Electronic supplementary information (ESI)

Synthesis and Characterization of a New Nonanuclear Ni(II) Cluster from a Pyridyl-Alcohol Ligand

Alexandre Massard,[a] Guillaume Rogez,[b] and Pierre Braunstein[a]

[a] *Laboratoire de Chimie de Coordination, UMR 7177 CNRS/Université de Strasbourg, Institut Le Bel 4, Rue Blaise Pascal, CS90032, 67081 Strasbourg Cedex, France. Fax: +33 368851322; Tel: 33 368851308; E-mail: braunstein@unistra.fr*

[b] *Institut de Physique et Chimie des Matériaux de Strasbourg & Labex NIE, University of Strasbourg, CNRS UMR 7504, F-67034 Strasbourg Cedex 2, France*

General methods

All manipulations were performed under N_2 or under Ar using standard Schlenk techniques, unless stated otherwise. Solvents were dried by passing through alumina columns (pentane, toluene) or by distillation from sodium benzophenone ketyl (ether, THF) and kept under Ar over carefully activated sieves until use. NMR solvents were dried and distilled from KH and stored in the glove box over activated molecular sieves. The ligands were prepared according to the literature procedures and commercially available $[Ni(OAc)_2{\cdot}4H_2O]$ was used as received. Elemental analyses were carried out by the "Service de microanalyse" of the Institute of Chemistry.

Crystallographic data

The data set was collected on a Enraf-Nonius Kappa CCD area detector diffractometer with an FR591 rotating anode (Mo-Kα radiation, λ = 0.71073 Å) and an Oxford Cryosystems low temperature device operating in ω scanning mode with ψ and ω scans to fill the Ewald sphere, OR was collected on a Bruker APEX II DUO Kappa-CCD diffractometer equipped with an Oxford Cryosystem liquid N_2 device, using Mo-Kα radiation. The cell parameters were determined using the APEX2 software.[1] The programs used for control and integration were Collect, Scalepack, and Denzo.[2] All solutions and refinements were performed using the WinGX[3] package and all software packages within. The refinement and all further calculations were carried out using SHELXL-97.[4] The non-H atoms were refined anisotropically, using weighted full-matrix least-squares on F^2. A MULTISCAN absorption correction was applied.[5] CCDC 964330 contain the supplementary crystallographic data for **1** and can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_requ-est/cif.

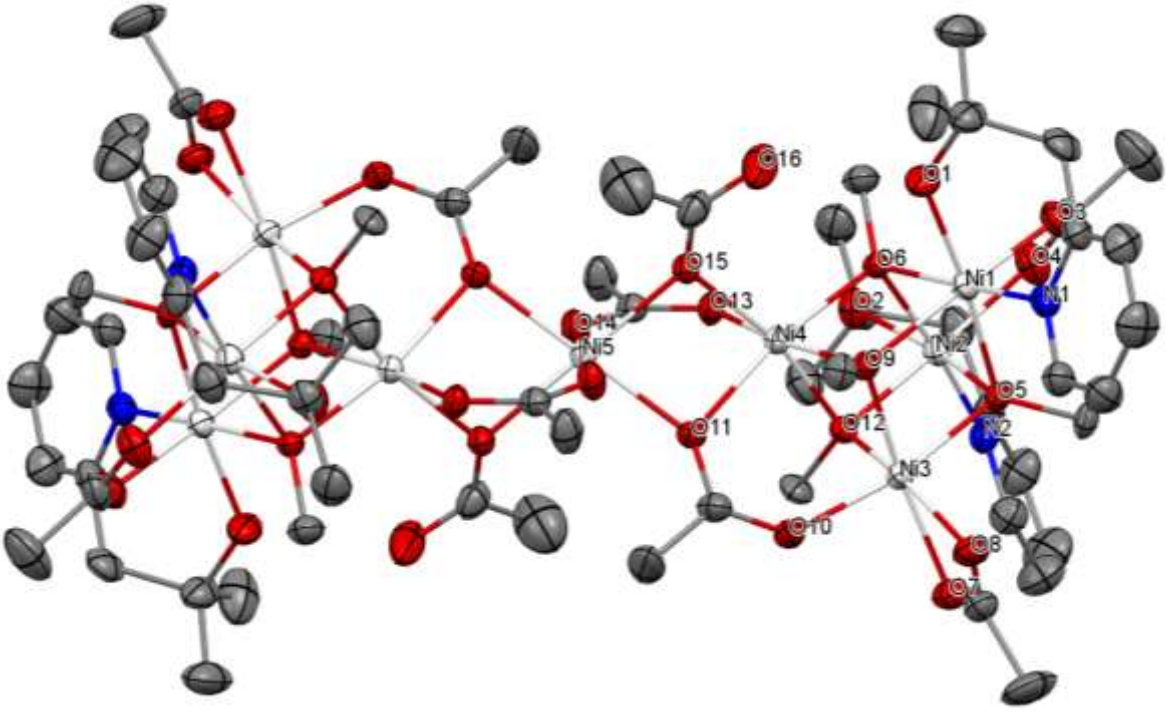


Figure S1: X-ray structure of $[Ni_9(HL\text{-}\kappa^2N,O)_4(OAc\text{-}\kappa^2O)_2(\mu_2\text{-}OAc\text{-}\kappa^2O)_2(\mu_2\text{-}OAc\text{-}\kappa^2O,O')_4(\mu_3\text{-}OAc\text{-}\kappa^2O;\kappa^2O,O')_2(\mu_3\text{-}OMe)_8]$ (**1**). Ellipsoids at 40% probability level.

Table S1: Crystallographic data for the complex **1**.

Identification code	$[Ni_9(HL\text{-}\kappa^2N,O)_4(OAc)_2(\mu\text{-}OMe)_8(\mu\text{-}OAc)_8]$ (**1**)	
Empirical formula	$C_{64}H_{106}N_4Ni_9O_{32}$	
Formula weight	1971.92	
Temperature	193(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	*a* = 11.8147(4) Å	α = 95.379(2)°
	b = 11.8639(3) Å	β = 101.262(2)°
	c = 18.4813(7) Å	γ = 115.172(2)°
Volume	2253.26(13) $Å^3$	
Z	1	
Density (calculated)	1.453 Mg/m^3	
Absorption coefficient	1.911 mm^{-1}	
F(000)	1026	
Theta range for data collection	1.15 to 27.48°	
Index ranges	-15< h< 11, -13< k< 15, -22< l< 23	
Reflections collected	26080	
Independent reflections	10282 [R(int) = 0.0550]	
Completeness to theta = 27.48°	99.5 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10282 / 0 / 506	
Goodness-of-fit on F^2	1.117	
Final R indices [$I>2\sigma(I)$]	R1 = 0.0599, wR2 = 0.1533	
R indices (all data)	R1 = 0.0870, wR2 = 0.1666	
Largest diff. peak and hole	0.786 and -1.332 $e.Å^{-3}$	

Table S2: Bond lengths [Å] and angles [°] for complex **1**.

C(1)-N(1)	1.338(6)	C(1)-C(2)	1.378(7)
C(1)-H(1)	0.9500	C(2)-C(3)	1.380(8)
C(2)-H(2)	0.9500	C(3)-C(4)	1.379(8)
C(3)-H(3)	0.9500	C(4)-C(5)	1.386(7)
C(4)-H(4)	0.9500	C(5)-N(1)	1.351(6)
C(5)-C(6)	1.505(7)	C(6)-C(7)	1.539(7)
C(6)-H(6A)	0.9900	C(6)-H(6B)	0.9900
C(7)-O(1)	1.443(5)	C(7)-C(9)	1.518(8)
C(7)-C(8)	1.527(8)	C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800	C(8)-H(8C)	0.9800
C(9)-H(9A)	0.9800	C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800	C(10)-N(2)	1.356(6)
C(10)-C(11)	1.379(7)	C(10)-H(10)	0.9500
C(11)-C(12)	1.377(9)	C(11)-H(11)	0.9500
C(12)-C(13)	1.374(8)	C(12)-H(12)	0.9500
C(13)-C(14)	1.386(7)	C(13)-H(13)	0.9500
C(14)-N(2)	1.356(6)	C(14)-C(15)	1.500(7)
C(15)-C(16)	1.532(7)	C(15)-H(15A)	0.9900
C(15)-H(15B)	0.9900	C(16)-O(2)	1.442(5)
C(16)-C(17)	1.517(7)	C(16)-C(18)	1.518(7)
C(17)-H(17A)	0.9800	C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800	C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800	C(18)-H(18C)	0.9800
C(19)-O(3)	1.259(6)	C(19)-O(4)	1.264(6)
C(19)-C(20)	1.526(7)	C(20)-H(20A)	0.9800
C(20)-H(20B)	0.9800	C(20)-H(20C)	0.9800
C(21)-O(5)	1.426(5)	C(21)-H(21A)	0.9800
C(21)-H(21B)	0.9800	C(21)-H(21C)	0.9800
C(22)-O(6)	1.435(5)	C(22)-H(22A)	0.9800
C(22)-H(22B)	0.9800	C(22)-H(22C)	0.9800
C(23)-O(7)	1.250(6)	C(23)-O(8)	1.259(6)
C(23)-C(24)	1.514(7)	C(23)-Ni(3)	2.445(5)
C(24)-H(24A)	0.9800	C(24)-H(24B)	0.9800
C(24)-H(24C)	0.9800	C(25)-O(9)	1.433(5)
C(25)-H(25A)	0.9800	C(25)-H(25B)	0.9800
C(25)-H(25C)	0.9800	C(26)-O(10)	1.241(6)

C(26)-O(11)	1.294(5)	C(26)-C(27)	1.503(7)
C(27)-H(27A)	0.9800	C(27)-H(27B)	0.9800
C(27)-H(27C)	0.9800	C(28)-O(12)	1.428(5)
C(28)-H(28A)	0.9800	C(28)-H(28B)	0.9800
C(28)-H(28C)	0.9800	C(29)-O(13)	1.250(5)
C(29)-O(14)	1.258(5)	C(29)-C(30)	1.519(6)
C(30)-H(30A)	0.9800	C(30)-H(30B)	0.9800
C(30)-H(30C)	0.9800	C(31)-O(16)	1.237(6)
C(31)-O(15)	1.278(6)	C(31)-C(32)	1.430(9)
C(32)-H(32A)	0.9800	C(32)-H(32B)	0.9800
C(32)-H(32C)	0.9800	N(1)-Ni(1)	2.110(4)
N(2)-Ni(2)	2.095(4)	O(1)-Ni(1)	2.037(3)
O(1)-H(10)	0.8590	O(2)-Ni(2)	2.070(3)
O(2)-H(20)	0.8606	O(3)-Ni(1)	2.046(3)
O(4)-Ni(2)	2.055(3)	O(5)-Ni(1)	2.073(3)
O(5)-Ni(3)	2.078(3)	O(5)-Ni(2)	2.083(3)
O(6)-Ni(4)	2.031(3)	O(6)-Ni(1)	2.054(3)
O(6)-Ni(2)	2.061(3)	O(7)-Ni(3)	2.111(3)
O(8)-Ni(3)	2.139(3)	O(9)-Ni(3)	2.035(3)
O(9)-Ni(4)	2.079(3)	O(9)-Ni(1)	2.091(3)
O(10)-Ni(3)	2.046(3)	O(11)-Ni(4)	2.088(3)
O(11)-Ni(5)	2.105(3)	O(12)-Ni(3)	2.016(3)
O(12)-Ni(2)	2.048(3)	O(12)-Ni(4)	2.061(3)
O(13)-Ni(4)	2.072(3)	O(14)-Ni(5)	2.021(3)
O(15)-Ni(5)	2.099(3)	O(15)-Ni(4)	2.117(3)
Ni(1)-Ni(2)	2.9528(7)	Ni(3)-Ni(4)	2.9094(7)
Ni(5)-O(14)#1	2.021(3)	Ni(5)-O(15)#1	2.099(3)
Ni(5)-O(11)#1	2.105(3)		
N(1)-C(1)-C(2)	124.0(5)	N(1)-C(1)-H(1)	118.0
C(2)-C(1)-H(1)	118.0	C(1)-C(2)-C(3)	118.3(5)
C(1)-C(2)-H(2)	120.8	C(3)-C(2)-H(2)	120.8
C(4)-C(3)-C(2)	118.1(5)	C(4)-C(3)-H(3)	120.9
C(2)-C(3)-H(3)	120.9	C(3)-C(4)-C(5)	120.9(5)
C(3)-C(4)-H(4)	119.5	C(5)-C(4)-H(4)	119.5
N(1)-C(5)-C(4)	120.6(5)	N(1)-C(5)-C(6)	118.1(4)
C(4)-C(5)-C(6)	121.3(4)	C(5)-C(6)-C(7)	114.2(4)
C(5)-C(6)-H(6A)	108.7	C(7)-C(6)-H(6A)	108.7
C(5)-C(6)-H(6B)	108.7	C(7)-C(6)-H(6B)	108.7

H(6A)-C(6)-H(6B)	107.6	O(1)-C(7)-C(9)	108.8(4)
O(1)-C(7)-C(8)	109.3(5)	C(9)-C(7)-C(8)	111.7(5)
O(1)-C(7)-C(6)	105.8(4)	C(9)-C(7)-C(6)	109.0(5)
C(8)-C(7)-C(6)	112.1(4)	C(7)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8B)	109.5	H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5	H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5	C(7)-C(9)-H(9A)	109.5
C(7)-C(9)-H(9B)	109.5	H(9A)-C(9)-H(9B)	109.5
C(7)-C(9)-H(9C)	109.5	H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5	N(2)-C(10)-C(11)	124.0(5)
N(2)-C(10)-H(10)	118.0	C(11)-C(10)-H(10)	118.0
C(12)-C(11)-C(10)	117.4(6)	C(12)-C(11)-H(11)	121.3
C(10)-C(11)-H(11)	121.3	C(13)-C(12)-C(11)	119.8(5)
C(13)-C(12)-H(12)	120.1	C(11)-C(12)-H(12)	120.1
C(12)-C(13)-C(14)	120.3(5)	C(12)-C(13)-H(13)	119.8
C(14)-C(13)-H(13)	119.8	N(2)-C(14)-C(13)	120.7(5)
N(2)-C(14)-C(15)	117.8(4)	C(13)-C(14)-C(15)	121.5(5)
C(14)-C(15)-C(16)	114.7(4)	C(14)-C(15)-H(15A)	108.6
C(16)-C(15)-H(15A)	108.6	C(14)-C(15)-H(15B)	108.6
C(16)-C(15)-H(15B)	108.6	H(15A)-C(15)-H(15B)	107.6
O(2)-C(16)-C(17)	109.7(4)	O(2)-C(16)-C(18)	107.8(4)
C(17)-C(16)-C(18)	111.5(4)	O(2)-C(16)-C(15)	104.6(4)
C(17)-C(16)-C(15)	113.1(4)	C(18)-C(16)-C(15)	109.7(4)
C(16)-C(17)-H(17A)	109.5	C(16)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5	C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(17B)-C(17)-H(17C)	109.5
C(16)-C(18)-H(18A)	109.5	C(16)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5	C(16)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5	H(18B)-C(18)-H(18C)	109.5
O(3)-C(19)-O(4)	125.9(4)	O(3)-C(19)-C(20)	116.4(5)
O(4)-C(19)-C(20)	117.7(5)	C(19)-C(20)-H(20A)	109.5
C(19)-C(20)-H(20B)	109.5	H(20A)-C(20)-H(20B)	109.5
C(19)-C(20)-H(20C)	109.5	H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5	O(5)-C(21)-H(21A)	109.5
O(5)-C(21)-H(21B)	109.5	H(21A)-C(21)-H(21B)	109.5
O(5)-C(21)-H(21C)	109.5	H(21A)-C(21)-H(21C)	109.5
H(21B)-C(21)-H(21C)	109.5	O(6)-C(22)-H(22A)	109.5
O(6)-C(22)-H(22B)	109.5	H(22A)-C(22)-H(22B)	109.5

O(6)-C(22)-H(22C)	109.5	H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5	O(7)-C(23)-O(8)	120.7(4)
O(7)-C(23)-C(24)	119.8(5)	O(8)-C(23)-C(24)	119.5(5)
O(7)-C(23)-Ni(3)	59.7(2)	O(8)-C(23)-Ni(3)	61.0(2)
C(24)-C(23)-Ni(3)	178.7(4)	C(23)-C(24)-H(24A)	109.5
C(23)-C(24)-H(24B)	109.5	H(24A)-C(24)-H(24B)	109.5
C(23)-C(24)-H(24C)	109.5	H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5	O(9)-C(25)-H(25A)	109.5
O(9)-C(25)-H(25B)	109.5	H(25A)-C(25)-H(25B)	109.5
O(9)-C(25)-H(25C)	109.5	H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5	O(10)-C(26)-O(11)	124.2(4)
O(10)-C(26)-C(27)	117.3(4)	O(11)-C(26)-C(27)	118.6(4)
C(26)-C(27)-H(27A)	109.5	C(26)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5	C(26)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5	H(27B)-C(27)-H(27C)	109.5
O(12)-C(28)-H(28A)	109.5	O(12)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5	O(12)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5	H(28B)-C(28)-H(28C)	109.5
O(13)-C(29)-O(14)	125.7(4)	O(13)-C(29)-C(30)	117.9(4)
O(14)-C(29)-C(30)	116.4(4)	C(29)-C(30)-H(30A)	109.5
C(29)-C(30)-H(30B)	109.5	H(30A)-C(30)-H(30B)	109.5
C(29)-C(30)-H(30C)	109.5	H(30A)-C(30)-H(30C)	109.5
H(30B)-C(30)-H(30C)	109.5	O(16)-C(31)-O(15)	124.2(5)
O(16)-C(31)-C(32)	112.5(5)	O(15)-C(31)-C(32)	123.3(5)
C(31)-C(32)-H(32A)	109.5	C(31)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5	C(31)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5	H(32B)-C(32)-H(32C)	109.5
C(1)-N(1)-C(5)	118.0(4)	C(1)-N(1)-Ni(1)	120.0(3)
C(5)-N(1)-Ni(1)	121.8(3)	C(10)-N(2)-C(14)	117.6(4)
C(10)-N(2)-Ni(2)	120.3(3)	C(14)-N(2)-Ni(2)	122.0(3)
C(7)-O(1)-Ni(1)	131.4(3)	C(7)-O(1)-H(10)	109.2
Ni(1)-O(1)-H(10)	118.6	C(16)-O(2)-Ni(2)	130.2(3)
C(16)-O(2)-H(20)	109.3	Ni(2)-O(2)-H(20)	111.6
C(19)-O(3)-Ni(1)	128.5(3)	C(19)-O(4)-Ni(2)	125.3(3)
C(21)-O(5)-Ni(1)	117.3(3)	C(21)-O(5)-Ni(3)	124.8(3)
Ni(1)-O(5)-Ni(3)	98.81(12)	C(21)-O(5)-Ni(2)	119.9(3)
Ni(1)-O(5)-Ni(2)	90.53(12)	Ni(3)-O(5)-Ni(2)	98.54(12)
C(22)-O(6)-Ni(4)	120.0(3)	C(22)-O(6)-Ni(1)	119.2(3)

Ni(4)-O(6)-Ni(1)	102.76(13)	C(22)-O(6)-Ni(2)	118.9(3)
Ni(4)-O(6)-Ni(2)	99.02(12)	Ni(1)-O(6)-Ni(2)	91.71(11)
C(23)-O(7)-Ni(3)	89.6(3)	C(23)-O(8)-Ni(3)	88.0(3)
C(25)-O(9)-Ni(3)	117.6(3)	C(25)-O(9)-Ni(4)	121.5(3)
Ni(3)-O(9)-Ni(4)	90.00(11)	C(25)-O(9)-Ni(1)	121.9(3)
Ni(3)-O(9)-Ni(1)	99.60(12)	Ni(4)-O(9)-Ni(1)	99.85(12)
C(26)-O(10)-Ni(3)	129.8(3)	C(26)-O(11)-Ni(4)	124.3(3)
C(26)-O(11)-Ni(5)	134.2(3)	Ni(4)-O(11)-Ni(5)	97.17(12)
C(28)-O(12)-Ni(3)	116.9(3)	C(28)-O(12)-Ni(2)	123.7(3)
Ni(3)-O(12)-Ni(2)	101.77(13)	C(28)-O(12)-Ni(4)	118.7(3)
Ni(3)-O(12)-Ni(4)	91.05(11)	Ni(2)-O(12)-Ni(4)	98.49(12)
C(29)-O(13)-Ni(4)	128.5(3)	C(29)-O(14)-Ni(5)	131.0(3)
C(31)-O(15)-Ni(5)	127.9(3)	C(31)-O(15)-Ni(4)	132.5(3)
Ni(5)-O(15)-Ni(4)	96.45(11)	O(1)-Ni(1)-O(3)	93.95(14)
O(1)-Ni(1)-O(6)	90.21(12)	O(3)-Ni(1)-O(6)	91.55(13)
O(1)-Ni(1)-O(5)	174.46(13)	O(3)-Ni(1)-O(5)	90.46(13)
O(6)-Ni(1)-O(5)	86.32(11)	O(1)-Ni(1)-O(9)	94.95(13)
O(3)-Ni(1)-O(9)	166.50(12)	O(6)-Ni(1)-O(9)	78.29(11)
O(5)-Ni(1)-O(9)	80.14(11)	O(1)-Ni(1)-N(1)	83.58(14)
O(3)-Ni(1)-N(1)	93.22(14)	O(6)-Ni(1)-N(1)	172.41(14)
O(5)-Ni(1)-N(1)	99.54(13)	O(9)-Ni(1)-N(1)	97.85(13)
O(1)-Ni(1)-Ni(2)	132.91(10)	O(3)-Ni(1)-Ni(2)	79.01(9)
O(6)-Ni(1)-Ni(2)	44.24(8)	O(5)-Ni(1)-Ni(2)	44.87(8)
O(9)-Ni(1)-Ni(2)	87.49(8)	N(1)-Ni(1)-Ni(2)	142.74(11)
O(12)-Ni(2)-O(4)	169.78(12)	O(12)-Ni(2)-O(6)	80.79(11)
O(4)-Ni(2)-O(6)	91.47(13)	O(12)-Ni(2)-O(2)	90.15(12)
O(4)-Ni(2)-O(2)	96.10(13)	O(6)-Ni(2)-O(2)	86.81(12)
O(12)-Ni(2)-O(5)	79.41(12)	O(4)-Ni(2)-O(5)	93.47(13)
O(6)-Ni(2)-O(5)	85.87(11)	O(2)-Ni(2)-O(5)	168.08(12)
O(12)-Ni(2)-N(2)	97.04(14)	O(4)-Ni(2)-N(2)	91.45(15)
O(6)-Ni(2)-N(2)	172.61(13)	O(2)-Ni(2)-N(2)	86.13(14)
O(5)-Ni(2)-N(2)	100.73(13)	O(12)-Ni(2)-Ni(1)	88.69(8)
O(4)-Ni(2)-Ni(1)	81.11(9)	O(6)-Ni(2)-Ni(1)	44.05(8)
O(2)-Ni(2)-Ni(1)	130.30(9)	O(5)-Ni(2)-Ni(1)	44.60(8)
N(2)-Ni(2)-Ni(1)	143.25(11)	O(12)-Ni(3)-O(9)	88.08(11)
O(12)-Ni(3)-O(10)	92.62(12)	O(9)-Ni(3)-O(10)	90.39(12)
O(12)-Ni(3)-O(5)	80.27(12)	O(9)-Ni(3)-O(5)	81.36(12)
O(10)-Ni(3)-O(5)	169.24(12)	O(12)-Ni(3)-O(7)	102.86(13)

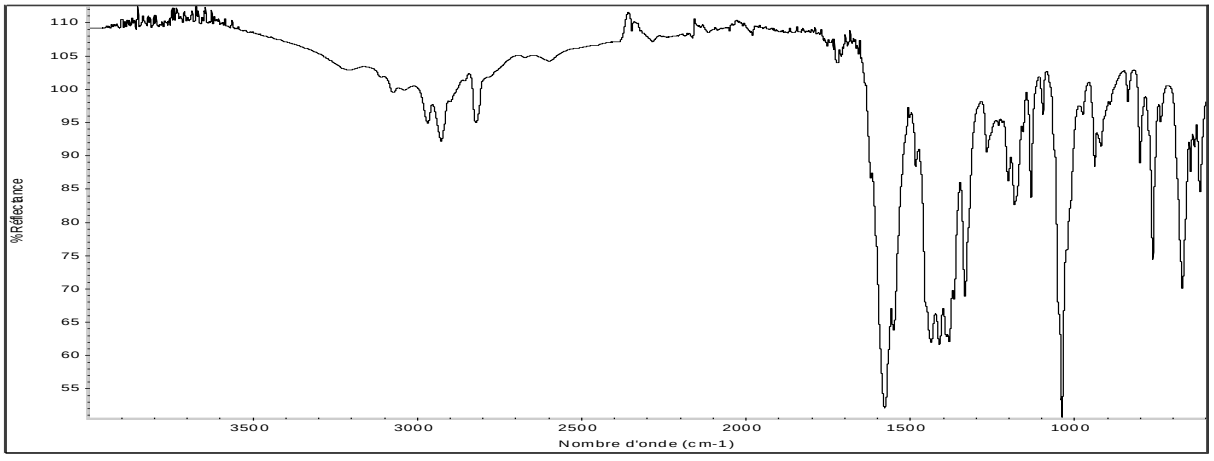
O(9)-Ni(3)-O(7)	168.84(13)	O(10)-Ni(3)-O(7)	91.30(13)
O(5)-Ni(3)-O(7)	98.13(13)	O(12)-Ni(3)-O(8)	164.51(12)
O(9)-Ni(3)-O(8)	107.25(12)	O(10)-Ni(3)-O(8)	89.57(13)
O(5)-Ni(3)-O(8)	99.44(13)	O(7)-Ni(3)-O(8)	61.74(13)
O(12)-Ni(3)-C(23)	133.61(15)	O(9)-Ni(3)-C(23)	138.23(15)
O(10)-Ni(3)-C(23)	90.16(15)	O(5)-Ni(3)-C(23)	100.59(15)
O(7)-Ni(3)-C(23)	30.76(15)	O(8)-Ni(3)-C(23)	30.98(14)
O(12)-Ni(3)-Ni(4)	45.09(8)	O(9)-Ni(3)-Ni(4)	45.62(8)
O(10)-Ni(3)-Ni(4)	79.92(9)	O(5)-Ni(3)-Ni(4)	89.36(8)
O(7)-Ni(3)-Ni(4)	145.47(10)	O(8)-Ni(3)-Ni(4)	150.12(10)
C(23)-Ni(3)-Ni(4)	169.68(12)	O(6)-Ni(4)-O(12)	81.19(11)
O(6)-Ni(4)-O(13)	89.81(12)	O(12)-Ni(4)-O(13)	87.81(12)
O(6)-Ni(4)-O(9)	79.08(12)	O(12)-Ni(4)-O(9)	85.70(11)
O(13)-Ni(4)-O(9)	167.87(12)	O(6)-Ni(4)-O(11)	169.46(12)
O(12)-Ni(4)-O(11)	90.67(12)	O(13)-Ni(4)-O(11)	96.59(12)
O(9)-Ni(4)-O(11)	93.73(12)	O(6)-Ni(4)-O(15)	111.13(11)
O(12)-Ni(4)-O(15)	167.29(12)	O(13)-Ni(4)-O(15)	89.04(11)
O(9)-Ni(4)-O(15)	99.44(11)	O(11)-Ni(4)-O(15)	77.46(12)
O(6)-Ni(4)-Ni(3)	88.17(8)	O(12)-Ni(4)-Ni(3)	43.85(8)
O(13)-Ni(4)-Ni(3)	131.30(8)	O(9)-Ni(4)-Ni(3)	44.38(8)
O(11)-Ni(4)-Ni(3)	81.31(8)	O(15)-Ni(4)-Ni(3)	136.33(8)
O(14)-Ni(5)-O(14)#1	180.00(15)	O(14)-Ni(5)-O(15)#1	88.21(12)
O(14)#1-Ni(5)-O(15)#1	91.79(12)	O(14)-Ni(5)-O(15)	91.79(12)
O(14)#1-Ni(5)-O(15)	88.21(12)	O(15)#1-Ni(5)-O(15)	180.00(15)
O(14)-Ni(5)-O(11)#1	86.53(12)	O(14)#1-Ni(5)-O(11)#1	93.47(12)
O(15)#1-Ni(5)-O(11)#1	77.50(11)	O(15)-Ni(5)-O(11)#1	102.50(11)
O(14)-Ni(5)-O(11)	93.47(12)	O(14)#1-Ni(5)-O(11)	86.53(12)
O(15)#1-Ni(5)-O(11)	102.50(11)	O(15)-Ni(5)-O(11)	77.50(11)
O(11)#1-Ni(5)-O(11)	180.000(1)		

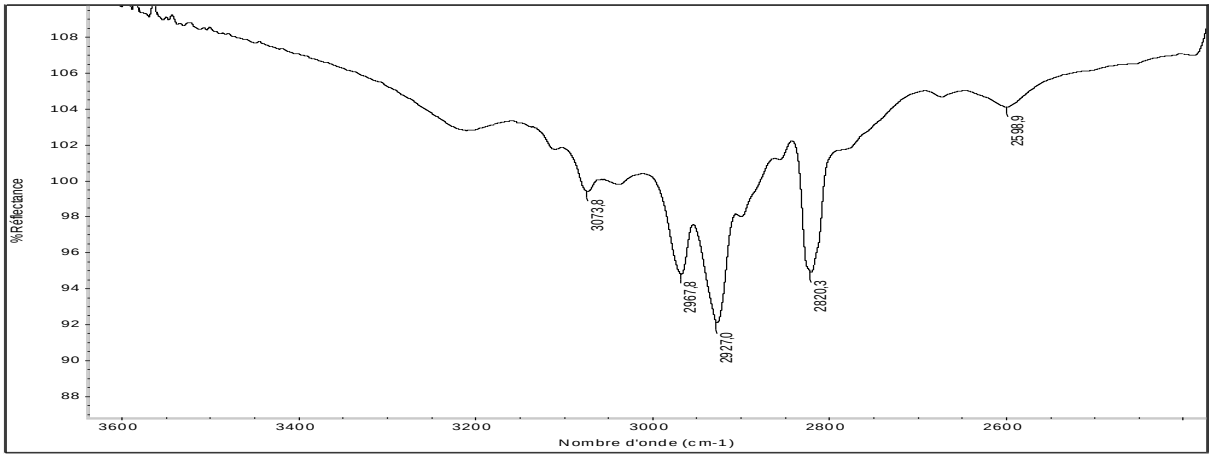
Symmetry transformations used to generate equivalent atoms:
#1 -x,-y,-z+1

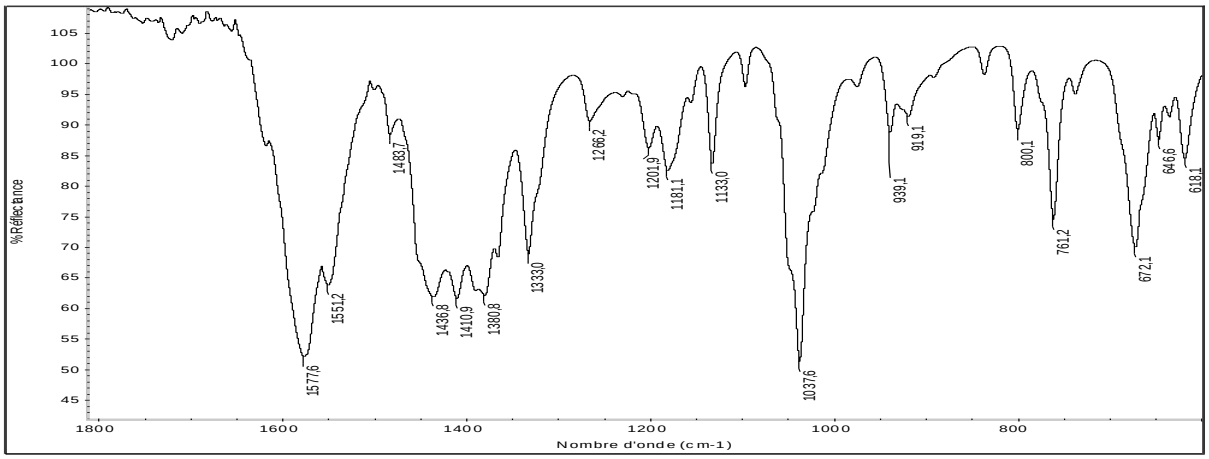
Magnetic measurements. They were performed at the Institut de Physique et Chimie des Matériaux de Strasbourg (UMR CNRS-UdS 7504) using a Quantum Design SQUID-VSM magnetometer. Magnetization measurements at different fields at a given temperature confirm the absence of ferromagnetic impurities. Data were corrected for the sample holder and diamagnetism was estimated from Pascal constants.

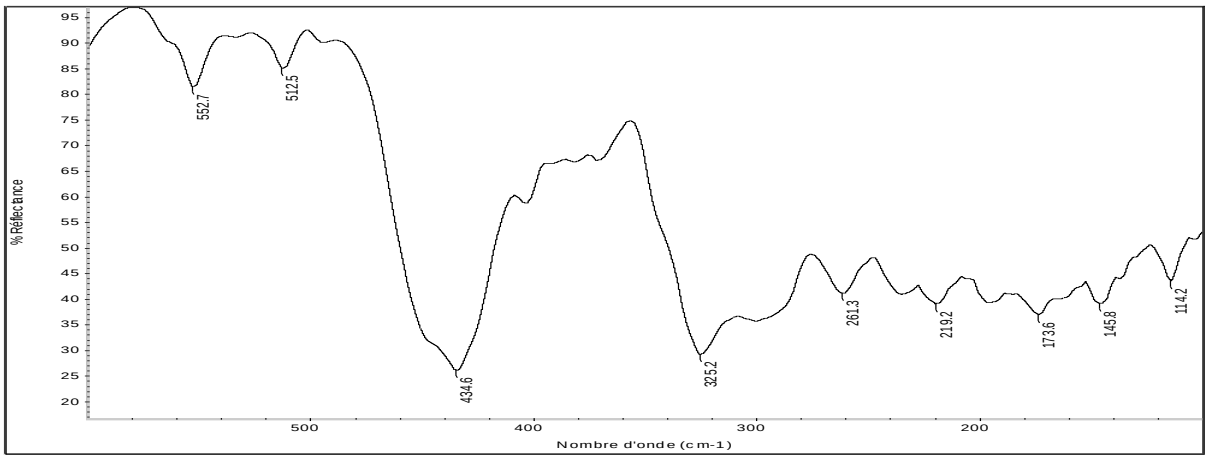
Catalytic oligomerization of ethylene. The catalytic reactions were performed in a magnetically stirred (1200 rpm) 145 mL stainless steel autoclave. A 125 mL glass container was used to avoid corrosion of the autoclave walls. The precatalyst solutions were prepared by dissolving **1** (8.7 mg, 4 x 10^{-5} mol of Ni) in chlorobenzene (10 mL). $AlEtCl_2$ was used as cocatalyst (8 x 10^{-5} mol in toluene). The solution of the precatalyst was injected into the reactor under an ethylene flux, then 5 mL of the cocatalyst toluene solution (10 equiv.) was added (total volume: 15 mL). All catalytic reactions were started between 20 and 30 °C. No cooling of the reactor was done during the reaction. After injection of the catalyst and cocatalyst solutions under a constant low flow of ethylene, the reactor was pressurized to 10 bars. The temperature increased, due solely to the exothermicity of the reaction. The 10 bars working pressure was maintained through a continuous feed of ethylene from a bottle placed on a balance to allow continuous monitoring of the ethylene uptake. At the end of each test (35 min, unless otherwise specified) a dry ice bath was used to rapidly cool the reactor. When the inner temperature reached 0 °C, the ice bath was removed, allowing the temperature to slowly rise to 18 °C. The gaseous phase was then transferred into a 10 L polyethylene tank filled with water. An aliquot of this gaseous phase was transferred into a Schlenk flask, previously evacuated, for GC analysis. The amount of ethylene not consumed was thus determined. Although this method is of limited accuracy, it was used throughout and gave satisfactory reproducibility. The reaction mixture in the reactor was quenched in situ by the addition of ethanol (10 mL), transferred into a Schlenk flask, and separated from the metal complexes by trap-to-trap evaporation (20 °C, 0.8 mbar) into a second Schlenk flask previously immersed in liquid nitrogen in order to avoid loss of product.

Infrared Spectra of complex 1









1. Bruker AXS Inc Madison USA, 2006.
2. (a) R. Hooft, COLLECT; Nonius BV, 1997-2000. (b) Z. Otwinoski, W. Minor, SCALEPACK, DENZO. *Methods Enzymol*. **1997**, *276*, 307.
3. L. J. Farrugia, *J. Appl. Crystallogr*., **1999**, *32*, 837-838.
4. G. M. Sheldrick, University of Göttingen: Göttingen, **1999**.
5. R. H. Blessing, *Acta Crystallogr*., Sect A **1995**, *51*, 33.