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# Isonitrile substitution reactions of $Co_4(\mu_4\text{-}SiR)_2(CO)_{11}$ clusters and the structure of $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$

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## Abstract

The substitution of carbonyl ligands by isonitriles on $Co_4(\mu_4\text{-}SiC_6H_4R)_2(CO)_{11}$ (R = H, OMe, $NMe_2$) clusters has been examined. The reactions could be monitored by electrospray ionisation mass spectrometry, identifying the substituted products as either protonated $[M+H]^+$ ($R = NMe_2$) or oxidised $[M]^+$ (R = H, OMe) ions. Evidence for substitution of up to nine carbonyl ligands was found and the structure of tetra-substituted example, $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$, was determined.

*Keywords:* Cluster; Electrospray mass spectrometry; Isonitrile; Crystal structure determination

## 1. Introduction

The chemistry of pseudo-octahedral $E_2M_4$ clusters (e.g. $Co_4(\mu_4\text{-}SiR)_2(CO)_{11}$ (**1**) [R = Co(CO)$_4$, Me, Ph]) [1,2] has generated significant interest, attributable in part to the catalytic [3] and electrochemical properties [4] displayed by these materials. The clusters contain a $M_4$ plane, capped on both faces by a quadruply-bridging main group element (either alone or as part of a ligand). The clusters typically contain Group 8 or 9 transition metal elements, with Group 14, 15 or 16 main group elements in the capping positions. Examples include $Fe_4(\mu_4\text{-}Se)_2(CO)_{11}$ [5], $Co_4(\mu_4\text{-}AsPh)_2(CO)_{10}$ [6] and $Co_4(\mu_4\text{-}GeR)_2(CO)_{11}$ [R = Co(CO)$_4$, Me] [7,8].

The clusters usually possess eight SEP (skeletal electron pairs) rather than the seven SEP expected from electron counting procedures [9]. Despite the higher than expected value, few $E_2M_4$ clusters display facile ligand loss. $Fe_4(\mu_4\text{-}PR)_2(CO)_x$ ($x = 11$, 12; $R = {}^t$Bu, Ph, tolyl) clusters are an exception, with both seven and eight SEP forms readily available [10].

Although ligand loss is rare, reports of carbonyl substitution by phosphine, phosphite, or isonitrile ligands are well documented. Electrochemical analysis of the phosphine- and phosphite-substituted derivatives of $Co_4(\mu_4\text{-}PPh_2)_2(CO)_{10}$ has been reported [4], with the phosphine-substituted derivatives displaying improved catalytic activity towards hydroformylation of olefins [3]. The substitution of carbonyl ligands by isonitriles and phosphites has been reported for $Fe_4(\mu_4\text{-}PR_2)_2(CO)_{11}$ (R = Ph, tolyl), with the use of difunctionalised ligands allowing linking of the pseudo-octahedral cluster cores [10–12]. The maximum degree of substitution reported to date is the tetrakis-phosphite cluster $Co_4(\mu_4\text{-}PPh)_2(CO)_6[P(OMe)_3]_4$ (**2**) where the phosphite ligands were shown to be evenly distributed over the cobalt atoms [13]. No isonitrile derivatives of $E_2M_4$ clusters have been structurally characterised.

We report herein ligand-substitution of $Co_4(\mu_4\text{-}SiC_6H_4R)_2(CO)_{11}$ (R = H, OMe, $NMe_2$) clusters using electrospray ionisation mass spectrometry (ESI-MS) to monitor the course of the reaction. The structure of one of the products, $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$ (**3**), was established by X-ray crystallography.

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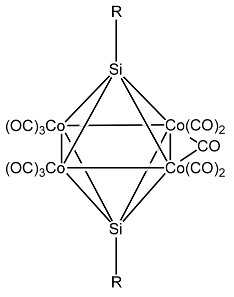
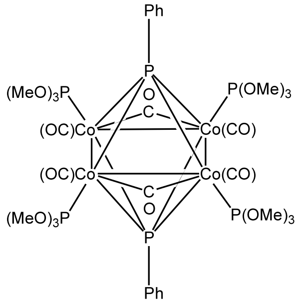


**(1)**



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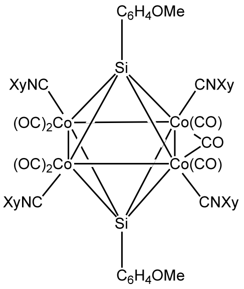

**(3)**

## 2. Results and discussion

### 2.1. Reactions

Treatment of a dichloromethane solution of $Co_4(\mu_4\text{-}SiC_6H_4NMe_2)_2(CO)_{11}$ with an excess of $^t$BuNC or XyNC resulted in facile substitution of the carbonyl ligands at slightly elevated temperatures (30–35 °C). The formation of the substituted derivatives was monitored by ESI-MS and (less effectively) IR spectroscopy (through analysis of the carbonyl stretching frequencies).

The substitution reactions were initially examined using ESI-MS to follow the course of the reaction. As reported earlier [2], the presence of the basic dimethylamino functionality assists protonation of $Co_4(\mu_4\text{-}SiC_6H_4NMe_2)_2(CO)_{11}$ in solution, allowing detection of the parent cluster as a $[M+H]^+$ ion (the technique of incorporating basic functionalities onto ligands to assist protonation and hence ESI-MS detection has been exploited previously [14]). Likewise, any substituted derivatives of this cluster would also be amenable to protonation allowing the use of ESI-MS to monitor reactions of this cluster in a similar (though more informative) manner to IR monitoring of the carbonyl stretching frequencies.

ESI-MS analysis of a crude reaction solution containing $Co_4(\mu_4\text{-}SiC_6H_4NMe_2)_2(CO)_{11}$ and $^t$BuNC indicated facile substitution of the carbonyl ligands over a 5-h period, with $[M+H]^+$ ions identified for all clusters in the series $Co_4(\mu_4\text{-}SiC_6H_4NMe_2)_2(CO)_{11-n}(^tBuNC)_n$ ($n=0$–7) as summarised in Table 1. Equivalent treatment of the parent clusters with XyNC (Xy = xylyl, 2,6 dimethylphenyl) afforded a higher degree of substitution, with a maximum of nine ligands replaced (Fig. 1). In each case, the major product of the reaction (as determined by the relative intensity of the ESI-MS

Table 1
ESI-MS data for $Co_4(\mu_4\text{-}SiC_6H_4R)_2(CO)_{11-n}(R'NC)_n$ clusters

| $n$ [a] | R = H [b] | | R = OMe [b] | | R = NMe$_2$ [c] | |
|---|---|---|---|---|---|---|
| | R′ = $^t$Bu | R′ = Xy | R′ = $^t$Bu | R′ = Xy | R′ = $^t$Bu | R′ = Xy |
| 1 | – | – | – | – | 896 ( < 1) | 944 (4) |
| 2 | – | – | – | – | 951 ( < 1) | 1047 (10) |
| 3 | 919 ( < 1) | 1063 ( < 1) | 979 ( < 1) | 1123 (3) | 1006 (2) | 1150 (28) |
| 4 | 974 (4) | 1166 (2) | 1034 (3) | 1226 (10) | 1051 (4) | 1253 (36) |
| 5 | 1029 (12) | 1269 (8) | 1089 (10) | 1329 (8) | 1116 (52) | 1356 (48) |
| 6 | 1084 (100) | 1372 (100) | 1144 (100) | 1432 (100) | 1171 (100) | 1459 (100) |
| 7 | 1139 (6) | 1475 (32) | 1199 (5) | 1535 (50) | 1226 (5) | 1562 (13) |
| 8 | – | 1578 (8) | – | 1638 (2) | – | 1665 (2) |
| 9 | – | 1682 (4) | – | 1742 ( < 1) | – | 1769 ( < 1) |

*m*/*z* values (% intensity).
[a] $n$ = degree of carbonyl replacement.
[b] $[M]^+$ ions arising from oxidation in the ESI-MS.
[c] $[M+H]^+$ ions arising from protonation of the NMe$_2$ group.

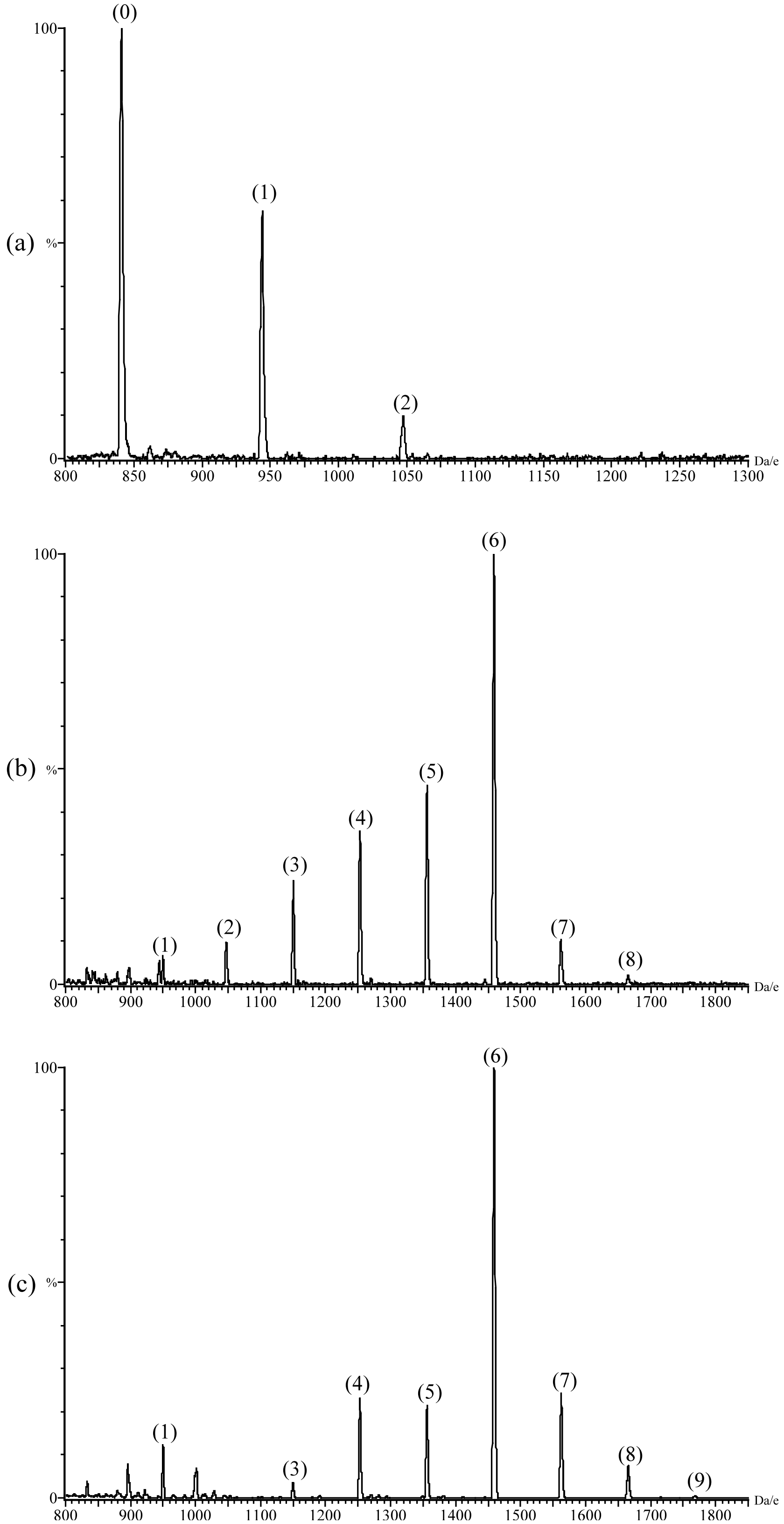

Fig. 1. Electrospray ionisation mass spectra of the xylylisonitrile-substituted derivatives, $Co_4(\mu_4\text{-}SiC_6H_4NMe_2)_2(CO)_{11-n}(XyNC)_n$ after (a) 1 h, (b) 3 h and (c) 5 h. All signals correspond to $[M+H]^+$ ions and are labelled with respect to the degree of substitution.

signals) corresponded to the hexa-substituted derivative. As a general observation, the overall intensity of the spectra increased with the degree of substitution experienced.

Subsequent to reactions of the $Co_4(\mu_4\text{-}SiC_6H_4R)_2(CO)_{11}$ ($R = NMe_2$) cluster, equivalent treatment of the R = H, OMe analogues was examined. The absence of the dimethylamino functionality hampers chemical ionisation of these analogues in solution (hence hindering ESI-MS detection) but the clusters are available in higher yield than the $R = NMe_2$ variant [2]. Although un-, mono- and di-substituted clusters were absent from the resultant spectra, signals corresponding to oxidised ions, $[M]^+$, of the more highly substituted derivatives were readily observed. As summarised in Table 1, after treatment with $^t$BuNC, signals associated with all clusters in the series $Co_4(\mu_4\text{-}SiC_6H_4R)_2(CO)_{11-n}(RNC)_n$ ($n = 3$–7; R = H, OMe) were observed, while equivalent reactions involving XyNC provided evidence for a higher degree of substitution ($n = 3$–9).

The oxidation of the substituted clusters from the neutral parent molecules is considered to occur within the spectrometer at the tip of the metal capillary. Equivalent *in situ* oxidation has been reported for metallocenes [15] and fullerenes [16], with a low redox potential common to all identified species.

The change in ESI-MS character of the clusters through substitution is attributed to the electronic character of the isonitrile ligand. Isonitriles are stronger σ-donors (poorer π-acceptors) than carbonyls, with substitution effectively increasing the electron-rich character and accentuating the clusters propensity towards oxidation. An equivalent effect has been noted in electrochemical studies of isonitrile-substituted $Fe_4(\mu_4\text{-}PPh)_2(CO)_{11}$ clusters, with the parent clusters displaying higher reductive potentials than the substituted derivatives [12]. Similarly, phosphine- and phosphite-substituted derivatives of $Co_4(\mu_4\text{-}PR)_2(CO)_{10}$ are more readily oxidised than the parent carbonyl cluster [4].

The substitution of carbonyl ligands by isonitriles was also monitored by IR spectroscopy. The signals observed were typically broad and became less distinct with increasing substitution. Despite the limited resolution, trends in the spectra were apparent. The parent clusters display a characteristic series of signals within the carbonyl region of the spectrum [ca. 2040 (s), 2030 (m,sh), 2010 (m,sh), 1850 (w,br)] [2]. Treatment with the isonitrile introduced an additional signal (ca. 2150 $cm^{-1}$) attributable to $\nu_{C-N}$. The isonitrile-substituted derivatives retained the IR pattern of the parent cluster although shifted to lower wavenumber (a shift of ca. 10 $cm^{-1}$ was observed for the mono- and di-substituted derivatives compared to ca. 40 $cm^{-1}$ for the penta- and hexa-substituted products). Likewise, the signal attributed to the isonitrile functionality shifted to lower wavenumber as the degree of substitution increased (ca. 2125 $cm^{-1}$ in the penta- and hexa-substituted derivatives). A similar effect has been noted in the IR spectra of the $Fe_4(\mu_4\text{-}PPh_2)_2(CO)_{11-n}(RNC)_n$ ($n = 1, 2$; $R = {}^t$Bu, tolyl) clusters [11].

The mechanism of substitution is presumed to be similar to that reported [13] for phosphite-substitution on the isoelectronic $Co_4(\mu_4\text{-}PPh)_2(CO)_{10}$ cluster, i.e. ligand addition accompanied by Co–Co bond scission for the mono- and di-substituted products as compared to carbonyl loss, ligand scrambling and subsequent ligand addition for the tri- and tetra-substituted products.

Since mixtures of products were always obtained, full characterisation was not possible, except in one case where fractional crystallisation gave a pure sample of the $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$ example from a mixture which also contained the penta- and hexa-substituted variants. The substituted clusters were not amenable to separation using standard chromatographic techniques, with a degree of air-sensitivity noted. The air-sensitivity was not unexpected since (as shown by ESI-MS analysis) the substituted clusters show a significantly higher propensity towards oxidation than the parent cluster.

This study has shown that ESI-MS is a useful technique for monitoring ligand-substitution reactions of clusters, especially for non-specific reactions where mixtures of products are formed. The ability to sample directly from reaction solutions and the observation of parent ions without fragmentation makes interpretation straightforward. The only proviso is that signal intensity will only generally correlate with the abundance of species (since different ionisation efficiencies will be involved).

### 2.2. Structure

$Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$ crystallised in the *C*2/*c* space group on a twofold rotation axis [coincident with the C(2)–O(2) bond] with one half of the molecule unique. The structure of the molecule is shown in Fig. 2 with relevant bond lengths and angles provided in Table 2.

The isonitrile ligands are located on each of the four cobalt atoms, arranged in a syn/syn/anti/anti configuration. This arrangement allows for maximum interligand separation, with a *trans* configuration of the isonitrile ligands noted across the carbonyl bridged Co–Co bond. A similar arrangement was reported for $Co_4(\mu_4\text{-}PPh)_2(CO)_6[P(OMe)_3]_4$, although in this case the ligand configuration was *cis* across the bridged Co–Co bonds [13].

The $Co_4$ square is distorted by the presence of the bridging [CO(2)] and semi-bridging [CO(4)] ligands which lie within the $Co_4$ plane. A similar distortion is

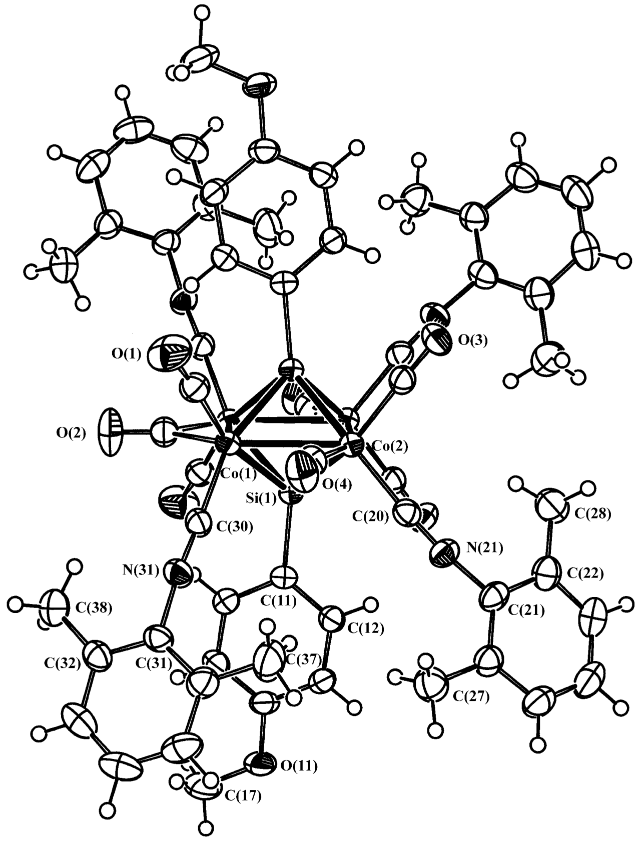

Fig. 2. Labelled diagram of $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$ (**3**). Ellipsoids are drawn at the 50% probability level and hydrogen atoms presented as arbitrary spheres.

Table 2
Selected bond lengths (Å) and angles (°) for $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$

| | | | |
|---|---|---|---|
| *Bond lengths* | | | |
| Co(1)–Co(2) | 2.6624(5) | Co(1)–Co(1)′ | 2.5594(6) |
| Co(2)–Co(2)′ | 2.7468(7) | Si(1)–Co(1) | 2.3039(7) |
| Si(1)–Co(1)′ | 2.3284(7) | Si(1)–Co(2) | 2.3087(7) |
| Si(1)–Co(2)′ | 2.3208(7) | Co(1)–C(2) | 1.905(3) |
| C(2)–O(2) | 1.178(5) | Co(2)–C(4) | 1.783(3) |
| C(4)–O(4) | 1.150(3) | $Co–C_{CO}$ | 1.790–1.792 |
| C–O | 1.139–1.143 | $Co–C_{XyNC}$ | 1.858–1.865 |
| N–C | 1.167–1.168 | | |
| *Bond angles* | | | |
| Co(1)–C(2)–O(2) | 137.79(8) | Co(1)–C(2)–Co(1) | 84.41(16) |
| Co(2)–C(4)–O(4) | 163.1(3) | Co–C–O | 176.1–177.9 |

′ Symmetry related atom.

noted in all $Co_4(\mu_4\text{-}ER)_2(CO)_{11}$ structures, and the Co–Co bond distances [2.5594(6), 2.6624(5) and 2.7468(7) Å for the bridged, semi-bridged and unbridged bonds respectively] are similar to those reported [2] for $Co_4(\mu_4\text{-}SiPh)_2(CO)_{11}$ [2.566, 2.647, 2.684 and 2.725 Å respectively]. The non-bonded Si(1)–Si(1)′ distance [2.7036(13) Å] is comparable to that reported for $Co_4(\mu_4\text{-}SiPh)_2(CO)_{11}$ [2.705 Å]. The semi-bridging character of the CO(4) ligand is apparent from the C–O bond distance and Co–C–O angle, which are intermediate between those found for the fully-bridged and terminal ligands.

Although $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$ is a crowded molecule, the bond parameters associated with the isonitrile ligands are similar to those found for other substituted clusters. The C–N–C–Co arrangements are approximately linear (171–178°) while the N–C bond distances [1.168(3) and 1.167(3) Å for N(21)–C(20) and N(31)–C(30) respectively] are typical of a coordinated isonitrile. The XyNC planes bisect the $Co_4$ plane at an average angle of 62.01°.

There are no obvious analogues to this structure reported, with the closest potential examples being $Fe_4(\mu_4\text{-}PPh)_2(CO)_{11-n}(^tBuNC)_n$ ($n = 1, 2$), neither of which was structurally characterised, while the more highly-substituted $Fe_4(\mu_4\text{-}PPh)_2(CO)_8(^tBuNC)_3$ cluster was unable to be isolated in a pure form [10].

## 3. Experimental

All reactions were carried out under an inert atmosphere (either argon or nitrogen) using standard Schlenk techniques. Dichloromethane and petroleum spirits (60–80 °C fraction) were freshly distilled over calcium hydride under a nitrogen atmosphere. $Co_4(\mu_4\text{-}SiPh)_2(CO)_{11}$, $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_{11}$, $Co_4(\mu_4\text{-}SiC_6H_4NMe_2)_2(CO)_{11}$ [2], XyNC [17], and $^t$BuNC [17] were prepared according to published methods.

ESI-MS spectra were recorded on a VG platform II instrument in positive-ion mode using methanol as the mobile phase. Samples of the reaction solution were diluted in methanol immediately prior to injection. Spectra were acquired with a 10 V potential-difference across the skimmer cones. Observed isotope patterns recorded under high-resolution conditions were compared with those theoretically calculated by the Isotope program [18]. Stated $m/z$ values correspond to the most intense peak in the isotope pattern.

IR spectra of solutions were acquired on a Digilab FTS-40 instrument.

### 3.1. Synthesis of isonitrile-substituted $Co_4(\mu_4\text{-}SiC_6H_4R)_2(CO)_{11}$ clusters

To a dichloromethane solution of the appropriate $Co_4(\mu_4\text{-}SiC_6H_4R)_2(CO)_{11}$ (R = H, OMe, $NMe_2$) cluster (0.05 g, ca. 0.06 mmol) was added a tenfold excess of the isonitrile (0.6 mmol) either directly or in a dichloromethane solution. The solution was heated to 30–35 °C for ca. 5 h, with the composition of the solution monitored by ESI-MS. Details relating to ESI-MS signals observed and their relative intensities are summarised in Table 1. Analysis indicated that the hexa-substituted derivative was the major product in each reaction, though $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_{17}(XyNC)_4$ was the only product isolated by fractional crystallisation as discussed below.

### 3.2. *Isolation of* $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$

To a dichloromethane solution of $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_{11}$ (0.05 g, 0.06 mmol) was added an excess of XyNC (0.047 g, 0.36 mmol) and the mixture heated to 35 °C for 4 h, at which point ESI-MS analysis of the solution suggested the main components were the penta- and hexa-substituted products (*m*/*z* 1329 and 1432 respectively). Solvent was removed in vacuo and the crude material dissolved in a 4:1 petroleum spirits:toluene solution. Cooling of the mixed solution to −20 °C afforded a deep red crystalline product determined to be $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$ by X-ray crystallography.

### 3.3. *X-ray structure determination of* $Co_4(\mu_4\text{-}SiC_6H_4OMe)_2(CO)_7(XyNC)_4$

Crystal structure data were obtained using a Siemens SMART CCD diffractometer with Mo–$K_\alpha$ X-radiation ($\lambda = 0.71073$ Å).

*Crystal data:* $C_{57}Co_4H_{50}N_4O_9Si_2$, $M_r = 1226.91$, monoclinic, space group $C2/c$, $a = 21.5814(4)$, $b = 15.3852(3)$, $c = 17.4610(2)$ Å, $\beta = 110.106(1)^\circ$, $U = 5444.33(16)$ Å$^3$, $Z = 4$, $D_c = 1.497$ g cm$^{-3}$, μ(Mo–$K_\alpha$) = 1.302 mm$^{-1}$, $F(000) = 2512$, $T = 203$ K, crystal size = 0.35 × 0.30 × 0.18 mm$^3$.

A total of 14736 reflections were collected to $\theta = 25.38^\circ$, 4939 of which were unique ($R_{int} = 0.0215$). Reflections were corrected for absorption using SADABS [19] ($T_{max,\ min}$ 0.9152, 0.8075). The structure was solved and refined on $F^2$. Hydrogen atoms were placed in calculated positions ($U_{iso}$ 1.2 times that of the atom to which they were attached) using a riding model. Refinement converged to $R_1 = 0.0336$ [for $I > 2\sigma(I)$ data] $wR_2 = 0.0912$ for all data with GoF = 1.052.

All calculations used the SHELX-97 programs [20] manipulated through WinGx [21].

## 4. Supplementary material

Full crystallographic data have been deposited at the Cambridge Crystallographic Data Centre, CCDC reference number 195758. Copies of this information may be obtained free of charge on application to The Director, CCDC, 12 Union Rd, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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## References

[1] (a) M.L. Van Tiel, K.M. Mackay, B.K. Nicholson, J. Organomet. Chem. 326 (1987) C101;
(b) S.G. Anema, S.K. Lee, K.M. Mackay, B.K. Nicholson, J. Organomet. Chem. 444 (1993) 211.

[2] B.K. Nicholson, C. Evans, J.S. McIndoe, M.L. Van Tiel, G.J. Harfoot, K.M. Mackay, C.J. McAdam, B.H. Robinson, J. Chem. Soc. Dalton Trans., in press.

[3] (a) C.U. Pittman, G.M. Wileman, W.D. Wilson, R.C. Ryan, Angew. Chem. Int. Ed. Engl. 19 (1980) 478;
(b) R.C. Ryan, C.U. Pittman, J.P. O'Connor, J. Am. Chem. Soc. 99 (1977) 1986.

[4] M.G. Richmond, J.K. Kochi, Inorg. Chem. 25 (1986) 656.

[5] P. Mathur, M.M. Hossain, R.S. Rashid, J. Organomet. Chem. 467 (1994) 245.

[6] R.M. De Silva, M.J. Mays, J.E. Davies, P.R. Raithby, M.A. Rennie, G.P. Shields, J. Chem. Soc. Dalton Trans. (1998) 439.

[7] S.P. Foster, K.M. Mackay, B.K. Nicholson, Inorg. Chem. 24 (1985) 909.

[8] (a) S.P. Foster, K.M. Mackay, B.K. Nicholson, Chem. Commun. (1982) 1156;
(b) S.G. Anema, S.K. Lee, K.M. Mackay, L.C. McLeod, B.K. Nicholson, M. Service, J. Chem. Soc. Dalton Trans. (1991) 1209.

[9] J.-F. Halet, Coord. Chem. Rev. 143 (1995) 637.

[10] J.T. Jaeger, S. Aime, H. Vahrenkamp, Organometallics 5 (1986) 245.

[11] J.T. Jaeger, H. Vahrenkamp, Organometallics 7 (1988) 1746.

[12] J.T. Jaeger, J.S. Field, D. Collison, G.P. Speck, B.M. Peake, J. Hähnle, H. Vahrenkamp, Organometallics 7 (1988) 1753.

[13] M.G. Richmond, J.K. Kochi, Inorg. Chem. 25 (1986) 1334.

[14] C. Decker, W. Henderson, B.K. Nicholson, J. Chem. Soc. Dalton Trans. (1999) 3507.

[15] X. Xu, S.P. Nolan, R.B. Cole, Anal. Chem. 66 (1994) 119.

[16] (a) A. Dupont, J.-P. Gisselbrecht, E. Leize, L. Wagner, A. Van Dorsselaer, Tet. Lett. 35 (1994) 6083;
(b) T.-Y. Liu, L.-L. Shiu, T.-Y. Luh, G.-R. Her, Rapid Commun. Mass Spectrom. 9 (1995) 604.

[17] G.W. Gokel, R.P. Widera, W.P. Weber, Org. Synth. 55 (1976) 98.

[18] L.J. Arnold, J. Chem. Educ. 69 (1992) 811.

[19] R.H. Blessing, Acta Crystallogr. A51 (1995) 33.

[20] G.M. Sheldrick, SHELX-97, Programs for the solution and refinement of crystal structures, University of Göttingen, Göttingen, Germany, 1997.

[21] L.J. Farrugia, J. Appl. Cryst. 32 (1999) 837.