

Preparation of Bisdiazoalkane and Related Complexes from the Reactions of Diazo Compounds with the Dinitrogen Complexes *trans*-[$M(N_2)_2(Ph_2PCH_2CH_2PPh_2)_2$] ($M = Mo$ or W)

Armando J. L. Pombeiro^{1,*} and Raymond L. Richards²

¹ Centro de Química Estrutural, Complexo I, Instituto Superior Técnico, Av. Rovisco Pais, 1096 Lisbon Codex, Portugal

² AFRC-IPSR Nitrogen Fixation Laboratory, The University of Sussex, Brighton, BN1 9RQ, U.K.

Summary. Reactions of *trans*-[$M(N_2)_2(dppe)_2$] (A ; $M = Mo, W$; $dppe = Ph_2PCH_2CH_2PPh_2$) with ethyldiazoacetate, $N_2CHCOOEt$, yield the bisdiazoalkane species *trans*-[$M(N_2CHCOOEt)_2(dppe)_2$], upon simple replacement of the dinitrogen ligand by ethyldiazoacetate. However, diazomethane, N_2CH_2 , reacts with A with loss of N_2 to give products which we tentatively formulate as containing methylene ligands, *trans*-[$M(CH_2)_2(dppe)_2$].

Keywords. Diazoalkane complexes; Dinitrogen complexes.

Herstellung von Bisdiazoalkan- und ähnlichen Komplexen aus den Reaktionen von Diazoverbindungen mit Distickstoffkomplexen des Typs *trans*-[$M(N_2)_2(Ph_2PCH_2CH_2PPh_2)_2$] mit $M = Mo$ oder W

Zusammenfassung. Die Reaktion von *trans*-[$M(N_2)_2(dppe)_2$] (A ; $dppe = Ph_2PCH_2CH_2PPh_2$ und $M = Mo$ oder W) mit Ethyldiazoacetat, $N_2CHCOOEt$, ergab nach einfachem Austausch des Distickstoffliganden mit Ethyldiazoacetat die Bisdiazoalkane *trans*-[$M(N_2CHCOOEt)_2(dppe)_2$]. Diazomethan (N_2CH_2) hingegen reagierte mit A unter Verlust von N_2 zu Produkten, die tentativ als *trans*-[$M(CH_2)_2(dppe)_2$] mit Methylenliganden formuliert wurden.

Introduction

The electron-rich site $\{M(dppe)_2\}$ ($M = Mo$ or W , $dppe = Ph_2PCH_2CH_2PPh_2$), generated from the corresponding dinitrogen complexes *trans*-[$M(N_2)_2(dppe)_2$] (A), has been shown to bind, apart from N_2 , a number of unsaturated molecules such as isocyanides and alkyne-derived species [1]. The strong electron release of the site provides a partial double bond character for the metal-ligand bonding with a concomitant weakening of the multiple bond of the unsaturated ligand which is rendered susceptible to electrophilic attack.

Hence, e.g., in the complexes *trans*-[$M(CNR)_2(dppe)_2$] the isocyanide ligands appear to display a partial carbene character and can be protonated [2] or alkylated

[3] yielding aminoalkylidyne ligands of the type $\equiv\text{C}-\text{NR}'\text{R}$ ($\text{R}'=\text{H}$ or Me). Similarly, alkynyl ligands ($-\text{C}\equiv\text{CR}$) at those sites are susceptible to β -electrophilic attack to give alkylidyne ligands ($\equiv\text{C}-\text{CH}_2\text{R}$) [4]. Therefore, these studies provided a variety of routes for complexes with ligands multiply bonded to the metal.

We were interested in extending this type of study to the binding of other reactive unsaturated species such as diazo compounds which are potential relevant intermediates for the syntheses of organonitrogenated products derived from dinitrogen [5]. In a previous study [6], we have detected the formation of benzene-azomethane ($\text{Ph}-\text{N}=\text{N}-\text{CH}_3$) and of a phosphazene species as organonitrogenated products of the reactions of ethyldiazoacetate ($\text{NN}=\text{CHCOOEt}$) with *trans*- $[\text{Mo}(\text{N}_2)_2(\text{dppe})_2]$, in benzene, or with the W analogue, respectively, but no organometallic compound was then characterized. We now report the formation of diazoalkane complexes in those reactions, and related studies with diazomethane ($\text{NN}=\text{CH}_2$).

Experimental Part

Experiments were carried out in the absence of air, through standard high-vacuum and inert gas flow techniques. Solvents were dried and degassed by standard procedures.

Infrared spectra were recorded on a 577 or 457 Perkin Elmer Grating Infrared Spectrophotometer. ^1H -NMR spectra were recorded on a Jeol JNM-PS-100 spectrometer (100 MHz), whereas ^{31}P -NMR spectra were recorded on a Jeol PFT 100 spectrometer operating at 40.49 MHz.

Conductivities were measured using a Portland Electronics conductivity bridge.

Complexes *trans*- $[\text{M}(\text{N}_2)_2(\text{dppe})_2]$ (A , $\text{M}=\text{Mo}$ or W) [7] and the diazomethane [8] were prepared by published methods. Ethyldiazoacetate was used as purchased from Aldrich.

a) Preparation of the Bis(diazo) Complexes

trans-Bis[1,2-bis(diphenylphosphino)ethane]bis(ethyldiazoacetate)tungsten(0),
trans- $[\text{W}(\text{N}_2\text{CHCOOEt})_2(\text{dppe})_2]$ (**B** and **C**, $\text{M}=\text{W}$)

Ethyldiazoacetate (0.0660 mol, 0.633 mmol) was added to a benzene solution (20 cm³) of *trans*- $[\text{W}(\text{N}_2)_2(\text{dppe})_2]$ (0.262 g, 0.253 mmol), and the resulting system was left stirring in a N_2 atmosphere, under irradiation by two 150 W tungsten filament bulbs.

The solution colour darkened and, after one day, infrared spectroscopy of the solution showed the absence of the starting compounds, and strong bands could be seen at 1730, 1685 and 1660 cm⁻¹.

The solution was then concentrated, filtered and a brown solid **X** precipitated upon slow addition of hexane. This solid was a mixture of both complexes **B** and **C** as evidenced by its infrared spectrum, and further crops (progressively richer in species **C**) were obtained upon concentration of the mother liquor and addition of hexane.

Methanol (11 cm³) was added to the total amount of solid **X**, the mixture was stirred for a few minutes, the solution was filtered and the remaining solid was washed with methanol; this solid was the complex **B** ($\text{M}=\text{W}$) (1675 cm⁻¹) in an analytically pure form (0.13 g, 0.113 mmol, 45% yield). Concentration of the filtered solution, addition of diethylether and cooling led to the precipitation of complex **C** ($\text{M}=\text{W}$) (1725 cm⁻¹) contaminated with a small amount of complex **B**; recrystallisation from methanol/diethylether afforded complex **C** in a more pure form, although still contaminated (0.050 g, ca. 0.043 mol, ca. 17% yield).

trans-Bis[1,2-bis(diphenylphosphino)ethane]bis(ethyldiazoacetate)molybdenum(0),
trans- $[\text{Mo}(\text{N}_2\text{CHCOOEt})_2(\text{dppe})_2]$ (**B** and **C**, $\text{M}=\text{Mo}$)

Complex **C** ($\text{M}=\text{Mo}$) (1730 cm⁻¹) may be prepared from a benzene solution of **A** ($\text{M}=\text{Mo}$) and ethyldiazoacetate using a variety of experimental conditions. These can be: with or without irradiation

(even in the dark); under N_2 or argon; using ethyldiazoacetate in variable proportions (relative amounts of 1 : 1 up to 1 : 15); suspending complex **A** in methanol with ethyldiazoacetate (1 : 15) under tungsten bulb light in a N_2 atmosphere. In a few experiments, complex **B** ($M = Mo$) (1690 cm^{-1}) is also formed, although in much lower yield than **C** and always contaminated with **C** ($M = Mo$).

As an example, the procedure described below may be used for the preparation of complex **C** and impure **B** ($M = Mo$).

Ethyldiazoacetate (0.26 cm^3 , 2.49 mmol) was added to a benzene (10 cm^3) solution of *trans*- $[Mo(N_2)_2(dppe)_2]$ (0.170 g , 0.179 mmol). The solution colour darkened and N_2 gas evolved.

After being stirred overnight the very dark solution was concentrated and a brown solid **X** separated out of the solution upon addition of hexane and cooling. A further crop of this solid was obtained upon concentration of the mother liquor, addition of hexane and cooling. Solid **X** was a mixture of species **B** and **C** ($M = Mo$) as evidenced by its infrared spectrum, and it was recrystallised from *THF*/hexane leading to complex **C** (1730 cm^{-1}) in an analytically pure form (0.130 g , 0.116 mmol , 65% yield) and to impure species **B** in very low yield.

b) Preparation of the Possible Bismethylene Complexes, trans-bis(methylene)bis[1,2-bis-(diphenylphosphino)ethane]molybdenum and tungsten, trans-[M(CH₂)₂(dppe)₂] (D, M = Mo and W)

The preparation of the **W** complex is analogous to the **Mo** species, and only the latter will be given in detail.

An ether solution (13 cm^3) of diazomethane (ca. 0.35 M) was added to a solution (17 cm^3) of *trans*- $[Mo(N_2)_2(dppe)_2]$ (0.209 g , 0.220 mmol) in benzene. The solution was stirred overnight under the light of two 150 W tungsten filament bulbs. The yellow suspension which precipitated out of the solution was filtered off, washed with benzene and dried under vacuum; further crops of the crude yellow solid were obtained upon concentration of the mother liquor and addition of ether.

The total amount (0.10 g) of the crude solid was then purified by recrystallisation from CH_2Cl_2/Et_2O : addition of 12 cm^3 of CH_2Cl_2 followed by filtration, concentration and slow addition of ether gave complex **D** ($M = Mo$) as a light yellow solid; new addition of ether to the solution separated from this complex by filtration yielded a further crop of such a complex as thin light yellow needles (total amount of 0.060 g , 0.065 mmol , 30% yield).

Results and Discussion

Reactions of trans-[M(N₂)₂(dppe)₂] (A) with Ethyldiazoacetate. Formation of the Bis(ethyldiazoacetate) Complexes trans-[M(N₂CHCOOEt)₂(dppe)₂] (B and C)

Reaction of *trans*- $[W(N_2)_2(dppe)_2]$ (**A**, $M = W$) in benzene with $N_2CHCOOEt$ (in a molar ratio of 1 : 2.5), under tungsten filament light and under N_2 for one day, led to the precipitation of a mixture of two species with strong bands at 1675 cm^{-1} (**B**, $M = W$) and 1725 cm^{-1} (**C**, $M = W$) in the infrared spectra, which could be separated by recrystallisation from methanol/ether, although the latter, which is the most soluble in methanol, was not obtained analytically pure.

A corresponding species (**C**, $M = Mo$) with a strong infrared band at 1730 cm^{-1} was obtained from reaction between *trans*- $[Mo(N_2)_2(dppe)_2]$ (**A**, $M = Mo$) and ethyldiazoacetate in benzene. This was the predominant product obtained, despite considerable variation of reaction conditions (see experimental), although another species (**B**, $M = Mo$) with a strong band at 1690 cm^{-1} was also formed in much lower yield. As isolated, **B** ($M = Mo$) was always contaminated by **C** ($M = Mo$).

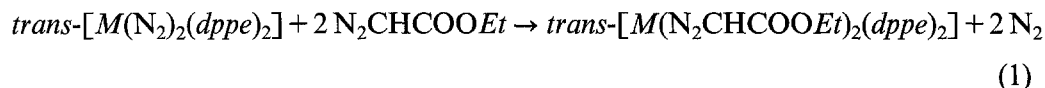
The formulation of species **B** and **C** as the bis(ethyldiazoacetate) complexes *trans*- $[M(N_2CHCOOEt)_2(dppe)_2]$, formed through replacement of N_2 in the parent

Table 1. Physical data for the bis(ethyl diazoacetate) complexes *trans*-[M(N₂CHCOOEt)₂(*dppe*)₂] (**B**, **C**) and the possible bismethylene complexes *trans*-[M(CH₂)₂(*dppe*)₂] (**D**)

Complex	Colour	Infrared data ^a ν (C=O)	Elemental Analysis ^b		
			%C	%H	%N
B (<i>M</i> = W)	Dark brown	1 675 s	59.1 (59.6)	5.1 (5.0)	4.4 (4.6)
B (<i>M</i> = Mo) ^c	Brown	1 690 s			
C (<i>M</i> = Mo)	Brown	1 730 s	63.7 (64.3)	5.7 (5.4)	4.6 (5.0)
C (<i>M</i> = W) ^c	Brown	1 725 s			
D (<i>M</i> = Mo)	Yellow		70.6 (70.4)	5.9 (5.7)	
D (<i>M</i> = W)	Yellow		63.5 (64.3)	5.3 (5.2)	

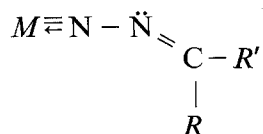
^a In Nujol mull^b Theoretical values in parenthesis^c Not obtained in an analytically form

complexes by the diazo compound (reaction 1), is based on analytical, infrared, ¹H-, ³¹P-NMR and conductivity data (Tables 1 and 2).



During the reaction, N₂ evolution is observed and it is assisted by photochemical excitation, a process which has been rationalized [9] in terms of a π-MO scheme.

Diazoalkane complexes related to **B** and **C**, [MX(NN=CRR')(diphos)₂]⁺ (*M* = Mo or W, *X* = halide, *diphos* = *dppe* or related diphosphine), have been prepared by the quite different routes of reaction of **A** with Br₂CRR' [10] or with THF/MeBr/HBr [11], as well as of [MX(NNH₂)(diphos)₂]⁺ with RR'C=O or RCHO [12, 13]. With other metal sites, however, diazoalkane complexes have been obtained by the ligand displacement route used here, e.g., [M(CO)(NNCHR)(S₂CNR'₂)₂] from [M(CO)₃(S₂CNR'₂)₂] [14]. In these diazoalkane complexes, the diazoalkane ligand has a linear MNN grouping and behaves as a four-electron donor



so that the effective atomic number rule is obeyed.

However, in the bisdiazo complexes of the present study, to maintain the 18-electron count, each diazo ligand must behave as a two-electron donor (Fig. 1); we

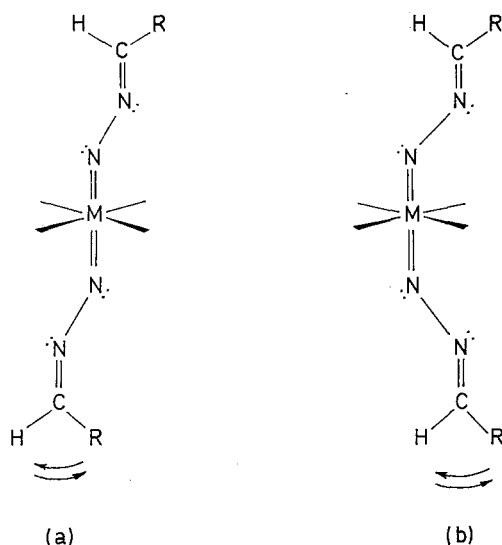


Fig. 1. Some possible isomers for complexes $trans\text{-}[M(N_2CHCOOEt)_2(dppe)_2]$ ($M = Mo$ or W)

note that the proposed structure (a) resembles that established [15] for $trans\text{-}[Mo(CNMe)_2(dppe)_2]$ and might therefore be also the most favoured configuration.

Absorptions in the $1675\text{--}1730\text{ cm}^{-1}$ region of the infrared spectra of **B** and **C** are assigned to $\nu(\text{CO})$ of the acetate groups, but no band could be assigned to $\nu(\text{C}=\text{N})$ (in the $1510\text{--}1600\text{ cm}^{-1}$ range); such a band was also absent from spectra of diazo complexes such as $[WBr(N-N=CH_2)(dppe)_2]Br$ [10, 11] and $[MoF(N-N=CH_2)(dppe)_2]BF_4$ [12].

The *trans* geometry in complexes **B** and **C** follows from the single resonance which is observed in the ^{31}P -NMR spectra (Table 2).

Solutions of these complexes in nitromethane are non-conductors, in agreement with neutral species.

Compounds of types **B** and **C** appear to be isomers, a main spectroscopic difference being the resonance of the $N_2CHCOOEt$ proton which in the ^1H -NMR spectra appears at δ 4.9 ppm ($M = W$, for type **B** species) and at δ 2.40 or 2.50 ppm (for $M = W$ or Mo , in compounds of type **C**).

In view of the number of possible isomers for **B** and **C** and the probability of restricted rotation about the $M=N$ and $N=C$ bonds, it is not surprising that the NMR spectra show isomers of **B** and **C** in solution, which would account for the observed broadening of the ethyl and the $CHCOOEt$ proton magnetic resonances. It is not possible *a priori* to decide which isomeric form should dominate.

*Reactions of $trans\text{-}[M(N_2)_2(dppe)_2]$ with Diazomethane. Formation of the Possible Bismethylene Complexes $trans\text{-}[M(CH_2)_2(dppe)_2]$, (**D**)*

If solutions of $trans\text{-}[M(N_2)_2(dppe)_2]$ (**A**, $M = Mo$ and W) in C_6H_6 with an excess of N_2CH_2 are left stirring overnight under the irradiation of a tungsten filament bulb, light yellow species **D** separate out of the solutions. They may be recrystallised

Table 2. ^1H and ^{31}P -NMR data for the bis(ethyldiazoacetate) complexes *trans*- $[M(\text{N}_2\text{CHCOOEt})_2(\text{dppe})_2]$ (**B**, **C**) and the possible bismethylene complexes *trans*- $[M(\text{CH}_2)_2(\text{dppe})_2]$ (**D**)

Complex	^1H -NMR ^a			$^{31}\text{P}\{^1\text{H}\}$ -NMR ^b
	δ	Integration	Assignment	δ
B ($M=\text{W}$)	7.5–6.8 m	40	–C ₆ H ₅ (40)	110.8 ^d
	4.9 m	2	–CHCOOEt (2)	
	4.08 q ^c	4	–CHCOOCH ₂ CH ₃ (4)	
	2.9–2.2	8	–CH ₂ CH ₂ – (8)	
	1.30 t ^c	6	–CH ₂ CH ₃ (6)	
C ($M=\text{Mo}$) ^e	8.6–6.4 m	40	–C ₆ H ₅ (40)	108.0
	4.15 q, br ^f	11	–CHCOOCH ₂ CH ₃ (4)	
	4.4–2.0 m		–CH ₂ CH ₂ – (8)	
	2.50 s	2	–CHCOOEt (2)	
	1.25 t, br	6	–CHCOOCH ₂ CH ₃ (6)	
C ($M=\text{W}$)	7.8–6.7 m	40	–C ₆ H ₅ (40)	107.8
	4.4–3.9 m, br	4	–CHCOOCH ₂ CH ₃ (4)	
	3.4–2.4 m, br	8	–CH ₂ CH ₂ – (8)	
	2.40 s, br	2.5	–CHCOOEt (2)	
	1.5–0.9 m, br	6	–CHCOOCH ₂ CH ₃ (6)	
D ($M=\text{Mo}$)	8.0–7.0 m	40	–C ₆ H ₅ (40)	59.8 ^e
	3.40 q ^c	1	O(CH ₂ CH ₃) ₂ ^g	
	3.3–1.4 m	11	–CH ₂ CH ₂ – (8)	
	2.44 s, br		=CH ₂ (4)	
	1.10 t ^c	1.5	O(CH ₂ CH ₃) ₂ ^g	
D ($M=\text{W}$)	7.8–7.0 m	40	–C ₆ H ₅ (40)	59.8
	3.40 q ^c	1	O(CH ₂ CH ₃) ₂ ^g	
	3.2–1.2 m	11	–CH ₂ CH ₂ – (8)	
	2.18 s ^h		=CH ₂ (2)	
	2.16 s ^h		=CH ₂ (2)	
1.04 t ^c	1.5	O(CH ₂ CH ₃) ₂ ^g		

^a In CD₂Cl₂ at 25°C, δ values in ppm relative to TMS

^b In CD₂Cl₂ at 25°C, δ values upfield from internal TMP

^c $^3J_{\text{H-H}} = 7.0$ Hz

^d $^1J_{^{183}\text{W}-^{31}\text{P}} = 283.2$ Hz

^e No appreciable difference was observed upon cooling at –70°C

^f $^3J_{\text{H-H}} = 6 \pm 1$ Hz

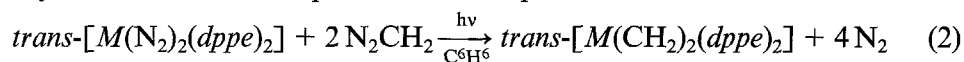
^g Solvent of crystallisation

^h Two equally intense singlets with total integration of ca. 5

from CH₂Cl₂/Et₂O as very thin needles which unfortunately are unsuitable for crystal structure determination.

Their postulated formulation as the bismethylene complexes *trans*- $[M(\text{CH}_2)_2(\text{dppe})_2]$, formed according to reaction (2), is supported by microana-

lytical, infrared, ^1H -, ^{31}P -NMR and conductivity data (Tables 1 and 2). The low solubility in common solvents precluded the acquisition of relevant ^{13}C -NMR data.



The solutions of complexes **D** in nitromethane are nonconductors and the singlet ^{31}P -NMR resonance agrees with a *trans* configuration for the *dppe* ligands.

In the ^1H -NMR spectra, two singlet resonances (each integrating for ca. 2.5 protons at δ 2.18 and 2.16 ppm) and a broad singlet resonance at δ 2.44 ppm (integrating for ca. 5 protons) are observed for complexes **D** ($M=\text{W}$) and **D** ($M=\text{Mo}$), respectively. These resonances are assigned to the carbene CH_2 protons and their splitting suggests a staggered configuration for these two CH_2 groups, the $\text{H}_2\text{C}=\text{M}=\text{CH}_2$ grouping possibly resembling allene in structure.

The ^1H -NMR resonances of the CH_2 groups are at higher field than those commonly observed at less electron-rich sites with transition metals of other groups, namely $[\text{Ta}(\eta^5\text{-C}_5\text{H}_5)_2(\text{CH}_3)(\text{CH}_2)]$ [16] or $[M(\text{CH}_2)\text{Cl}(\text{NO})(\text{PPh}_3)_2]$ ($M=\text{Ru}$ or Os) [17] which probably reflects the different type of methylene binding in the two types of complexes.

In complexes **D**, the bonding of each CH_2 to the metal may be regarded as formed from a σ component by donation of an electron pair from a sp_2 orbital of **C** to a metal vacant orbital, and a π component by backbonding from a filled metal d orbital to the vacant p carbon orbital; hence the ligating methylene may be envisaged as a π -acceptor singlet carbene. However, at the above mentioned less electron-rich metal centres, the methylene possibly may act as a $(\sigma + \pi)$ donor triplet carbene.

The use of diazomethane as a methylene ligand source has been applied to the syntheses of other d^6 metal complexes, e.g., $[M\text{Cl}(=\text{CH}_2)(\eta\text{-C}\{\text{O}\}R)(\text{PPh}_3)_2]$ ($M=\text{Ru}$ or Os ; $R=\text{aryl}$) derived from the methylene addition to the coordinatively unsaturated compounds $[M\text{RCl}(\text{CO})(\text{PPh}_3)_2]$ [17].

Final Comments

Although ethyldiazoacetate adds intact to the $\{M(\text{dppe})_2\}$ ($M=\text{Mo}$ or W) metal sites, the less stable diazomethane photochemically generates methylene which appears to be trapped and stabilized by the metal centre.

The formation of the diazo and the possible methylene complexes agrees with the known [1] ability of those metal sites to form multiple bonds to nitrogen and to carbon and this study demonstrates that diazoalkanes can be used as reagents for the synthesis of complexes with such types of bonds at the above mentioned electron-rich site.

The potential application of these types of complexes in the syntheses of organonitrogenated products will be investigated.

Acknowledgements

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