1882	–	–	–
VII	1937	–	–	–
	1866	–	–	–
CpMn(CO)$_3$	2026 [c]	2028 [d]	2026 [e]	2032 [f]
	1943, 1928	1943	1938	1946
CpMn(CO)$_2$	1957 [c], 1951 [g]	1961 [c]	1955 [e]	–
	1888, 1880	1893	1886	–
CpMn(CO)$_2$(THF)	1930 [c]	–	1916, 1942 [g]	1925 [h]
	1852	–	1834, 1867	1850
VIII	–	2024 [i]	–	2025 [j]
	–	1947	–	1956
IX	–	2025 [i]	–	2022 [k]
	–	1946	–	1941
X	–	1964 [i]	–	–
	–	1896	–	–
XI	–	1962 [i]	–	–
	–	1894	–	–

[a] Methylcyclohexane–isopentane glass (4 : 1) at 77 K; data from Ref. 8. [b] CH$_2$Cl$_2$ solution at 298 K. [c] T.E. Bitterwolf, A.J. Rest and K.A. Lott, J. Organomet. Chem., submitted. [d] Data from Ref. 7. [e] Data from Ref. 8. [f] Data from Ref. 8; methylcyclohexane–isopentane solution at 293 K. [g] Bands assigned as major and minor rotamers; data from Ref. 8. [h] THF solution. I.S. Butler, N.J. Coville and D. Cozak, J. Organomet. Chem., 133 (1977) 59. [i] Data from Ref. 11. [j] n-Hexane solution; data from Ref. 11. [k] CH$_2$Cl$_2$ solution; data from Ref. 11.

(THF) in 10% THF in hexane and 5% THF in methylcyclohexane–isopentane (4 : 1) [8] (see Table 1 for band positions in several media) together with the CO reversal and uptake behavior of reactive species in THF-doped poly(vinylchloride) films at 12–298 K [9] enable the bands at 1951 and 1882 cm^{-1} to be assigned to the single CO-loss product, VI, and the bands at 1937 and 1866 cm^{-1} may be assigned to the
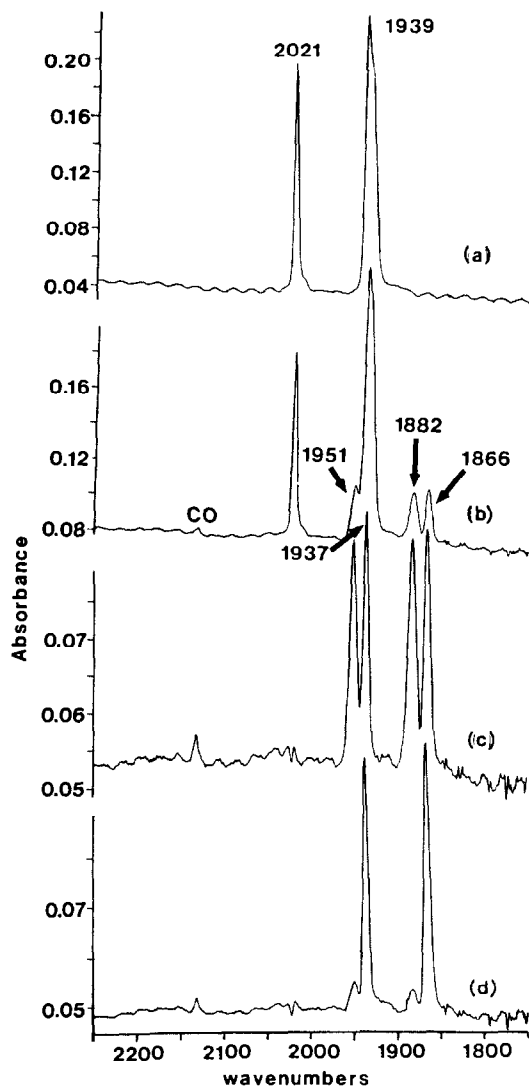

VI   VII
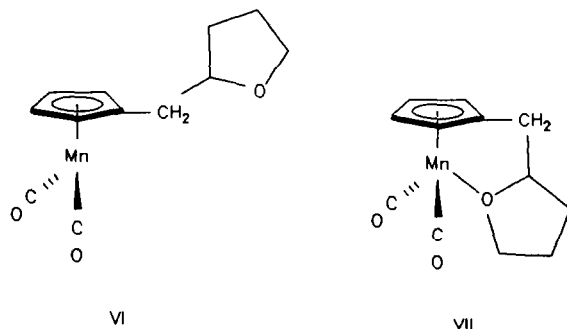
Fig. 3. Infrared spectra from an experiment with III in a Nujol matrix: (a) starting spectrum at 77 K, (b) after 15 min photolysis (filter 250 nm $< \lambda <$ 350 nm), (c) subtraction spectrum [(b)−(a)] (to remove parent peaks only), (d) after 15 min photolysis (filter $\lambda > 350$ nm) with parent peaks (a) removed by subtraction.

species, VII, stabilized by interaction with the pendant tetrahydrofurfuryl group (Table 1).

Discussion

Tetrahydrofurfurylcyclopentadiene, I, was prepared by reaction of sodium cyclopentadienide with tetrahydrofurfuryl chloride using a procedure analogous to that previously reported by us for the synthesis of benzylcyclopentadiene [10]. I was

converted into the versatile thallium synthon, II, by reaction with thallium ethoxide. Subsequent reaction of II with $BrMn(CO)_5$ yielded III, while reaction with $[ClRh(CO)_2]_2$ gave both IV and V. The spectral properties of these materials were unexceptional.

Previous matrix photochemical studies of organometallic species [5] have indicated that while unstable species can easily capture small ligands in the gas matrix cage, e.g. ^{13}CO, N_2, C_2H_4, the ability of molecules to rearrange so as to capture a remote center is very limited. For example, it was hoped that the bimetallic complexes VIII and IX, which afford the CO loss products X and XI, might rearrange in gas matrices at ca. 12 K to yield the corresponding Mn–Mn bonded dimers [11]. In fact, no such dimerization occurred and this was attributed to the likelihood that the dinuclear species adopted rigidly *anti* conformations as illustrated, setting the manganese carbonyl fragments as far away from one another as possible so that there was little possibility of reaction between the Mn atoms. It seemed, therefore, that gas matrices afford a "tight" matrix cage which restricts rotation, especially of larger species. On the basis that other media, e.g. poly(vinyl chloride) films and Nujol, have looser cages [12], and the known ability of THF to give metal complexes, it seemed possible that the photoproduct of III might undergo sufficient conformational change to bind the furfuryl unit to an electronically unsaturated manganese.

X

XI

VIII

IX
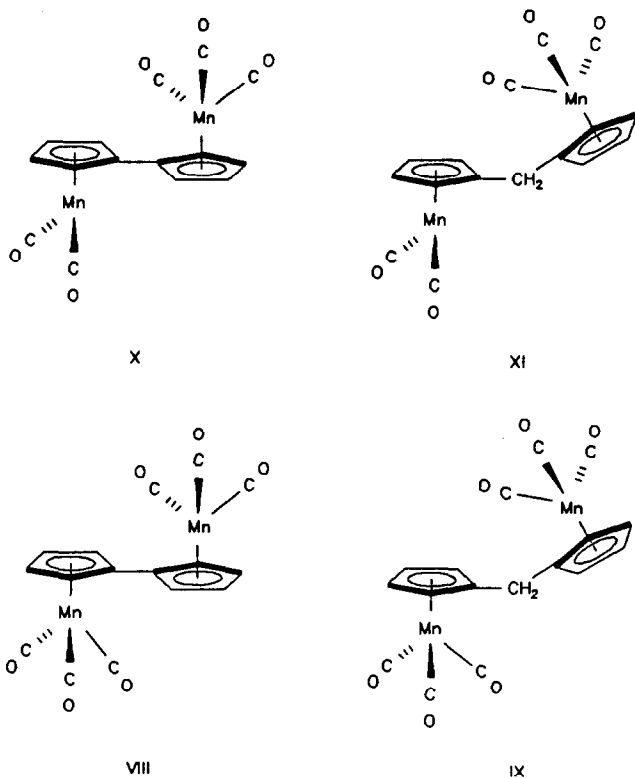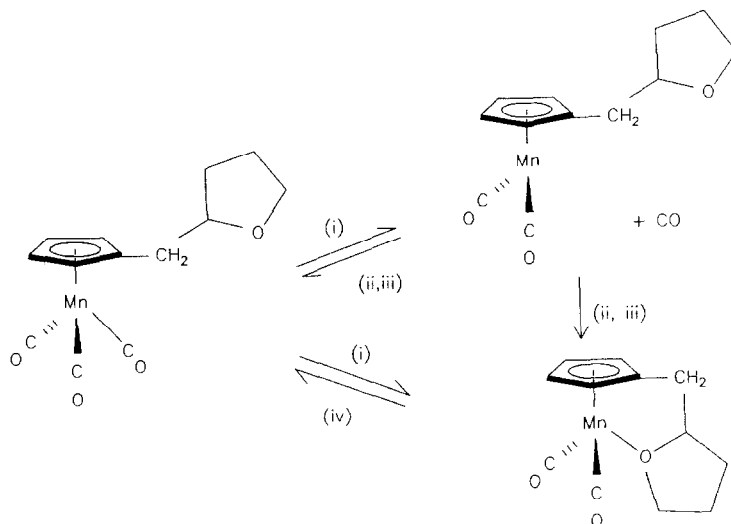
Scheme 1. (i) Photolysis with filter 250 < λ < 350 nm, (ii) photolysis with filter λ > 350 nm, (iii) initial annealing, (iv) annealing to 298 K.

Photolysis of III has been found to yield two species whose IR spectra are consistent with their assignment as a manganese dicarbonyl and a manganese dicarbonyl furfuryl, respectively. The CO reversal and the THF uptake behavior of the $Mn(CO)_2$ fragment (Scheme 1) are typical of species in THF doped glasses [8] and poly(vinyl-chloride) films [9,12], i.e. the $[M](CO)_n$ fragments ([M] indicates a metal and its other ligands) formed initially take up THF on modest warming 77 K to 100 K. The complete reversal to III on warming to higher temperatures arises because the CO which is trapped in the cage is a better ligand than the tetrahydrofurfuryl oxygen. The tetrahydrofurfuryl group in III is behaving much like THF in doped glasses in that it can bind to an electrophilic Mn group, and be ejected by CO on warm-up. It would appear that the tetrahydrofurfuryl which is initially bound on photolysis represents a fraction of molecules in which the tetrahydrofurfuryl group is fortuitously adjacent to the manganese when photolysis occurs. Those tetrahydrofurfuryl groups which become bound to the metal on warm-up most likely begin in a conformation distant from the metal, then rotate toward the metal as the matrix softens. The entire reaction sequence for the various species is illustrated in Scheme 1.

We intend to exploit compounds of this class to form heterobimetallic complexes in which the second metal atom is held by the pendant tetrahydrofurfuryl. We are presently examining the photolysis of these and other analogous compounds in solution in an effort to determine whether stable pendant tetrahydrofurfuryl complexes can be isolated. In addition, low temperature matrix studies employing a variety of media [13] for other compounds in this series are underway.

Experimental

Tetrahydrofurfuryl chloride was purchased from Aldrich and pentacarbonyl-manganesebromide was purchased from Strem. These materials were used as re-

ceived. Thallium ethoxide [14] and [ClRh(CO)$_2$]$_2$ [15] were prepared by standard literature routes. *Note*: **Thallium compounds are highly toxic and must be handled with rigorous safety precautions**. All solvents were dried and distilled under nitrogen. Preparative chromatography was conducted using nitrogen-flushed solvents and neutral (CAMAG) alumina.

Infrared spectra were recorded on a Bio-Rad Qualimatic FTIR Spectrometer, and a Bio-Rad FTS-15/80 operating at 2 cm^{-1} resolution. ^{1}H and ^{13}C NMR spectra were recorded on an IBM NR-300 MHz NMR Spectrometer and were referenced to appropriate solvent resonances. 2-D COSY NMR spectra and mass spectroscopy were carried out by Dr. Gary Knerr. Both electron impact and chemical ionization mass spectroscopies were recorded on a VG 7070-HS GC/MS using direct insertion. Elemental analyses were conducted by Desert Analytics of Tucson, AZ.

Low-temperature experiments were carried out using a simple glass cryostat, modified after the design of Hooker and Rest [16] and equipped with CaF$_2$ inner and outer windows. The cryostat was evacuated to a vacuum of ca. 10^{-4} Torr, and cooled with liquid N$_2$ to ca. 77 K.

The photolysis lamp used was a UVP 350 watt high pressure mercury arc. Irradiation was performed with the source 28 cm away from the matrix. A quartz cell (pathlength 2 cm) filled with water was placed in the path of the beam to absorb infrared radiation and prevent excess heating of the mulls. Photolysis into the desired wavelength bands was controlled with the following filters: (250 nm < λ < 390 nm) Hoya filter U330; (λ > 350 nm) Corion filter LG–350.

Nujol solutions of III were prepared by adding approximately 1 mg of III to 5 drops of Nujol (ca. 65 mg) and grinding with a mortar and pestle. Homogeneity of the solution was ensured by grinding the mixture for several minutes. In this type of experiment it has been observed that similar photochemistry is observed whether the sample is dispersed in the Nujol as a microcrystalline solid, e.g. W(CO)$_6$ [17], or seems to go into solution as is found in the present case. Nujol solutions were sealed between two CaF$_2$ plates which were mounted in a copper holder. Preliminary IR spectra were recorded using the Qualimatic FTIR on these samples to determine solution quality and concentration. The copper holder assembly was screwed into the "cryotip" of the cryostat. Good thermal conductivity in the window holder assembly and at the tip was ensured by using indium gaskets. Once the sample holder assembly was mounted, the apparatus was evacuated and the mull frozen with liquid nitrogen. When Teflon spacers were used between the CaF$_2$ plates in order to increase the pathlength, it became necessary to first flush the apparatus with dry nitrogen for 10–15 min, freeze the solution with liquid nitrogen (25 min) and then evacuate to avoid loss of the sample from the cell. The cells were warmed by removing the liquid nitrogen from the reservoir for measured periods of time, then refrozen prior to spectra being recorded.

Forward and reverse reactions were monitored by infrared spectroscopy taking care to record all the spectra at 77 K so as to enable meaningful subtraction spectra (absorbance mode) to be obtained.

Synthesis of tetrahydrofurfurylcyclopentadiene (I)

Sodium cyclopentadienide, 0.170 mol, was prepared from sodium dispersion and cyclopentadiene in THF as previously described [10]. Tetrahydrofurfuryl chloride,

25.0 g (0.207 mol), was added in one portion and the solution was maintained at 60°C with stirring for 1 day. During this time a white precipitate of NaCl formed. After cooling to room temperature, 3 mL of water was added to coagulate the fine salt precipitate. The reaction mixture was filtered through Celite, dried with MgSO$_4$, and stripped of solvent without heating. The resulting yellow oil was distilled under dynamic vacuum. The product was collected over the range 48–56°C (0.1 mmHg). Recovered product (11.36 g) was shown by NMR to be slightly contaminated. This material was used in subsequent syntheses without additional purification. ^{1}H NMR: (ppm, CDCl$_3$) 6.45 (d, 1H, olefinic Cp), 6.39 (s, 2H, olefinic Cp), 6.22 (d, 1H, olefinic Cp), 6.21 (s, 1H, olefinic Cp), 6.07 (s, 1H, olefinic Cp), 4.01 (septet, 1H, H-7), 3.87 (quartet, 1H, H-10), 3.70 (quartet, 1H, H-10), 2.93 (m, 2H, Cp CH$_2$), 2.65 (sextet, 1H, H-6), 2.51 (sextet, 1H, H-6), 1.90 (m, 1H, H-9), 1.86 (m, 2H, H-9), 1.50 ppm (m, 1H, H-8). ^{13}C NMR (ppm, CDCl$_3$): 145.6 and 143.4 (*ipso* Cp), 137.8, 134.4, 134.0, 133.2, 130.7, 127.7, 127.4 (olefinic Cp), 78.6 and 78.1 (C-7), 67.4 (C-10), 43.5 and 40.9 (Cp CH$_2$), 36.4 and 35.7 (C-6), 29.6 and 25.2 (C-8 and C-9).

Synthesis of tetrahydrofurfurylcyclopentadienyl thallium (II)

I, 4.07 g, was taken up in 50 mL of absolute ethanol under nitrogen, and thallium ethoxide, 6.8 g (27.3 mmol), was added dropwise with stirring. A white precipitate formed immediately. After addition was complete, the mixture was stirred for one hour. The solid was filtered and washed with ethanol, ethyl ether and pentane to give a white to off-white powder which was stored under nitrogen. Yield: 4.7 g (49%). ^{1}H NMR (ppm, DMSO-d_6): 5.77 (t, 2H, Cp), 5.64 (t, 2H, Cp), 3.80 (d of t, 1H, H-7), 3.71 and 3.53 (d of quartets, 2H, H-10), 1.92 (m, 1H, H-8), 1.80 (m, 2H, H-9), 1.47 (m, 1H, H-8). ^{13}C NMR (ppm, CDCl$_3$): 122.1 (*ipso* Cp), 107.1 and 105.4 (Cp), 80.8 (C-7), 66.4 (C-10), 35.5 (C-6), 31.0 and 25.1 (C-8 and C-9). Analysis: Found: C, 33.78; H, 3.75. C$_{10}$H$_{13}$OTl calcd.: 33.96; H, 3.68%.

Synthesis of tetrahydrofurfurylcymantrene (III)

II, 1.0 g (2.88 mmol) and BrMn(CO)$_5$, 0.90 g (3.28 mmol) were taken up in 50 mL of benzene and refluxed under nitrogen overnight. The resulting red suspension was filtered through Celite, stripped of solvent and chromatographed on a CYMAG alumina column with 10% dichloromethane in petroleum ether. A pale yellow band eluted with the solvent front and was shown to be Mn$_2$(CO)$_{10}$ by IR. A second yellow band was eluted which gave a yellow oil after removal of solvent. Yield: 0.49 g (59%). IR: (CH$_2$Cl$_2$) 2019, 1930 cm^{-1}. ^{1}H NMR (ppm, CDCl$_3$): 4.64 (4H), 3.84 (3H), 2.40 (2H), 1.86 (3H), 1.60 (1H). ^{13}C NMR: (CDCl$_3$) 225.3 (Mn–CO), 103.2 (*ipso* Cp), 83.4, 83.7, 82.2, and 81.6 (Cp), 79.4 (C-7), 68.0 (C-10), 34.5 (C-6), 31.3 and 25.7 ppm (C-8 and C-9). Mass Spectroscopy: (CI) 492 (2) [RCp$_2$Mn$_2$(CO)$_3$], 228 (3) M^+, 271 (15) M^+ – OH, 260 (30) M^+ – CO, 232 (45) M^+ – 2CO, 217 (24) M^+ – C$_4$H$_7$O, 214 (74), 205 (100) M^+ – 3CO. Analysis: Found: C, 54.53; H, 4.54. C$_{13}$H$_{13}$MnO$_4$ calcd.: C, 54.18; H, 4.51%.

Synthesis of tetrahydrofurfurylcyclopentadienyl rhodium dicarbonyl (IV) and its dinuclear species (V)

II, 0.5 g (1.44 mmol), and [ClRh(CO)$_2$]$_2$, 0.28 g (0.72 mmol), were taken up in 50 mL of benzene and refluxed under nitrogen overnight. The red oil which remained after filtration of the reaction mixture and solvent removal was columned on a

CYMAG alumina column. Upon elution with petroleum ether a yellow band was removed from the column which yielded a yellow-orange oil, IV. IR: (CH_2Cl_2) 2039, 1971 cm^{-1}. ^{1}H NMR (ppm, CDCl$_3$): 5.49 (d of d, 1H, Cp), 5.42 (d of d, 1H, Cp), 5.28 (m, 2H, Cp), 3.82 (m, 2H, H-7 multiplet buried within one of the H-10 multiplets), 3.69 (m, 1H, H-10), 2.48 (m, 2H, H-6), 1.98 (m, 1H, H-8), 1.83 (m, 2H, H-9), 1.50 (m, 1H, H-8). ^{13}C NMR (ppm, CDCl$_3$): 191.4 (d, J(C–Rh) = 66.5 Hz, Rh–CO), 108.5 (d, J = 2.6 Hz, $ipso$ Cp), 88.8 (d, J = 2.6 Hz, Cp), 88.7 (d, J = 2.5 Hz), 85.8 (d, J = 2.7 Hs), 85.3 (d, J = 2.8 Hz), 80.2 (C-7), 67.4 (C-10), 34.0 (C-6), 30.9 and 25.2 (C-8 and C-9).

Continued elution of the alumina column with 20% dichloromethane in petroleum ether yielded a red oil, V. IR (cm^{-1}): (CH_2Cl_2) 2012 (w), 1975 (s), 1807 (m). ^{1}H NMR (ppm, CDCl$_3$): 5.48 (m, 1H, Cp), 5.40 (m, 1H, Cp), 5.30 (m, 2H, Cp), 3.94 (pentet, 1H, H-7), 3.83 and 3.72 (complex ABXY, 2H, H-10), 2.54 (d of d, 2H, H-6), 1.99 (m, 1H, H-8), 1.84 (m, 2H, H-9), 1.53 (m, 1H, H-8) ^{13}C NMR (ppm, CDCl$_3$): 204.8 (t, J(C–Rh) = 33 Hz), 109.3 ($ipso$ Cp), 90.1 (1C), 89.7 (1C), and 88.6 (2C) (Cp), 79.5 (C-7), 67.5 (C-10), 33.9 (C-6), 30.9 and 25.2 (C-8 and C-9).

Acknowledgements

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