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Research paper

Mononuclear nickel(II) complexes with arylhydrazones of acetoacetanilide and their catalytic activity in nitroaldol reaction

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ABSTRACT

The new mononuclear nickel(II) complexes $[Ni(HL^1)(H_2O)_2(CH_3OH)]$ (**1**, NaH_2L^1 = sodium (*Z*)-2-(2-(1,3-dioxo-1-(phenylamino)butan-2-ylidene)hydrazinyl)benzenesulfonate) and $[Ni(H_2L^2)_2(H_2O)_4]$ $[(CH_3)_2S{=}O)_2]$ (**2**, H_3L^2 = (Z)-2-(2-(1,3-dioxo-1-(phenylamino)butan-2-ylidene)hydrazinyl)benzoic acid) were synthesized and characterized by IR and ESI-MS spectroscopies, elemental and X-ray crystal structural analyses. Both compounds **1** and **2** act as homogenous catalysts for the diastereoselective nitroaldol (Henry) reaction of aliphatic and aromatic aldehydes with nitroethane in different solvents such as acetonitrile, methanol or water. Complex **1** was found to be the more efficient catalyst for the Henry reaction in aqueous medium, providing β-nitroalcohols with good yields (67–82%) and diastereoselectivities (*syn/anti* 59:41–70:30).

1. Introduction

The use of nickel complexes as catalysts in homogeneous catalysis has received much attention in recent years [1,2]. Interestingly, the catalytic properties of nickel complexes can be manipulated and tuned by selecting organic ligands [1,2]. Our group has been interested in exploring the catalytic applications of transition metal complexes of arylhydrazone ligands in the oxidation or C—C coupling reaction [3]. Whereas, several Ni(II)-arylhydrazone complexes are reported [3], their catalytic behavior for C—C coupling reaction has not been investigated. Herein, we report the synthesis and structural characterization of two mononuclear Ni-arylhydrazone complexes and their use as the catalysts in C—C coupling reaction of nitroethane with aldehydes (Henry reaction). To the best of our knowledge, in comparison to Cu(II) complexes [4], only limited numbers of nickel(II) complexes have been applied in the Henry reaction under different conditions, namely in homogeneous systems [5–7], under solvent free microwave conditions [8] or in ionic liquids [9]. Thus, the search for efficient and environmentally friendly homogenous protocol for C—C coupling of nitroethane with aldehydes is still highly desirable.

Hence, on the basis of the above considerations, we focused this work on the following aims: *i*) to synthesize new Ni(II)-arylhydrazone complexes, $[Ni(HL^1)(H_2O)_2(CH_3OH)]$ (**1**, NaH_2L^1 = sodium (*Z*)-2-(2-(1,3-dioxo-1-(phenylamino)butan-2-ylidene)hydrazinyl)benzenesulfonate) and $[Ni(H_2L^2)_2(H_2O)_4]$ $[(CH_3)_2S{=}O)_2]$ (**2**, H_3L^2 = (*Z*)-2-(2-(1,3-dioxo-1-(phenylamino)butan-2-ylidene)hydrazinyl)benzoic acid); *ii*) to evaluate the catalytic activity of the prepared new Ni(II) complexes in the Henry reaction.

2. Experimental

2.1. Materials and instrumentation

All the chemicals were obtained from commercial sources (Aldrich) and used as received. The NaH_2L^1 and H_3L^2 were synthesized according to the reported procedures [10,11]. Infrared spectra (4000–400 cm^{-1}) were recorded on a Vertex 70 (Bruker) instrument in KBr pellets. Carbon, hydrogen, and nitrogen elemental analyses were done using a "2400 CHN Elemental Analyzer" (Perkin Elmer). The ^1H and ^{13}C NMR spectra were recorded at room

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temperature on a Bruker Avance II + 300 (UltraShield^{TM} Magnet) spectrometer operating at 300.130 and 75.468 MHz for proton and carbon-13, respectively. The chemical shifts are reported in ppm using tetramethylsilane as the internal reference. Electrospray mass spectra (ESI-MS) were run with an ion-trap instrument (Varian 500-MS LC Ion Trap Mass Spectrometer) equipped with an electrospray ion source. For electrospray ionization, the drying gas and flow rate were optimized according to the particular sample with 35 p.s.i. nebulizer pressure. Scanning was performed from *m*/*z* 0 to 1100 in methanol solution. The compounds were observed in the positive mode (capillary voltage = 80–105 V).

2.2. Synthesis of [Ni(HL^1)(H$_2$O)$_2$(CH$_3$OH)] (**1**)

0.1 mmol (38 mg) of NaH$_2$L^1 were dissolved in 20 mL of methanol, then 0.1 mmol (25 mg) of Ni(CH$_3$COO)$_2$ 4H$_2$O were added. The mixture was stirred for 5 min and left for slow evaporation. Greenish crystals of the product started to form after *ca.* 2 d at room temperature; they were filtered off and dried in air.

1: Yield, 52% (based on Ni). Calcd. for $C_{17}H_{21}N_3NiO_8S$ (*Mr.* = 486.12): C 42.00, H 4.35, N 8.64; found C 41.92, H 4.17, N 8.57. MS (ESI, positive ion mode), *m*/*z*: 455.06 [*Mr*–CH$_3$OH + H]$^+$. IR (KBr): 3380 (s, br) *ν*(OH), 3118 *ν*(NH), 1676 (s) *ν*(C=O), 1598 (s) *ν*(C=N) cm^{-1}.

2.3. Synthesis of [Ni(H$_2$L^2)$_2$(H$_2$O)$_4$] [(CH$_3$)$_2$S=O)$_2$] (**2**)

0.1 mmol (37 mg) of H$_3$L^2 were dissolved in 25 mL of methanol, then 0.1 mmol (25 mg) of Ni(CH$_3$COO)$_2$ 4H$_2$O were added and the system was stirred for 10 min. After *ca.* 3 d at room temperature, greenish crystals precipitated, which were then filtered off and dried in air.

2: Yield, 48% (based on Ni). Calcd. for $C_{38}H_{48}N_6NiO_{14}S_2$ (*Mr.* = 935.64): C 48.78, H 5.17, N 8.98; found C 48.67, H 5.06, N 8.79. MS (ESI, positive ion mode), *m*/*z*: 780.36 [*Mr*–2(CH$_3$)$_2$S=O + H]$^+$. IR (KBr): 3437 (s, br) *ν*(OH), 3212 *ν*(NH), 1669 (s) *ν*(C=O), 1611 *ν* (C=N) cm^{-1}.

2.4. Crystal structure determination

X-ray diffraction patterns of **1** and **2** were collected using a Bruker SMART APEX-II CCD area detector equipped with graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å) at room temperature. Absorption correction was applied by SADABS [12,13]. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares using Bruker's SHELXTL-97 [14]. All non-hydrogen atoms were refined anisotropically. The details of the crystallographic data, selected bond distances and angles for **1** and **2** are summarized in Tables 1 and 2. Crystallographic data for the structural analysis have been deposited to the Cambridge Crystallographic Data Center (CCDC 1564002 for **1** and 1564001 for **2**). Copy of this information can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: (+44) 1223–336033; E-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk/data_request/cif).

2.5. General procedure for the study of catalytic activity

To a 10 mL vial were added the catalyst (1.0–4.0 mol%) and 2 mL solvent (H$_2$O, MeOH or MeCN) and the solution was stirred for 2 min at room temperature. Then, the aldehyde (1 mmol) and nitroethane (4 mmol) were added and the resulting transparent homogeneous solution was stirred at room temperature for the appropriate time. After evaporation of the solvent, the residue was dissolved in CDCl$_3$ and this blurry solution filtrated, then analyzed by ^{1}H NMR. The yield of the β-nitroalkanol product (relative to the aldehyde) and the ratio between *syn* and *anti* isomers were established by ^{1}H NMR as reported previously [11,15–17]. The adequacy of this procedure was proved by blank ^{1}H NMR analyses with 1,2-dimethoxyethane as an internal reference.

Table 1
Crystallographic data and structure refinement details for **1** and **2**.

	1	2
Empirical formula	$C_{17}H_{21}N_3NiO_8S$	$C_{38}H_{48}N_6NiO_{14}S_2$
fw	486.14	935.65
Temperature (K)	296(2)	100(2)
Cryst. Syst.	Monoclinic	Orthorhombic
Space group	P 21/n	*Pbca*
a (Å)	14.471(4)	19.7783(9)
b (Å)	7.838(2)	9.6876(5)
c (Å)	18.439(6)	22.0023(10)
α, °	90	90
β, °	92.048(11)	90
γ, °	90	90
V (Å^3)	2090.1(10)	4215.7(3)
Z	4	4
ρ_{calc} (g cm^{-3})	1.545	1.474
μ(Mo Kα) (mm^{-1})	1.077	0.633
F (0 0 0)	1008	1960
R1[a] ($I \geq 2\sigma$)	0.0284	0.0774
wR2[b] ($I \geq 2\sigma$)	0.0684	0.1954
GOOF	1.081	1.207

[a] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.
[b] $wR2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$.

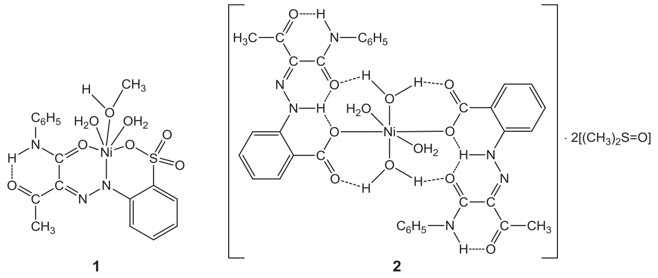
Table 2
Selected bond distances (**Å**) and angles (°) for **1** and **2**.

1		2	
Ni1–O1	1.9847 (15)	Ni1–O1	2.036 (3)
Ni1–O4	2.0550 (15)	Ni1–O1′	2.036 (3)
Ni1–O5	2.0445 (17)	Ni1–O5	2.080 (3)
Ni1–O6	2.0848 (17)	Ni1–O5′	2.080 (3)
Ni1–O8	2.0700 (16)	Ni1–O6	2.061 (3)
Ni1–N3	2.0302 (18)	Ni1–O6′	2.061 (3)
O1–Ni1–N3	89.79 (6)	O1–Ni1–O1	180.0
O1–Ni1–O5	94.15 (6)	O1–Ni1–O6	91.05 (12)
N3–Ni1–O5	90.76 (7)	O1–Ni1–O6′	88.95 (12)
O1–Ni1–O4	175.54 (7)	O1–Ni1–O6	88.95 (12)
N3–Ni1–O4	92.78 (6)	O1–Ni1–O6′	91.05 (12)
O5–Ni1–O4	89.46 (6)	O6–Ni1–O6	180.0
O1–Ni1–O8	92.97 (6)	O1–Ni1–O5	93.57 (11)
N3–Ni1–O8	177.04 (6)	O1–Ni1–O5′	86.43 (11)
O5–Ni1–O8	87.98 (7)	O6–Ni1–O5	89.28 (11)
O4–Ni1–O8	84.54 (6)	O6–Ni1–O5′	90.72 (11)
O1–Ni1–O6	85.49 (7)	O1–Ni1–O5′	86.43 (11)
N3–Ni1–O6	92.09 (7)	O1–Ni1–O5	93.57 (11)
O5–Ni1–O6	177.13 (6)	O6–Ni1–O5	90.72 (11)
O4–Ni1–O6	90.77 (6)	O6–Ni1–O5	89.28 (11)
O8–Ni1–O6	89.19 (7)	O5–Ni1–O5	180.0

3. Results and discussion

3.1. Synthesis and characterization of **1** and **2**

The known NaH$_2$L^1 and H$_3$L^2 were prepared according to our earlier works [10,11]. Reaction of Ni(CH$_3$COO)$_2$ 4H$_2$O with NaH$_2$L^1 or H$_3$L^2 in methanol in the absence or presence of dimethylsulfoxide led to the mononuclear Ni(II) complexes [Ni(HL^1)(H$_2$O)$_2$(CH$_3$OH)] (**1**) or [Ni(H$_2$L^2)$_2$(H$_2$O)$_4$] [(CH$_3$)$_2$S=O)$_2$] (**2**), respectively (Scheme 1). These complexes were characterized by both X-ray structural determination and spectroscopic methods. In the IR spectra of **1** and **2**, the *ν*(C=O) and *ν*(C=N) signals appear at 1676 and 1598, 1669 and 1611 cm^{-1}, respectively, values that are significantly shifted in relation to the corresponding signals of NaH$_2$L^1 or H$_3$L^2 [10,11]. Mass spectrometry of a methanol solution



Scheme 1. Schematic representation of **1** and **2**.

of **1** and **2** shows relevant peaks at m/z = 455.06 [Mr—CH_3OH + H]$^+$ (for **1**) and 780.36 [Mr—2$(CH_3)_2S{=}O$ + H]$^+$ (for **2**). Elemental analyses and X-ray crystallography are also in agreement with the proposed formulations.

In both crystal structures, the nickel ion has a distorted octahedral coordination geometry with the chelating hydrazone and water (or methanol in the case of **1**) ligands occupying the four equatorial positions and the apical sites being engaged with the water molecules (Fig. 1). Despite the fact that a nickel(II) acetate were used for the synthesis of **1** and **2**, the resonance assisted hydrogen bond (RAHB) system [18] was not destroyed in $(H^2L^2)^-$ and the hydrazone hydrogen atoms participate in intramolecular RAHB system, O3C8C7N2N1H(N1) (Fig. 1). This can conceivably be due to the involvement of coordinated carboxylic group in the intra- and intermolecular hydrogen bonding. Generally, both compounds present inter- and intramolecular noncovalent interactions [19] involving the NH, sulfonic, carboxylic groups as well as aromatic moieties of coordinated ligands (Fig. 2). In addition, the dimethylsulfoxide molecule in **2** contacts with coordinated water molecules and donor ($C_{aromatic}$—H) or acceptor ($C_{carboxylic}$=O) moieties of $(H^2L^2)^-$ (Fig. 2).

*3.2. Catalytic activity of **1** and **2** in the Henry reaction*

Initially, benzaldehyde was chosen as a test substrate to determine the best conditions for the Henry reaction with nitroethane to afford the corresponding β-nitroalcohol, *i.e.* (1*R*,2*R*)- or (1*R*,2*S*)-2-nitro-1-phenylpropan-1-ol (Scheme 2). During the first step in the optimization of the reaction conditions, the catalyst and solvent effects were examined and the observed results are summarized in Table 3. The obtained results clearly shows that the reaction is highly sensitive to the nature of the solvent employed and water is the best reaction media in terms of yield and diastereoselectivity (Table 3). Thus, catalyst **1** behaves as the most active and diastereoselective catalyst among $Ni(CH_3COO)_2{\cdot}4H_2O$, NaH_2L^1,

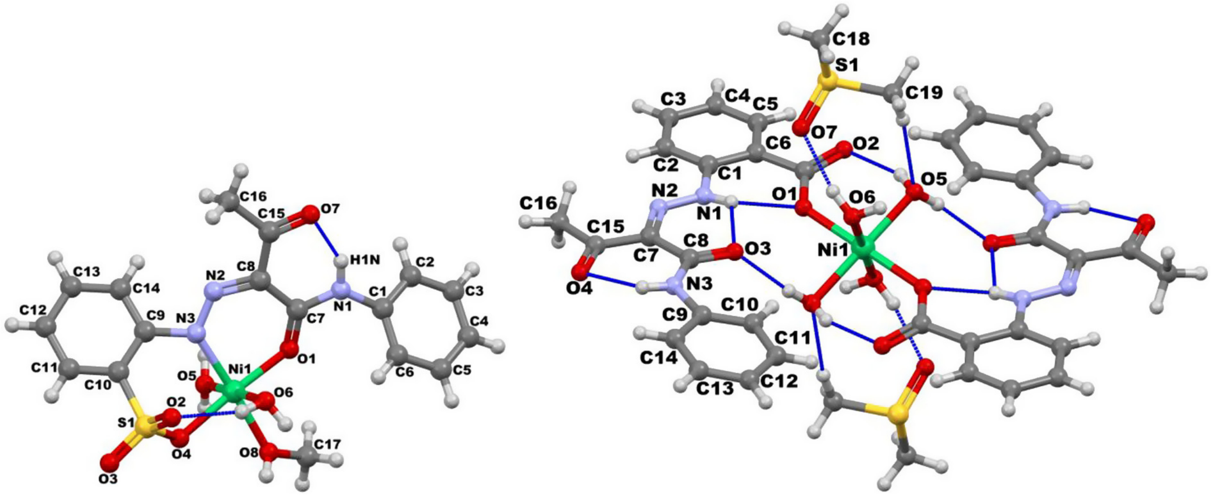


Fig. 1. Molecular structures of **1** and **2**.

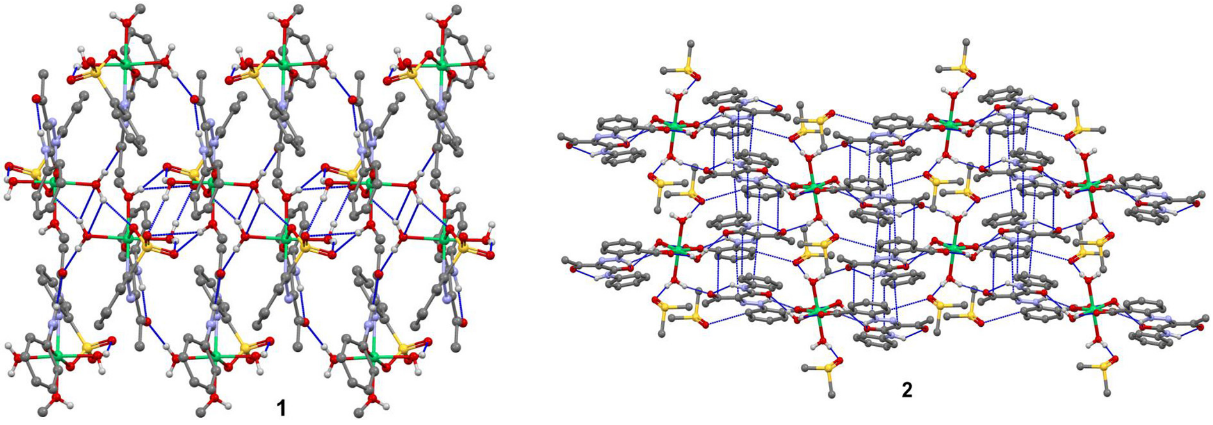
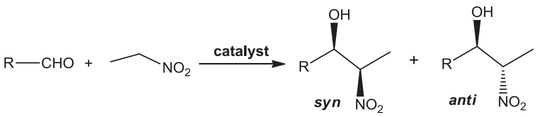


Fig. 2. Packing structures of **1** and **2**. Noncovalent interactions are shown as dashed blue lines. In **2** H atoms are omitted. Symmetry codes to generate equivalent atoms: (**1**) *i*) x, y, z; *ii*) −x, −y, −z; (**2**) *i*) x, y, z; *ii*) −x, −y, −z.



Scheme 2. The nitroaldol (Henry) reaction.

H_3L^2, **1** and **2** in the water medium as well as under catalyst- and solvent-free conditions (entry 16, Table 3). In the next step, to establish the optimized conditions, the variations of catalyst amount, reaction time and temperature were studied using **1** as a catalyst in water (Table 4). The increase of reaction time from 0.5 h to 24 h led to higher conversion (entries 1–5, Table 4), however the 5 h appears to be the optimal reaction time. The variation of the catalyst amount (entries 6–9, Table 4) allowed to conclude that 3 mol% loading is optimal for the given reaction conditions and thus this amount was used for the further studies. The increase in temperature led to decrease of yield and diasteroselectivity (entries 8, 10–12, Table 4). From the observed results, higher chemical yield and diasteroselectivity were obtained at room temperature (entry 8, Table 4). Due to poor solubility of **1** in nitroethane (solvent-free conditions, entry 13), a lower yield (49%) was observed.

Next, based on the above optimized results, we tested a variety of *para*-substituted [$N(CH_3)_2$, OCH_3, CH_3, Cl and NO_2] aromatic and aliphatic aldehydes to determine the generality of the method, in water at room temperature (Table 5). Thus, the electron-withdrawing substituents on the phenyl ring of the aromatic aldehydes favour the reaction, whereas electron-donating ones decrease the yield of β-nitroalcohols (entries 1–6, Table 5). To test the electronic and steric effects of the substrate on the reaction, aromatic aldehydes such as 2-nitrobenzaldehyde, 2-methylbenzaldehyde and 2,4,6-trimethylbenzaldehyde as the substrates (entries 7–9, Table 3) were employed. The slight decrease on the yields for the *ortho*-nitro or -methyl benzaldehyde relative to those for the

Table 3
Catalyst screening and optimization.[a]

Entry	Catalyst	Solvent	Yield, %[c]	Selectivity, *syn/anti*[d]
1[b]	Blank	No solvent	–	–
2	Blank	MeCN	–	–
3		MeOH	–	–
4		H_2O	–	–
5	$Ni(CH_3COO)_2·4H_2O$	MeCN	–	–
6		MeOH	5	60:40
7		H_2O	6	62:38
8	NaH_2L^1	MeCN	9	57:43
9		MeOH	27	60:40
10		H_2O	39	60:40
11	H_3L^2	MeCN	5	59:41
12		MeOH	26	59:41
13		H_2O	35	60:40
14	**1**	MeCN	33	62:38
15		MeOH	47	63:37
16		H_2O	62	69:31
17	**2**	MeCN	29	61:39
18		MeOH	36	63:37
19		H_2O	51	69:31

[a] Reaction conditions: catalyst precursor: $Ni(CH_3COO)_2·4H_2O$, NaH_2L, **1** and **2** (1.0 mol%), acetonitrile, methanol or H_2O (2 mL), nitroethane (4 mmol) and benzaldehyde (1 mmol), reaction time: 24 h, reaction temperature: 20 °C.
[b] Solvent-free conditions, using nitroethane as solvent (2 mL).
[c] Determined by ^1H NMR, based on the starting benzaldehyde.
[d] Calculated by ^1H NMR (see Experimental).

Table 4
Optimization of the Henry reaction condition catalyzed by **1**.[a]

Entry	Time, h	Amount of catalyst, mol%	Temp., °C	Yield, %[b]	Selectivity, *syn/anti*[c]
1	0.5	1.0	20	27	68:32
2	1	1.0	20	35	68:32
3	3	1.0	20	29	69:31
4	5	1.0	20	62	69:31
5	24	1.0	20	62	69:31
6	5	1.0	20	62	69:31
7	5	2.0	20	69	70:30
8	5	3.0	20	74	70:30
9	5	4.0	20	74	70:30
10	5	3.0	35	74	71:29
11	5	3.0	55	73	64:36
12	5	3.0	75	70	59:41
13[d]	5	3.0	20	49	61:39

[a] Reaction conditions: 1.0–4.0 mol% of catalyst precursor (typically 3.0 mol%), H_2O (2 mL), nitroethane (4 mmol) and aldehyde (1 mmol).
[b] Determined by ^1H NMR analysis (see Experimental).
[c] Calculated by ^1H NMR.
[d] Solvent-free conditions, using nitroethane as solvent (2 mL).

Table 5
Henry reaction of nitroethane with various aldehydes catalyzed by **1**.[a]

Entry	Substrate	Yield, %[b]	Selectivity, *syn/anti*[c]
1	4-(Dimethylamino)benzaldehyde	67	69:31
2	4-Methoxybenzaldehyde	71	69:31
3	4-Methylbenzaldehyde	72	70:30
4	Benzaldehyde	74	70:30
5	4-Chlorobenzaldehyde	76	69:31
6	4-Nitrobenzaldehyde	80	71:29
7	2-Nitrobenzaldehyde	73	69:31
8	2-Methylbenzaldehyde	73	69:31
9	2,4,6-Trimethylbenzaldehyde	70	66:34
10	Propionaldehyde	82	74:26
11	Butyraldehyde	81	73:27
12	Pentanal	81	73:27

[a] Reaction conditions: 3.0 mol% of catalyst **1**, H_2O (2 mL), nitroethane (4 mmol) and aldehyde (1 mmol), reaction time: 5 h.
[b] Determined by ^1H NMR analysis (see Experimental part).
[c] Calculated by ^1H NMR.

para-substituted ones can be due to the steric hindrance of the former.

4. Conclusions

Two new mononuclear Ni(II) complexes bearing an *ortho*-sulfonate (in **1**) or *ortho*-carboxylate (in **2**) functionalized arylhydrazone of acetoacetanilide have been synthesized and characterized. In both crystal structures, the nickel ion has a distorted octahedral coordination geometry. These Ni(II)-arylhydrazone complexes catalyze the Henry reaction between nitroethane and various aromatic and aliphatic aldehydes, providing in an efficient and simple method the desired β-nitroalkanols in moderate to good yields (up to 82%) and *syn/anti* diastereoselectivities (up to 74:26%) in water at room temperature.

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