Note

Reactions of [Y(BDI)(I)$_2$(THF)] [BDI = {HC(CMeNAr)$_2$}$^-$, Ar = 2,6-diisopropylphenyl] with Na[M(Cp)(CO)$_3$] (M = Cr, W): X-ray crystal structures of [{Y(BDI)[Cr(Cp)(CO)$_3$]$_2$(THF)}$_2$] and [W(Cp)(CO)$_3$][Na(THF)$_2$]

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A R T I C L E   I N F O

A B S T R A C T

Reaction of [Y(BDI)(I)$_2$(THF)] (**1**) with two equivalents of Na[Cr(Cp)(CO)$_3$] affords the dimeric complex [{Y(BDI)[Cr(Cp)(CO)$_3$]$_2$(THF)}$_2$] (**2**). Complex **2** contains two yttrium-BDI units which are each linked by two isocarbonyl bridging [Cr(Cp)(CO)$_3$]$^-$ anions; a terminal, isocarbonyl bound [Cr(Cp)(CO)$_3$]$^-$ anion and THF molecule completes the coordination sphere at each yttrium. This results in formation of a centrosymmetric, 12-membered C$_4$O$_4$Cr$_2$Y$_2$ ring. Forcing conditions were required to produce carbonyl metallate derivatives such as **2**, as exemplified by the isolation of crystals of [W(Cp)(CO)$_3$][Na(THF)$_2$] (**3**) from the analogous reaction between **1** and two equivalents of Na[W(Cp)(CO)$_3$]. Complex **3** loses coordinated THF very easily and all isolated samples exhibit spectra consistent with the known, un-solvated form of Na[W(Cp)(CO)$_3$]. The crystal structure of **3** shows dimeric sodium units bridged by two THF molecules and one isocarbonyl group. Each sodium centre is further coordinated by one THF molecule and two isocarbonyl ligands. There are two crystallographically distinct [W(Cp)(CO)$_3$]$^-$ units; one exhibits two bridging isocarbonyl groups and the other exhibits three bridging isocarbonyl groups to different sodium dimer units. This results in a 2-dimensional polymeric sheet network in the solid state. Complex **2** was characterised by single crystal X-ray diffraction, NMR spectroscopy, FTIR spectroscopy and CHN microanalysis; complex **3** was characterised by single crystal X-ray diffraction only.

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1. Introduction

Heterobimetallic lanthanide (Ln)-transition metal assemblies are of much fundamental and practical interest [1]. For example, bi-metallic arrays may be employed in NIR-emission [2], and luminescence [3]. Additionally, they have potential to act as catalysts in processes as diverse as hydrogenation of phenol [4] and hydrodechlorination of chlorobenzenes [5]. When the transition metal fragment incorporates carbonyl groups a number of structural combinations become available, due to the possibility of bridging and isocarbonyl linkages, and the resulting complexes become relevant to Fischer–Tropsch reactions [6] and as precursors to perovskite-type oxides, LnMO$_3$, which are used as methane oxidation catalysts [7].

Recently, we reported a series of yttrium complexes supported by the sterically demanding β-diketiminate ligand {HC(CMeNAr)$_2$}$^-$ (BDI, Ar = 2,6-diisopropylphenyl) [8]. One of these complexes, [Y(BDI)(I)$_2$(THF)] (**1**) is an excellent start point for derivatisation so we decided to investigate the synthesis of chromium and tung-

sten carbonyl metallates. Herein, we report the synthesis and structure of [{Y(BDI)[Cr(Cp)(CO)$_3$]$_2$(THF)}$_2$] (**2**). Additionally, we report the crystal structure of the *bis*-THF adduct of Na[W(Cp)(CO)$_3$], namely [W(Cp)(CO)$_3$][Na(THF)$_2$] (**3**), which could not be isolated in its solvated form for further analysis due to facile and complete loss of coordinated THF.

2. Results and discussion

The reaction between **1** and two equivalents of Na[Cr(Cp)(CO)$_3$] [9] proceeds sluggishly, and refluxing of solutions was found to be necessary in order to obtain isolable products. Following work-up and recrystallisation, the dimeric complex [{Y(BDI)[Cr(Cp)(CO)$_3$]$_2$(THF)}$_2$] (**2**) was isolated in moderate yield; crystals of **2** rapidly collapse to a powder under dynamic vacuum. In order to record well resolved NMR spectra it was necessary to use deuterated THF, which implies that the dimeric structure observed in the solid state is maintained in non-coordinating solvents (*vide infra*) since we have observed this phenomenon before with other yttrium-BDI complexes which are dimeric in the solid state [8]. The simplicity of the ^{1}H and ^{13}C{^{1}H} NMR spectra in THF indicates a complex possessing C_s symmetry on the NMR timescale with

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equivalent $[Cr(Cp)(CO)_3]^-$ groups. The carbonyl groups appear as a broad resonance at 242 ppm in the $^{13}C\{^1H\}$ NMR spectrum which most likely implies dynamic exchange in solution, as well as long relaxation times [10]. However, cooling a solution in THF to the lowest accessible temperature did not result in de-coalesce of the resonance, and the complex precipitates at low temperature in non-coordinating solvents. Raising the temperature of either THF or toluene solutions of **2** does not resolve the resonance. The FTIR spectrum (Nujol) exhibits five identifiable carbonyl stretches which may be categorised as bridging [1604.4 (s), 1677.2 (s), 1697.5 (s) cm^{-1}] and terminal [1839.1 (s), 1930.0 (s) cm^{-1}]. There are no Ln-$[Cr(Cp)(CO)_3]$ complexes for comparison; however, these bands compare with v_{CO} stretches of 1660, 1740, 1800, 1900, and 2000 cm^{-1} for $[La\{(OC)Mo(Cp)(CO)_2\}_3(THF)_5]$ THF [1i], and stretches of 1687, 1745, 1834, and 1920 cm^{-1} for $[\{Ln(Tp^{tBuMe})(THF)[Mo(C_5H_4Me)(CO)_3]\}_2]$ (Ln = Sm, Yb) [1b]. However, the interpretation of the FTIR data for **2** is not straightforward; for example, the stretch at 1697 cm^{-1} appears to have a shoulder but this is not well resolved. We therefore carried out an X-ray diffraction study on a yellow crystal of **2**.

The molecular structure of **2** is illustrated in Fig. 1 and selected bond lengths and angles are in Table 1. The structure is dimeric, and is constructed around a crystallographic centre of inversion. Each yttrium centre is coordinated to a BDI ligand, a THF molecule, a terminally bound $[Cr(Cp)(CO)_3]^-$ group via a bridging isocarbonyl and to two other bridging $[Cr(Cp)(CO)_3]^-$ groups via a bridging isocarbonyl group from each. Thus, a 12-membered $C_4O_4Cr_2Y_2$ ring is formed. The presence of $[Cr(Cp)(CO)_3]^-$ groups which utilise two isocarbonyl groups in bridging is noteworthy as it has previously been noted [3a] to be unusual since formation of one isocarbonyl bridge renders the whole metallate fragment more electron poor and therefore less able to engage in further bridging to other metal centres. However, a similar arrangement was reported in $[\{Ce(C_5H_3R_2)_2[W(Cp)(CO)_3]\}_2]$ (R = SiMe$_3$) [3a] and $[\{Ln(Tp^{tBuMe})(THF)[Mo(C_5H_4Me)(CO)_3]\}_2]$ (Ln = Sm, Yb) [1b] which reflects the oxophilic nature of group 3 and lanthanide elements. The effects of the terminal versus bridging natures of the $[Cr(Cp)(CO)_3]^-$ groups in **2** is apparent from inspection of the Y–O bond lengths since the Y(1)–O(2) bond length of 2.190(2) Å is significantly shorter

than the Y(1)–O(4A) and Y(1)–O(5) bond lengths of 2.298(2) and 2.313(2) Å, respectively. Within each $[Cr(Cp)(CO)_3]^-$ fragment the presence of bridging isocarbonyl and terminal carbonyl groups is reflected in the disparity of the Cr–C$_{carbonyl}$ distances. For example, the bridging isocarbonyl Cr(1)–C(31) distance of 1.742(4) Å is markedly shorter than the terminal carbonyl Cr(1)–C(30) and Cr(1)–C(32) distances of 1.812(5) and 1.815(5) Å, respectively, and the bridging isocarbonyl Cr(2)–C(38) and Cr(2)–C(39) bond distances of 1.765(4) and 1.770(4) Å, respectively, are significantly
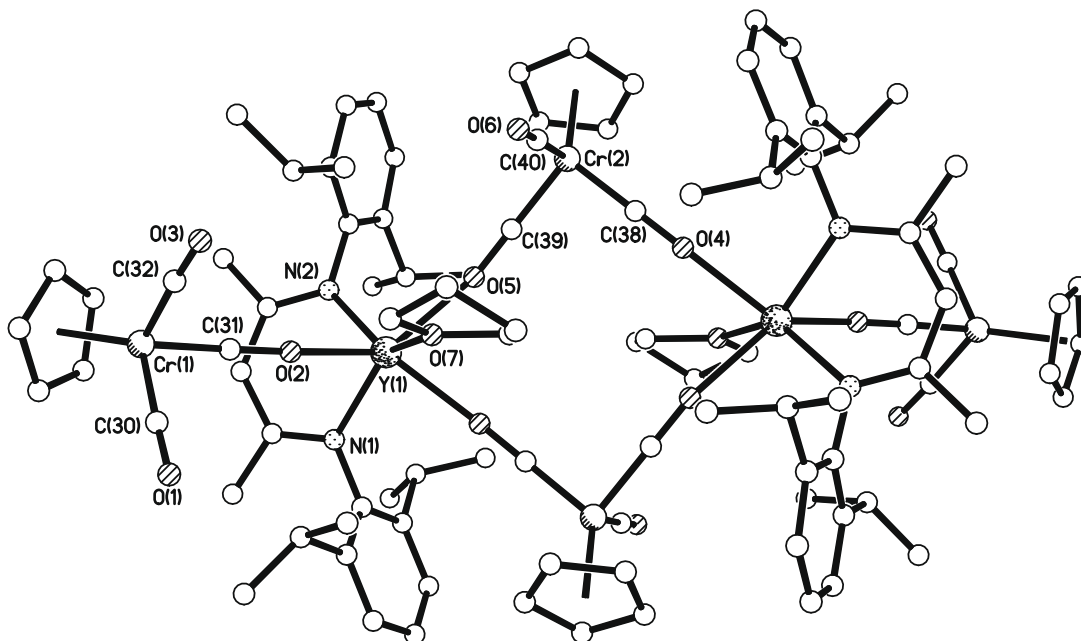
Table 1
Selected bond lengths (Å) and angles (°) for **2** and **3**.

2			
Y(1)–N(1)	2.320(3)	Y(1)–N(2)	2.313(3)
Y(1)–O(2)	2.190(2)	Y(1)–O(4A)	2.298(2)
Y(1)–O(5)	2.313(2)	Y(1)–O(7)	2.387(2)
Cr(1)–C(30)	1.812(5)	Cr(1)–C(31)	1.742(4)
Cr(1)–C(32)	1.815(5)	Cr(2)–C(38)	1.765(4)
Cr(2)–C(39)	1.770(4)	Cr(2)–C(40)	1.827(5)
Cr(1)–Cp$_{cent}$	1.856(4)	Cr(2)–Cp$_{cent}$	1.845(4)
O(1)–C(30)	1.170(5)	O(2)–C(31)	1.219(4)
O(3)–C(32)	1.161(5)	O(4)–C(38)	1.207(4)
O(5)–C(39)	1.207(4)	O(6)–C(40)	1.158(5)
Y(1)–O(2)–C(31)	169.8(3)	Y(1)–O(5)–C(39)	155.2(2)
Y(1)–O(4A)–C(38A)	158.5(3)		
3			
W(1)–C(6)	1.933(4)	W(1)–C(7)	1.935(4)
W(1)–C(8)	1.922(4)	O(1)–C(6)	1.175(5)
O(2)–C(7)	1.171(5)	O(3)–C(8)	1.186(5)
W(2)–C(14)	1.923(4)	W(2)–C(15)	1.930(5)
W(2)–C(16)	1.919(4)	O(4)–C(14)	1.177(5)
O(5)–C(15)	1.174(5)	O(6)–C(16)	1.181(5)
W(1)–Cp$_{cent}$	2.037(4)	W(2)–Cp$_{cent}$	2.053(4)
Na(1)–O(2)	2.478(3)	Na(1)–O(4A)	2.344(3)
Na(1)–O(6)	2.386(3)	Na(1)–O(7)	2.416(3)
Na(1)–O(8)	2.427(3)	Na(1)–O(9)	2.328(4)
Na(2)–O(2A)	2.477(3)	Na(2)–O(3)	2.313(3)
Na(2)–O(5A)	2.348(3)	Na(2)–O(7A)	2.463(3)
Na(2)–O(8A)	2.407(3)	Na(2)–O(10)	2.261(3)
Na(1)–O(2)–C(7)	128.7(3)	Na(1)–O(4A)–C(14A)	159.3(3)
Na(1)–O(6)–C(16)	141.0(3)	Na(2)–O(2A)–C(7A)	136.6(3)
Na(2)–O(3)–C(8)	157.5(3)	Na(2)–O(5A)–C(15A)	164.0(3)

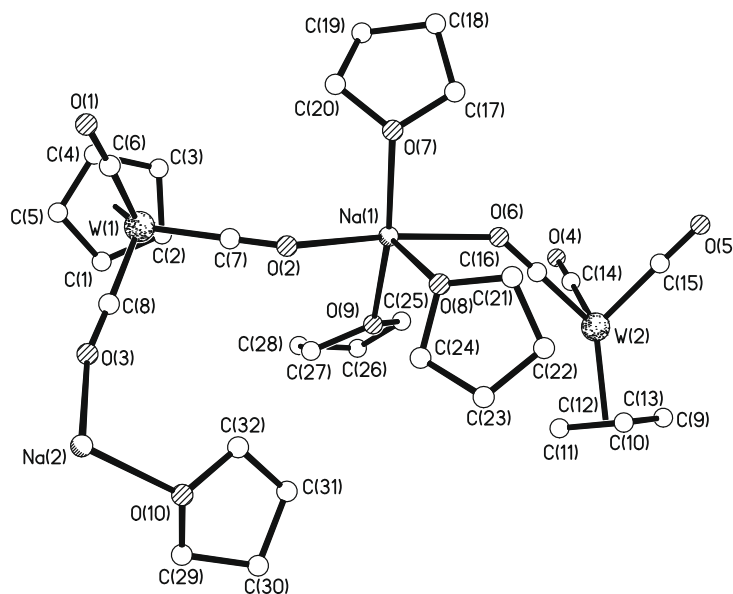**Fig. 1.** Molecular structure of **2**.

Fig. 2. Asymmetric unit of **3**.

shorter than the terminal carbonyl Cr(2)–C(40) distance of 1.827(5) Å. In each case, the shorter Cr–C distance in bridging isocarbonyl groups is consistent with increased back-bonding of chromium to carbon as a consequence of charge polarisation towards the electropositive yttrium centre. Also in line with this observation is the higher average Cr–C distance for the bridging isocarbonyls in the bridging [Cr(Cp)(CO)$_3$]$^-$ group (1.768 Å) compared to the bridging isocarbonyl group in the terminal [Cr(Cp)(CO)$_3$]$^-$ fragment (1.742 Å) since the charge is polarised via Cr–C back-bonding to two isocarbonyl groups in the former and only one in the latter. Furthermore, the expected elongation of the O–C$_{carbonyl}$ bond distances in the bridging isocarbonyls, as a result of increased Cr–C back-bonding, is evident in **2**. For example, whereas the bridging isocarbonyl groups exhibit O(2)–C(31), O(4)–C(38), and O(5)–C(39) bond distances of 1.219(4), 1.207(4), and 1.207(4) Å, respectively, the terminal carbonyl groups exhibit shorter O(3)–C(32), O(1)–C(30), and O(6)–C(40) distances of 1.170(5), 1.161(5), and 1.158(5) Å, respectively. The Y(1)–N(1) and Y(1)–N(2) bond lengths of 2.320(3) and 2.313(3) Å, respectively are longer than in **1** (cf. average of 2.278 Å) [8] which reflects the replacement of soft iodide ligands in **1** with the harder isocarbonyl groups in **2**.

We noted above that the Na[Cr(Cp)(CO)$_3$] complex reacted sluggishly with **1**. We have thus far been unable to isolate any tractable yttrium containing products from the corresponding reaction of Na[W(Cp)(CO)$_3$] [9] with **1**. However, from one reaction we isolated a small crop of colourless crystals. The crystals collapse to a pyrophoric powder rapidly when removed from their mother liquor and/or are dried under vacuum; however, we were able to transfer one crystal to an X-ray diffractometer and collect data from it, which revealed it to be [W(Cp)(CO)$_3$][Na(THF)$_2$] (**3**). However, spectroscopic data on the powdered form of **3** are identical to literature values for Na[W(Cp)(CO)$_3$] [1n,9] and we conclude the THF is loosely bound and thus easily removed, which accounts for the rapid degradation of crystals to a powder. Consequently, we do not report any characterisation data other than the single crystal X-ray diffraction structure.

The asymmetric unit of **3** is illustrated in Fig. 2. Two crystallographically independent [W(Cp)(CO)$_3$][Na(THF)$_2$] units are present in the asymmetric unit, which reflects the different coordination modes of the [W(Cp)(CO)$_3$]$^-$ anions in the solid state. The asymmetric unit 'dimerises' to form the basic building block of the

extended polymeric sheet structure which **3** exhibits in the solid state (Fig. 3). The structure is built around sodium dimers which are held together by two bridging THF molecules and a bridging isocarbonyl group. The coordination sphere of each sodium centre is further augmented by a terminal THF molecule, and two isocarbonyl groups. In each case, one isocarbonyl is part of a [W(Cp)(CO)$_3$]$^-$ unit which employs two isocarbonyl bridges and the other isocarbonyl is part of a triply bridging [W(Cp)(CO)$_3$]$^-$ unit. The latter bonding mode is noteworthy because, as noted above, formation of one isocarbonyl bridge renders the whole metallate fragment more electron poor and subsequent bridging to other metal centres is increasingly disfavoured; therefore, three isocarbonyl bridges is very rare [11]. In contrast to **2**, no concrete conclusions regarding the extent of W–C back-bonding can be gleaned from inspection of W–C and C–O bond lengths since the observed ranges (1.919–1.935 Å and 1.171–1.186 Å, respectively) are narrow and the differences between bridging and terminal W–C and C–O bond lengths are not statistically significant. This reflects the fact that sodium is softer than yttrium and consequently does not polarise the electron density in the [W(Cp)(CO)$_3$]$^-$ units as much as the observed polarisation of the [Cr(Cp)(CO)$_3$]$^-$ units in **2**.

3. Conclusions

We have prepared the new complex [{Y(BDI)[Cr(Cp)(CO)$_3$]$_2$ (THF)}$_2$] (**2**) which is dimeric in the solid state. Complex **2** exhibits two yttrium-BDI units which are linked by two bridging isocarbonyl groups in two [Cr(Cp)(CO)$_3$]$^-$ anions; a terminally bound, isocarbonyl-bridged [Cr(Cp)(CO)$_3$]$^-$ anion and THF molecule completes the coordination sphere at each yttrium. This results in formation of a centrosymmetric, 12-membered C$_4$O$_4$Cr$_2$Y$_2$ ring. A small crop of crystals of the complex [W(Cp)(CO)$_3$][Na(THF)$_2$] (**3**) were isolated, which was shown to form an extended polymeric sheet network with doubly and triply bridging [W(Cp)(CO)$_3$]$^-$ anions via isocarbonyl linkages. Complex **3** readily loses the coordinated THF and consequently all analytical data match that of the known, solvent free complex Na[W(Cp)(CO)$_3$]. We are currently investigating the synthesis of other lanthanide transition metal complexes.
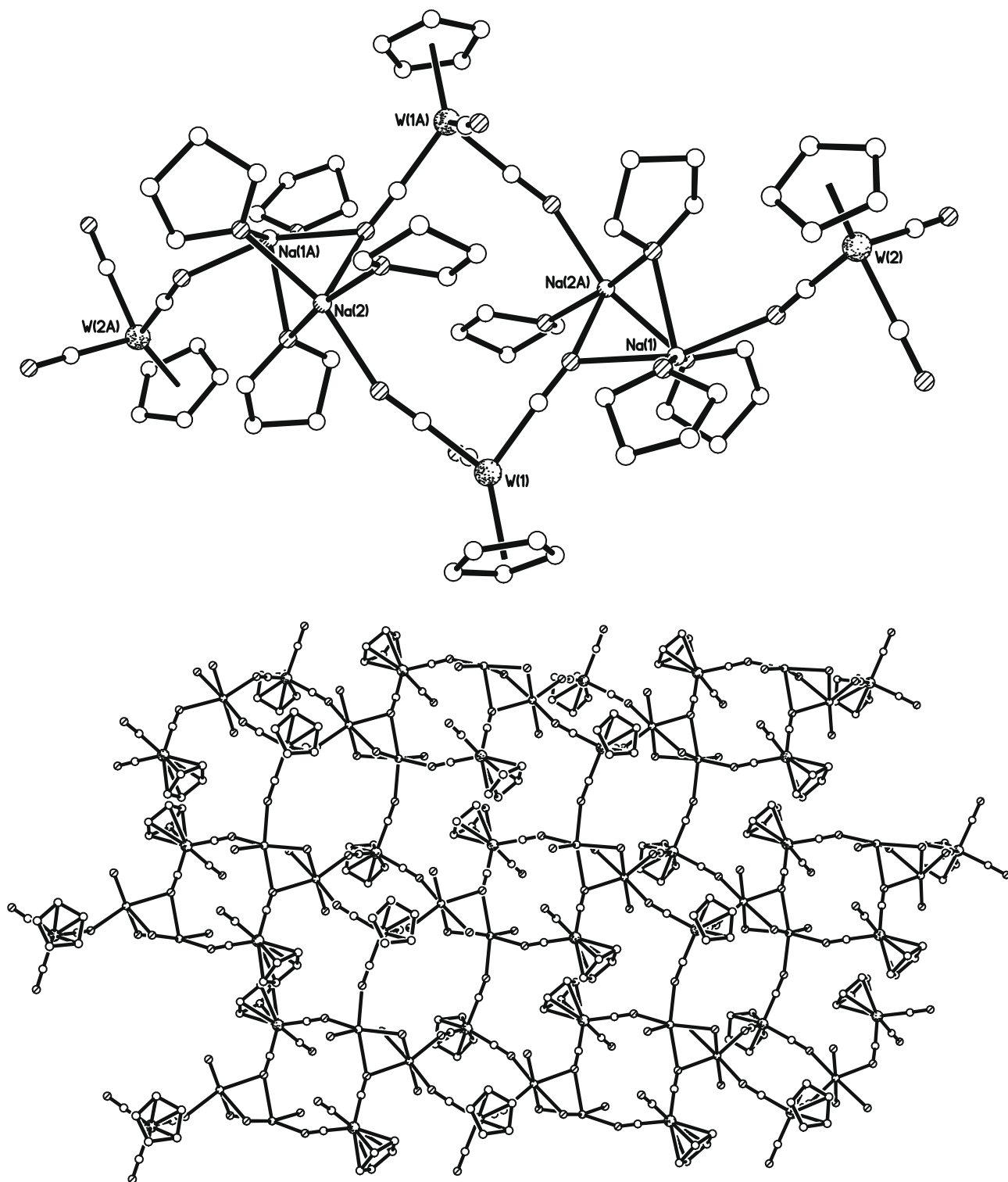
Fig. 3. 'Dimer' and extended polymeric network structures of **3**.

4. Experimental

4.1. General

All manipulations were carried out using standard Schlenk techniques, or an MBraun UniLab glovebox, under an atmosphere of dry nitrogen. Toluene and THF were dried by passage through activated alumina towers and degassed before use. Deuterated THF was distilled from potassium, degassed by three freeze-pump-thaw cycles and stored under nitrogen. The compounds **1** [8], Na[Cr(Cp)(CO)₃] [9], and Na[W(Cp)(CO)₃] [9] were prepared by published literature procedures. ¹H, and ¹³C{¹H} NMR spectra were recorded on a Bruker 400 spectrometer operating at 400.2 and 100.6 MHz, respectively, chemical shifts are quoted in parts

per million and are relative to TMS. FTIR spectra were recorded on a Bruker Tensor 27 spectrometer. CHN analyses were carried out by Mr. Stephen Boyer at London Metropolitan University.

4.2. Preparation of [{Y(BDI)[Cr(Cp)(CO)₃]₂(THF)}₂] (2)

THF (25 ml) was added to a cold (-78 °C) mixture of **1** (2.14 g, 2.60 mmol) and Na[Cr(Cp)(CO)$_3$] (1.05 g, 5.20 mmol). The mixture was allowed to warm to room temperature slowly with stirring over 18 h. The solution was refluxed for 1 h the cooled to room temperature. Volatiles were removed *in vacuo*. The product was extracted into toluene (10 ml), filtered, concentrated and stored at 5 °C for 5 days to afford yellow crystals of **2**. The crystals were isolated, washed with cold (0 °C) toluene (3×5 ml) and dried *in vacuo* to afford **2** as a yellow powder. Yield: 1.12 g, 46%. Anal. Calc. for $C_{39}H_{55}I_2N_2O_2P_2Si_2Y$: C, 60.00; H, 6.06; N, 2.86. Found: C, 59.09; H, 5.86; N, 2.83%. ^{1}H NMR (C_4D_8O, 300 K): δ 1.23 (d, $^3J_{HH}$ = 6.9 Hz, 24H, Pr^i-CH_3), 1.50 (d, $^3J_{HH}$ = 6.9 Hz, 24H, Pr^i-CH_3), 1.78 (m, 8H, CH_2-THF), 1.83 (s, 12H, CH_3), 3.30 (sept, $^3J_{HH}$ = 6.9 Hz, 8H, Pr^i-CH), 3.67 (m, 8H, OCH_2-THF), 4.59 (s, 20H, C_5H_5), 5.39 (s, 2H, CH), 7.34 (m, 12H, Ar-CH). ^{13}C{^{1}H} NMR (C_4D_8O, 300 K): δ 22.17 (Pr^i-CH$_3$), 22.49 (CH$_2$-THF), 22.75 (Pr^i-CH$_3$), 26.90 (Pr^i-CH), 64.90 (OCH$_2$-THF), 82.43 (C_5H_5), 97.21 (CH), 121.22 (*meta-C*), 125.35 (*para-C*), 141.54 (*ipso-C*), 144.55 (*ortho-C*), 166.20 (C=N), 242.0 (br, CO). IR ν/cm^{-1} (Nujol): 514.5 (m), 588.3 (w), 665.7 (w), 796.3 (w), 845.4 (w), 930.5 (w), 1007.7 (w), 1056.8 (w), 1254.5 (m), 1604.4 (s), 1677.2 (s), 1697.5 (s), 1839.1 (s), 1930.0 (s).

4.3. Isolation of [W(Cp)(CO)₃][Na(THF)₂] (3)

Colourless crystals of **3** were obtained from the reaction between two equivalents of Na[W(Cp)(CO)$_3$] and **1** carried out analogously to **2**. Crystals of **3** de-solvate rapidly and collapse to a powder when removed from their mother liquor and/or are dried *in vacuo*. The spectroscopic data of the powder match exactly that of Na[W(Cp)(CO)$_3$] [9].

4.4. Crystal structure determinations of 2

Data were collected at 150 K on a Bruker SMART Apex diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). Crystal data: $C_{112}H_{134}Cr_4N_4O_{14}Y_2$, M = 2146.05, yellow plate, crystal size $0.05 \times 0.23 \times 0.31$ mm, monoclinic, space group $P2_1/c$, a = 14.6682(9), b = 32.745(2), c = 14.3009(9) Å, β = 117.537(2)°, V = 6090.7(7) Å^3, Z = 2, μ = 1.341 mm^{-1}, R (F; $F^2 > 2\sigma$) = 0.0483, R_w (F^2; all data) = 0.127, S = 1.02 for 10675 unique, absorption corrected data and 560 parameters; final difference map extremes +0.58 and -0.50 e Å^{-3}. Programs: Bruker SMART (diffractometer control), Bruker SAINT (data integration), and Bruker SHELXTL (structure solution and refinement).

4.5. Crystal structure determinations of 3

Data were collected at 150 K on a Bruker SMART Apex diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). Crystal data: $C_{16}H_{21}NaO_5W$, M = 500.17, colourless tablet, crystal size $0.11 \times 0.29 \times 0.39$ mm, triclinic, space group $P\bar{1}$, a = 10.7966(6), b = 11.2563(6), c = 15.2166(8) Å, α = 103.717(2),

β = 103.122(2), γ 91.087(2)°, V = 6090.7(7) Å^3, Z = 4, μ = 6.667 mm^{-1}, R (F; $F^2 > 2\sigma$) = 0.0274, R_w (F^2; all data) = 0.0716, S = 1.03 for 7844 unique, absorption corrected data and 425 parameters; final difference map extremes +2.243 and -1.093 e Å^{-3}. Programs: Bruker SMART (diffractometer control), Bruker SAINT (data integration), and Bruker SHELXTL (structure solution and refinement).

Appendix A. Supplementary material

CCDC 712132 and 712133 contains the supplementary crystallographic data for **2** · 2toluene and **3**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.01.029.

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