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Chromium complexes with mixed Group 15 elements as ligands. Synthesis and characterisation of the first *cyclo*-P_2As ligand in $[CrCp(CO)_2(\eta^3\text{-}P_2As)]$ †

Shubhangi Umbarkar, Perumal Sekar and Manfred Scheer *

Institute of Inorganic Chemistry, University of Karlsruhe, D-76128 Karlsruhe, Germany.
E-mail: mascheer@achibm6.chemie.uni-karlsruhe.de

Received 9th December 1999, Accepted 17th February 2000
Published on the Web 13th March 2000

Reaction of $[\{CrCp(CO)_2\}_2(\mu\text{-}\eta^2\text{-}P_2)]$ **1** with PCl_5 or ECl_3 (E = P, As or Sb) led to *cyclo*-P_2E ligand complexes $[CrCp(CO)_2(\eta^3\text{-}P_3)]$ **2** and $[CrCp(CO)_2(\eta^3\text{-}P_2As)]$ **4**. The eliminated $[CpCr(CO)_2]$ fragment of **1** is converted into $[CrCp(\mu\text{-}Cl)Cl]_2$ **3**. Co-crystals of **4** and **2** were characterised by Total Reflection X-ray Fluorescence Analysis and by single crystal X-ray diffraction analysis, revealing the tetrahedral CrP_2As core.

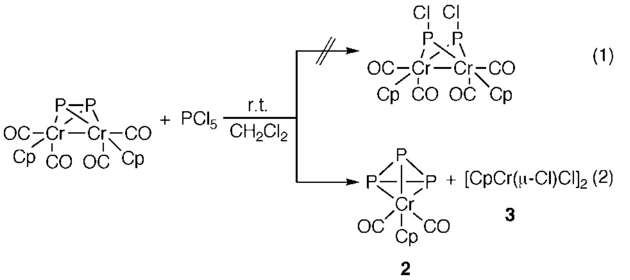
Introduction

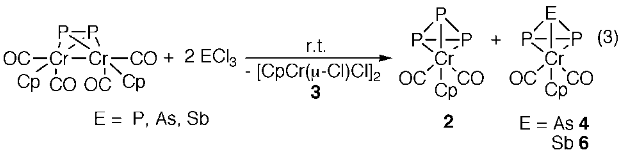
The last few decades have witnessed a surge in activity in the area of co-ordination chemistry of naked Group 15 elements.[1] Although a large number of polyhedrons, containing such elements of different co-ordination numbers have been discovered, the reactivity of these complexes is much less explored.[2] In continuation of our interest to explore unusual reactivity features of metal complexes containing naked Group 15 elements,[3] we have focused our attention on using the tetrahedral complex $[\{CrCp(CO)_2\}_2(\mu\text{-}\eta^2\text{-}P_2)]$ **1**[4] as potential starting material. Compound **1** is a reactive complex in which the P–P bond is considerably more inert as compared to the Cr–P and Cr–Cr bonds. By starting from this complex, we tried first to cleave its P–P bond by using $LiBEt_3H$ to generate a dianionic complex, which however led to the isolation of the novel PH containing complexes $[\{CrCp(CO)_2\}_2(\mu\text{-}PH_2)(\mu\text{-}H)]$, $[\{CrCp(CO)_2\}_2(\mu\text{-}PH_2)_2]$, and $[\{[CrCp(CO)_2]_2(\mu\text{-}PH)\}\{(CrCp)_2(\mu\text{-}\eta^1{:}\eta^1{:}\eta^5{:}\eta^5\text{-}P_5)\}]$.[5] In further investigations we attempted to open the P–P bond in **1** oxidatively by chlorination and our unexpected results are reported herein.

Results and discussion

In the past, we have successfully used PCl_5 to chlorinate the W–P bond in the *cyclo*-P_4 complex $[\{W(CO)_4\}\{PW(CO)_5\}_4]$[3a] as well as to chlorinate metal carbonyls to obtain various transition metal chlorides and Cp containing metal chlorides, respectively.[6]

Therefore the chlorination of the P–P bond in compound **1** was attempted by using PCl_5 as chlorinating agent at ambient temperature to obtain the possible dichloro-complex [eqn. (1)]. However, this reaction led to the replacement of one $[Cp(CO)_2Cr]$ unit by a phosphorus atom and $[CrCp(CO)_2(\eta^3\text{-}P_3)]$ **2** was isolated as shown in eqn. (2). Surprisingly, we found the same reaction pattern when PCl_3 was used [eqn. (3)], a reaction pattern never found before.





Further isolated from these reaction mixtures was the paramagnetic compound $[CrCp(\mu\text{-}Cl)Cl]_2$ **3**, which was identified by crystal structure analysis at 203 K. In 1983 Köhler *et al.*[7] first synthesized this compound by chlorination of $CrCp_2$ with $CHCl_3$ at high temperature (230 °C) and its structure was determined at room temperature. When **1** was treated with $AsCl_3$ in toluene at room temperature, along with compounds **2** and **3**, a mixed P/As analogue of **2**, $[CrCp(CO)_2(\eta^3\text{-}P_2As)]$ **4**, was formed [eqn. (3)]. Compounds **2** and **4** could not be separated by column chromatography or thin layer chromatography as the R_f values for both compounds are the same. Thus, **2** and **4** were obtained as a mixture from n-hexane. Furthermore, a brown solid was obtained from toluene. The fragmentation pattern in the mass spectrum shows it to be a mixture of the triple decker sandwich complexes $[(CrCp)_2(\eta^5\text{-}P_xAs_{5-x})]$ **5** ($x = 1$–5). However, there were no signals observed in the EPR (130 K) and ^{31}P NMR spectra (300 K) of this mixture at ambient temperature. When separation of compounds **2** and **4** by fractional crystallisation was attempted, red co-crystals of **2** and **4** (referred to as **4a** hereafter) were obtained and characterised by single crystal X-ray diffraction analysis. The percentage of phosphorus and arsenic in **4a** was determined by TRFA (Total Reflection X-ray Fluorescence Analysis) measurements, which showed the crystals to contain P : As in a ratio of 20 : 1, which comes to a ratio of complexes **2** to **4** of about 6 : 1.

Complex **4a** crystallises in the monoclinic space group $P2_1/c$ with two independent molecules in the asymmetric unit cell, similar to pure compound **2**.[4] The molecular structure of one of the molecules of **4a** is shown in Fig. 1, which depicts the tetrahedral CrP_2As core. Selected bond lengths and bond angles are given in Table 1. The Cr–E/E′ and E–E′ (E, E′ = P or As) bond lengths in **4a** are slightly longer than the Cr–P and P–P bond

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† Dedicated to Professor Reinhard Ahlrichs on the occasion of his 60th birthday.

DOI: 10.1039/a909717a

Table 1 Selected bond lengths (Å) and bond angles (°) for compound **4a**

Cr(1)–P/As(1)	2.4630(11)	P/As(1)–P/As(2)	2.1528(14)
Cr(1)–P/As(2)	2.4441(12)	P/As(2)–P/As(3)	2.356(16)
Cr(1)–P/As(3)	2.5154(13)	P/As(1)–P/As(3)	2.1560(15)
P/As(2)–P/As(1)–P/As(3)	59.42(5)	P/As(2)–Cr(1)–P/As(3)	50.99(4)
P/As(3)–P/As(2)–P/As(1)	60.36(5)	P/As(2)–Cr(1)–P/As(1)	52.04(4)
P/As(2)–P/As(3)–P/As(1)	60.21(5)	P/As(1)–Cr(1)–P/As(3)	51.31(4)
P/As(2)–P/As(1)–Cr(1)	63.53(4)	P/As(3)–P/As(1)–Cr(1)	65.60(4)
P/As(3)–P/As(2)–Cr(1)	66.23(4)	P/As(1)–P/As(2)–Cr(1)	64.43(4)
P/As(2)–P/As(3)–Cr(1)	62.78(4)	P/As(1)–P/As(3)–Cr(1)	63.09(5)

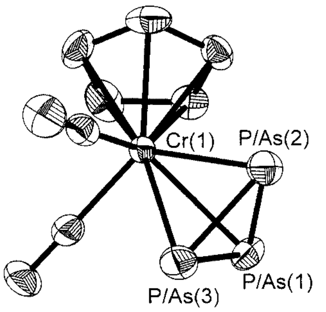

Fig. 1 Molecular structure of one of the two independent molecules in the asymmetric unit of complex **4a** (ellipsoids drawn at 50% probability level).

lengths in **2** and shorter than the Cr–As and As–As bond lengths in [CrCp(CO)$_2$(η^3-As$_3$)].[8] The average P–P bond length in **2** is 2.123 Å and average As–As bond length in [CrCp(CO)$_2$(η^3-As$_3$)] is 2.338 Å whereas the average E–E′ bond length in **4a** is 2.1445 Å. The E–E′ bond lengths in **4a** are also found to be longer than the average P–P bond distances in [NiCp*(η^3-P$_3$)],[9] 2.1003 Å, [MoWCp$_2$(CO)$_4$(μ,η^2-P$_2$)],[10] 2.104(4) Å, and [{WCp(CO)$_2$}$_2$(μ,η^2-P$_2$)],[10] 2.087(6) Å, but shorter than As–As bond distances in [Co(CO)$_3$(η^3-As$_3$)],[11] 2.372(5) Å, [MoCp*(CO)$_2$(η^3-As$_3$)],[12] 2.372(1)–2.377(2) Å, [CrCp*(CO)$_2$(η^3-As$_3$)],[13] 2.347(2)–2.361(2) Å, [{CrCp(CO)$_2$}$_2$(μ-η^2-As$_2$)],[14] 2.276(1) Å, [{MoCp(CO)$_2$}$_2$(μ-η^2-As$_2$)],[15] 2.312(3) Å, and [MoCp(CO)$_2$(η^3-As$_3$)],[12] 2.372(1) and 2.377(2) Å, respectively. The average Cr–P bond distance in **2** is 2.454 Å and average Cr–As bond length in [CrCp(CO)$_2$(η^3-As$_3$)] is 2.5796 Å, whereas the average Cr–E/E′ bond distance in **4a** is 2.474 Å which is shorter than the Cr–As bond distances in [CrCp*(CO)$_2$(η^3-As$_3$)],[13] 2.542(1)–2.635(2) Å and [{CrCp(CO)$_2$}$_2$(μ-η^2-As$_2$)],[14] 2.597(1) and 2.452(1) Å. The crystals of **4a** were further characterised by ^{1}H and ^{31}P NMR spectroscopy and mass spectrometry. The ^{31}P NMR spectrum of **4a** shows the presence of two singlets, one at δ −284.7, which corresponds to complex **2**, and the second at δ −241.6, which corresponds to **4**. The mass spectrum of these crystals shows molecular ion peaks at *m*/*z* 265.9 for **2** and 309.9 for **4**. It also shows the presence of $[M - CO]^+$ and $[M - 2CO]^+$ fragment peaks for both of the compounds.

When complex **1** was treated with SbCl$_3$ in dichloromethane at room temperature the antimony analogue of complex **4**, [CrCp(CO)$_2$(η^3-P$_2$Sb)] **6**, was observed along with **2** and **3** [eqn. (3)]. However **6** could not be isolated either in a pure form or in a co-crystalline form and was characterised by ^{31}P NMR spectroscopy only, which showed a singlet at δ −72, besides the singlet at δ −284.7 for **2**. Generally, it was found that the mixed E$_2$E′ ligand containing complexes **4** and **6** are much less stable than the phosphorus analogue **2**. Based on the recorded ^{31}P NMR spectra, during their synthesis and work-up considerable decomposition occurred.

Conclusion

The results show the unusual reaction pattern between [{CrCp(CO)$_2$}$_2$(μ-η^2-P$_2$)] **1** and ECl$_3$, resulting in the novel *cyclo*-P$_2$E ligand complexes. Complex **4** represents the first example of a *cyclo*-E$_3$ ligand complex containing different Group 15 elements. The general first example of this novel class of mixed element Group 15 ligand complexes was discovered by Mays and co-workers[2i] with the synthesis of the EE′ ligands in [{MoCp(CO)$_2$}$_2$(μ-η^2-PE)] (E = As or Sb). At present there are insufficient data to speculate about the pathway of reactions (2) and (3) leading to the formation of **2** and **4**, respectively. Either the Cr–Cr bond in **1** is first opened by the nucleophilic attack of ECl$_3$, or CO substitution occurs followed by so far unknown subsequent transformations to give the final products.

Experimental

Reactions and manipulations were carried out under an inert atmosphere of dry argon using standard Schlenk techniques. All solvents were dried by common methods and distilled prior to use. The complex [{CrCp(CO)$_2$}$_2$(μ-η^2-P$_2$)] was prepared by literature methods;[4] PCl$_3$, PCl$_5$, AsCl$_3$ and SbCl$_3$ were purchased from Aldrich Chemical Co. and used as such without any further purification. The NMR spectra were recorded on Bruker AC 250 (^{1}H, ^{13}C, ^{29}Si) and AMX 300 (^{1}H), spectrometers using TMS as external standard, IR spectra on a Bruker IFS 28, and mass spectra on a Varian MAT 711. Elemental analyses were made on an Elementar Vario EL instrument. The TRFA measurements were performed on an Atomika Extra IIa instrument with Mo-Kα radiation.

Reaction of [{CrCp(CO)$_2$}$_2$(μ-η^2-P$_2$)] 1

With PCl$_5$. The compound PCl$_5$ (0.208 g, 1 mmol) was added to a stirred solution of [{CrCp(CO)$_2$}$_2$(μ-η^2-P$_2$)] **1** (0.408 g, 1 mmol) in 30 ml CH$_2$Cl$_2$ at room temperature. After 15 min all of the volatile materials were evaporated in high *vacuo*. The reaction mixture was extracted with n-hexane. The resulting yellow solution was reduced to 10 ml and stored at 0 °C. After a few days dark crystals of [CrCp(CO)$_2$(η^3-P$_3$)] **2** were isolated (125 mg, 0.47 mmol, 23.5% based on **1**). The green residue was recrystallised from CH$_2$Cl$_2$ and found to be [CrCp(μ-Cl)Cl]$_2$ **3** (188 mg, 0.5 mmol, 50% based on **1**). Compound **2** (Found: C, 31.80; H, 1.70. C$_7$H$_5$CrO$_2$P$_3$ requires C, 31.60; H, 1.89%): ^{1}H NMR (C$_6$D$_6$) δ 3.92 (C$_5$H$_5$); ^{31}P NMR (C$_6$D$_6$) δ −284. Compound **3** (Found: C, 31.61; H, 2.44. C$_5$H$_5$Cl$_2$Cr requires C, 31.94; H, 2.68%); MS (70 eV EI) *m*/*z* (%) 377.7 (15) $[M]^+$, 338.8 (76), $[M - Cl]^+$; 310.8 (6), $[M - Cp]^+$; 273.8 (15), $[M - CpCl]^+$ and 151.9 (100), $[M - CrCpCl_3]^+$.

With PCl$_3$. The compound PCl$_3$ (0.18 ml, 2 mmol) was added to a stirred solution of [{CrCp(CO)$_2$}$_2$(μ-η^2-P$_2$)] **1** (0.408 g, 1 mmol) in 30 ml CH$_2$Cl$_2$ at room temperature. The work-up was done as described above giving 133 mg (0.5 mmol, 25% based on **1**) of [CrCp(CO)$_2$(η^3-P$_3$)] **2** and 150 mg (0.4 mmol, 40% based on **1**) of [CrCp(μ-Cl)Cl]$_2$ **3**.

With AsCl$_3$. The compound AsCl$_3$ (0.27 ml, 3.19 mmol) was added to a solution of [{CrCp(CO)$_2$}$_2$(μ-η^2-P$_2$)] **1** (650 mg, 1.59 mmol) in 30 ml toluene at room temperature. After stirring for 2.5 h all of the solvent was evaporated in high *vacuo*. The reaction mixture was first extracted with n-hexane which gave a

Table 2 Crystallographic data for compound **4a**

Empirical formula	$C_7H_5As_{0.12}CrO_2P_{2.88}$
T/K	190(1)
M_r	271.46
Crystal system	Monoclinic
Space group	$P2_1/c$
a/Å	11.287(2)
b/Å	14.365(3)
c/Å	13.237(7)
β/°	114.99(3)
V/Å^3	1945.3(7)
Z	8
μ(Mo-Kα)/mm^{-1}	2.025
Reflections collected	8261
Independent reflections	3414 (R_{int} = 0.0541)
Reflections with $I > 2\sigma(I)$	2692
Final $R1$, $wR2$ ($I > 2\sigma(I)$)	0.0403, 0.1044
(all data)	0.0560, 0.1162

yellow fraction containing a mixture of $[CrCp(CO)_2(\eta^3\text{-}P_3)]$ **2** and $[CrCp(CO)_2(\eta^3\text{-}P_2As)]$ **4** from which 90 mg (10% based on **1**) of co-crystals **4a** were obtained. Further extraction with toluene gave a brown solution from which a brown solid **5** precipitated, which was shown by mass spectroscopy to be a mixture of the compounds $[(CrCp)_2(\eta^5\text{-}P_xAs_{5-x})]$ ($x = 1$–5) (100 mg). Extraction with CH_2Cl_2 gave a green solution, from which green crystals of $[CrCp(\mu\text{-}Cl)Cl]_2$ **3** (90 mg, 0.24 mmol, 15% based on **1**) crystallised. Compound **4a**: IR (hexane) $\tilde{\nu}$ (CO) 1993s and 1947s; ^{1}H NMR (C_6D_6) δ 3.909 ($C_5H_5CrP_3$, **2**), 3.912 ($C_5H_5CrP_2As$, **4**); ^{31}P NMR (C_6D_6) δ −284.7 (**2**), −241.6 (**4**); MS (70 eV, EI) m/z (%) 265.9 (62) (M$^+$ for **2**), 237.9 (82), $[M - CO]^+$; 209.9 (100), $[M - 2CO]^+$; 309.9 (11) (M$^+$ for **4**), 281.9 (3), $[M - CO]^+$; 253.9 (28), $[M - 2CO]^+$; 188.8 (2) $[M - 2CO - Cp]^+$. Compound **5**: MS (EI, 70 eV) 564.5 (2), $[Cp_2Cr_2PAs_4]^+$; 520.5 (2), $[Cp_2Cr_2P_2As_3]^+$; 476.6 (6), $[Cp_2Cr_2P_3As_2]^+$; 413.9 (10), $[Cp_2Cr_2PAs_2]^+$; 432.7 (13), $[Cp_2Cr_2P_4As]^+$; 370.8 (8), $[Cp_2Cr_2P_2As]^+$; 388.7 (16), $[Cp_2Cr_2P_5]^+$; 326.8 (20), $[Cp_2Cr_2P_3]^+$. Compound **3**: MS (EI, 70 eV) 377.7 (15), $[M]^+$; 338.8 (76), $[M - Cl]^+$; 310.8 (6), $[M - Cp]^+$; 273.8 (15), $[M - CpCl]^+$; 151.9 (100), $[M - CrCpCl_3]^+$.

With $SbCl_3$. The compound $SbCl_3$ (0.662 mg, 2.9 mmol) was added to a solution of $[\{CrCp(CO)_2\}_2(\mu\text{-}\eta^2\text{-}P_2)]$ (600 mg, 1.47 mmol) in 30 ml CH_2Cl_2 at room temperature. After stirring the mixture for 10 min all of the solvent was evaporated in high *vacuo*. Either by chromatographic separation or by extraction with different solvents no separation of the products was achieved. The presence of compound **2** and an antimony analogue of **4**, $[CrCp(CO)_2(\eta^3\text{-}P_2Sb)]$ **6**, was detected by ^{31}P NMR spectroscopy. The presence of **3** in the reaction mixture was confirmed on the basis of the mass spectrometric data. ^{31}P NMR (C_6D_6): δ −284.7 (**2**), −72 (**6**).

Crystal structure determinations

Data were collected on a STOE IPDS (**4a**) and a STOE STADI4 (**3**) diffractometer using Mo-Kα (λ = 0.71069 Å) radiation with empirical absorption corrections for **3** (ψ-scans). The structures were solved by direct methods using SHELXS 86,[16] full-matrix least-squares refinement on F^2 in SHELXL 93[17] with anisotropic displacement for non-H atoms located in idealised positions and refined isotropically according to the riding model. All non-hydrogen atoms were refined anisotropically The occupancies of the atomic positions of P and As in both independent molecules of **4a** were refined and were similar to those obtained from the TRFA analysis, which led to a further improvement of the final values of the refinement. The same anisotropic displacement parameters were used for the refinement of the same positions of a P and an As atom, respectively. Crystallographic data of **4a** are given in Table 2.

Crystal data of compound 3. $C_{10}H_{10}Cl_4Cr_2$, $M = 375.98$, orthorhombic, space group $Pbca$ (no. 61), $a = 11.144(2)$, $b = 9.853(2)$, $c = 12.139(2)$ Å, $V = 1332.7(5)$ Å^3, $T = 203(2)$ K, $Z = 4$, μ(Mo-Kα) = 2.399 mm^{-1}, 2892 reflections measured, 1530 unique ($R_{int} = 0.0388$) which were used in all calculations. The final $wR(F^2)$ was 0.0765. $R1 = 0.0299$.

CCDC reference number 186/1862.

See http://www.rsc.org/suppdata/dt/a9/a909717a/ for crystallographic files in .cif format.

Acknowledgements

Authors thank the Deutsche Akademische Austauschdienst (S. U.), the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support. Dr M. von Wagner is kindly acknowledged for the TRFA measurements.

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Paper a909717a