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Synthesis and characterization of zinc and cupper MOFs derived from diazo and oxim ligands for wastewater treatment

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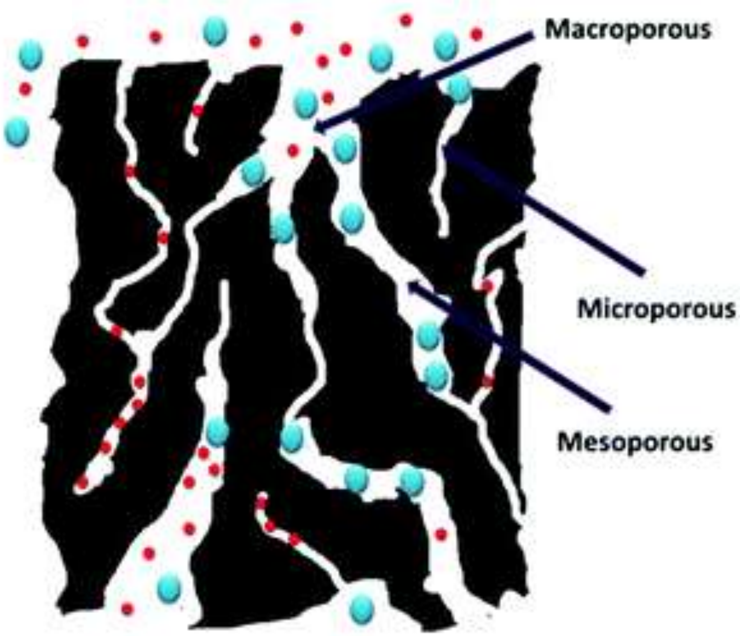
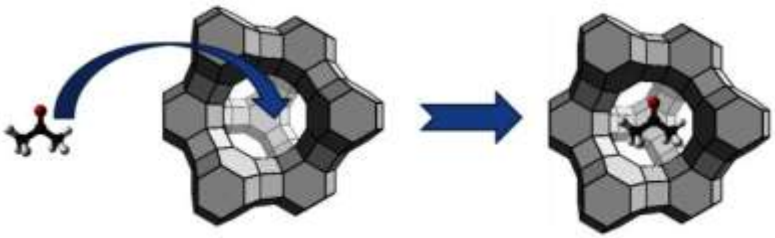
ADSORPTION



Li et al. 2015



Zheng et al. 2012

Adsorbent characteristics

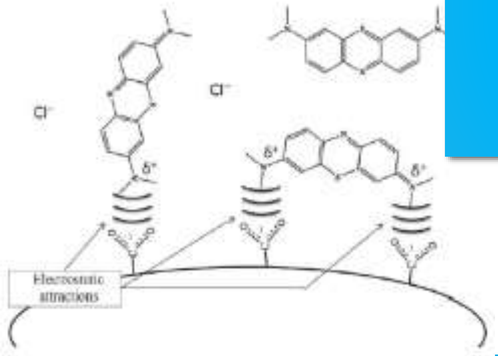
High surface area

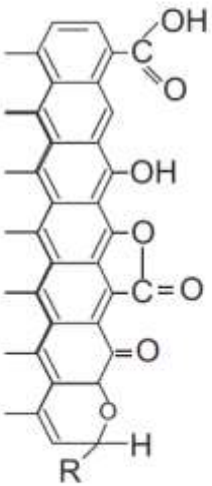
Specific adsorption sites

High adsorption capacity

Selectivity for specific compounds

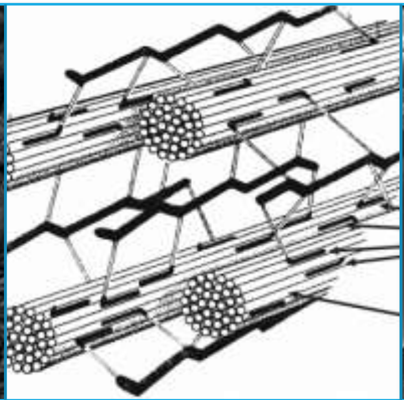
Regenerability



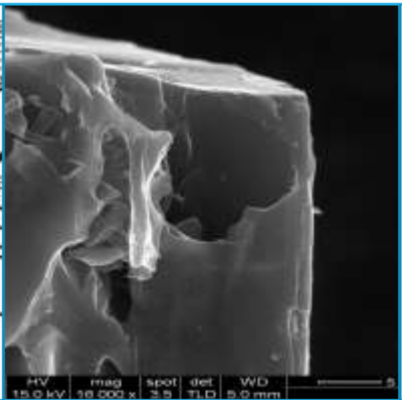


Adsorbents

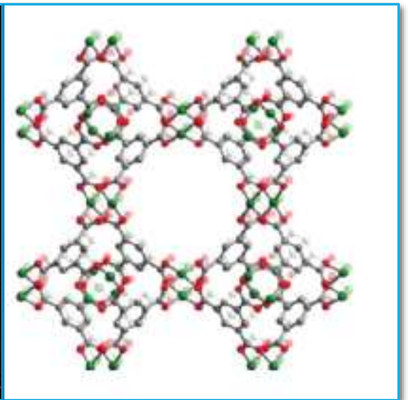
Commercial activated carbon



Raw and modified lignocellulosic material

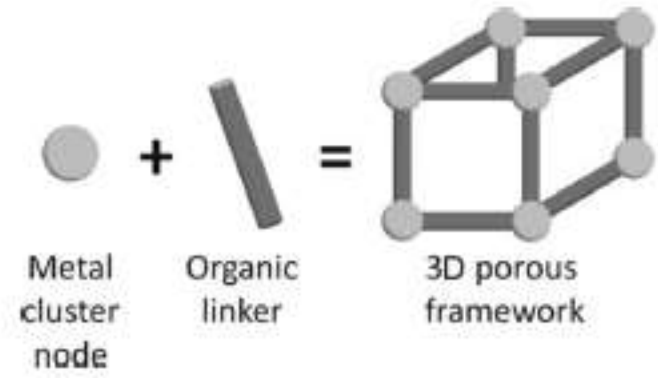


Lignocellulosic activated carbon



Metal organic framework (MOF)

MOF



Xu et al. 2014, Rouquerol et al. 2014

Metal ion and an organic molecule

3D structures

High surface area (1500-7000 m^2/g)

Several analogous MOFs

Easy tunability

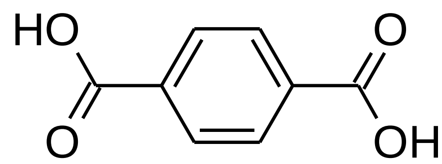
MOF

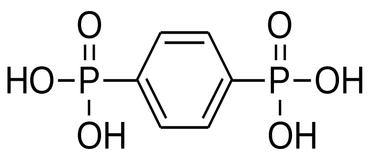
Metal

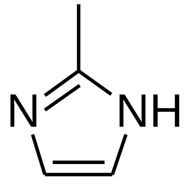
- Divalent (**Cu, Zn**, Mg, etc.)
- Trivalent (Al, Cr, Ga, Fe, In, etc.)
- Tetravalent (V, Zr, Ti, etc.)

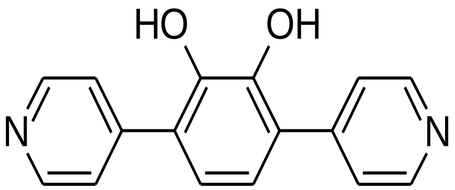
Organic linker

- Carboxylate
- Imidazolate
- Phosphonate
- Pyrazolate









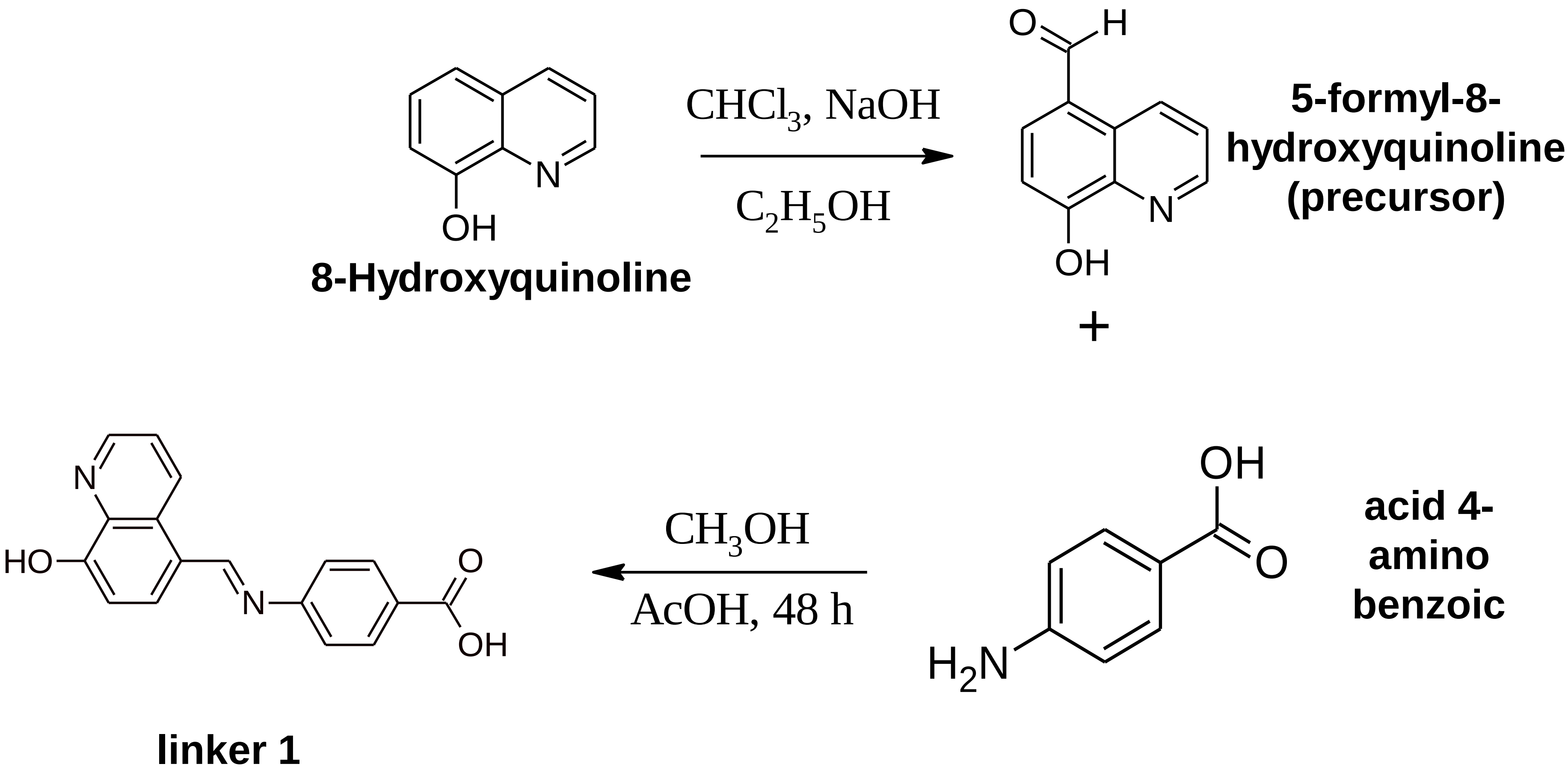
Summary of MOFs as colorant adsorbent

Adsorbent	Surface area (m^2/g)	Adsorbate	q_{max} (mg/g)	Reference
Cu-BTC	279	Methylene blue	303	Lin et al., 2014
MOF-235 (Fe)	NR	Methyl orange	477	Haque et al., 2011
MIL-101 (Fe)	3200-3400	Acid orange	153.4	X. Li et al., 2016
MIL-100 (Fe)	2800	Congo red	597.85	Moradi et al., 2015
MIL-101-Cr	3873	Methyl orange	194	Haque et al., 2010
Amino-MIL-101 (Al)	1980	Methylene blue	762	Haque et al., 2014

Objective

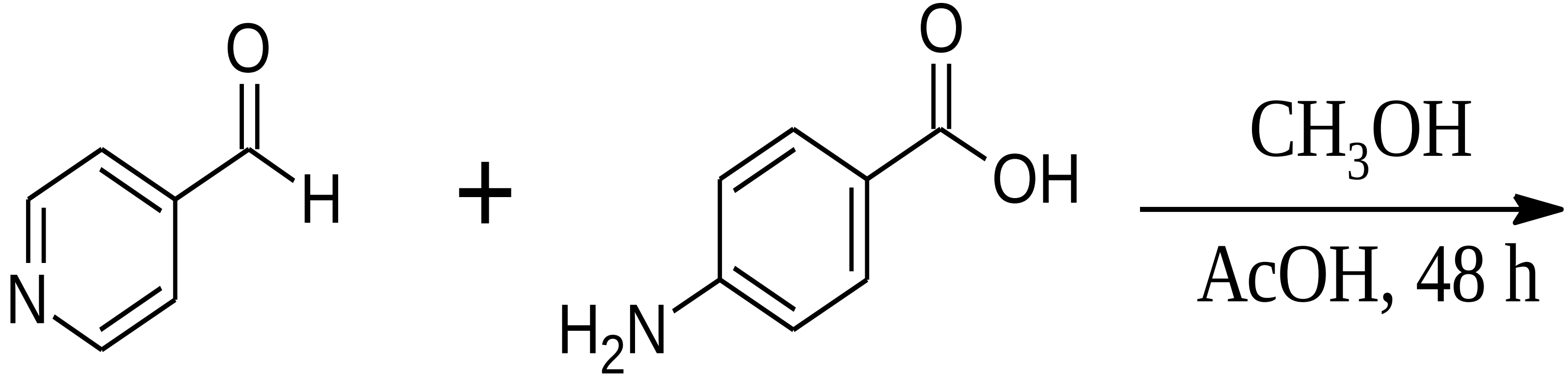
Synthesis and characterization of a new MOF for wastewater treatment

Linker 1



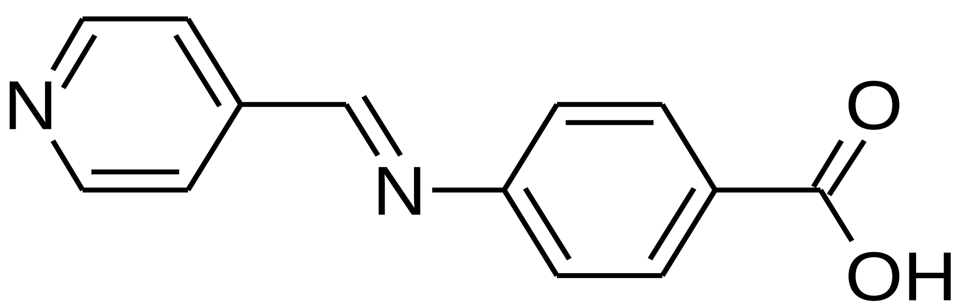
8-Hydroxyquinoline
CHCl3, NaOH
C2H5OH
5-formyl-8-hydroxyquinoline (precursor)
+
acid 4-amino benzoic
CH3OH
AcOH, 48 h
linker 1

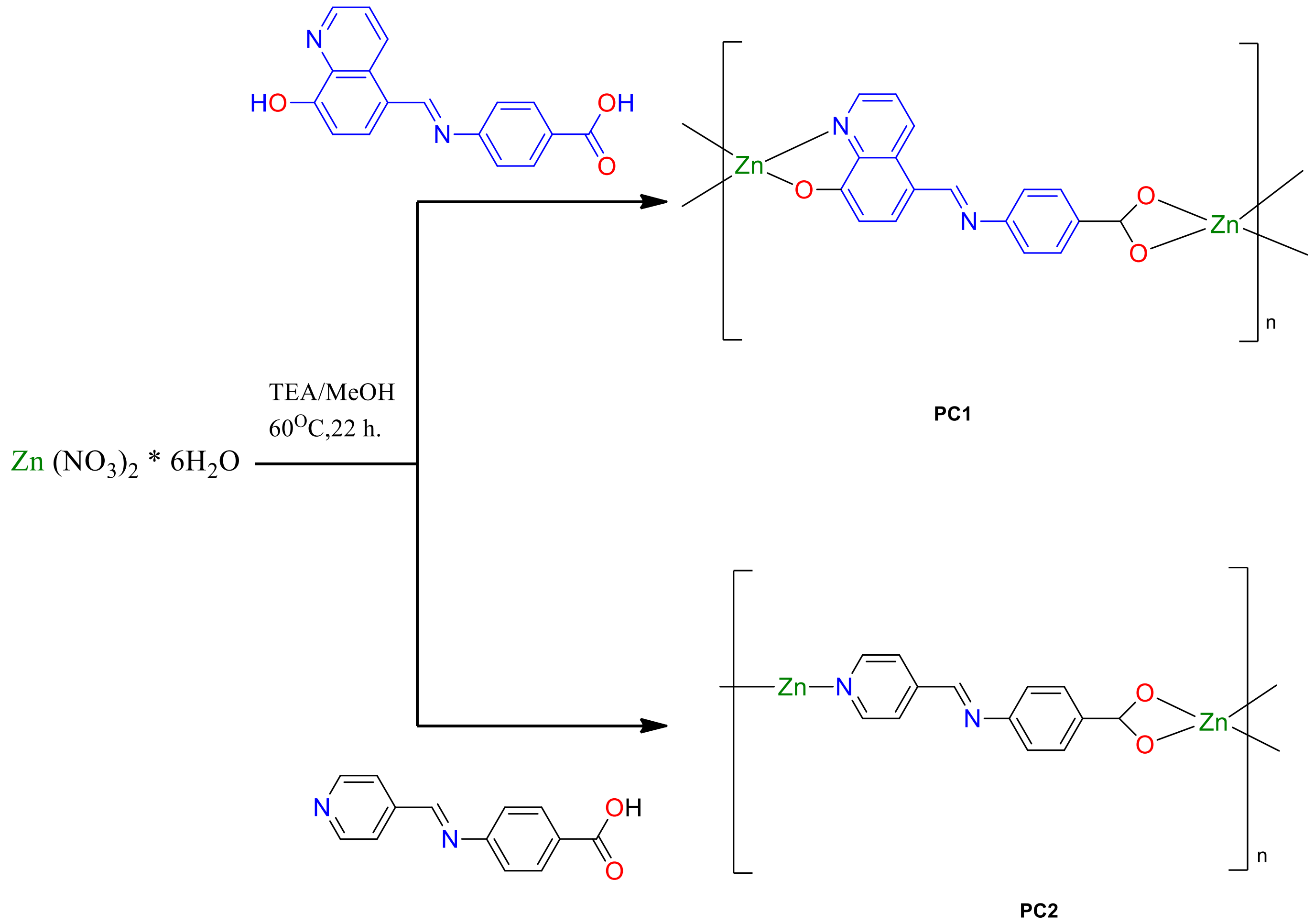
Linker 2



4-formyl pyridine

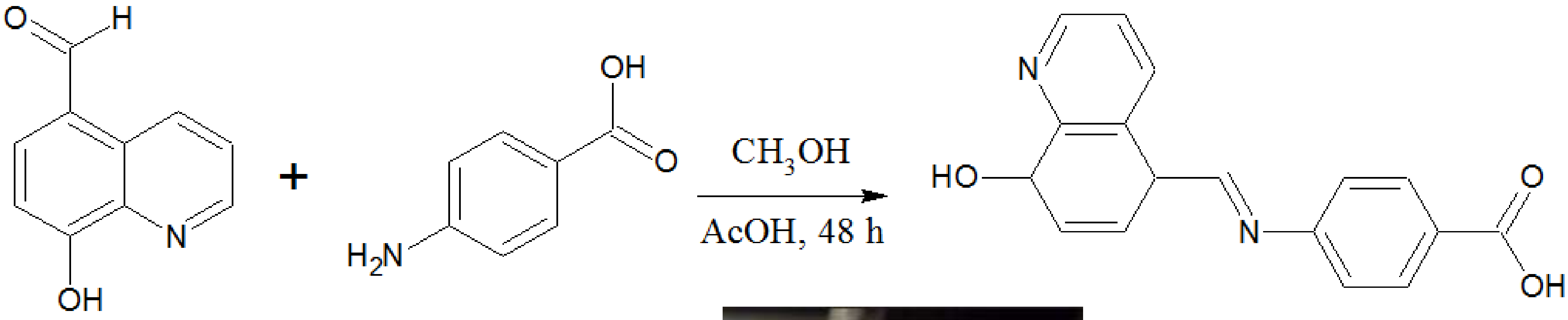
acid 4-amino benzoic





Synthesis of coordination polymers

Linker 1



5-formyl-8-hidroxyquinoline

4-amino acid benzoic
P.F.=187-189 °C

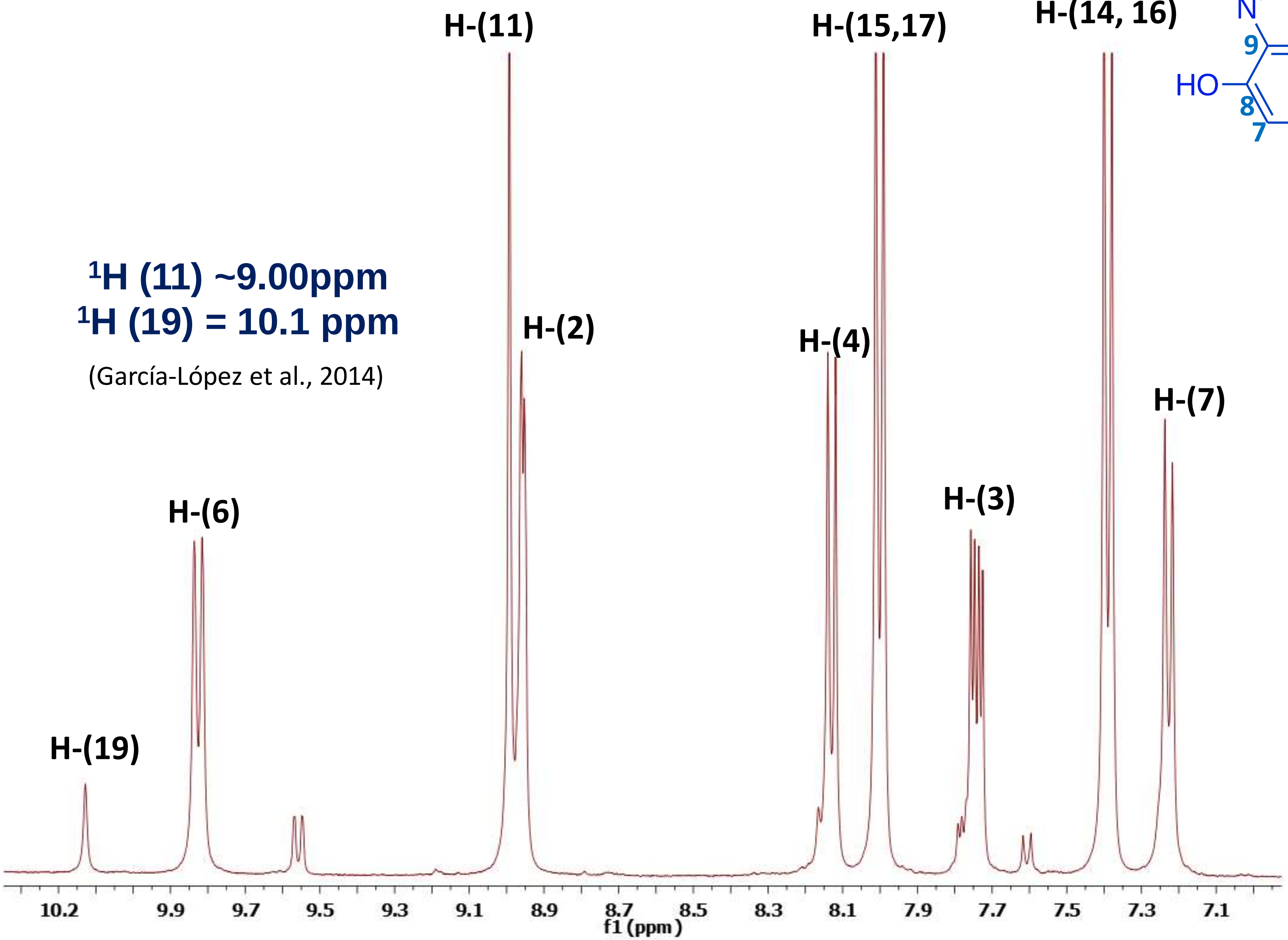
Linker 1

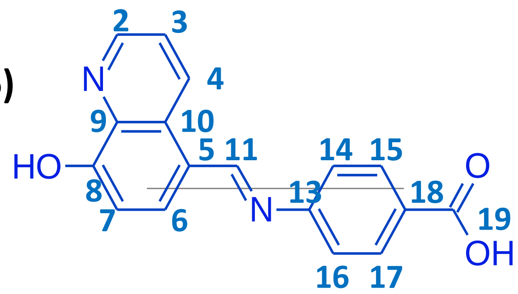
Yield = 92%
Melting point = 300°C
Color = yellow

^{1}H PROTON NMR SPECTRA OF LINKER 1

^{1}H (11) ~9.00ppm
^{1}H (19) = 10.1 ppm

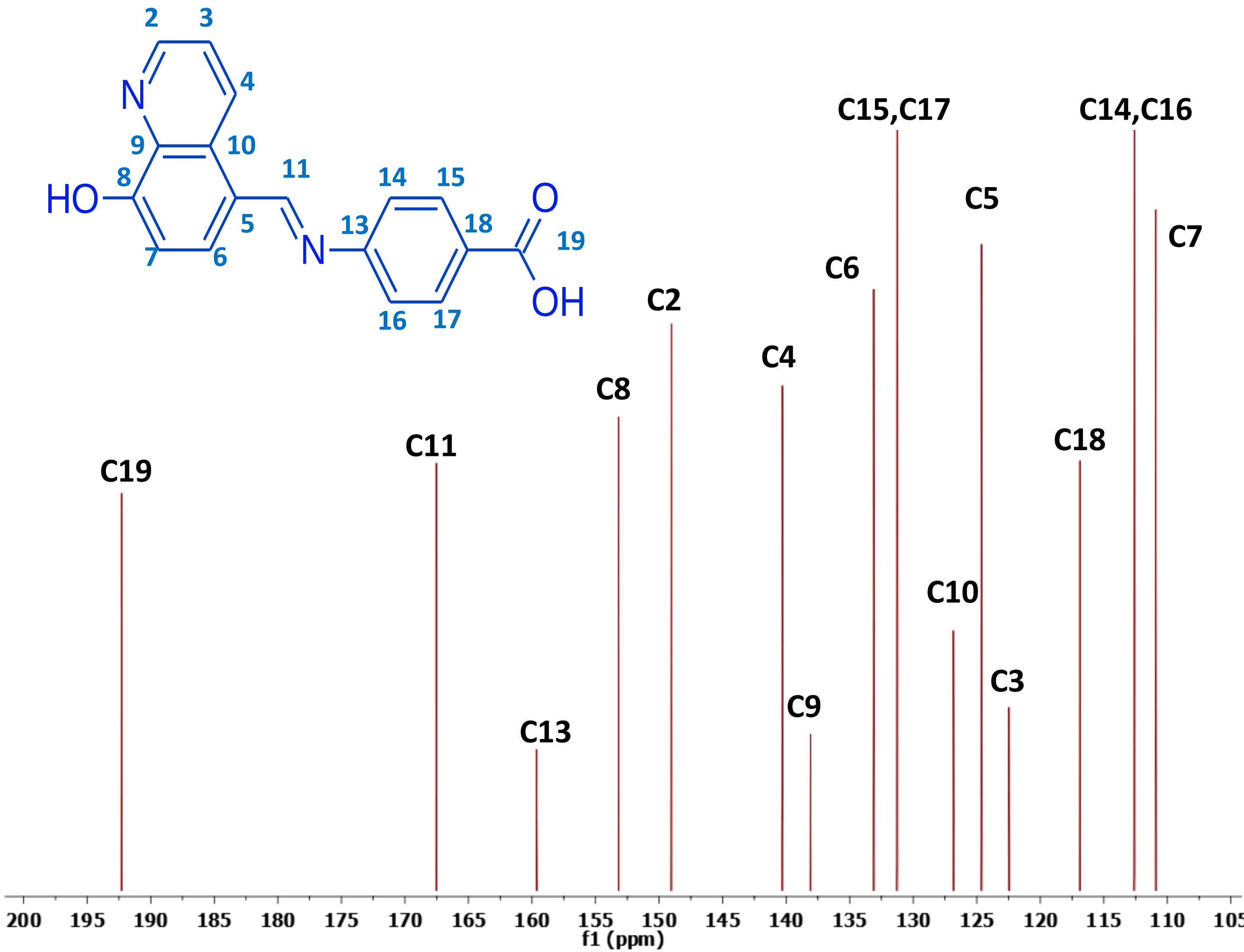
(García-López et al., 2014)





Proton	ppm
H-(7)	7.22
H-(14, H16)	7.39
H-(3)	7.73-7.76
H-(15,17)	8.00
H-(4)	8.12-8.13
H-(2)	8.96
H-(11)	**8.99**
H-(6)	9.84
H-(19)	**10.1**

^{13}C PROTON NMR SPECTRA OF LINKER 1

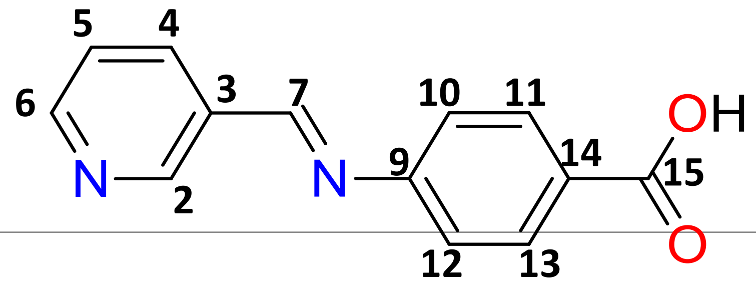


$C_{17}H_{12}N_2O_3$
^{13}C (11) ~ 167 ppm

(García-López et al., 2014)

Carbón	ppm
C7	111
C14, C16	112
C18	117
C5	122
C3	125
C10	127
C15, C17	131
C6	138
C9	140
C4	149
C8	153
C13	160
C11	**167**
C19	**192**

CHARACTERIZATION OF LINKER 2



Linker 2

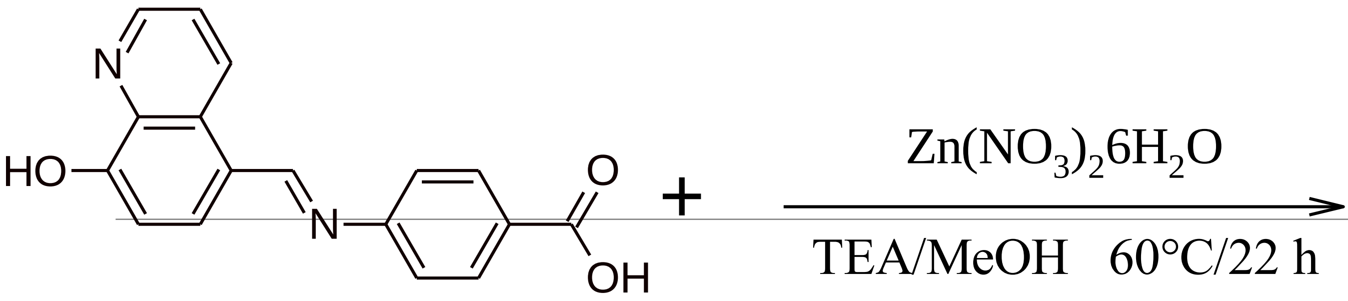
Yield = 83%
Melting point = 260 °C
Color = white

^{1}H and ^{13}C PROTON NMR SPECTRA OF LINKER 2

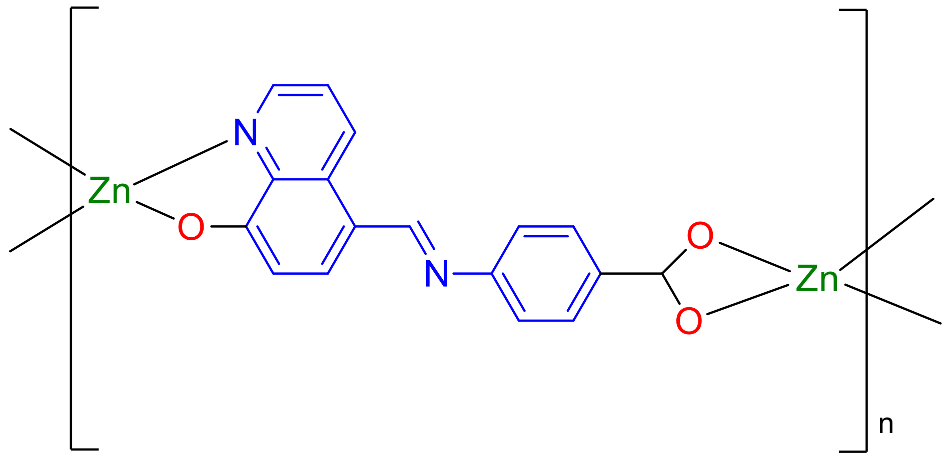
Protón	ppm
H-(10,12)	7.30
H-(5)	7.50-7.51
H-(11,13)	7.75
H-(4)	7.90
H-(6)	8.2
H-(2)	8.97
H-(7)	**9.04**
H-(15)	**10.1**

Carbón	ppm
C10,12	113
C5	117
C14	121
C3	124
C11,C13	131
C4	136
C2	148
C9	155
C6	160
C7	**167**
C15	**190**

COORDINATION POLYMER 1



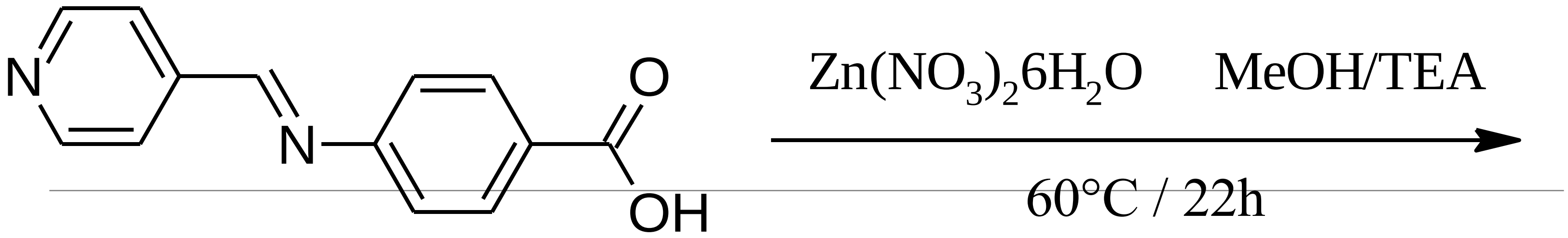
Ligante 1



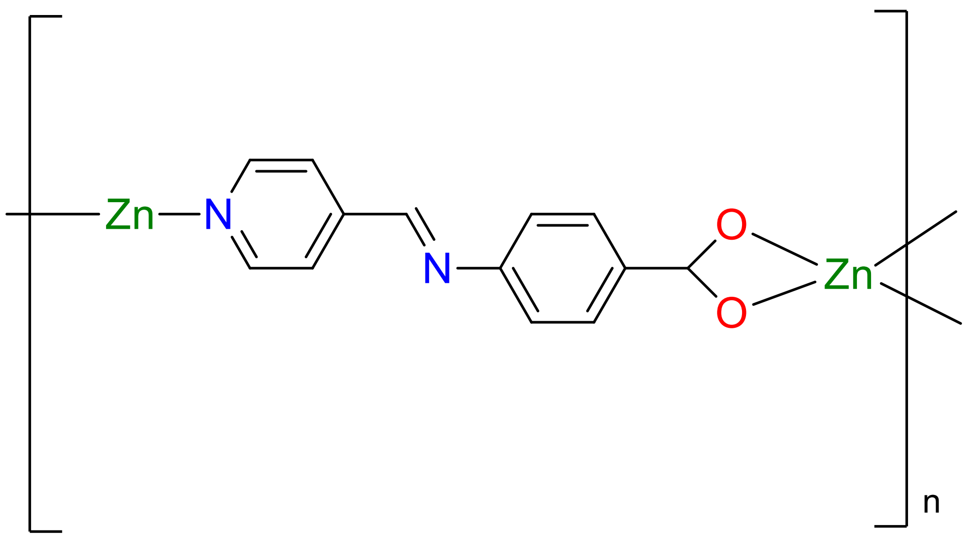
PC 1

Yield = 83%
Melting point > 300 °C
Color = Yellow

Coordination polymer 2



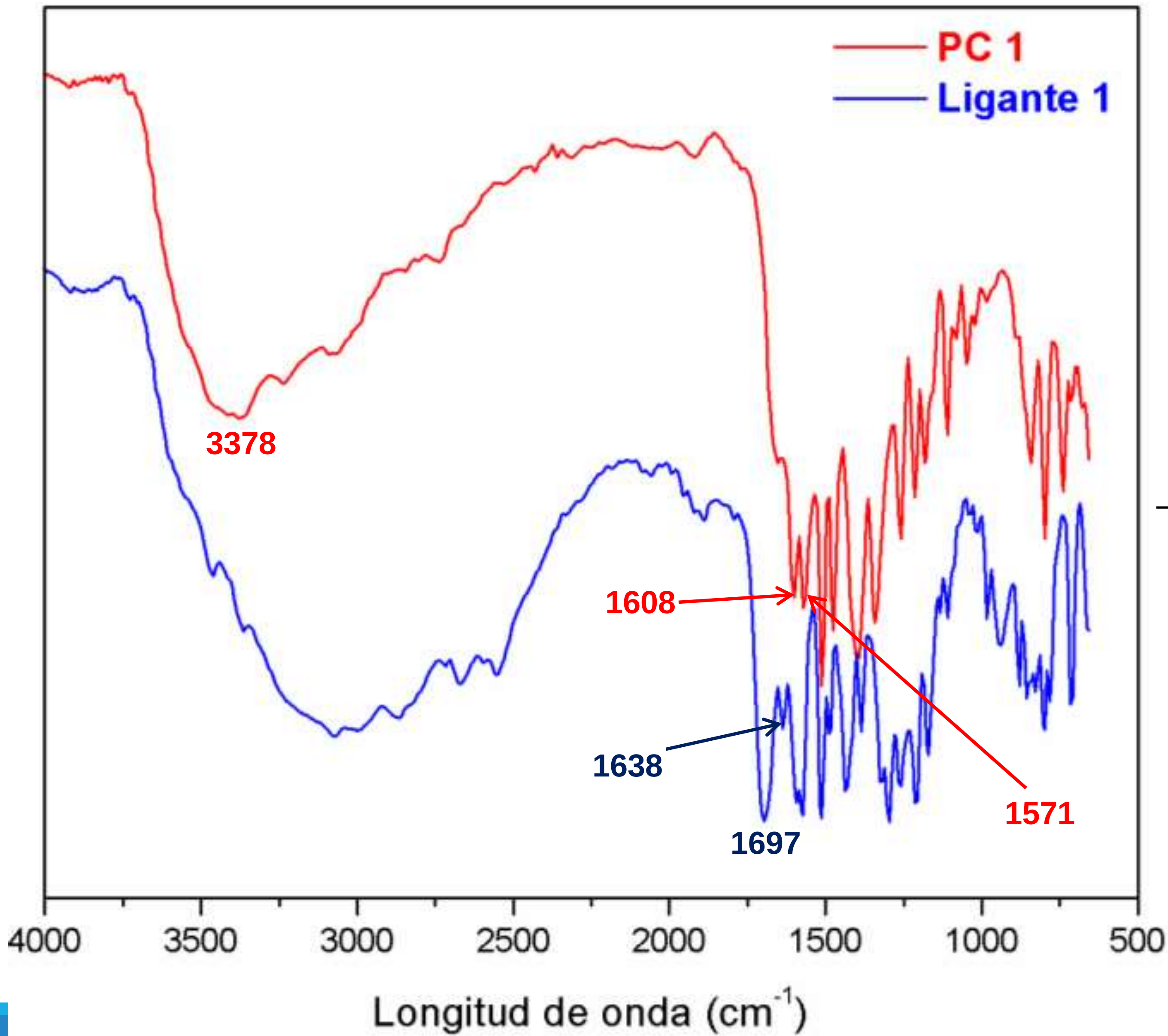
Ligante 2



PC 2

Yield = 85%
Melting point > 300 °C
Color= white

FT-IR SPECTRA OF LINKER 1 AND PC 1



(Reiss et al., 2015)

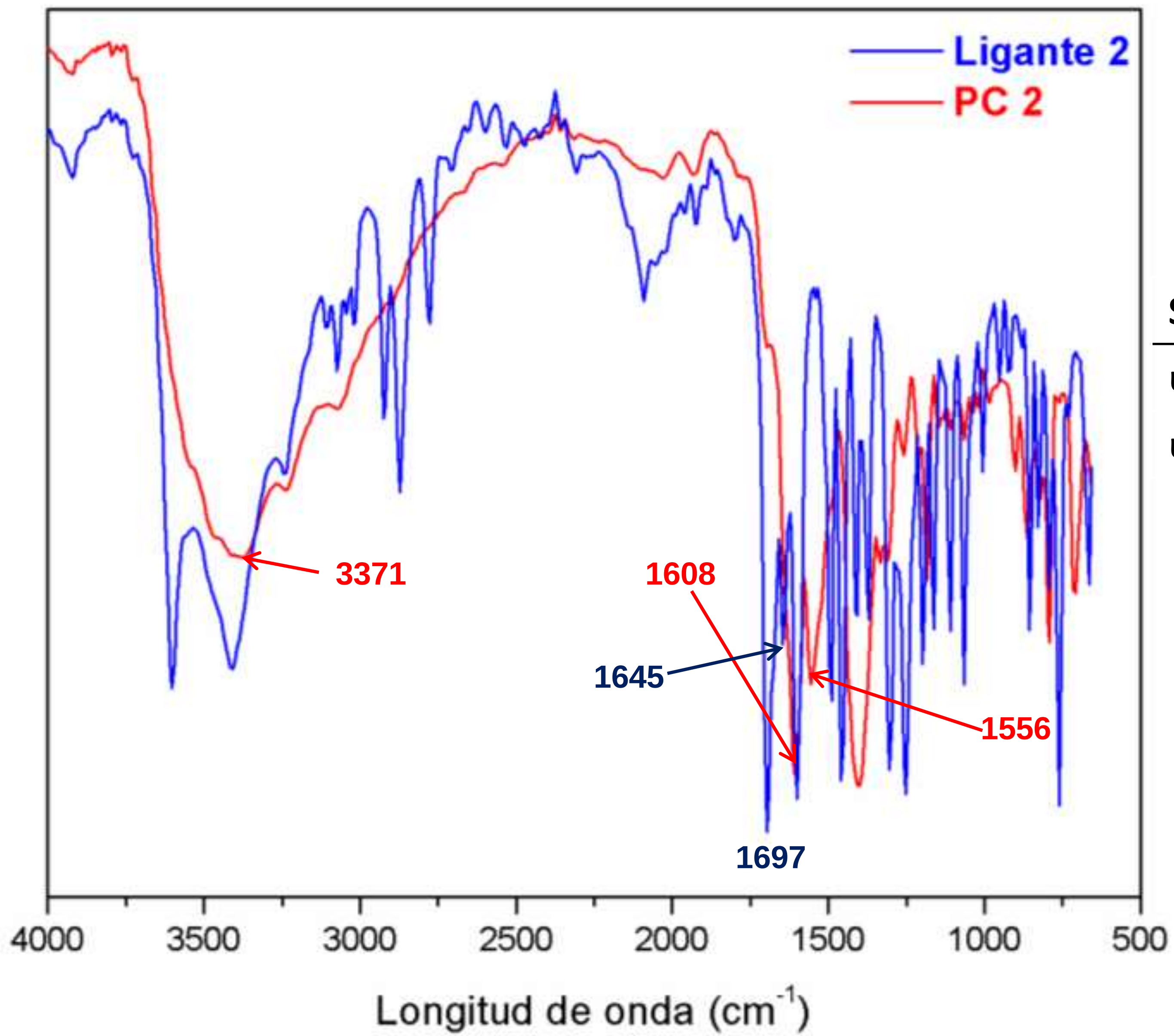
~~(Mohammadikish, 2017)~~

SIGNAL	L1	PC1
υ(C=O)	1697	1608
υ(C=N)	1637	1570

υ (O-H)=3500-3000 cm^{-1}

υ (H_2O)=3378 cm^{-1}

FT-IR SPECTRA OF LINKER 2 AND PC 2



(Reiss et al., 2015)

(~~Mohammadikish, 2017~~)

SIGNAL	L1	PC1
υ(C=O)	1697	1608
υ(C=N)	1645	1556

υ (O-H)=3500-3000 cm^{-1}

υ (H_2O)=3371 cm^{-1}

SUMMARY

Linkers were successfully syntethized with yield above 80%.

Precursor formation was verified via NMR ^{1}H y ^{13}C.

Zinc-linker coordination was achieved and verified with the displacement of C=O y C=N bands, as reported in literature.

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¡THANKS FOR YOUR ATENTION!

Purificación del precursor

Extracción Soxlet

Cromatografía de columna

Revelado cromatografía de capa fina