

Supporting information

Selective ligand-free cobalt catalysed hydrosilylation of esters to aldehydes or alcohols.

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I) General remarks.

All solvents were dried using standard methods and stored over molecular sieves (4 Å). All sensitive salts were weighted in a glovebox. All reactions were carried out under a dry nitrogen atmosphere and were repeated at least twice. Analytical thin layer chromatography (TLC) was performed on Merck pre-coated 0.20 mm silica gel Alugram Sil 60 G/UV₂₅₄ plates. Flash chromatography was carried out with Macherey silica gel (Kieselgel 60). ¹H (300 MHz), ¹³C (75 or 125 MHz) NMR spectra were acquired on Bruker Avance spectrometers. Chemical shifts (δ) are reported downfield of Me₄Si in ppm and coupling constants are expressed in Hz. Infra-red analyses were acquired by Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) on a ThermoScientific-Nicolet 6700 spectrometer. X-ray diffraction analyses were performed in reflexion mode using a Bruker D8 Advance diffractometer and in transmission mode using a Rigaku Smartlab diffractometer. XPS measurements were acquired on a XPS KRATOS, AXIS UltraDLD (2009). Ester substrates were purchased and used as received. Alternatively, ester reagents **3a**, **3b**, **3c**, **3d**, **3e**, **3f**, **3g**, **3h**, **3k**, **3l**, **3m**, **3n**, **3p**, **3q** were prepared from the corresponding carboxylic acids following the general procedure (vide infra). Cobalt salts were purchased and used as received, dry compounds being manipulated in a glovebox: CoCl₂ [anhydrous, 99+% Strem, Lot 26235300], CoCl₂ [anhydrous, 99.998%, AlfaAesar, Lot Q21D126], Co(acac)₂ [99% Acros, Lot A0273018], Co(OAc)₂ .4 H₂O [reagent grade, Aldrich], Co(OAc)₂ [anhydrous, 99.995%, Aldrich, Lot MKBW0775V], Co(BF₄)₂.4 H₂O [96%, AlfaAesar, Lot T11D001].

II) General procedures.

1) General procedure for the synthesis of esters.

In a round bottom flask, carboxylic acid (1 eq.), sulfuric acid (5 mol%) and ethanol (solvent of reaction) were added and refluxed during one night under stirring. The ethanol was then evaporated under vacuum through the use of a rotary evaporator. The crude product was subsequently dissolved with dichloromethane and washed three times with NaOH 1M. The organic layer was then dried with magnesium sulfate and evaporated to dryness under vacuum. All esters were obtained without any further purification.

2) General procedure for the catalysis using $\text{Co}(\text{OAc})_2$, e.g. cat. A:

$\text{Co}(\text{OAc})_2 \cdot 4 \text{H}_2\text{O}$ (3.8 mg, 5 mol%, 0.015 mmol) was introduced into a Schlenk tube and dried under vacuum during 15 minutes. Under nitrogen, dry DME (2 mL) was transferred and the resulting solution was stirred at 60 °C. After 15 minutes, NaBHET_3 (30 μL , 1M in toluene, 10 mol%, 0.03 mmol) was added. After 5 minutes of additional stirring, PhSiH_3 (74 μL , 2 eq., 0.6 mmol) was added followed by the ester (0.3 mmol, 1 eq.). The reaction mixture was then heated at 25 °C under stirring during 15 hours. Reactions proceeded also on a 3 mmol scale of ester.

General procedure for the catalysis using CoCl_2 , e.g. cat. B:

In a glovebox, CoCl_2 (1.95 mg, 5 mol%, 0.015 mmol), KO^tBu (3.36 mg, 10 mol%, 0.03 mmol) were introduced into a Schlenk tube. Under nitrogen, dry dioxane (2 mL) was transferred and the resulting mixture was heated at 60°C. After 15 minutes, NaBHET_3 (30 μL , 1M in toluene, 10 mol%, 0.03 mmol) and water (2.7 μL , 50 mol%, 0.15 mmol) were added. After 5 additional minutes of stirring, PhSiH_3 (74 μL , 2 eq., 0.6 mmol) was added followed by the ester (0.30 mmol, 1 eq.). The reaction mixture was then heated at 60°C under stirring during 15 hours. Reactions proceeded also on a 3 mmol scale of ester.

To monitor the progress of the reactions, aliquots (0.1 mL) were taken at defined times, filtered through Celite with a CH_2Cl_2 wash (3 mL), and analysed by GC. At the end of each reaction, the crude product was filtered through Celite with a CH_2Cl_2 wash (3 mL) then purified by flash chromatography or preparative TLC.

Volatile aldehyde products were isolated as 2,4-dinitrophenylhydrazoneadducts as follows. After reaction, the resulting crude mixture was dried over MgSO_4 and diluted with ethanol (1 mL). The resulting solution was treated with 0.5 mL of Brady's reagent [solution of 2,4-dinitrophenylhydrazine (0.3 g), H_2O (1.5 mL), ethanol (5 mL) and H_2SO_4 conc. (1 mL)] for 30 min with vigorous stirring. The resulting orange precipitate was isolated by filtration through filter paper, washed and triturated with ethanol (10 mL) and finally dried under vacuum.

III) Optimization of the reaction conditions: additional experimental data.

Table S1. Screening of cobalt salts for cat. A

$$\text{Ph-CH}_2\text{-CO}_2\text{Et} \xrightarrow[\text{DME, T}^\circ\text{C), t (h)}]{\begin{array}{l} 1) \text{ Cobalt salt (5 mol\%)} \\ 2) \text{ NaBHET}_3 \text{ (10 mol\%)} \\ 3) \text{ PhSiH}_3 \text{ (2 eq.)} \end{array}} \text{Ph-CH}_2\text{-CHO} + \text{Ph-CH}_2\text{-CH}_2\text{-OH}$$

Entry	Cobalt salt	T (°C)	Time (h)	Yield (%) ^a	Selectivity (%) ^a 6a / 7a	
1	Co(OAc) ₂ .4 H ₂ O	25	0.5	9	100	-
2	Co(OAc) ₂ .4 H ₂ O	25	5	89	100	-
3	Co(OAc) ₂ 99.995%	25	0.5	9	100	-
4	Co(OAc) ₂ 99.995%	25	5	93	100	-
5	Co(acac) ₂	25	15	78	100	-
6	CoCl ₂ 99+%	25	15	0	-	-
7	CoCl ₂ 99+%	60	15	0	-	-
8	Co(BF ₄) ₂ .4 H ₂ O 96%	60	15	2	traces	traces
9 ^b	Co(OAc) ₂ .4 H ₂ O	25	15	100	100	-
10	none	25	15	75	-	100

a) determined by GC. b) on a 3 mmol scale (instead 0.3 mmol).

Table S2. Screening of solvents for cat. A.

$$\text{Ph-CH}_2\text{-CO}_2\text{Et} \xrightarrow[\text{solvent, 40}^\circ\text{C, 15 h}]{\begin{array}{l} 1) \text{ Co(OAc)}_2 \text{ (5 mol\%)} \\ 2) \text{ NaBHET}_3 \text{ (10 mol\%)} \\ 3) \text{ PhSiH}_3 \text{ (2 eq.)} \end{array}} \text{Ph-CH}_2\text{-CHO} + \text{Ph-CH}_2\text{-CH}_2\text{-OH}$$

Entry	Solvent	Yield (%) ^a	Selectivity (%) ^a 6a / 7a	
1	THF	100	81	19
2	Dioxane	78	100	0
3	TBME	100	10	-
4	DBE	100	6	-
5	Anisole	0	-	-
6	CPME	0	-	-
7	DME	100	100	-

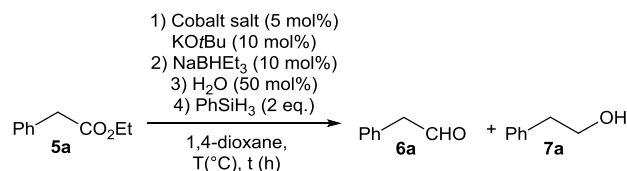
a) determined by GC.

Table S3. Screening of silanes for cat. A.

$$\text{Ph-CH}_2\text{-CO}_2\text{Et} \xrightarrow[\text{DME, 40}^\circ\text{C, 15 h}]{\begin{array}{l} 1) \text{ Co(OAc)}_2 \text{ (5 mol\%)} \\ 2) \text{ NaBHET}_3 \text{ (10 mol\%)} \\ 3) \text{ silane (x eq.)} \end{array}} \text{Ph-CH}_2\text{-CHO} + \text{Ph-CH}_2\text{-CH}_2\text{-OH}$$

Entry	Silane	Yield (%) ^a	Selectivity (%) ^a 6a / 7a	
1	PhSiH ₃	100	100	-
2	Ph ₂ SiH ₂	0	-	-
3	Ph ₃ SiH	0	-	-
4	PhMe ₂ SiH	0	-	-
5	Et ₃ SiH	0	-	-
6	TMDS	0	-	-
7	PMHS	0	-	-
8	Hexylsilane	100	100	-

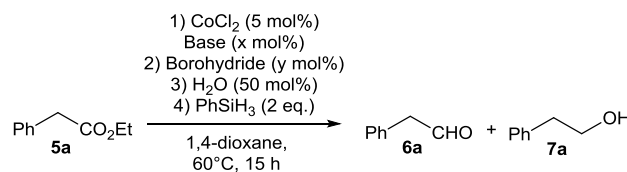
a) determined by GC.

Table S4. Screening of cobalt salts for cat. B.

Entry	Cobalt salt	T (°C)	Time (h)	Yield (%) ^a	Selectivity (%) ^a 6a / 7a	
1	CoCl ₂ 99+%	25	5	46	2	98
2	CoCl ₂ 99+%	25	15	95	6	94
3	CoCl ₂ 99+%	60	5	99	-	100
4	CoCl ₂ 99+%	60	15	100	-	100
5	CoCl ₂ 99.99%	60	5	100	-	100
6	Co(OAc) ₂ .4 H ₂ O	60	5	100	2	98
7	Co(acac) ₂	60	15	88	80	20
8	Co(BF ₄) ₂ .4 H ₂ O	60	15	83	-	100
9 ^b	CoCl ₂ 99+%	60	15	100	-	100
10 ^c	none	60	5	39	-	100

a) determined by GC. b) on a 3 mmol scale (instead 0.3 mmol).

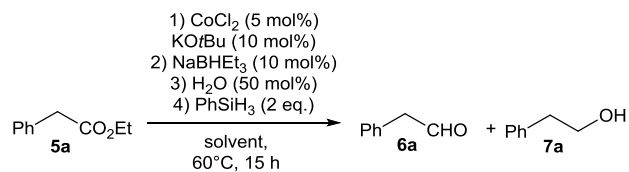
c) reaction without CoCl₂ and base.

Table S5. Screening of bases and hydrides for cat. B.

Entry	Base (mol%)	Borohydride (mol%)	Yield (%) ^a	Selectivity (%) ^a 6a / 7a	
1	KOtBu (10)	NaBHET ₃ (10)	100	-	100
2	KOtBu (5)	NaBHET ₃ (10)	85	-	100
3	KOtBu (10)	NaBHET ₃ (5)	81	-	100
4	KOtBu (10)	-	0	-	-
5 ^b	KOtBu (10)	-	0	-	-
6 ^c	none	NaBHET ₃ (10)	73	-	100
7	KOtBu (10)	NaBH ₄ (10)	0	-	-
8	KOtBu (10)	DIBAL-H (10)	0	-	-
9	KHMDS (10)	NaBHET ₃ (10)	98	-	100
10	K ₂ CO ₃ (10)	NaBHET ₃ (10)	0	-	-
11	CS ₂ CO ₃ (10)	NaBHET ₃ (10)	100	38	62
12	KOH (10)	NaBHET ₃ (10)	93	28	72
13	KOAc (10)	NaBHET ₃ (10)	25	-	100

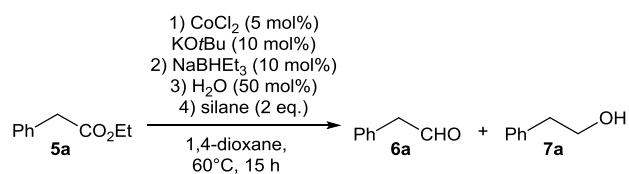
a) determined by GC. b) only KOtBu used with 5a, no other reagents implied.

c) reaction without CoCl₂ and base.

Table S6. Screening of solvents for cat. B.

Entry	Solvent	Yield (%) ^a	Selectivity (%) ^a 6a / 7a	
1	DME	71	-	100
2	CPME	98	-	34 + by-products
3	THF	100	54	46
4	TBME	7	36	64
5	DBE	92	3	97
6	1,4-dioxane	100	-	100
7	anisole	0	-	-

a) determined by GC.

Table S7. Screening of silanes for cat. B.

Entry	Silane	Yield (%) ^a	Selectivity (%) ^a 6a / 7a	
1	PhSiH ₃	100	-	100
2	Ph ₂ SiH ₂	0	-	-
3	Ph ₃ SiH	0	-	-
4	Me ₂ PhSiH	0	-	-
5	Et ₃ SiH	0	-	-
6	TMDS	0	-	-
7	PMHS	0	-	-
8	Hexylsilane	100	-	100

a) determined by GC.

Table S8: recycling tests for catalyst A.

$$\text{Ph-CH}_2\text{-CO}_2\text{Et (5a)} \xrightarrow[\text{DME, 25}^\circ\text{C, 15 h}]{\begin{array}{l} 1) \text{Co(OAc)}_2 \text{ (5 mol\%)} \\ 2) \text{NaBHET}_3 \text{ (10 mol\%)} \\ 3) \text{PhSiH}_3 \text{ (2 eq.)} \end{array}} \text{Ph-CH}_2\text{-CHO (6a)} + \text{Ph-CH}_2\text{-OH (7a)}$$

Cycle ^a	Yield ^b	Selectivity (%) ^b 6a / 7a	
1	Quant.	100	-
2	Quant.	78	21
3	92	76	24
4	93	77	23

a) After a cycle, all volatiles were evaporated under vacuum and condensed in a side trap cooled by liquid nitrogen. Afterwards, the reaction vessel was filled with solvent under nitrogen gas and NaBHET₃ (10 mol%) was subsequently added followed by phenylsilane (2 eq.). The new reaction cycle was then started for 15 h. b) Determined by GC.

Table S9: recycling tests for catalyst B.

$$\text{Ph-CH}_2\text{-CO}_2\text{Et (5a)} \xrightarrow[\text{1,4-dioxane, 60}^\circ\text{C, 15 h}]{\begin{array}{l} 1) \text{CoCl}_2 \text{ (5 mol\%)} \\ \text{KOtBu (10 mol\%)} \\ 2) \text{NaBHET}_3 \text{ (10 mol\%)} \\ 3) \text{H}_2\text{O (50 mol\%)} \\ 4) \text{PhSiH}_3 \text{ (2 eq.)} \end{array}} \text{Ph-CH}_2\text{-CHO (6a)} + \text{Ph-CH}_2\text{-OH (7a)}$$

Cycle ^a	Yield ^b	Selectivity (%) ^b 6a / 7a	
1	Quant.	-	100
2	Quant.	-	100
3	Quant.	3	97
4	Quant.	3	97

a) After a cycle, all volatiles were evaporated under vacuum and condensed in a side trap cooled by liquid nitrogen. Afterwards, the reaction vessel was filled with solvent under nitrogen gas and NaBHET₃ (10 mol%) was subsequently added followed by phenylsilane (2 eq.). The new reaction cycle was then started for 15 h. b) Determined by GC.

IV) TEM analyses.

All samples were handled in a glovebox. They were ground in a mortar before being deposited as solids (no solvent used) on Copper grids compatible with MET analyses. Each prepared grid was then stored in an object holder under argon and later under vacuum before the analyses. The machine used was a transmission electron microscope JEOL JEM-2100F. This field emission microscope allowed the user to work under the following modes:

- TEM, HRTEM: conventional microscopy, high resolution and micro-diffraction
- EDS: microanalysis by energy dispersion of the photons emitted by the sample under the impact effect of the incident electrons.

Main characteristics:

- Field emission source
- Acceleration voltage: 200 kV
- Resolution: 2.3 Å
- EDX analysis: SDD Bruker (XFlash 5030, 127 eV resolution)

Help for indexation was provided by CaRInE, JEEMS and CrysTool softwares in order to rebuild and verify all orientations.^{S1}

Regarding catalyst **A** resulting from the reaction of $\text{Co}(\text{OAc})_2$ with NaBHET_3 and phenylsilane, nanoparticles appeared crystallised as plans and diffraction patterns were observed at HRTEM (ie $>400\text{kx}$) (see manuscript Figure 1d and Figure S1). However, indexation was not completely fitting with NaOAc (which was observed by DRX) as the diffraction data were few in number and of average quality. Other possible but not obvious indexations would be for: Co in $(-5,-3,7)$ or in $(-13,-4, 16)$, or CoFe_2O_4 in $(-4,-3,-16)$ or CoO in $(9,-7,6)$.

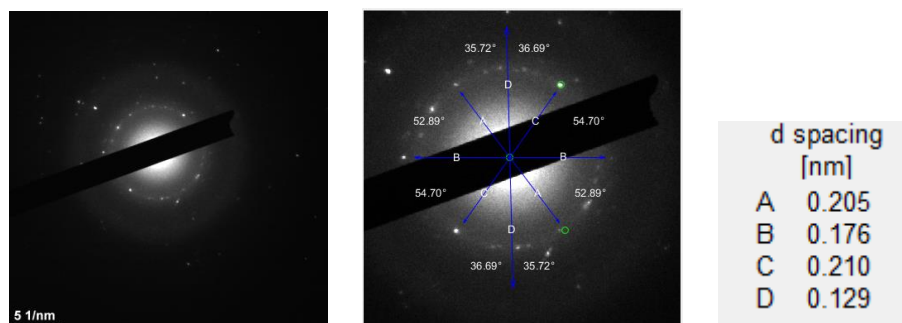


Figure S1. Diffraction patterns of catalyst **A** nanoparticles observed in Figure 1d.

The EDX analysis of the nanoparticles observed in Figure 1d showed piles were made of Co, Fe, Ti traces and Cu which was likely coming from the TEM grid on which the solid was deposited (Figure S2).

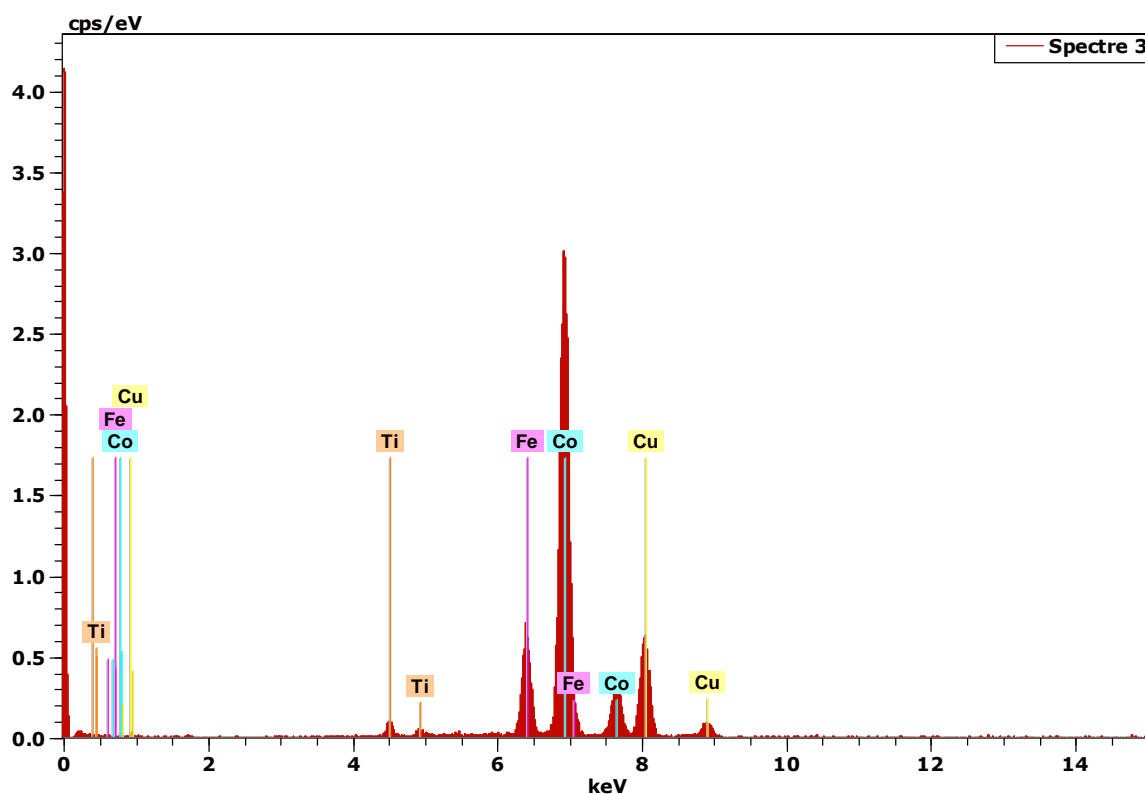


Figure S2. EDX analysis of catalyst **A** nanoparticles observed in Figure 1d.

The dispersion of the nanoparticles was studied by STEM EDS elemental mapping without quantification and sizing (Figures 1e-f, S3). The nanoparticles appearing as dark piles on the electronic picture were predominantly appearing as green piles of Co on the mapping.

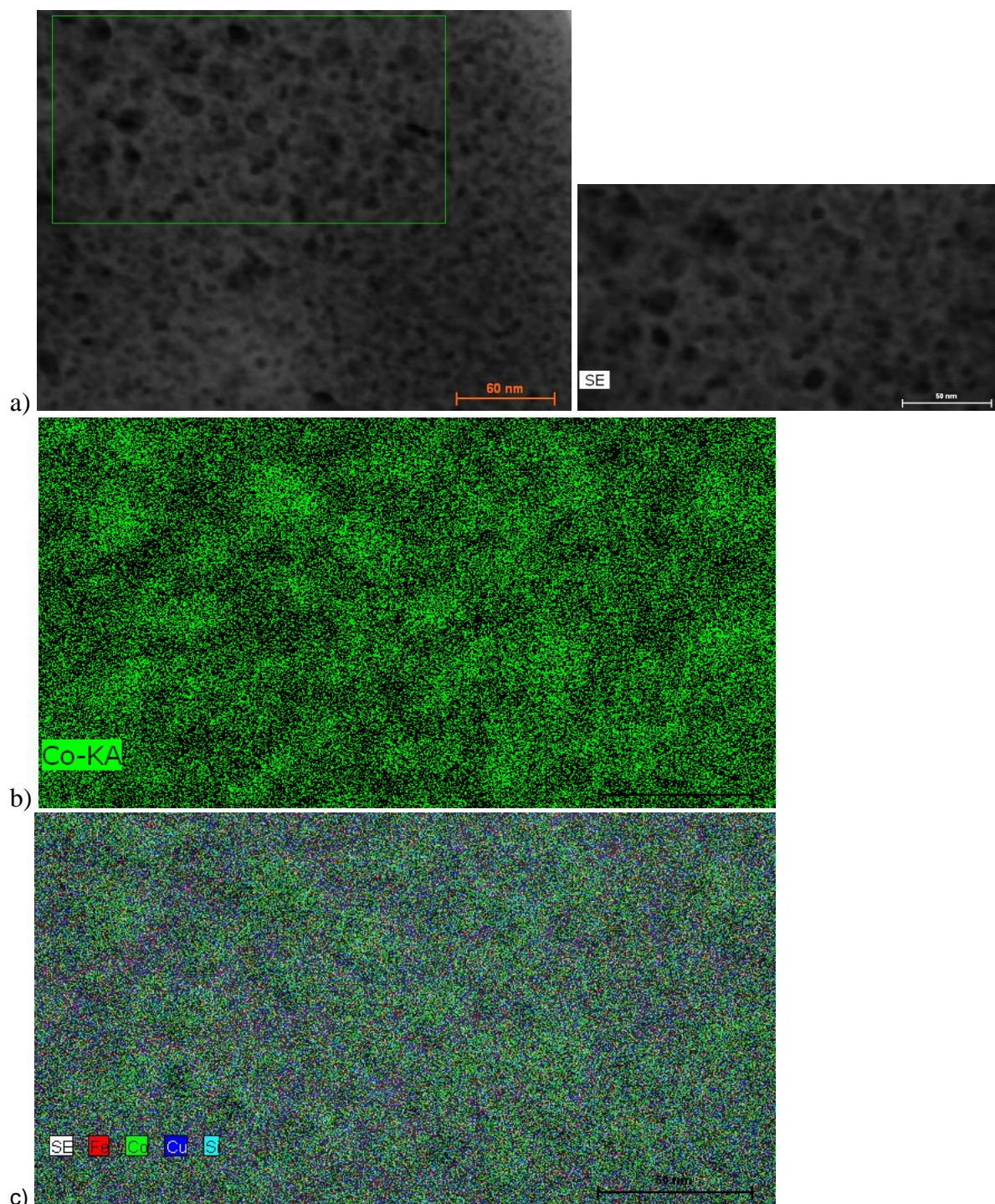


Figure S3. STEM EDS elemental mapping of catalyst A nanoparticles observed in Figure 1d. a) Electronic picture of the mapping area; the green area (SE, secondary electron) was scanned. b) Elemental mapping area for Co (K-alpha ray @6.925keV). c) Elemental mapping area for Fe, Co, Cu, Si.

Regarding catalyst **B** resulting from the reaction of CoCl_2 with KO^tBu , NaBHET_3 , water and phenylsilane, nanoparticles appeared crystallised by diffraction or at HRTEM (Figures 1h as well as 1i-1j) and the observed diffraction patterns (Figures S4-S6) could be indexed for $\text{Co}(\text{OH})_2$. Distances fitted with the cif file of $\text{Co}(\text{OH})_2$. This was also verified by using VESTA software and a possible indexation was found for (-1, 4, 2) with small differences.

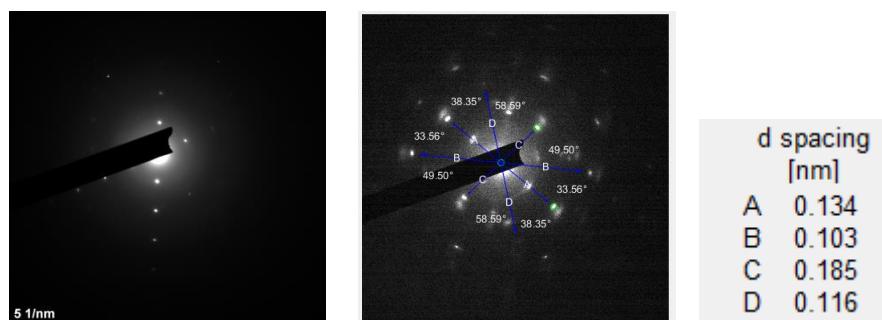


Figure S4. Diffraction patterns of catalyst **B** nanoparticles observed in Figure 1h.

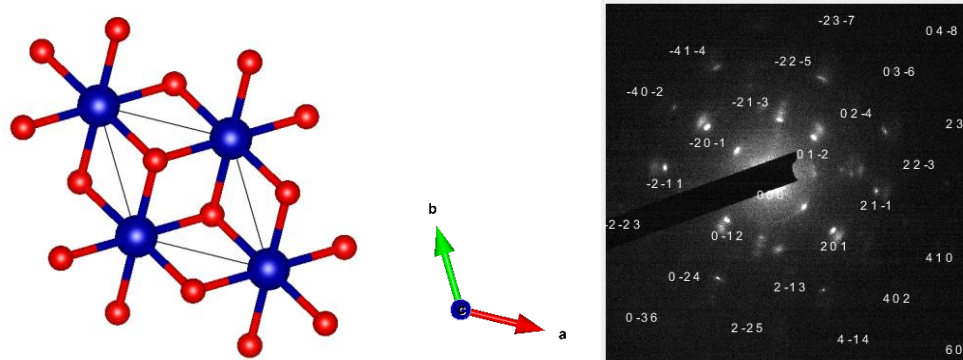


Figure S5. Possible indexation of diffraction patterns of catalyst **B** nanoparticles observed in Figure 1h.

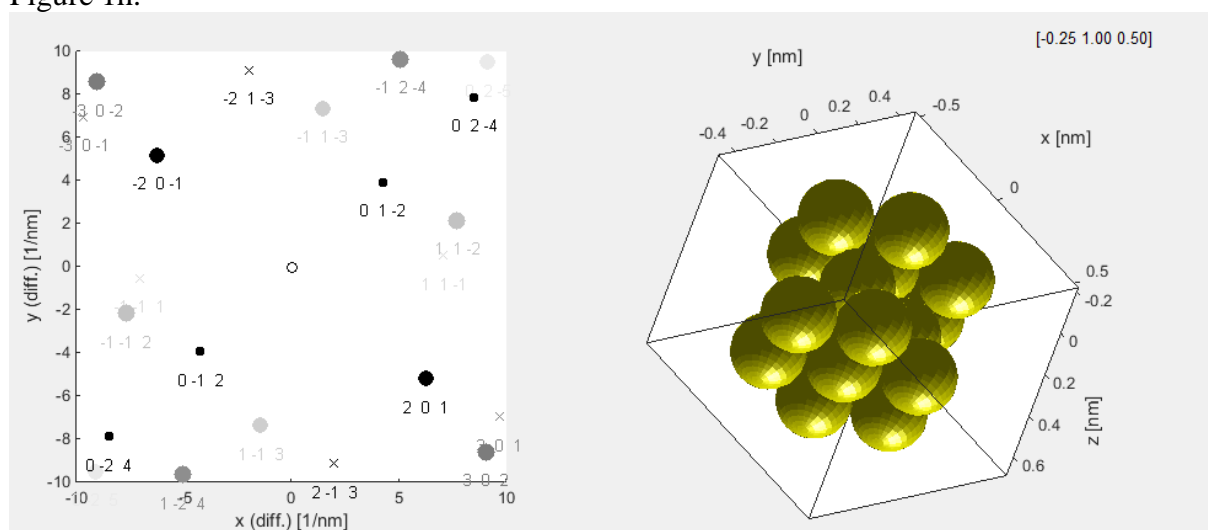


Figure S6. Possible indexation of diffraction patterns of catalyst **B** nanoparticles observed in Figure 1h.

However, sample was not stable enough to allow any STEM-EDS analysis as nanoparticles were rapidly becoming amorphous or even melting upon effect of the 200kV beam (Figure S7).

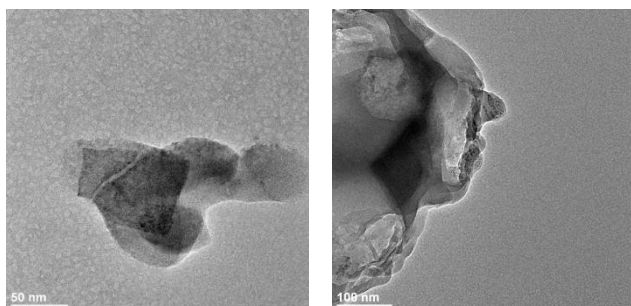


Figure S7. TEM images of catalyst **B** nanoparticles - negative effect of the 200kV beam.

EDX analysis of the nanoparticles observed in Figure 1h (see manuscript) showed the piles were composed of Co, Fe, Si, K and Cl with the presence of Ti traces and Cu from the TEM grid holding the sample (Figure S8).

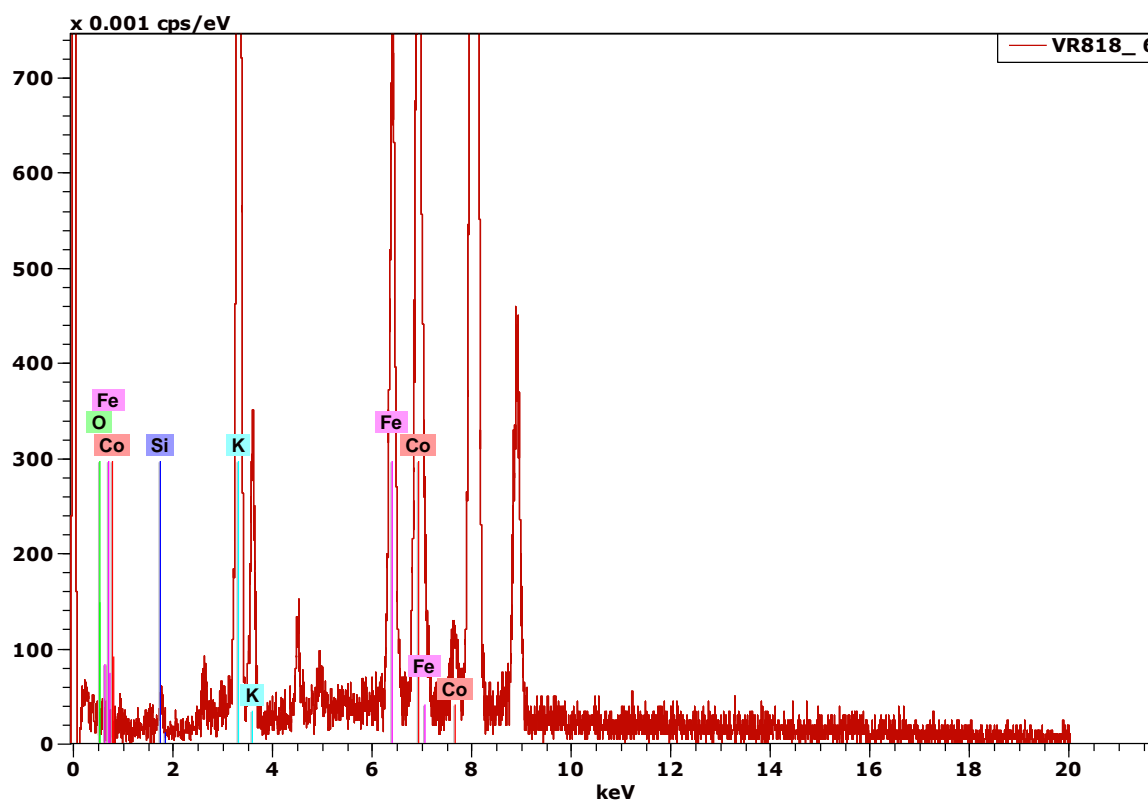


Figure S8. EDX analysis of catalyst **B** nanoparticles observed in Figure 1h.

When catalyst **A** was prepared without silane, from the reaction of $\text{Co}(\text{OAc})_2$ with NaBHET_3 , piles of various sizes and thicknesses were also observed with various kinds of shapes, some area being crystalline (Figure S9).

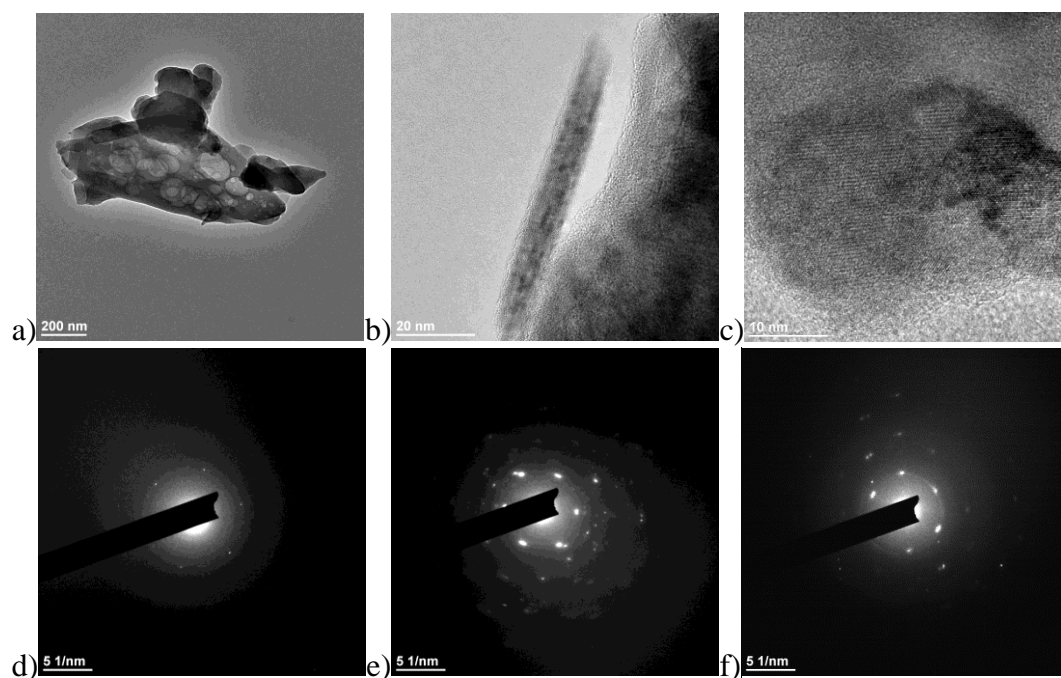


Figure S9. TEM images and diffraction patterns of nanoparticles of catalyst **A** prepared without silane (correlations between a and d, b and e, c and f).

If nanoparticles appeared crystallised at HRTEM, indexations of the diffraction patterns were not obvious because of other orientation. Indexation for CoFe_2O_4 (-1,-3,1) was possible (Figure S10) but not for Co alone.

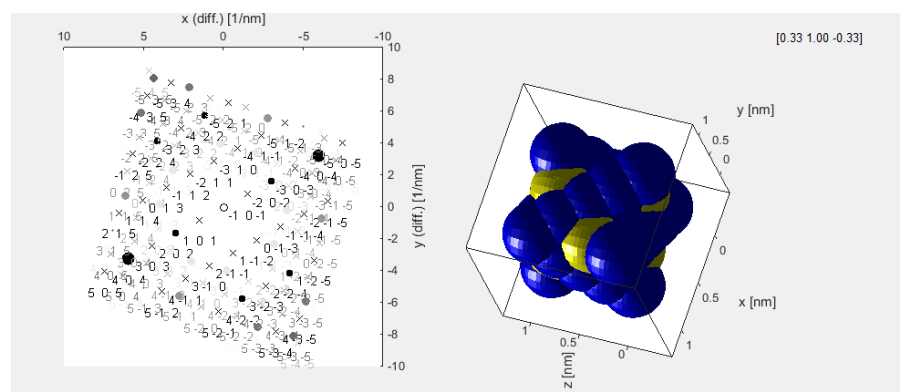


Figure S10. Possible indexation for CoFe_2O_4 (-1,-3,1) of diffraction patterns of nanoparticles observed in Figure S8 of catalyst **A** prepared without silane.

An indexation for CoO (-1, 4,2) was also possible (Figure S11).

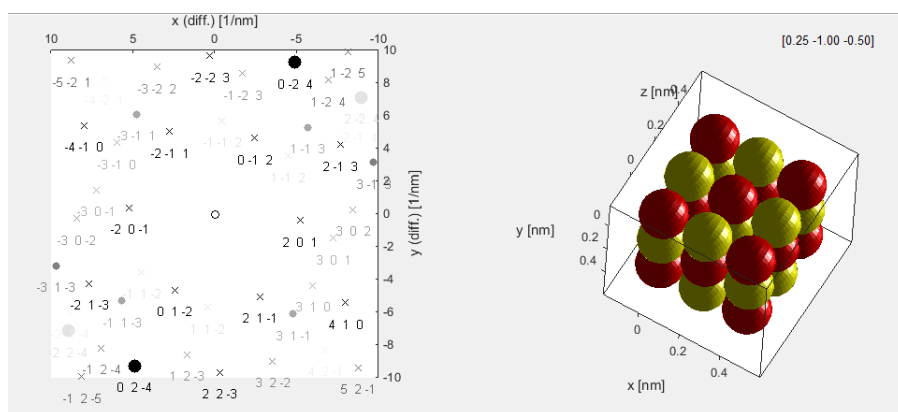


Figure S11. Possible indexation for CoO (-1, 4,2) of diffraction patterns of nanoparticles observed in Figure S8 of catalyst A prepared without silane.

When catalyst **B** was prepared without silane, from the reaction of CoCl_2 with $\text{KO}t\text{Bu}$, NaBHET_3 and water, piles of various sizes and thicknesses were also observed at low magnification with various kinds of shapes. In most cases, crystalline piles (Figure S12a), needles (Figure S12b) and sheets (Figure S12c) were observed.

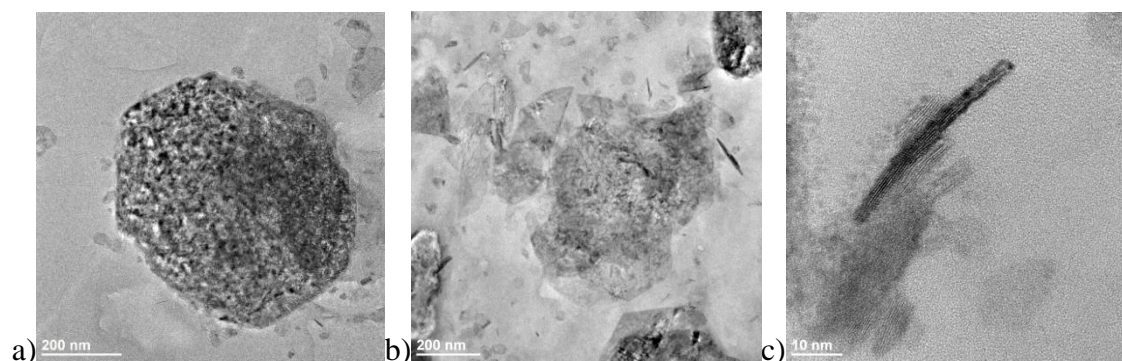


Figure S12. TEM images of nanoparticles of catalyst **B** prepared without silane.

If piles and needles were formed of various but randomly orientated crystals, sheets were rather big areas with 1 to 3 orientations. It was difficult to define the crystals composition as EDX spectra showed the presence of various elements (Co, Cl, Fe, K, Na, Si...) with different ratios depending on the analysed areas. While magnifying, the crystalline aspect was confirmed by the appearance of fringes and atomic arrangements on the different piles, sheets and needles (Figure S13). Nanoparticles appeared also crystallised by diffraction (Figure S14). Quality of the diffractions was highly dependent of the sample orientation. Modification of the later was only possible along the X axis due to the type of sample holder that was used. Because of the presence of many elements according EDX analyses, these well-defined diffraction patterns were not allowing to retrieve the crystals orientation. Possible indexations were found for Co (0,0,1) (Figure S15) and for CoCl_2 (-4,-5,0) (Figure S16).

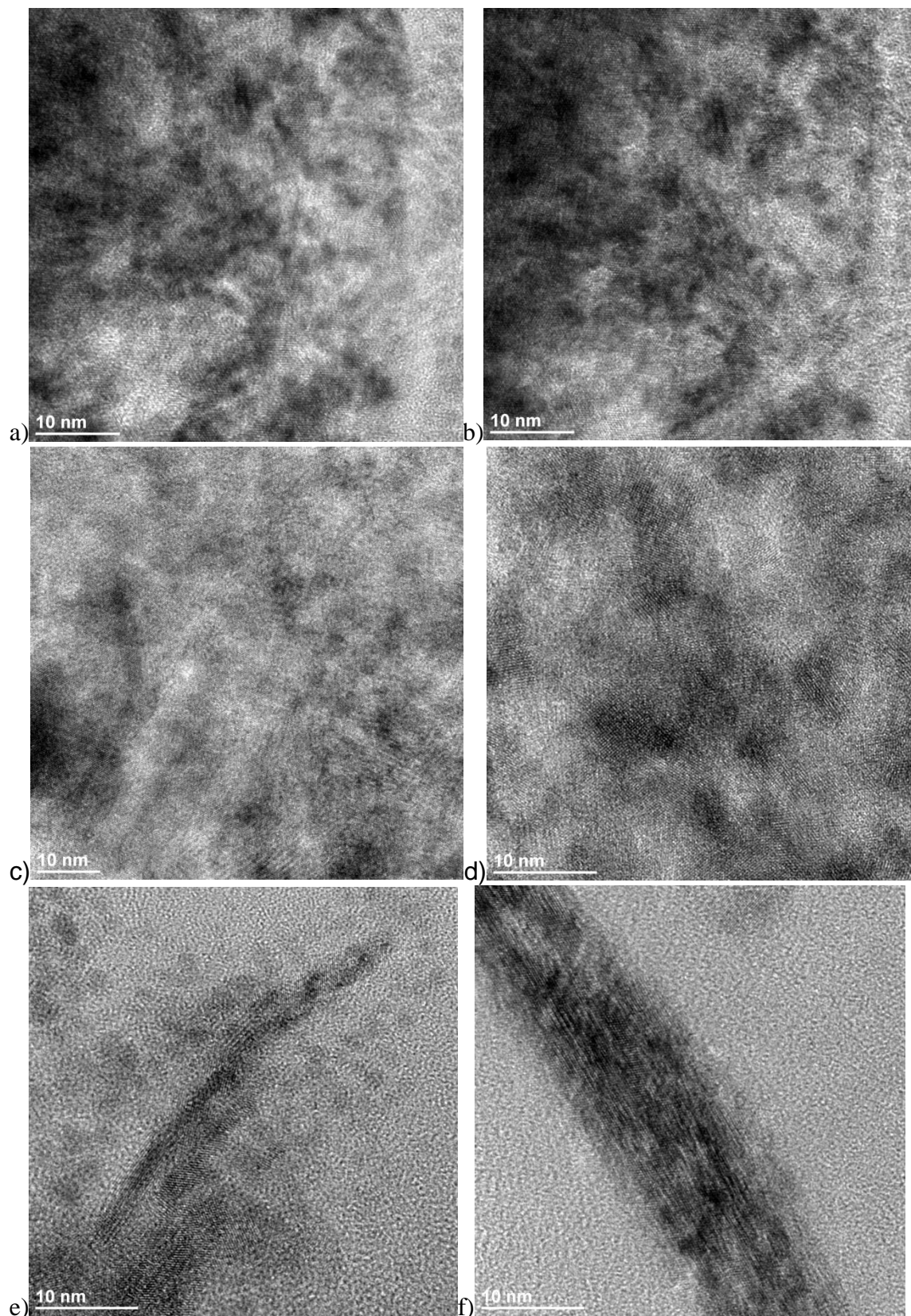


Figure S13. TEM images of nanoparticles of catalyst **B** prepared without silane (images a) and b) for piles, c) and d) for sheets, e) and f) for needles).

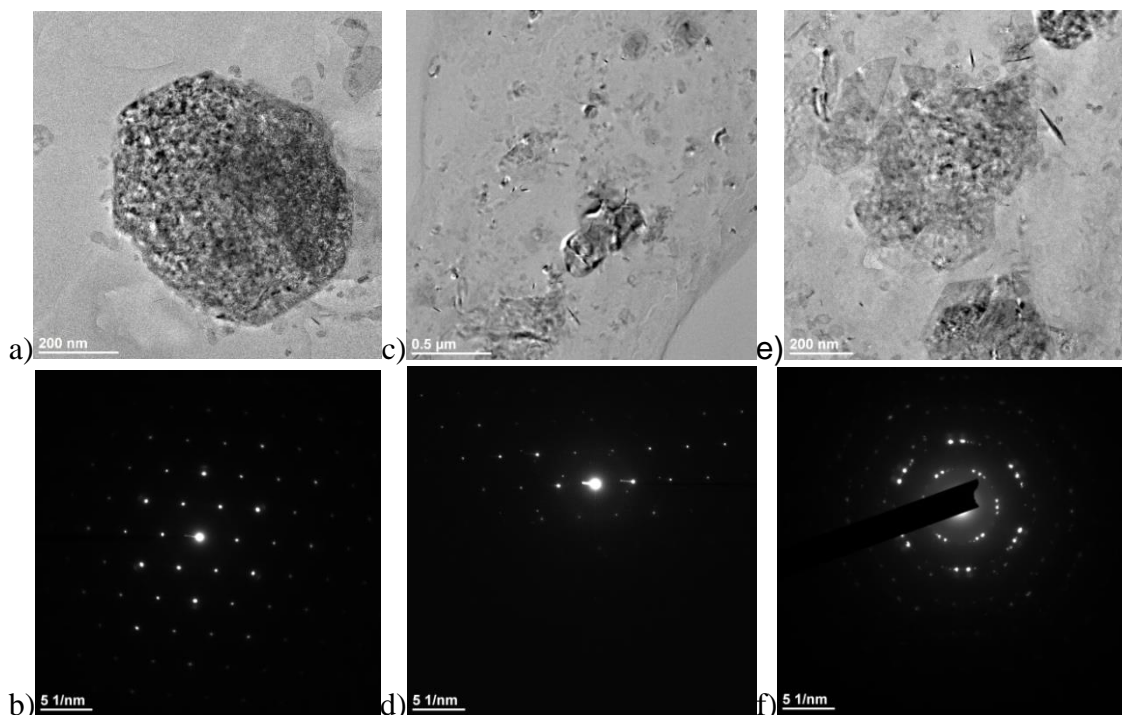


Figure S14. TEM images and diffraction patterns of nanoparticles of catalyst **B** prepared without silane (correlations between a and b, c and d, e and f).

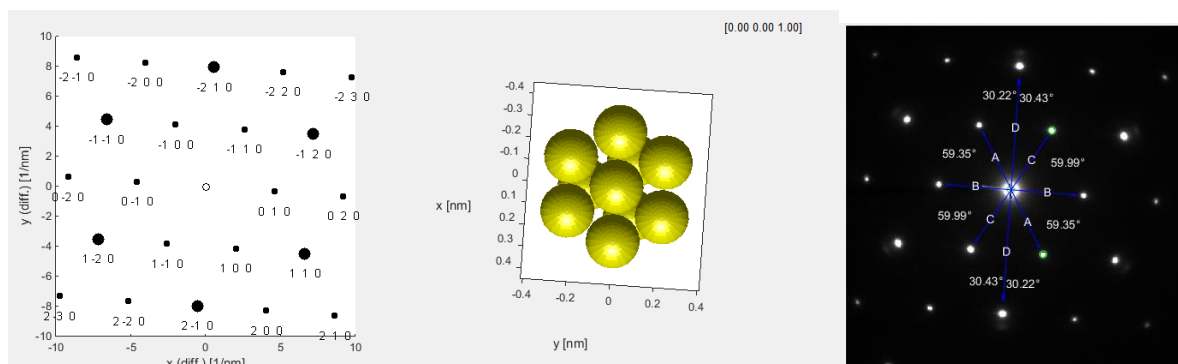


Figure S15. Possible indexing for Co (0,0,1) of diffraction patterns of nanoparticles observed for catalyst **B** prepared without silane.

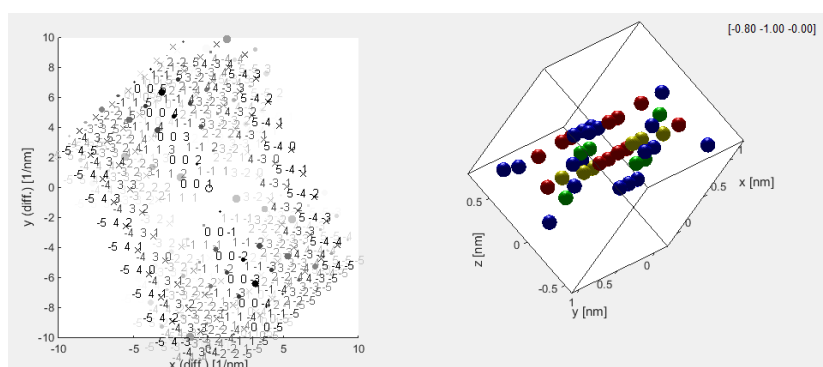


Figure S16. Possible indexing for CoCl_2 (-4,-5,0) of diffraction patterns of nanoparticles observed for catalyst **B** prepared without silane.

It was worth to note sample was not stable enough as nanoparticles were rapidly becoming amorphous or even melting upon effect of the 200kV beam (Figure S17a before analysis and S17b after analysis).

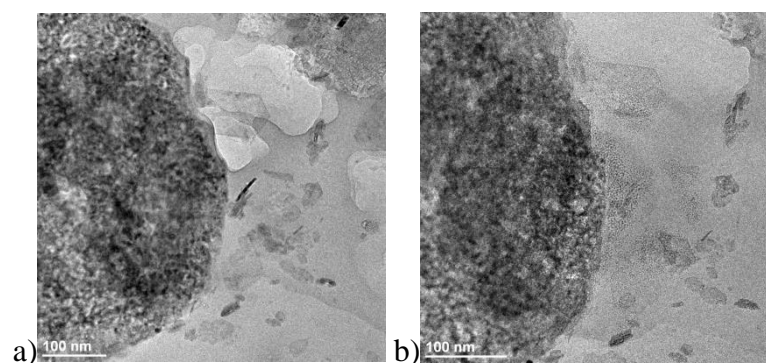


Figure S17. Negative effect of the 200kV beam on nanoparticles observed for catalyst **B** prepared without silane.

V) X-ray fluorescence analyses.

The presence of Co and Si elements as well as Fe, P and Cl impurities was also detected by X-ray fluorescence analyses of catalysts **A** and **B** (Figure S18). It was worth to note K element value was not reliable here. Indeed, the signal was shifting at the same value as one peak of the spectrometer's Pd anode.

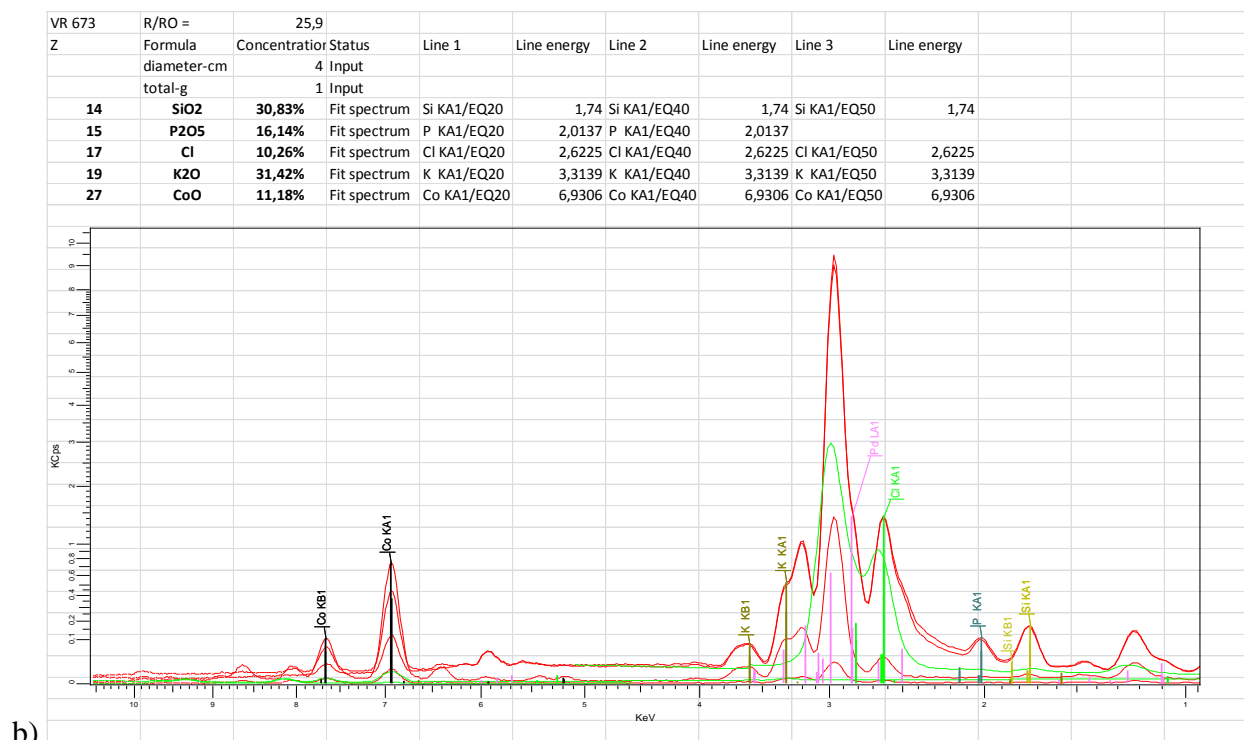
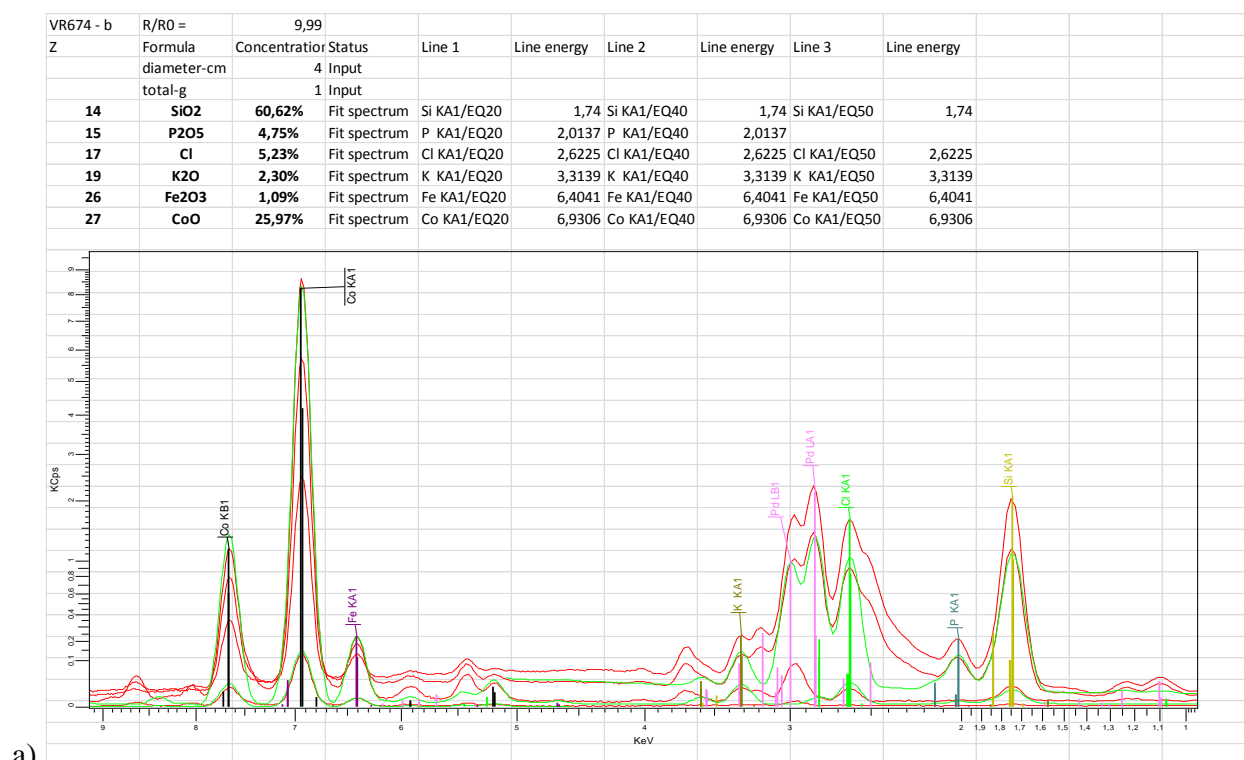


Figure S18. a) X-ray fluorescence analyses of **cat. A** based on $\text{Co}(\text{OAc})_2$, NaBHET_3 and phenylsilane; b) X-ray fluorescence analyses of **cat. B** based on CoCl_2 , $\text{KO}t\text{Bu}$, NaBHET_3 , water and phenylsilane.

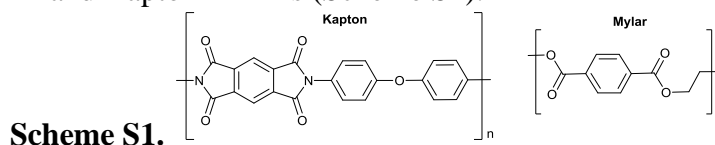
VI) X-ray diffraction analyses.

Two samples were first analysed:

- Catalyst **A** resulting from the reaction of $\text{Co}(\text{OAc})_2$ with NaBHET_3 and phenylsilane.

- Catalyst **B** resulting from the reaction of CoCl_2 with KO^tBu , NaBHET_3 , water and phenylsilane

Analyses were first performed in reflexion mode by protecting sample powders from oxygen and water of air using Mylar™ and Kapton™ films (Scheme S1).



Absorbance of the Mylar™ film was too important while measuring the catalyst **B** based on CoCl_2 (Figure S19).

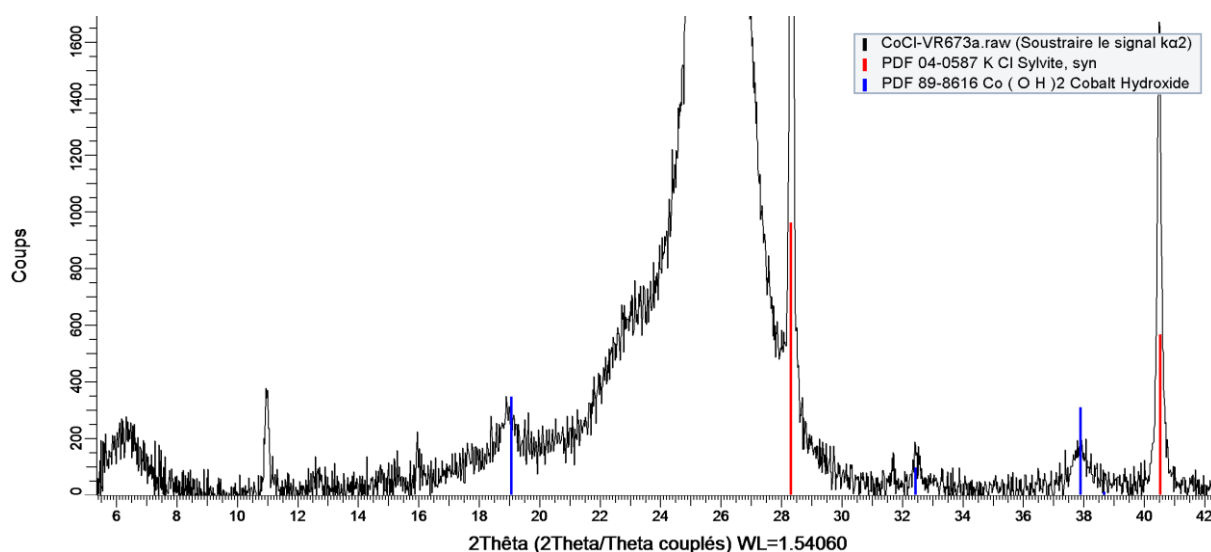


Figure S19. Diffraction by reflexion of catalyst **B** based on CoCl_2 using a Mylar™ film.

The use of a Kapton™ film proved to reduce significantly the absorbance but the diffraction patterns remained unclear between 10 to 30 2θ (Figure S20).

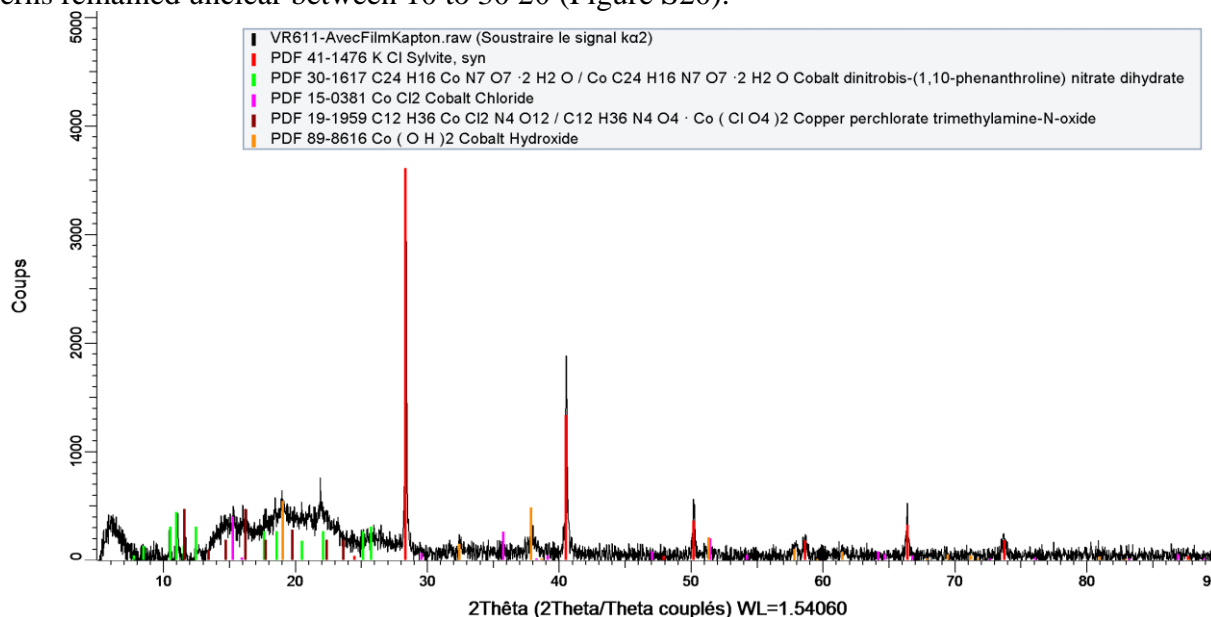


Figure S20. Diffraction by reflexion of catalyst **B** based on CoCl_2 using a Kapton™ film.

Finally, without any film, the diffraction analysis by reflexion of catalyst **B** proved to be better resolved without any significant decomposition of the sample (Figure S21). Diffraction patterns were better identified and likely to be ascribed to KCl, $\text{Co}(\text{OH})_2$ and CoCl_2 .

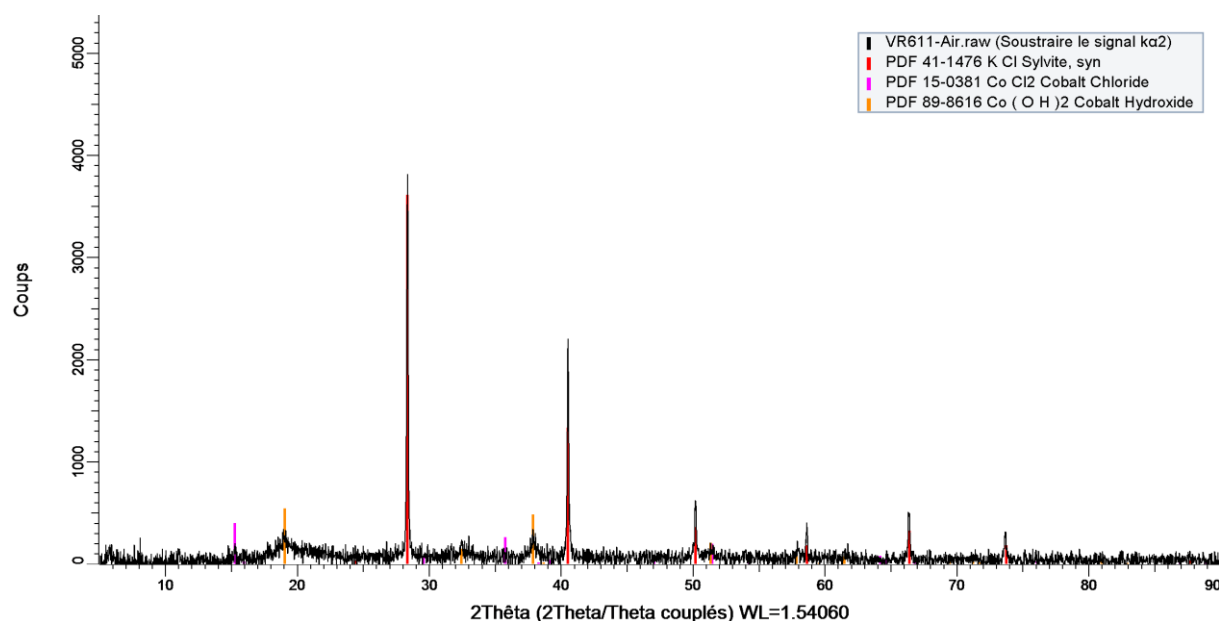


Figure S21. Diffraction by reflexion of catalyst **B** based on CoCl_2 using no film.

In the case of catalyst **A** based on $\text{Co}(\text{OAc})_2$, the diffraction analysis by reflexion showed only diffraction patterns of poor intensity that were likely to be ascribed to sodium acetate (Figure S22).

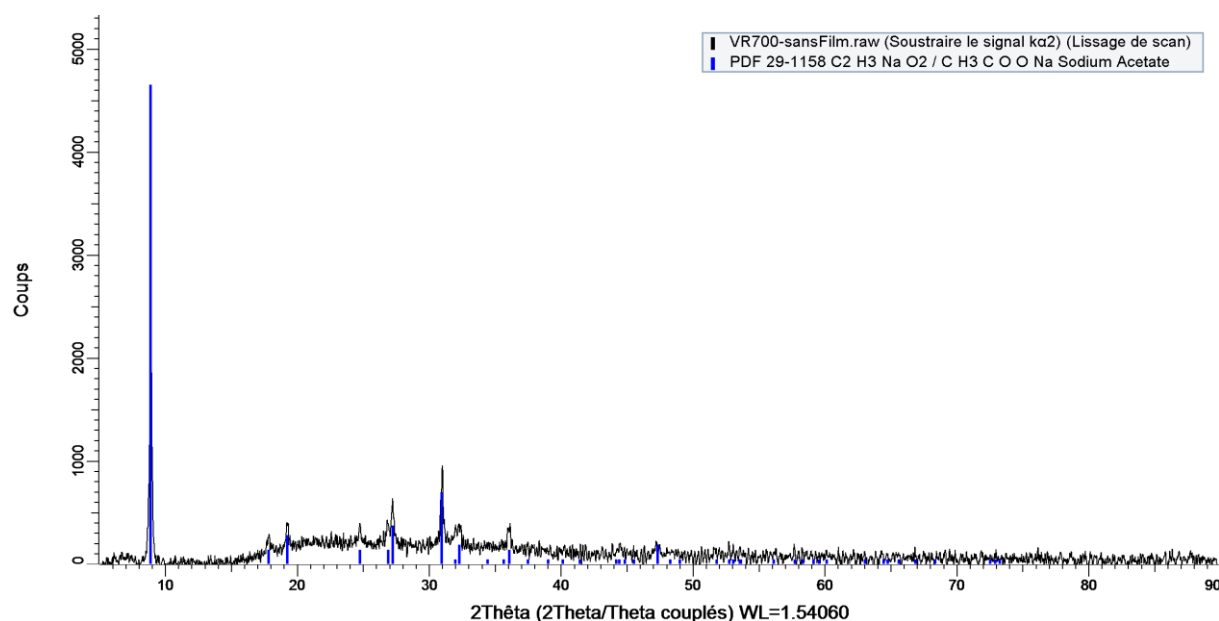


Figure S22. Diffraction by reflexion of catalyst **A** based on $\text{Co}(\text{OAc})_2$ using no film.

A better resolution was obtained for both samples using another diffractometer operating by transmission mode (see manuscript Figures 2a and 2b). Samples were prepared in a glovebox by deposition of the catalyst powders in degassed mineral oil between two sheets of KaptonTM film.

Two other samples were then analysed by transmission mode:

- Catalyst **A** resulting from the reaction of $\text{Co}(\text{OAc})_2$ with NaBHET_3 but no phenylsilane (Figure S23).

- Catalyst **B** resulting from the reaction of CoCl_2 with $\text{KO}t\text{Bu}$, NaBHET_3 and water but no phenylsilane (Figure S24).

The observed diffraction patterns were similar to the catalysts **A** and **B** prepared with phenylsilane.

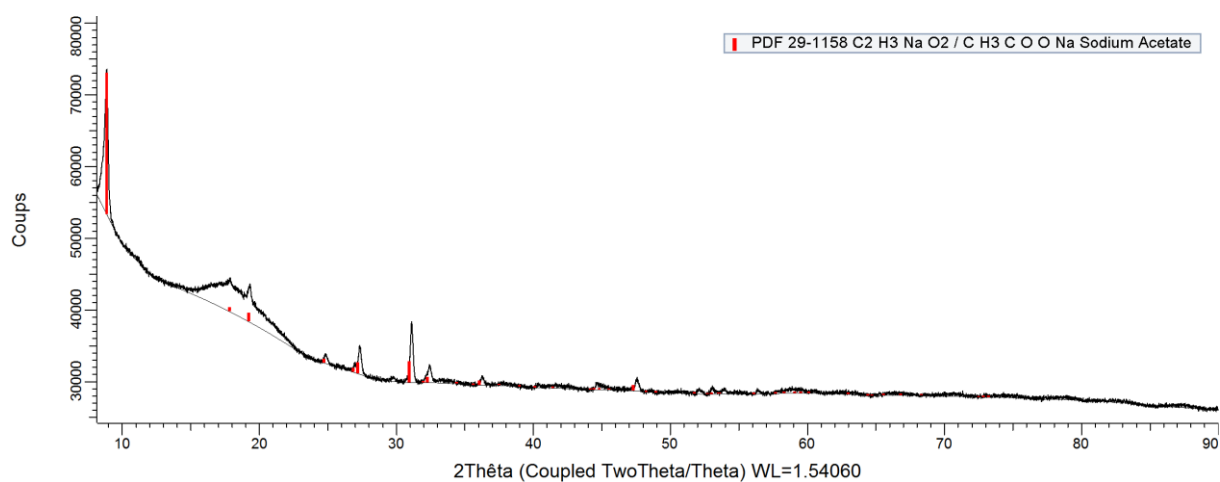


Figure S23. Diffraction by transmission of catalyst **A** based on $\text{Co}(\text{OAc})_2$ prepared without silane.

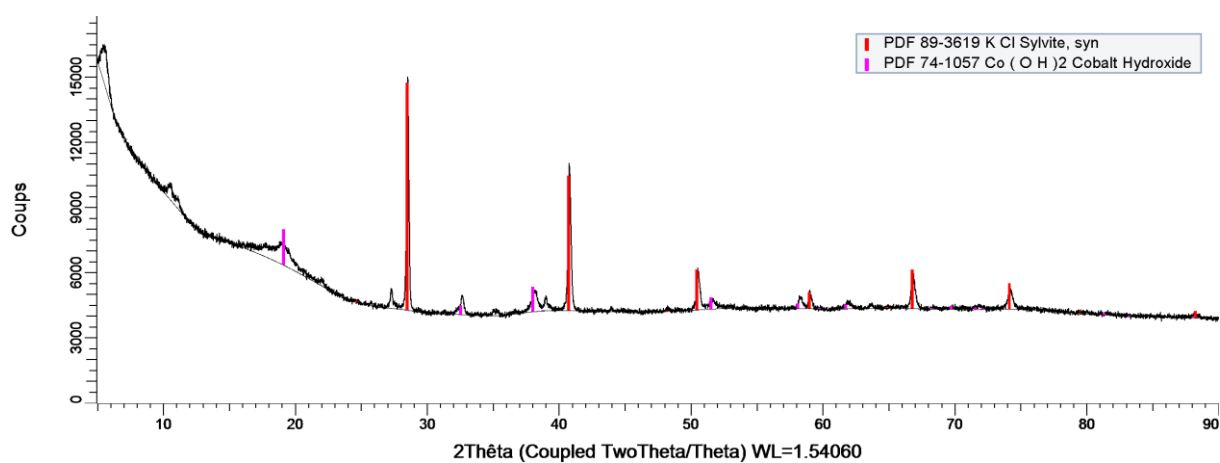


Figure S24. Diffraction by transmission of catalyst **B** based on CoCl_2 prepared without silane.

VII) Infra-red analyses.

Infra-red analyses were realised by Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS). In the case of the catalyst **B** resulting from the reaction of CoCl_2 with $\text{KO}t\text{Bu}$, NaBHET_3 , water and phenylsilane, a strong band is observed at 3631 cm^{-1} corresponding to the stretches of O-H bonds from Co(OH)_2 (Figure S25). Stretches of aromatic and aliphatic C-H bonds were observed between 3100 and 2800 cm^{-1} .

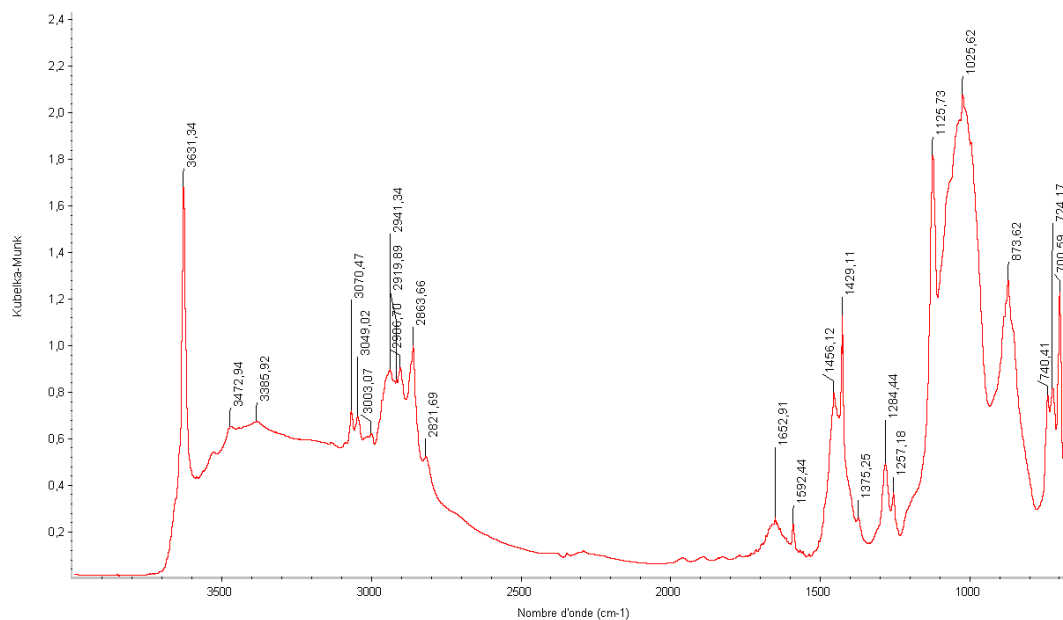


Figure S25. DRIFTS spectrum of the catalyst **B** resulting from the reaction of CoCl_2 with $\text{KO}t\text{Bu}$, NaBHET_3 , water and phenylsilane

In the case of the catalyst **A** resulting from the reaction of Co(OAc)_2 with NaBHET_3 and phenylsilane, stretches of aromatic and aliphatic C-H bonds were observed between 3100 and 2800 cm^{-1} (Figure S26). Carbonyl stretches from the acetate were observed at 1578 cm^{-1} .

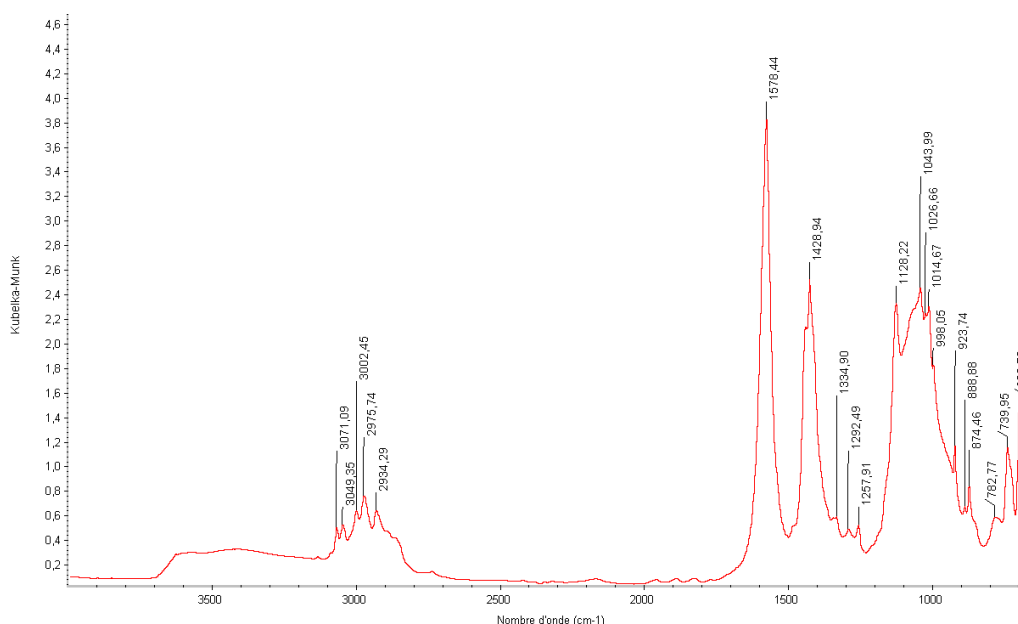


Figure S26. DRIFTS spectrum of the catalyst **A** resulting from the reaction of Co(OAc)_2 with NaBHET_3 and phenylsilane.

VIII) XPS analyses.

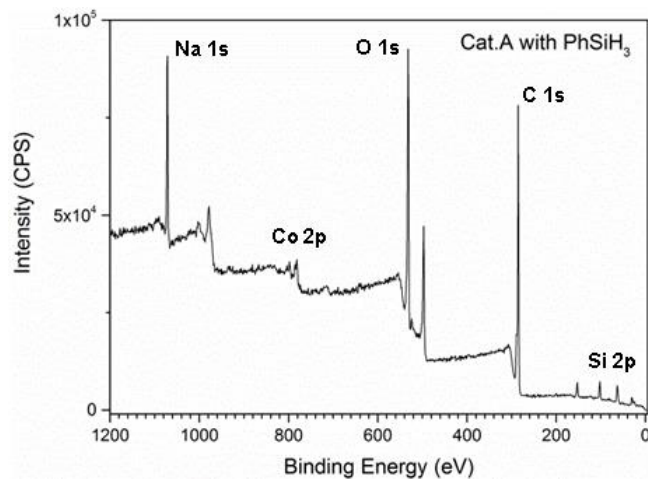


Figure S27. XPS full-scan spectrum of the catalyst **A** resulting from the reaction of Co(OAc)₂ with NaBHET₃ and phenylsilane.

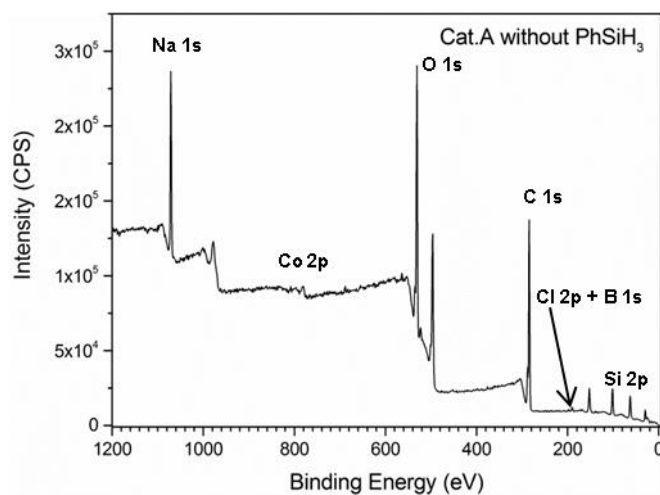


Figure S28. XPS full-scan spectrum of the catalyst **A** resulting from the reaction of Co(OAc)₂ with NaBHET₃ but without phenylsilane.

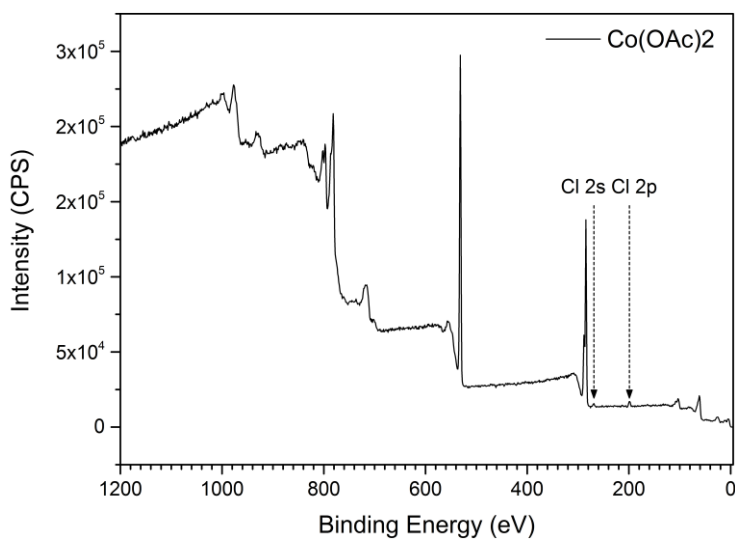


Figure S29. Pollution of Co(OAc)₂ (reagent grade) used in the catalysis by chloride

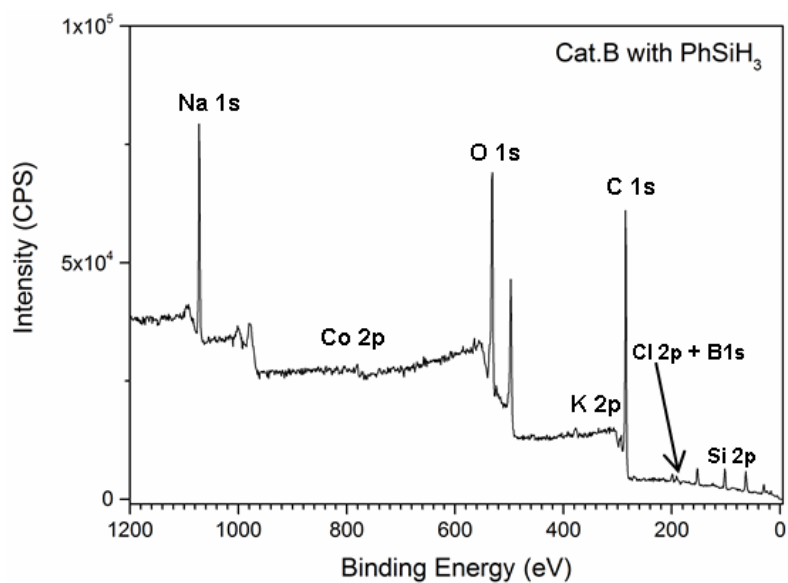


Figure S30. XPS full-scan spectrum of the catalyst **B** resulting from the reaction of CoCl_2 with $\text{KO}t\text{Bu}$, NaBHET_3 , water and phenylsilane.

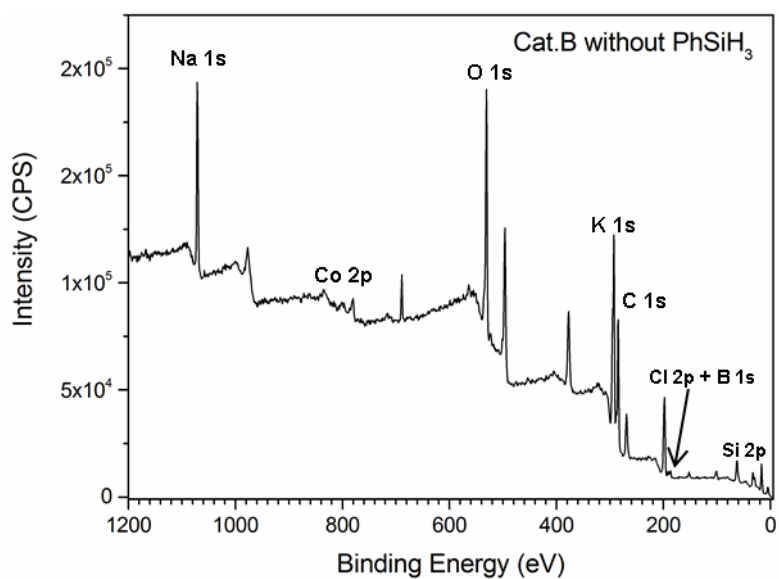
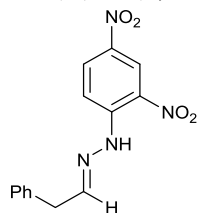


Figure S31. XPS full-scan spectrum of the catalyst **B** resulting from the reaction of CoCl_2 with $\text{KO}t\text{Bu}$, NaBHET_3 , water and without phenylsilane.

IX) Characterization of compounds.

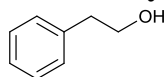
6a (*E*)-1-(2,4-dinitrophenyl)-2-(2-phenylethylidene)hydrazine CAS [2074-04-6]^{S2}



Isolated as the 2,4-dinitrophenylhydrazone adduct by precipitation, 92% yield.

¹H NMR (300 MHz, CDCl₃): δ = 11.05 (s, 1H, NH), 9.11 (d, J = 1.0 Hz, 1H_{Ar}), 8.31 (ddd, J = 9.6, 2.6, 0.7 Hz, 1H_{Ar}), 7.99 (d, J = 9.6 Hz, 1H_{Ar}), 7.60 (td, J = 5.8, 0.6 Hz, 1H_{Ar}), 7.33 (m, 4H_{Ar}), 3.76 (d, J = 5.8 Hz, 2H). ¹³C NMR (75 MHz, CDCl₃): δ = 150.4 (CH), 145.3 (C), 138.1 (C), 135.6 (C), 133.4 (C), 130.1 (CH_{Ar}), 129.2 (2CH_{Ar}), 129.1 (2CH_{Ar}), 127.4 (CH_{Ar}), 123.6 (CH_{Ar}), 116.7 (CH_{Ar}), 39.2 (CH₂).

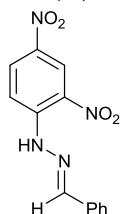
7a 2-Phenylethan-1-ol CAS [60-12-8]^{S3}



Isolated as a colorless oil after flash chromatography on silica gel using a (85/15) petroleum ether and diethyl ether mixture (R_f = 0.75), 94% yield.

¹H NMR (300 MHz, CDCl₃): δ = 7.40 (m, 2H_{Ar}), 7.32 (m, 3H_{Ar}), 3.87 (t, J = 6.8 Hz, 2H), 2.92 (t, J = 6.8 Hz, 2H), 2.73 (s, 1H). ¹³C NMR (75 MHz, CDCl₃): δ = 138.6 (C), 129.0 (2CH), 128.5 (2CH), 126.4 (CH), 63.5 (CH₂), 39.1 (CH₂).

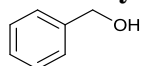
6e (*E*)-1-benzylidene-2-(2,4-dinitrophenyl)hydrazine CAS [1157-84-2]^{S4}



Isolated as the 2,4-dinitrophenylhydrazone adduct by precipitation, 95% yield.

¹H NMR (300 MHz, DMSO): δ = 11.68 (s, 1H, NH), 8.89 (d, J = 2.6 Hz, 1H), 8.72 (s, 1H_{Ar}), 8.37 (dd, J = 9.6, 2.7 Hz, 1H_{Ar}), 8.13 (s, J = 9.6 Hz, 1H_{Ar}), 7.81 (dd, J = 7.7, 5.1 Hz, 2H_{Ar}), 7.51 (dd, J = 5.2, 1.7 Hz, 3H_{Ar}). ¹³C NMR (75 MHz, DMSO): δ = 149.4 (CH), 144.6 (C), 137.0 (C), 133.8 (C), 130.5 (CH_{Ar}), 129.7 (CH_{Ar}), 129.53 (C), 128.9 (2CH_{Ar}), 127.3 (2CH_{Ar}), 123.0 (CH_{Ar}), 116.8 (CH_{Ar}).

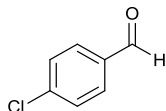
7e Phenylmethanol CAS [100-51-6]^{S5}



Isolated as a colorless oil after flash chromatography on silica gel using a (9/1) dichloromethane and diethylether mixture (R_f = 0,6), 96% yield.

¹H NMR (300 MHz, CDCl₃): δ = 7.40 (m, 5H_{Ar}), 4.64 (d, J = 5.5 Hz, 2H), 3.48 (m, 1H). ¹³C NMR (75 MHz, CDCl₃): δ = 140.9 (C), 128.4 (2CH), 127.4 (CH), 127.0 (2CH), 64.8 (CH₂).

6g 4-chlorobenzaldehyde CAS [104-88-1]^{S6}

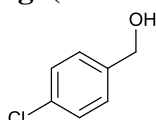


Isolated as a colorless oil after flash chromatography on silica gel using a (25/75) petroleum ether and dichloromethane mixture ($R_f = 0.6$), 84% yield.

^1H NMR (300 MHz, CDCl_3): $\delta = 9.96$ (d, $J = 1.0$ Hz, 1H, CHO), 7.79 (m, 2H), 7.48 (m, 2H).

^{13}C NMR (75 MHz, CDCl_3): $\delta = 190.9$ (CHO), 141.0 (C), 134.8 (C), 131.0 (2CH), 129.5 (2CH).

7g (4-chlorophenyl)methanol CAS [873-76-7]^{S7}

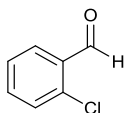


Isolated as a colorless oil after flash chromatography on silica gel using dichloromethane ($R_f = 0.4$), 98% yield.

RMN ^1H (CDCl_3 , 300 MHz): $\delta = 1.66$ (bs, 1H, OH), 4.67 (s, 2H, CH_2), 7.31 (m, 4H_{Ar}).

RMN ^{13}C (CDCl_3 , 75 MHz): $\delta = 64.6$ (CH_2), 128.4 (2 CH), 128.8 (2 CH), 133.4 (C), 139.3 (C).

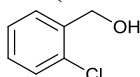
6h 2-chlorobenzaldehyde CAS [89-98-5]^{S6}



Isolated as a colorless oil after flash chromatography on silica gel using a (25/75) petroleum ether and dichloromethane mixture ($R_f = 0.5$), 49% yield.

^1H NMR (300 MHz, CDCl_3): $\delta = 10.37$ (s, 1H, CHO), 7.81 (d, $J = 7.7$ Hz, 1H_{Ar}), 7.44 (m, 1H_{Ar}), 7.32 (m, 2H_{Ar}). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 189.3$ (CHO), 137.6 (C), 134.9 (CH), 132.3 (C), 130.4 (CH), 129.1 (CH), 127.1 (CH).

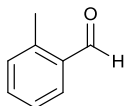
7h (2-Chlorophenyl)methanol CAS [17849-38-6]^{S8}



Isolated as a white solid after flash chromatography on silica gel using a (85/15) petroleum ether and diethylether mixture ($R_f = 0.5$), 97% yield.

^1H NMR (300 MHz, CDCl_3): $\delta = 7.34$ (dd, $J = 7.0, 2.3$ Hz, 1H), 7.23 (dd, $J = 7.3, 1.7$ Hz, 1H), 7.13 (m, 2H), 4.62 (s, 2H), 2.67 (s, 1H). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 138.3$ (C), 132.7 (C), 129.4 (CH), 128.8 (CH), 128.7 (CH), 127.0 (CH), 62.7 (CH_2).

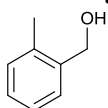
6i 2-methylbenzaldehyde CAS [529-20-4]^{S6}



Isolated as a colorless oil after flash chromatography on silica gel using a (9/1) petroleum ether and diethyl ether mixture ($R_f = 0.65$), 89% yield.

$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 10.27$ (s, 1H, CHO), 7.80 (dd, $J = 7.6\text{-}1.4$ Hz, 1H_{Ar}), 7.47 (td, $J = 7.5\text{-}1.5$ Hz, 1H_{Ar}), 7.35 (t, $J = 7.5$ Hz, 1H_{Ar}), 7.25 (d, $J = 3.3$ Hz, 1H_{Ar}), 2.67 (s, 3H, CH_3).
 $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 192.9$ (CHO), 140.7 (C), 134.5 (C), 133.9 (CH), 132.3 (CH), 132.1 (CH), 126.6 (CH), 19.7 (CH_3).

7i o-tolylméthanol CAS [89-95-2]^{S9}

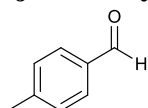


Isolated as a colorless oil after flash chromatography on silica gel using a (9/1) dichloromethane and diethylether mixture ($R_f = 0.5$), 92% yield.

RMN ^1H (CDCl_3 , 300 MHz): $\delta = 1.65$ (bs, 1H, OH), 2.37 (s, 3H), 4.70 (s, 2H), 7.23 (m, 3H), 7.35 (m, 1H).

RMN ^{13}C (CDCl_3 , 75 MHz): $\delta = 18.8$ (CH_3), 63.7 (CH_2), 126.2 (CH), 127.7 (CH), 127.9 (CH), 130.5 (CH), 136.3 (C), 138.8 (C).

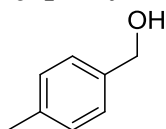
6j 4-methylbenzaldehyde CAS [104-87-0]^{S10}



Isolated as a colorless oil after flash chromatography on silica gel using a (9/1) petroleum ether and diethyl ether mixture ($R_f = 0.7$), 92% yield.

$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 9.96$ (s, 1H, CHO), 7.78 (d, $J = 8.1$ Hz, 2H_{Ar}), 7.32 (d, $J = 8.1$, 2H_{Ar}), 2.43 (s, 3H, CH_3). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 192.1$ (CHO), 145.7 (C), 134.6 (C), 130.0 (4CH), 22.0 (CH_3).

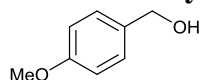
7j p-tolylméthanol CAS [589-18-4]^{S11}



Isolated as a colorless oil after flash chromatography on silica gel using a (9/1) petroleum ether and diethyl ether mixture ($R_f = 0.4$), 86% yield.

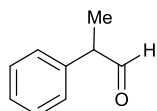
RMN ^1H (CDCl_3 , 300 MHz): $\delta = 1.62$ (bs, 1H, OH), 2.35 (s, 3H), 4.65 (s, 2H), 7.18 (d, $J = 7.2$ Hz, 2H), 7.27 (d, $J = 8.0$ Hz, 2H).

RMN ^{13}C (CDCl_3 , 75 MHz): $\delta = 21.3$ (CH_3), 65.4 (CH_2), 127.3 (2CH), 129.4 (2CH), 137.6 (C), 138.1 (C).

7k 4-Methoxyphenylmethanol CAS [105-13-5]^{S5}

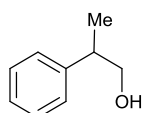
Isolated as a white solid after flash chromatography on silica gel using a (3/7) petroleum ether and dichloromethane ($R_f = 0.7$), 83% yield.

$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.20$ (m, 2H_{Ar}), 6.80 (m, 2H_{Ar}), 4.52 (s, 2H), 3.72 (s, 3H), 1.80 (s, 1H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 159.3$ (C), 133.2 (C), 128.8 (2CH), 114.0 (2CH), 65.1 (CH_2), 55.4 (CH_3).

6m 2-Phenylpropionaldehyde CAS [93-53-8]^{S12}

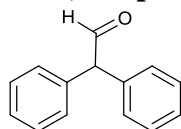
Isolated as a white solid after flash chromatography on silica gel using a (9/1) petroleum ether and diethyl ether mixture ($R_f = 0.2$), 83% yield

$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 9.74$ (d, $J = 1.4$ Hz, 1H, CHO), 7.40 (m, 3H_{Ar}), 7.27 (m, 2H_{Ar}), 3.69 (dq, $J = 7.1, 1.3$ Hz, 1H), 1.50 (d, $J = 7.1$ Hz, 3H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 201.0$ (CHO), 137.8 (C), 129.1 (2CH), 128.4 (2CH), 127.5 (CH), 53.0 (CH), 14.6 (CH_3).

7m 2-Phenylpropionaldehyde CAS [93-53-8]^{S13}

Isolated as a white solid after flash chromatography on silica gel using a (6/4) petroleum ether and diethyl ether mixture ($R_f = 0.5$), 92% yield

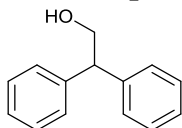
$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.40$ (m, 2H, 2CH_{Ar}), 7.31 (m, 3H_{Ar}), 3.72 (m, 2H), 2.98 (q, $J = 6.9$ Hz, 1H), 2.66 (s broad, OH), 1.35 (d, $J = 7.0$ Hz, 3H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 143.9$ (C), 128.5 (2CH), 127.5 (2CH), 126.5 (CH), 68.5 (CH_2), 42.3 (CH), 17.6 (CH_3).

6n 2,2-Diphenylacetaldehyde CAS [947-91-1]^{S14}

Isolated as a white solid after flash chromatography on silica gel using a (95/5) petroleum ether and dichloromethane mixture ($R_f = 0.5$), 90% yield.

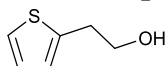
$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 10.04$ (m, 1H, CHO), 7.45 (m, 6H_{Ar}), 7.33 (m, 4H_{Ar}), 4.99 (b, 1H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 198.6$ (CHO), 136.5 (C), 129.3 (4CH), 129.1 (4CH), 127.7 (2CH), 64.17 (CH).

7n 2,2-diphenylethanol CAS [1883-32-5]^{S15}



Isolated as a colorless oil after flash chromatography on silica gel using a (60/40) petroleum ether and diethyl ether mixture ($R_f = 0.5$), 51% yield $^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.38$ (m, 10H_{Ar}), 4.30 (m, 1H), 4.22 (m, 2H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 141.6$ (C), 128.7 (4CH), 128.4 (4CH), 126.8 (2CH), 66.0 (CH_2), 53.6 (CH).

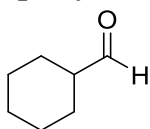
7o 2-Thiophenethanol CAS [5402-55-1]^{S16}



Isolated as a colorless oil after flash chromatography on silica gel using a (6/4) petroleum ether and diethyl ether mixture ($R_f = 0.5$), 92% yield.

$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 7.18$ (dd, $J = 5.1, 1.2$ Hz, 1H_{Ar}), 6.96 (dd, $J = 5.1, 3.3$ Hz, 1H_{Ar}), 6.88 (m, 1H_{Ar}), 3.87 (t, $J = 6.3$ Hz, 2H), 3.09 (dt, $J = 6.3, 0.8$ Hz, 2H), 2.10 (b, 1H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 140.9$ (C), 127.2 (CH), 125.8 (CH), 124.2 (CH), 63.7 (CH_2), 33.5 (CH_2).

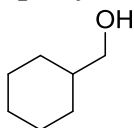
6p Cyclohexanecarbaldehyde CAS [2043-61-0]^{S17}



Isolated as a colorless oil after flash chromatography on silica gel using a (6/4) petroleum spirit and dichloromethane mixture ($R_f = 0.4$), 82 % yield.

$^1\text{H NMR}$ (300 MHz, CDCl_3): $\delta = 9.52$ (s, 1H, CHO), 2.12 (m, 1H), 1.68 (m, 6H), 1.23 (m, 3H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): $\delta = 204.7$ (CHO), 49.8 (CH), 25.9 (2CH_2), 25.9 (CH_2), 24.95 (2CH_2).

7p Cyclohexylmethanol CAS [100-49-2]^{S18}

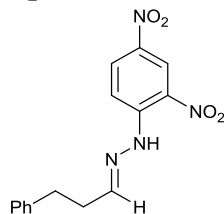


Isolated as a colorless oil after flash chromatography on silica gel using a (8/2) petroleum ether and diethyl ether mixture ($R_f = 0.8$), 84% yield.

RMN ^1H (CDCl_3 , 300 MHz): $\delta = 0.94$ (m, 2H), 1.21 (m, 3H), 1.48 (m, 2H), 1.71 (m, 5H), 3.43 (d, $J = 6.4$ Hz, 2H).

RMN ^{13}C (CDCl_3 , 75 MHz): $\delta = 26.0$ (2CH_2), 26.7 (CH_2), 29.7 (2CH_2), 40.6 (CH), 68.9 (CH_2).

6q (E)-1-(2,4-dinitrophenyl)-2-(3-phenylpropylidene)hydrazine CAS [1237-68-9]^{S19}

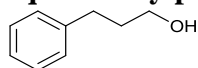


Isolated as the 2,4-dinitrophenylhydrazone adduct by precipitation, 92% yield.

¹H NMR (300 MHz, CDCl₃): δ= 11.00 (s, 1H, NH), 9.11 (d, *J*= 2.6 Hz, 1H), 8.29 (ddd, *J*= 9.6, 2.6, 0.6 Hz, 1H_{Ar}), 7.90 (d, *J*= 9.6 Hz, 1H_{Ar}), 7.56 (t, *J*= 4.9 Hz, 1H_{Ar}), 7.34 (m, 2H_{Ar}), 7.24 (m, 4H_{Ar}), 2.97 (t, *J*= 4.5 Hz, 2H), 2.79 (m, 2H).

RMN ¹³C (75 MHz, CDCl₃): δ= 151.4 (CH), 145.2 (C), 140.4 (C), 138.1 (C), 130.1 (CH_{Ar}), 129.1 (C), 128.8 (2CH_{Ar}), 128.5 (2CH_{Ar}), 126.6 (CH_{Ar}), 123.6 (CH_{Ar}), 34.1 (CH₂), 32.6 (CH₂).

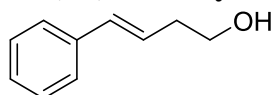
7q 3-Phenylpropan-1-ol CAS [122-97-4]^{S20}



Isolated as a colorless oil after flash chromatography on silica gel using a (85/15) petroleum ether and diethyl ether mixture (R_f = 0.8), 93% yield.

¹H NMR (300 MHz, CDCl₃): δ= 7.22 (m, 2H_{Ar}), 7.12 (m, 3H_{Ar}), 3.58 (t, *J*= 6.5 Hz, 2H), 2.62 (t, *J*= 7.7 Hz, 2H), 1.81 (m, 2H), 1.70 (s, 1H). ¹³C NMR (75 MHz, CDCl₃): δ= 141.9 (C), 128.6 (2CH), 128.5 (2CH), 126.0 (CH), 62.3 (CH₂), 34.3 (CH₂), 32.2 (CH₂).

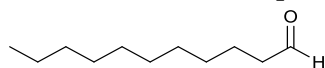
7r (3E)-4-Phenyl-3-buten-1-ol CAS [770-36-5]^{S21}



Isolated as a colorless oil after flash chromatography on silica gel using a (6/4) petroleum ether and diethyl ether mixture (R_f = 0.5), 92% yield

¹H NMR (300 MHz, CDCl₃): δ= 7.22 (m, 5H_{Ar}), 6.43 (d, *J*= 15.9 Hz, 1H), 6.13 (dt, *J*= 15.9, 7.1 Hz, 1H), 3.68 (t, *J*= 6.3 Hz, 2H), 2.42 (ddt, *J*= 7.1, 6.3, 1.3 Hz, 2H), 1.48 (s, 1H). ¹³C NMR (75 MHz CDCl₃): δ= 137.4 (C), 132.9 (CH), 128.7 (2CH_{Ar}), 127.4 (CH_{Ar}), 126.5 (CH), 126.2 (2CH_{Ar}), 62.2 (CH₂), 36.5 (CH₂).

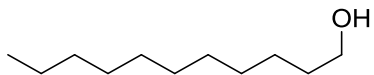
6s Undecanal CAS [112-44-7]^{S22}



Isolated as a colorless oil after flash chromatography on silica gel using a (7/3) petroleum ether and diethyl ether mixture (R_f = 0.5), 90% yield.

¹H NMR (300 MHz, CDCl₃): δ= 9.76 (t, *J*= 1.9 Hz, 1H, CHO), 2.41 (td, *J*= 7.3, 1.9 Hz, 2H), 1.63 (m, 2H), 1.29 (m, 14H), 0.88 (t, *J*= 1.3 Hz, 3H). ¹³C NMR (75 MHz, CDCl₃): δ= 203.1 (CHO), 44.1 (CH₂), 32.0 (CH₂), 29.7 (CH₂), 29.6 (CH₂), 29.5 (CH₂), 29.4 (CH₂), 29.3 (CH₂), 22.8 (CH₂), 22.3 (CH₂), 14.2 (CH₃).

7s Undecan-1-ol CAS [112-42-5]^{S23}

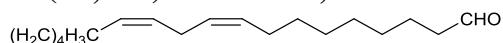


Isolated as a colorless oil after flash chromatography on silica gel using a (7/3) petroleum ether and diethyl ether mixture ($R_f = 0.3$), 92% yield.

RMN ^1H (CDCl_3 , 300 MHz): $\delta = 0.81$ (t, $J = 6.5$ Hz, 3H), 1.19 (m, 16H), 1.58 (m, 2H), 2.91 (bs, 1H, OH), 3.56 (t, $J = 6.7$ Hz, 2H, CH_2O).

RMN ^{13}C (CDCl_3 , 75 MHz): $\delta = 14.3$ (CH_3), 22.9 (CH_2), 25.9 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.8 (3CH_2), 32.1 (CH_2), 33.0 (CH_2), 63.2 ($\text{CH}_2\text{-OH}$).

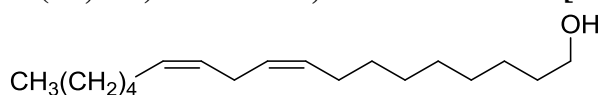
6t (9Z,12Z)-octadeca-9,12-dienal 4s CAS [2541-61-9]^{S24}



Isolated as a colorless oil after flash chromatography on silica gel using a (9/1) dichloromethane and diethyl ether mixture ($R_f = 0.6$), 98% yield.

^1H NMR (300 MHz, CDCl_3): $\delta = 9.77$ (t, $J = 1.9$ Hz, 1H, CHO), 5.35 (m, 4H), 2.77 (t, $J = 6.2$ Hz, 2H), 2.42 (td, $J = 7.3, 1.9$ Hz, 2H), 2.05 (m, 4CH), 1.63 (m, 2H), 1.33 (m, 14H), 0.89 (t, $J = 1.4$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3): $\delta = 203.0$ (CHO), 130.4 (CH), 130.1 (CH), 128.3 (CH), 128.1 (CH), 44.1 (CH_2), 31.7 (CH_2), 29.7 (CH_2), 29.5 (CH_2), 29.4 (CH_2), 29.3 (CH_2), 29.2 (CH_2), 27.4 (CH_2), 27.3 (CH_2), 25.8 (CH_2), 22.7 (CH_2), 22.2 (CH_2), 14.2 (CH_3).

7t (9Z,12Z)-octadeca-9,12-dien-1-ol CAS [506-43-4]^{S25}



Isolated as a colorless oil after flash chromatography on silica gel using a (9/1) dichloromethane and diethyl ether mixture ($R_f = 0.3$), 70% yield.

RMN ^1H (CDCl_3 , 300 MHz): $\delta = 0.89$ (m, 3H), 1.26 (m, 16H), 1.54 (m, 2H, $-\text{CH}_2\text{-CH}_2\text{OH}$), 2.06 (m, 4H, $=\text{CH-CH}_2-$), 2.77 (t, $J = 5.9$ Hz, 2H, $=\text{CH-CH}_2\text{-CH=}$), 3.64 (t, $J = 6.6$ Hz, 2H, $-\text{CH}_2\text{-OH}$), 5.36 (m, 4H, $=\text{CH-}$).

RMN ^{13}C (CDCl_3 , 75 MHz): $\delta = 14.2$ (CH_3), 22.7 (CH_2), 25.8 (CH_2), 25.9 (CH_2), 27.4 (CH_2), 29.3 (CH_2), 29.4 (CH_2), 29.5 (CH_2), 29.6 (CH_2), 29.7 (CH_2), 29.8 (CH_2), 31.7 (CH_2), 33.0 (CH_2), 63.3 (CH_2), 128.1 (CH), 128.2 (CH), 130.3 (CH), 130.4 (CH).

X) References.

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XI) ^1H , ^{13}C NMR spectra of isolated compounds.

