

Supporting Information for:

Synthesis of arsenic-rich As_n ligand complexes from yellow arsenic

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General Procedures and Starting Materials

All reactions were performed under an atmosphere of dry argon with standard vacuum, Schlenk and glove box techniques. Solvents were purified and degassed by standard procedures. Mass spectra were recorded on a Finnigan MAT SSQ 710 A mass spectrometer. NMR spectra were recorded at 25°C on a Bruker Avance 400 [¹H: 400.132 MHz standard tetramethylsilane, ¹³C: 100.627 MHz standard tetramethylsilane] or on a Bruker Avance 300 [¹H: 300.130 MHz standard tetramethylsilane, ¹³C: 75.468 MHz standard tetramethylsilane]. The chemical shifts are reported relative to tetramethylsilane (¹H, ¹³C).

Experimental Section

Synthesis of **1** at 70°C

Freshly prepared As₄¹ (excess) solution (300 mL) in toluene was kept at 70°C and [$\{\eta^5\text{-Cp}^*\text{Co}\}_2\text{-}\mu\text{-}\{\eta^{4:4}\text{-toluene}\}\}^2$ (0.560 g, 0.83 mmol) was added in toluene (50 mL) in the darkness. The solution was stirred for two hours at 70°C yielding a green solution. The solvent was removed completely under vacuum. The residue was extracted with dichloromethane and filtrated over diatomaceous earth. **1** was yielded as green powder (600 mg, 0.68mmol, 82%).

Synthesis of **1**, **2** and **3** at room temperature

Freshly prepared As₄¹ (excess) solution (300 mL) in toluene was cooled down to room temperature and [$\{\eta^5\text{-Cp}^*\text{Co}\}_2\text{-}\mu\text{-}\{\eta^{4:4}\text{-toluene}\}\}^2$ (0.350 g, 0.5 mmol) was added in toluene (50 mL) in the darkness. The solution was stirred for two hours at room temperature. The solvent was removed completely under vacuum. The residue was extracted with 10 mL of dichloromethane, 2g of silica was added and the solvent was evaporated from the mixture in vacuum until it was free-flowing. Column chromatography (column: 25 x 3 cm, silica, temperature: -20°C) with hexane/toluene (9:1) a green fraction (240 mg, 54%) of **1** followed by a brown zone containing 120 mg (14%) of **3**. Finally elution of a brown zone with tetrahydrofuran yielded a fraction from which 77 mg (8 %) of **2** was isolated.

Analytical Data for **1**:

¹H-NMR (300 MHz, CD₂Cl₂, 300K): 1.29 (s, 9 H, C(CH₃)₃), 1.42 (s, 18 H, C(CH₃)₃), 4.25 (s, 2 H, CH);

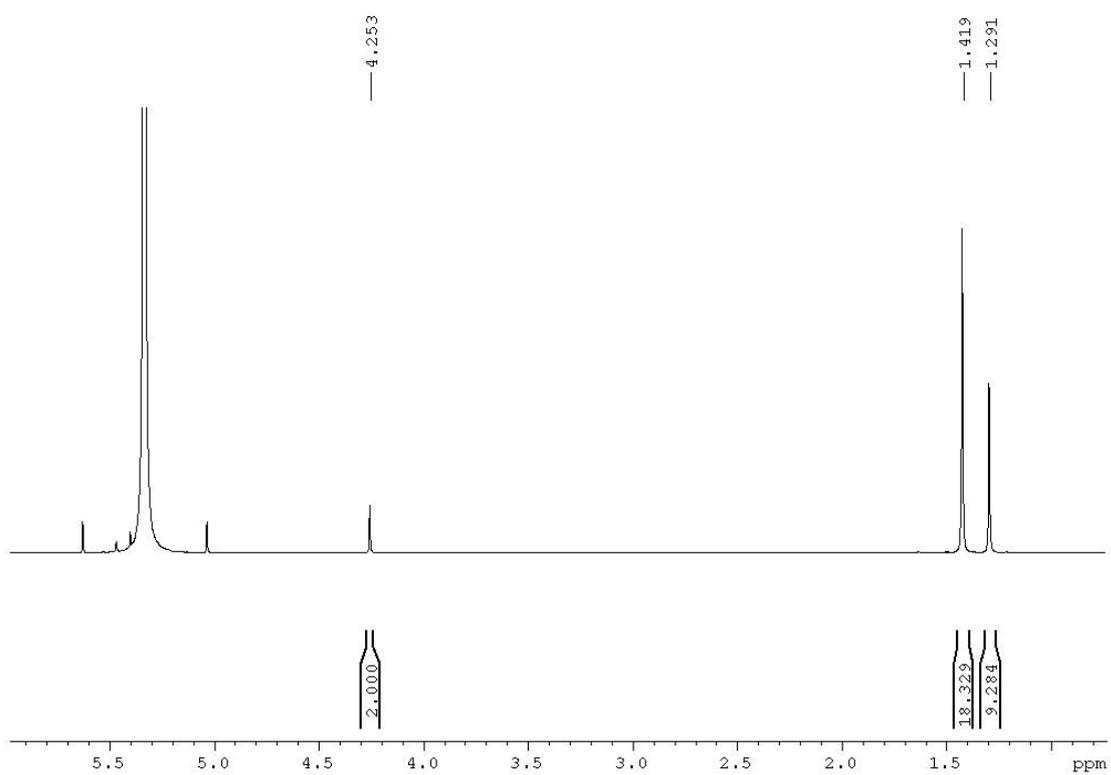


Figure S1: ¹H-NMR of **1** in CD₂Cl₂.

¹³C{¹H}-NMR (300 MHz, CD₂Cl₂, 300K): 31.8 (s, C(CH₃)₃), 32.1 (s, CH₃), 33.8 (s, C(CH₃)₃), 34.4 (s, CH₃), 79.2 (s, C(CH₃)₃), 106.3 (s, CH), 107.6 (s, C(CH₃)₃);

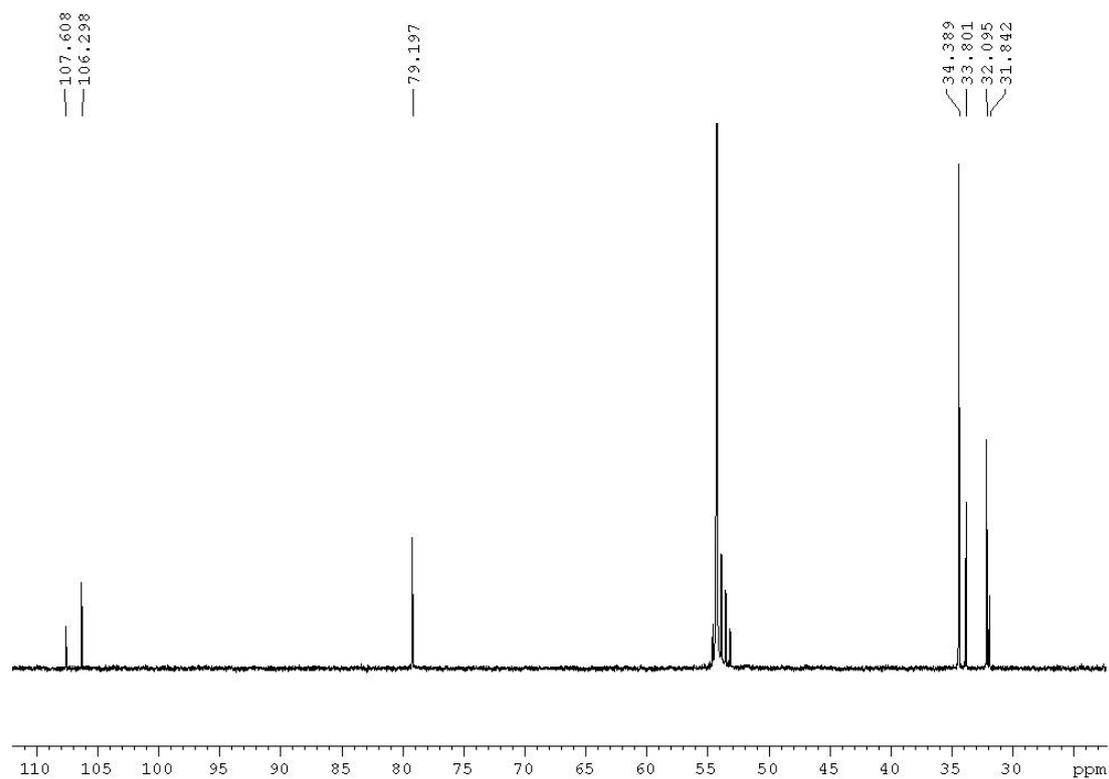


Figure S2: ^{13}C -NMR of **1** in CD_2Cl_2 .

MS (FDI): m/z (%): 883.6 (100) [M^+]; **Calculated (%) for $\text{C}_{34}\text{H}_{58}\text{Co}_2\text{As}_4$** (884.01 g/mol) C 46.18, H 6.61; found: C 45.96, H 6.92.

Analytical Data for 2:

^1H NMR (300 MHz, CD_2Cl_2 , 300K): 1.05 (br s, 9 H, $\text{C}(\text{CH}_3)_3$), 1.12 (br s, 9 H, $\text{C}(\text{CH}_3)_3$), 1.25 (br s, 18 H, $\text{C}(\text{CH}_3)_3$), 1.43 (br s, 18 H, $\text{C}(\text{CH}_3)_3$), 4.25 (s, 1 H, CH), 4.28 (s, 2 H, CH), 4.30 (s, 2 H, CH), 5.03 (br s, 2 H, CH);

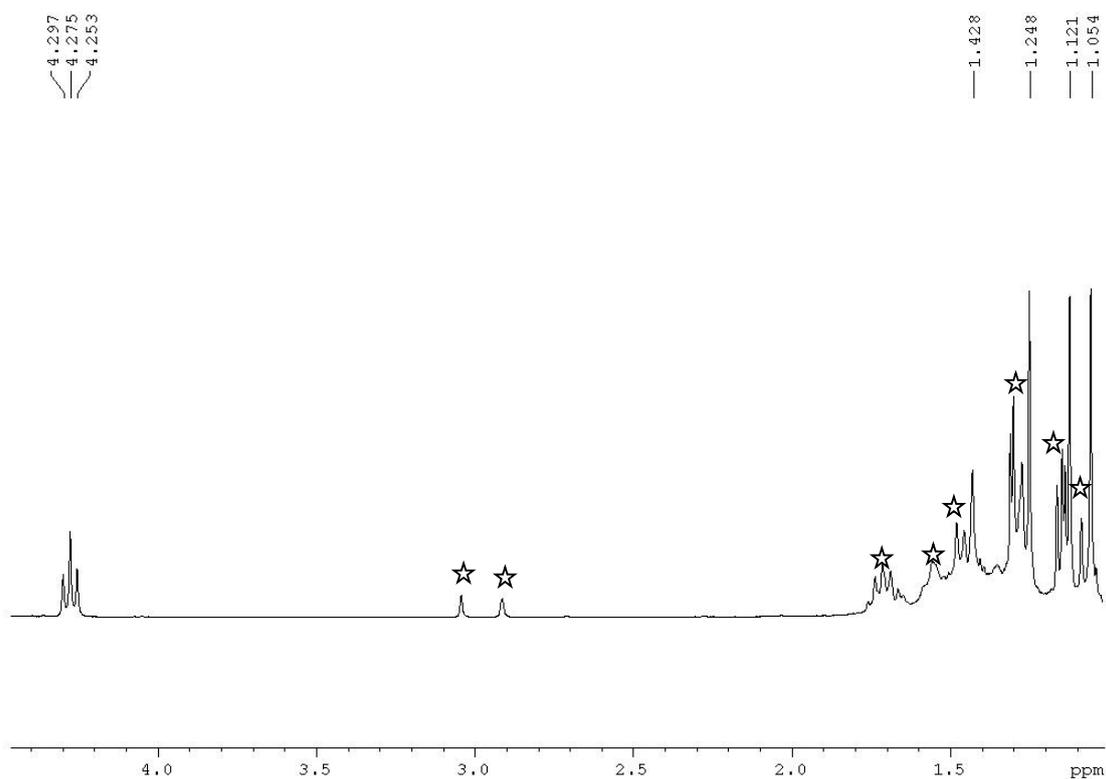


Figure S3: $^1\text{H-NMR}$ of **2** in CD_2Cl_2 . Signals marked with a star are impurities which cannot be removed through column chromatographic workup.

MS (FDI): m/z (%): 1918.4 (100) $[\text{M}^+]$, 1326.3 (100) $[\text{M}^+-\text{Cp}'''\text{CoAs}_4]$, 884.2 (100) $[\text{M}^+(\text{Cp}'''\text{Co})_2\text{As}_6]$;

Calculated (%) for $\text{C}_{68}\text{H}_{116}\text{Co}_4\text{As}_{10}$ (1917.86 g/mol) C 42.57, H 6.09; found: C 42.73, H 6.22.

Analytical Data for 3:

$^1\text{H-NMR}$ (400 MHz, CD_2Cl_2 , 300K): 1.07 (s, 9 H, $\text{C}(\text{CH}_3)_3$), 1.30 (s, 9 H, $\text{C}(\text{CH}_3)_3$), 1.39 (s, 18 H, $\text{C}(\text{CH}_3)_3$), 1.42 (s, 9 H, $\text{C}(\text{CH}_3)_3$), 1.43 (s, 18 H, $\text{C}(\text{CH}_3)_3$), 1.55 (s, 18 H, $\text{C}(\text{CH}_3)_3$), 4.36 (s, 2 H, CH), 4.80 (s, 2 H, CH), 5.03 (s br, 2H, CH);

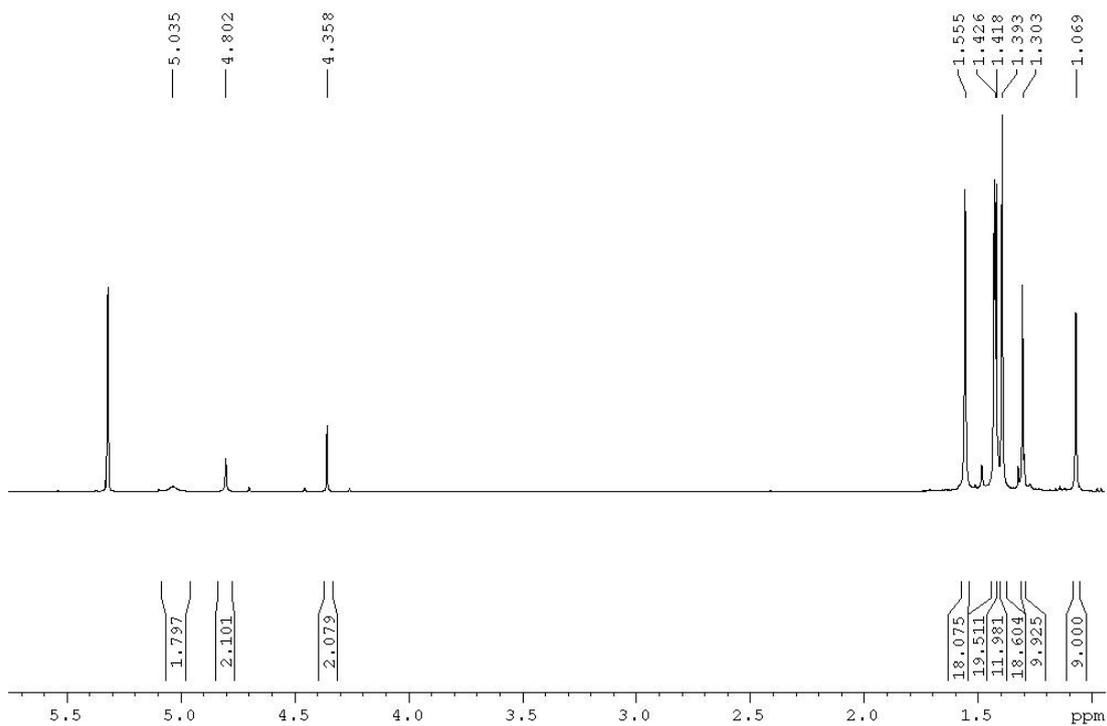


Figure S4: $^1\text{H-NMR}$ of **3** in CD_2Cl_2 .

$^{13}\text{C}\{^1\text{H}\}$ -NMR (400 MHz, CD_2Cl_2 , 300K): 31.8 (s, $\text{C}(\text{CH}_3)_3$), 32.2 (s, $\text{C}(\text{CH}_3)_3$), 32.5 (s br, $\text{C}(\text{CH}_3)_3$), 32.8 (s, CH_3), 33.5 (s, CH_3), 33.7 (s, CH_3), 33.8 (s, $\text{C}(\text{CH}_3)_3$), 33.9 (s, $\text{C}(\text{CH}_3)_3$), 34.2 (s br, $\text{C}(\text{CH}_3)_3$), 34.3 (s, CH_3), 34.4 (s, CH_3), 34.7 (s, CH_3), 79.2 (s, $\text{C}(\text{CH}_3)_3$), 82.2 (s, $\text{C}(\text{CH}_3)_3$), 82.6 (s, $\text{C}(\text{CH}_3)_3$), 85.8 (s, $\text{C}(\text{CH}_3)_3$), 107.4 (s, CH), 109.1 (s, CH), 110.1 (s, CH), 112.8 (s, $\text{C}(\text{CH}_3)_3$), 113.6 (s, $\text{C}(\text{CH}_3)_3$);

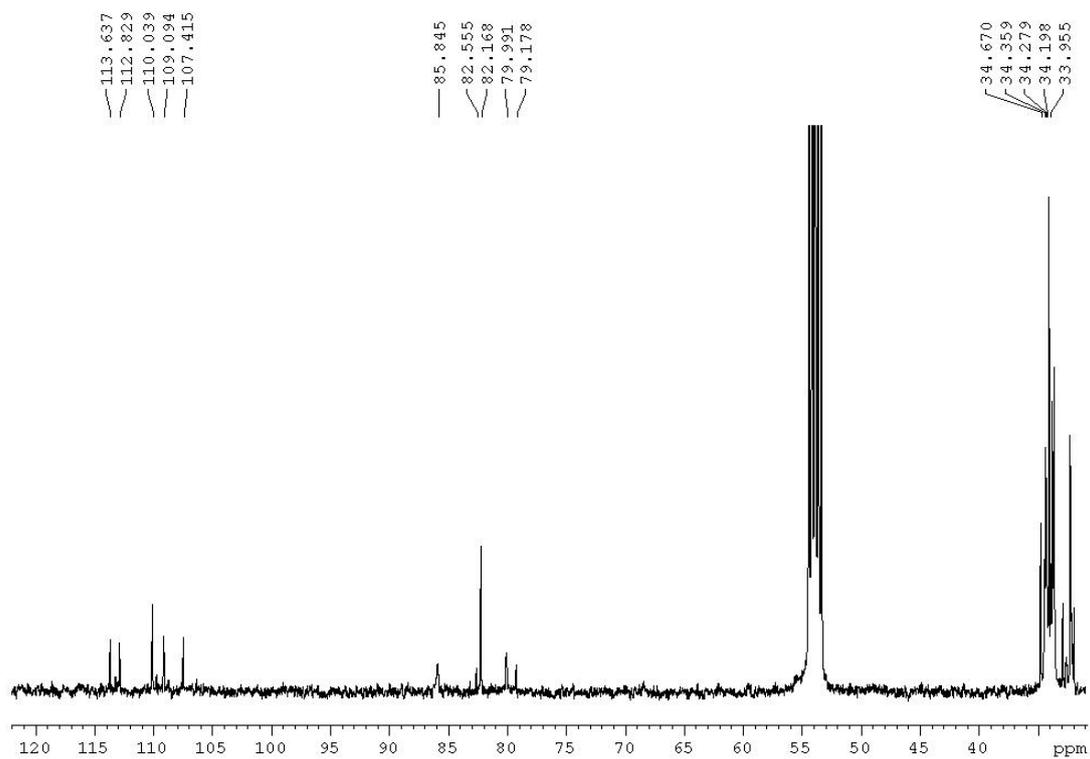


Figure S5: ^{13}C -NMR of **3** in CD_2Cl_2 .

MS (FDI): m/z (%): 1775.5 (100) $[\text{M}^+]$, 1033.9 (40) $[\text{M}^+-\text{Cp}'''\text{CoAs}_6]$, 884.3 (68) $[\text{M}^+-\text{Cp}''\text{CoAs}_8]$, 662.9 (100) $[\text{M}^+-\text{Cp}'''\text{CoAs}_{11}]$; **Calculated (%) for $\text{C}_{51}\text{H}_{87}\text{Co}_3\text{As}_{12}$** (1775.54 g/mol) C 34.49, H 4.94; found: C 34.81, H 4.91.

Crystal Data for 1.

Experimental. Single green block-shaped crystals of (**1**) were recrystallized from a saturated hexane solution at -25°C . A suitable crystal ($0.23 \times 0.12 \times 0.08 \text{ mm}^3$) was selected and mounted on a MITIGEN holder oil on an Aigent Technologies (formerly Oxford Diffraction) Gemini R Ultra diffractometer. Using **Olex2**,³ the structure was solved with the **SIR-97**⁴ structure solution program, using the Direct Methods solution method. The model was refined with version 2014/6 of **ShelXL**⁵ using Least Squares minimization. Absorption correction was applied empirically using SCALE3 program implemented to *CrysAlis PRO* software.

Crystal Data. $\text{C}_{34}\text{H}_{58}\text{As}_4\text{Co}_2$, $M_r = 884.34$, monoclinic, $\text{P}2_1/\text{n}$ (No. 14), $a = 8.43860(10) \text{ \AA}$, $b = 13.76760(10) \text{ \AA}$, $c = 16.02760(10) \text{ \AA}$, $\beta = 100.2700(10)^{\circ}$, $\alpha = \gamma = 90^{\circ}$, $V = 1832.24(3) \text{ \AA}^3$, $T = 123(2) \text{ K}$, $Z = 2$, $Z' = 0.5$, $\mu(\text{CuK}\alpha) = 11.249$, 19971 reflections measured, 3181 unique ($R_{int} = 0.0879$) which were used in all calculations. The final wR_2 was 0.0926 (all data) and R_I was 0.0331 ($I > 2(I)$).

Compound	1
CCDC	1028573
Formula	C ₃₄ H ₅₈ As ₄ Co ₂
$D_{calc.}/\text{g cm}^{-3}$	1.603
μ/mm^{-1}	11.249
Formula Weight	884.34
Colour	green
Shape	block
Max Size/mm	0.23
Mid Size/mm	0.12
Min Size/mm	0.08
T/K	123(2)
Crystal System	monoclinic
Space Group	P2 ₁ /n
$a/\text{\AA}$	8.4386(1)
$b/\text{\AA}$	13.7676(1)
$c/\text{\AA}$	16.0276(1)
α°	90
β°	100.270(1)
γ°	90
$V/\text{\AA}^3$	1832.24(3)
Z	2
Z'	0.5
θ_{min}°	4.263
θ_{max}°	66.683
Measured Refl.	19971
Independent Refl.	3181
Reflections Used	3018
R_{int}	0.0879
Parameters	181
Restraints	0
Largest Peak	0.900
Deepest Hole	-0.871
GooF	1.052
wR_2 (all data)	0.0926
wR_2	0.0915
R_1 (all data)	0.0348
R_1	0.0331

Crystal Data for 1*CH₂Cl₂.

Experimental. Single green block-shaped crystals of 1* CH₂Cl₂ were recrystallized from a saturated CH₂Cl₂ solution at -25°C. A suitable crystal (0.58 × 0.37 × 0.35 mm³) was selected and mounted on a MITIGEN holder oil on an Agilent Technologies (formerly Oxford Diffraction) Gemini R Ultra diffractometer. Using **Olex2**,³ the structure was solved with the **SIR-97**⁴ structure solution program, using the Direct Methods solution method. The model was refined with version 2014/6 of **ShelXL**⁵ using Least Squares minimization. Absorption correction was applied empirically using SCALE3 program implemented to *CrysAlis PRO* software. For the refinement of the disorder solvent CH₂Cl₂ molecule (90:10) displacement parameter restraints (SIMU) were applied.

Crystal Data. C₃₅H₆₀As₄Cl₂Co₂, $M_r = 969.27$, monoclinic, C2/c (No. 15), $a = 17.8930(10)$ Å, $b = 19.9249(11)$ Å, $c = 11.6299(5)$ Å, $\beta = 107.903(5)^\circ$, $\alpha = \gamma = 90^\circ$, $V = 3945.5(4)$ Å³, $T = 123(2)$ K, $Z = 4$, $Z' = 0.5$, $\mu(\text{MoK}\alpha) = 4.329$, 13850 reflections measured, 6515 unique ($R_{int} = 0.0241$) which were used in all calculations. The final wR_2 was 0.0998 (all data) and R_1 was 0.0387 ($I > 2(I)$).

Compound	1*CH ₂ Cl ₂
CCDC	1028574
Formula	C ₃₅ H ₆₀ As ₄ Cl ₂ Co ₂
$D_{calc.}/\text{g cm}^{-3}$	1.632
μ/mm^{-1}	4.329
Formula Weight	969.27
Colour	green
Shape	block
Max Size/mm	0.58
Mid Size/mm	0.37
Min Size/mm	0.35
T/K	123(2)
Crystal System	monoclinic
Space Group	C2/c
$a/\text{\AA}$	17.8930(10)
$b/\text{\AA}$	19.9249(11)
$c/\text{\AA}$	11.6299(5)
α°	90
β°	107.903(5)
γ°	90
$V/\text{\AA}^3$	3945.5(4)
Z	4
Z'	0.5
θ_{min}°	2.686
θ_{max}°	32.651
Measured Refl.	13850
Independent Refl.	6515
Reflections used	5548
R_{int}	0.0241
Parameters	204
Restraints	12
Largest Peak	1.679
Deepest Hole	-0.865
GooF	1.087
wR_2 (all data)	0.0998
wR_2	0.0948
R_1 (all data)	0.0490
R_1	0.0387

Crystal Data for 2*4 CH₂Cl₂.

Experimental. Single black block-shaped crystals of (2*4 CH₂Cl₂) were recrystallized from a saturated CH₂Cl₂ solution at -25°C. A suitable crystal (0.15 × 0.10 × 0.04 mm³) was selected and mounted on a MITIGEN holder oil on an Agilent Technologies (formerly Oxford diffraction) Gemini R Ultra diffractometer. Using **Olex2**,³ the structure was solved with the **SIR-97**⁴ structure solution program, using the direct methods solution method. The model was refined with version 2014/6 of **ShelXL**⁵ using by full matrix least squares on **F**² minimization. Absorption correction was applied empirically using **SCALE3** program implemented to *CrysAlis PRO* software. Distance and displacement parameter restraints (DFIX, SIMU) were applied to the two solvent CH₂Cl₂ molecules. The third solvent CH₂Cl₂ is located at the inversion center and could not be modelled. Therefore the Squeeze⁶ function of the Platon⁷ software was applied to back calculate its contribution to the structure factors.

Crystal Data. C₇₂H₁₂₄As₁₀Cl₈Co₄, $M_r = 2258.22$, triclinic, P-1 (No. 2), $a = 13.7956(8)\text{Å}$, $b = 14.0892(8)\text{Å}$, $c = 15.0382(10)\text{Å}$, $\alpha = 62.971(6)^\circ$, $\beta = 69.895(6)^\circ$, $\gamma = 61.292(6)^\circ$, $V = 2254.0(1)\text{Å}^3$, $T = 123(2)\text{K}$, $Z = 1$, $Z' = 0.5$, $\mu(\text{CuK}\alpha) = 12.199$, 11135 reflections measured, 6133 unique ($R_{\text{int}} = 0.0672$) which were used in all calculations. The final wR_2 was 0.1770 (all data) and R_I was 0.0787 ($I > 2(I)$).

Compound	2*4 CH ₂ Cl ₂
CCDC	1028576
Temperature/K	123(1)
Crystal system	triclinic
Space group	P-1
a/Å	13.7956(8)
b/Å	14.0892(8)
c/Å	15.0382(10)
$\alpha/^\circ$	62.971(6)
$\beta/^\circ$	69.895(6)
$\gamma/^\circ$	61.292(6)
Volume/Å ³	2254.0(3)
Z	1
$\rho_{\text{calc}}/\text{cm}^3$	1.664
μ/mm^{-1}	12.199
F(000)	1130.0
Crystal size/mm ³	0.15 × 0.1 × 0.035
Radiation	CuK α ($\lambda = 1.54184$)
2 Θ range for data collection/ $^\circ$	6.686 to 124.232
Index ranges	-15 ≤ h ≤ 15, -14 ≤ k ≤ 15, -13 ≤ l ≤ 17
Reflections collected	11135
Independent reflections	6133 [$R_{\text{int}} = 0.0672$, $R_{\text{sigma}} = 0.0879$]

Data/restraints/parameters 6133/231/452
Goodness-of-fit on F^2 1.108
Final R indexes [$I \geq 2\sigma(I)$] $R_1 = 0.0787$, $wR_2 = 0.1658$
Final R indexes [all data] $R_1 = 0.1088$, $wR_2 = 0.1770$
Largest diff. peak/hole / $e \text{ \AA}^{-3}$ 0.97/-1.02

Crystal Data for 2.

Experimental. Single black block-shaped crystals of (**2**) were recrystallised from a saturated hexane solution at 8°C. A suitable crystal ($0.11 \times 0.10 \times 0.04 \text{ mm}^3$) was selected and mounted on a MITIGEN holder oil on an Agilent Technologies (formerly Oxford diffraction) SuperNova A diffractometer. Using **Olex2**,³ the structure was solved with the **SIR-97**⁴ structure solution program, using the direct methods solution method. The model was refined with version 2014/6 of **ShelXL**⁵ using by full matrix least squares on F^2 minimization. Absorption correction was applied empirically using SCALE3 program implemented to *CrysAlis PRO* software.

The crystals appeared to possess a large mosaicity. Several attempts to refine the structure including disorder failed. Therefore, we assume dynamic rotation of the tert-butyl groups, probably together with two disordered positions of the cyclopentadienyl rings.

Crystal Data. $C_{68}H_{116}As_{10}Co_4$, $M_r = 1918.52$, triclinic, P-1 (No. 2), $a = 10.3921(5) \text{ \AA}$, $b = 14.3707(7) \text{ \AA}$, $c = 14.8262(8) \text{ \AA}$, $\alpha = 62.530(5)^\circ$, $\beta = 76.535(4)^\circ$, $\gamma = 79.819(4)^\circ$, $V = 1904.35(19) \text{ \AA}^3$, $T = 123(1) \text{ K}$, $Z = 1$, $Z' = 0.5$, $\mu(\text{CuK}\alpha) = 11.799$, 22490 reflections measured, 7152 unique ($R_{int} = 0.0363$) which were used in all calculations. The final wR_2 was 0.2212 (all data) and R_1 was 0.0781 ($I > 2(I)$).

Compound	2
CCDC	1028575
Formula	C ₆₈ H ₁₁₆ As ₁₀ Co ₄
$D_{calc.}/\text{g cm}^{-3}$	1.673
μ/mm^{-1}	11.799
Formula Weight	1918.52
Colour	black
Shape	block
Max Size/mm	0.11
Mid Size/mm	0.10
Min Size/mm	0.04
T/K	123(1)
Crystal System	triclinic
Space Group	P-1
$a/\text{\AA}$	10.3921(5)
$b/\text{\AA}$	14.3707(7)
$c/\text{\AA}$	14.8262(8)
α°	62.530(5)
β°	76.535(4)
γ°	79.819(4)
$V/\text{\AA}^3$	1904.35(19)
Z	1
Z'	0.5
θ_{min}°	3.411
θ_{max}°	70.824
Measured Refl.	22490
Independent Refl.	7152
Reflections Used	6108
R_{int}	0.0363
Parameters	295
Restraints	6
Largest Peak	1.719
Deepest Hole	-0.926
GooF	1.072
wR_2 (all data)	0.2212
wR_2	0.2137
R_1 (all data)	0.0864
R_1	0.0781

Crystal Data for 3.

Experimental. Single black plate-shaped crystals of (**3**) were recrystallized from a saturated hexane solution at -25°C . A suitable crystal ($0.07 \times 0.04 \times 0.01 \text{ mm}^3$) was selected and mounted on a MITIGEN holder oil on an Agilent Technologies (formerly Oxford diffraction) SuperNova A diffractometer. Using **Olex2**,³ the structure was solved with the **SIR-97**⁴ structure solution program, using the direct methods solution method. The model was refined with version 2014/6 of **ShelXL**⁵ using by full matrix least squares on \mathbf{F}^2 minimization. Absorption correction was applied empirically using SCALE3 program implemented to *CrysAlis PRO* software.

Crystal Data. $\text{C}_{51}\text{H}_{87}\text{As}_{12}\text{Co}_3$, $M_r = 1776.03$, monoclinic, $\text{C2}/c$ (No. 15), $a = 28.9202(3) \text{ \AA}$, $b = 15.78990(10) \text{ \AA}$, $c = 29.9375(3) \text{ \AA}$, $\beta = 112.7520(10)^{\circ}$, $\alpha = \gamma = 90^{\circ}$, $V = 12607.1(2) \text{ \AA}^3$, $T = 123.00(10) \text{ K}$, $Z = 8$, $Z' = 1$, $\mu(\text{CuK}\alpha) = 13.346$, 38088 reflections measured, 11768 unique ($R_{int} = 0.0451$) which were used in all calculations. The final wR_2 was 0.0589 (all data) and R_1 was 0.0271 ($I > 2(I)$).

Compound	3
CCDC	1028577
Formula	C ₅₁ H ₈₇ As ₁₂ Co ₃
<i>D</i> _{calc.} / g cm ⁻³	1.871
<i>μ</i> /mm ⁻¹	13.346
Formula Weight	1776.03
Colour	black
Shape	plate
Max Size/mm	0.07
Mid Size/mm	0.04
Min Size/mm	0.01
<i>T</i> /K	123(1)
Crystal System	monoclinic
Space Group	C2/c
<i>a</i> /Å	28.9202(3)
<i>b</i> /Å	15.7899(1)
<i>c</i> /Å	29.9375(3)
<i>α</i> [°]	90
<i>β</i> [°]	112.752(1)
<i>γ</i> [°]	90
<i>V</i> /Å ³	12607.1(2)
<i>Z</i>	8
<i>Z</i> '	1
<i>θ</i> _{min} [°]	3.201
<i>θ</i> _{max} [°]	70.494
Measured Refl.	38088
Independent Refl.	11768
Reflections Used	10045
<i>R</i> _{int}	0.0451
Parameters	622
Restraints	0
Largest Peak	0.914
Deepest Hole	-0.489
GooF	1.032
<i>wR</i> ₂ (all data)	0.0589
<i>wR</i> ₂	0.0555
<i>R</i> ₁ (all data)	0.0363
<i>R</i> ₁	0.0271

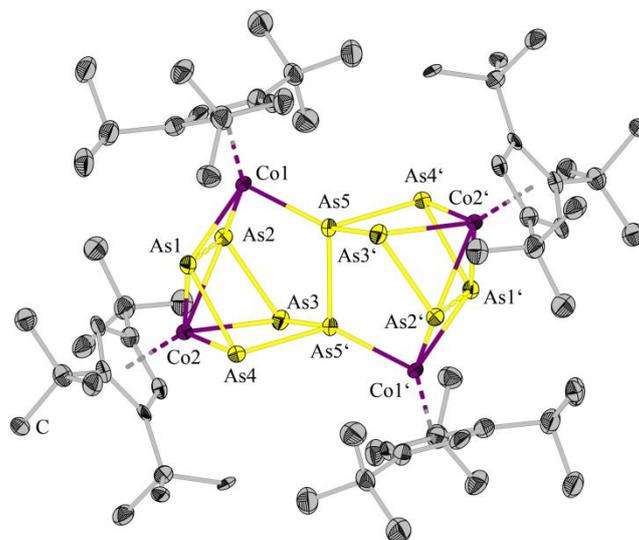


Figure S6: Molecular structure of **2*4 CH₂Cl₂**. The H atoms and CH₂Cl₂ solvent molecules are omitted for clarity. Anisotropic displacement parameters are depicted at 50% probability level.

Selected Bond Distances and Angles for **2*4 CH₂Cl₂**

Atom	Atom	Length/Å	Atom	Atom	Length/Å
As5	As5'	2.402(3)	As2	Co1	2.351(3)
As5	As4'	2.424(2)	As1	Co2	2.479(2)
As5	As3'	2.435(2)	As1	Co1	2.349(3)
As5	Co1	2.273(2)	As3	As5'	2.435(2)
As4	As5'	2.424(2)	As3	Co2	2.447(2)
As4	As1	2.379(2)			
As4	Co2	2.432(3)			
As2	As1	2.705(2)			
As2	As3	2.377(2)			
As2	Co2	2.465(3)			

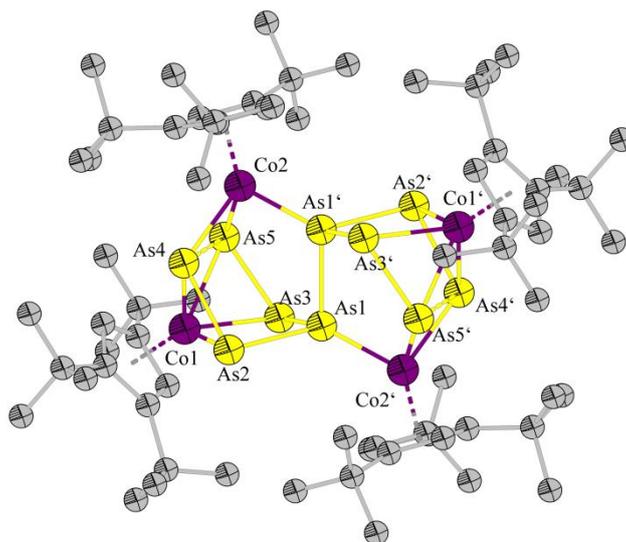


Figure S7: Molecular structure of **2**. The H atoms are omitted for clarity.

Selected Bond Distances and Angles for **2**

Co1	As2	2.431(2)
Co1	As3	2.437(2)
Co1	As4	2.4763(19)
Co1	As5	2.4656(19)
Co2	As1 [']	2.2816(15)
Co2	As4	2.3476(18)
Co2	As5	2.3448(18)
As1	Co2 [']	2.2816(15)
As1	As1 [']	2.4085(16)
As1	As2	2.4265(14)
As1	As3	2.4225(14)
As2	As4	2.3673(14)
As3	As5	2.3755(14)
As4	As5	2.6723(19)

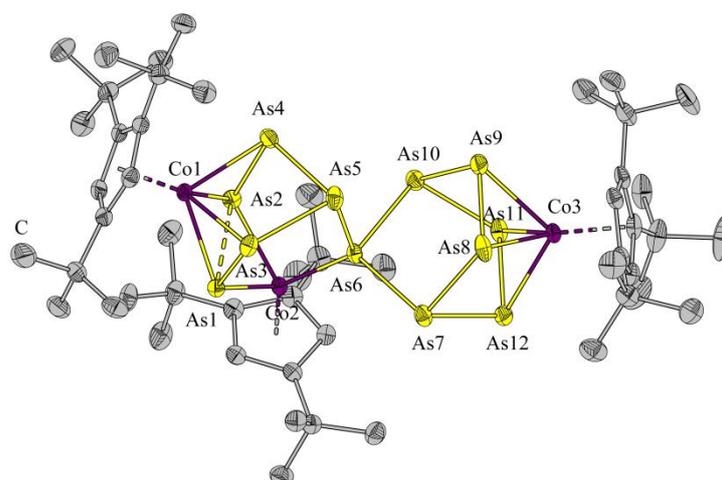


Figure S8: Molecular structure of **3**. The H atoms are omitted for clarity. Anisotropic displacement parameters are depicted at 50% probability level.

Selected Bond Distances and Angles for **3**

Co1 As1 2.4702(6)	Co1 As2 2.4627(6)	Co1 As3 2.4383(6)	Co1 As4 2.4284(6)
Co2 As1 2.3628(6)	Co2 As2 2.3610(6)	Co2 As6 2.2628(6)	Co3 As8 2.4525(6)
Co3 As9 2.4554(6)	Co3 As11 2.4402(6)	Co3 As12 2.4423(6)	As1 As2 2.6684(5)
As1 As3 2.3809(5)	As2 As4 2.3821(5)	As3 As5 2.4156(5)	As4 As5 2.4350(6)
As5 As6 2.4352(4)	As6 As7 2.4312(5)	As6 As10 2.4472(5)	As7 As8 2.4130(5)
As7 As12 2.4282(5)	As8 As9 2.3478(6)	As9 As10 2.4277(6)	As10 As11 2.4288(5)
As11 As12 2.3638(6)			

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