Journal of Fluorine Chemistry xxx (2013) xxx–xxx

Contents lists available at SciVerse ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

Iron(II) complexes are suitable catalysts for the isomerization of trifluoromethylated allylic alcohols. Synthesis of trifluoromethylated dihydrochalcones

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ARTICLE INFO

Article history:
Received 6 May 2013
Received in revised form 27 May 2013
Accepted 29 May 2013
Available online xxx

Keywords:
Iron
Fluorinated compounds
Catalysis
Isomerization
Dihydrochalcone

ABSTRACT

We demonstrated that iron(II) complexes can substitute platinum metals as well as iron(0) carbonyls for the isomerization of γ-trifluoromethylated allylic alcohols into β-trifluoromethylated ketones. In particular, iron(II)-tetra(isonitrile) complexes were employed for the synthesis of a series of trifluoromethylated dihydrochalcones variously decorated on each aromatic ring.

1. Introduction

The isomerization of allylic alcohols into the corresponding saturated carbonyl compounds, often referred to as redox isomerization is an efficient, selective, redox- and atom-economical, one-pot isomerization process [1]. Second- and third-row transition metals, such as Ru, Rh, and Ir, have been widely used in isomerization of allylic alcohols [2]. Faced with an ever-increasing demand for precious metal, their replacement by abundant, less expensive and environmentally benign first-row transition metals is eagerly sought after. In this context, iron salts, which are very abundant on Earth and usually non-toxic, are the subject of current intense research [3]. It has been demonstrated that various iron(0) carbonyls that include homoleptic [$Fe(CO)_5$] [4], [$Fe_2(CO)_9$] [5], [$Fe_3(CO)_{12}$] [6] as well as heteroleptic [(bda)$Fe(CO)_3$] (bda = *trans*-benzylideneacetone) [7] and [(COT)$Fe(CO)_3$] (COT = cyclooctatetraene) [7] are catalytically active in the isomerization of allylic alcohols under irradiation conditions. Good evidence was provided that photodissociation of these complexes gave [$Fe(CO)_3$] that would act as the true catalytic species [8]. However, as a source of carbon monoxide, iron(0) carbonyls are toxic and not really appropriate for the development of an asymmetric variant of the isomerization reaction. Consequently, we focused our attention on iron(II) catalysts which we could not find precedence in the literature as far as isomerization of allylic alcohols is concerned. In addition, a number of chiral iron(II)-catalysts have been successfully applied in asymmetric transfer hydrogenation and would be definitely evaluated in the isomerization reaction that is also a hydride transfer reaction [9,10,11]. We recently reported the first involvement of trifluoromethylated allylic alcohols in ruthenium-catalyzed isomerization [12]. The presence of the CF_3 group is beneficial to accelerate the hydride insertion step and thus allows higher reactivity in particular for trisubstituted C=C bond of allylic alcohols which isomerizations are conducted under mild conditions. We and others demonstrated that a ruthenium hydride intermediate is generated from an allylic alcohol and a ruthenium complex in basic medium with concomitant formation of the corresponding α,β-unsaturated carbonyl derivative [13]. Thus, we hypothesized that: (i) such a discrete metal hydride might be an intermediate in iron-catalyzed allylic alcohol isomerization and (ii) iron complexes, able to catalyze transfer hydride reduction, might also be active in isomerization of allylic alcohols. For this study, we focused on three types of iron(II) complexes. One of the most efficient and easily amenable to structural modification type of iron complexes are the modular Morris complexes **C1**–**C3** (Fig. 1)

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http://dx.doi.org/10.1016/j.jfluchem.2013.05.028

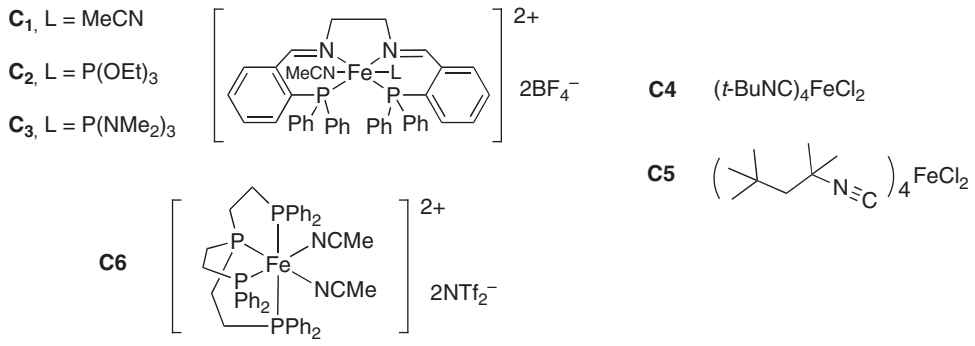


Fig. 1. Iron(II) catalysts.

[10]. Such complexes are able to reduce carbonyl functions through hydride transfer hydrogenation in high yields. Moreover, it is worth mentioning that reduction of enones in the presence of such iron complexes led to the corresponding saturated alcohols through reduction of the activated C=C double bond [10d,e]. Variation of the apical ligand nature (L = MeCN, $P(OEt)_3$, $P(NMe_2)_3$...) can also modify the reactivity of the iron complex. The second family of iron complexes are achiral analogues of the chiral iron-tetra(isonitrile) complexes reported by Reiser and coworkers (complexes **C4**–**C5**, Fig. 1) [11]. We also evaluated the complex $[(PP_3)Fe(NCMe)_2][NTf_2]_2$ ($PP_3 = P(CH_2CH_2PPh_2)_3$) **C6** [14].

Obviously, switching from ruthenium to iron in isomerization of allylic alcohols would create a fully cost-effective reaction. In order to demonstrate the ability of iron(II) complexes to catalyze the isomerization reaction, we targeted trifluoromethylated dihydrochalcones as fluoro analogues of these members of the flavonoid family [15]. Indeed, dihydrochalcones are key intermediates for the synthesis of bioactive molecules that possess a wide range of properties including anticancer, antiviral, antibacterial, antioxydant among others [16]. The search for novel substitution patterns for dihydrochalcones also included fluorinated motifs. Towards this goal, Surya Prakash, Mathew and coworkers have recently described a synthetic route to CF_3-dihydrochalcones through intermolecular Friedel-Crafts acylation and alkylation of 4,4,4-trifluorocrotonic acid with various arenes in the presence of excess triflic acid (Fig. 2, top) [17] However, this methodology is limited in that only CF_3-dihydrochalcones bearing identically substituted aryls at C_1 and C_3 positions can be synthesized. Moreover, in this synthetic approach, dihydrochalcones are sometimes accompanied by other regioisomers. Konno and coworkers obtained some CF_3-dihydrochalcones through asymmetric rhodium-catalyzed 1,4-conjugate arylation of 4,4,4-trifluoro-1-phenyl-2-buten-1-one, and hence aryl variety was generated only at C_3 (the aryl at C_1 was constantly a phenyl group) [18]. We herein propose an alternative route to single regioisomers of CF_3-dihydrochalcones that feature variously decorated aromatic rings through iron(II)-catalyzed isomerization of γ-CF_3 allylic alcohols (Fig. 2, bottom).

2. Results and discussion

In a first series of experiments, we examined the reaction conditions optimized for the ruthenium-catalyzed isomerization: 1 mol% catalyst and 1 equivalent of Cs_2CO_3 in toluene (0.5 M) at 25–50 °C. Under these conditions in the presence of allylic alcohol **1a**, the isomerization took place in the presence of iron(II) catalysts **C1**–**C5** but failed with **C6** (Table 1). With catalyst **C6**, we recovered the starting material quantitatively without any isomerized product. With Morris type catalysts **C1**-**C3**, the isomerization required a temperature of 50 °C to obtain full conversion of **1a**. The isomerization performed best with the tetra(isonitrile) catalysts **C4** and **C5** at 25 °C for 22 h providing the desired β-trifluoromethylated ketone **2a** in up to 72% yield after silica gel column chromatography (Table 1, entry 5). When the reaction was run at 50 °C with **C5**, full conversion was reached within 7 h, albeit in a much lower isolated yield due to the concomitant formation of ketolisation byproducts that were favoured at higher temperature (Table 1, entry 6). Advantageously, catalysts **C4** and **C5** are easily synthesized by treatment of the corresponding isonitriles with $FeCl_2{\cdot}4H_2O$ in methanol. We selected the iron(II)-tetra(isonitrile) catalyst **C5** for further investigation of reaction parameters and substrate scope.

The solvent effect was evaluated next (Table 2, entries 1–6). The reaction failed in $CHCl_3$ and MeOH but the isomerization reaction

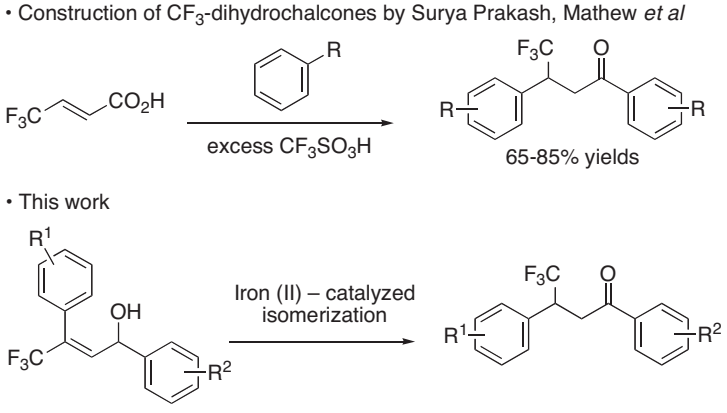
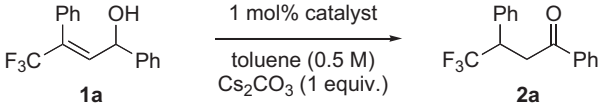


Fig. 2. Proposed investigation.

Table 1
Catalyst screening.



Entry	Catalyst	*T* (°C)	Time (h)	Conv. (%)[a]	**2** (Yield %)[b]
1	**C1**	50	18	93	70
2	**C2**	50	22	67	24
3	**C3**	50	22	88	40
4	**C4**	25	21	100	69
5	**C5**	25	22	100	72
6	**C5**	50	7	100	35
7	**C6**	50	27	0	–

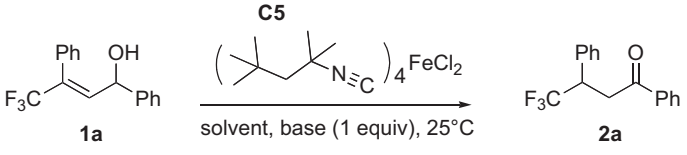
[a] Conversion was determined by ^{19}F NMR using trifluorotoluene as internal standard.
[b] Yield of isolated product by column chromatography.

took place in toluene, CH_2Cl_2, THF and CH_3CN with preference for toluene that afforded the β-trifluoromethylated ketone **2a** in the highest isolated yield. The isomerization was conducted with and without base and we found that base-free conditions are not appropriate for the isomerization process (Table 2, entry 7). This observation may indicate that the reaction proceeds through iron alkoxide intermediate by displacement of a chloride ligand. The conversions were not complete with *t*-BuOK and K_2CO_3 whereas full conversion was obtained in the presence of Cs_2CO_3 (Table 2, entries 8,9 *vs* 1). The molar ratio of catalyst **C5** could be reduced to 0.1 mol% (Table 2, entry 10); however, although full conversion was reached, the reaction yield is lower compared to the reaction run with 1 mol% of catalyst. A test experiment without catalyst but with Cs_2CO_3 confirmed that the catalyst is required for the isomerization; nevertheless, with *t*-BuOK alone the reaction, although very messy, produced *ca.* 15% of **2** [19].

Under the optimal conditions, the substrate scope was investigated by employing a variety of bis-aryl allylic alcohols in order to synthesize β-CF_3 dihydrochalcones that feature diversely decorated Ar^1 and Ar^2 aryl groups. The results are summarized in Table 3. Substrates featuring Ar^1 substituted with electron-donating, electron-neutral, and electron-withdrawing groups gave the dihydrochalcones in a similar range of yield 65–76%. Halogen and electron-donating substituents on Ar^2 are suitable with the exception of the 2-methoxy substituent (substrate **1j**, Table 3, entry 10) that gave only 28% yield. This poor yield might result of steric hindrance or, more likely, a chelation between the oxygen atom of the methoxy group and the iron alkoxide. In addition, a 4-nitro substituent (substrate **1l**, Table 3, entry 12) did not react at all. In this latter case, the strong electron-withdrawing NO_2 group renders more acidic the hydrogen atom at C_1. In other words, this hydrogen atom has a lower hydride character and could be responsible for the poor reactivity of substrate **1l**.

The stereocontrol of C(sp^3)–CF_3 stereogenic centres at the β-position of the carbonyl function in dihydrochalcone motif would be of great added value to the method [18,20]. Towards this goal, we have applied our recently published approach consisting in the enantiospecific *syn*-specific 1,3-hydride transfer starting from optically enriched allylic alcohols **1a** [12a]. The tetra(isonitrile) catalysts **C4** and **C5** gave the isomerized product in only 34% ee with 36% enantiospecificity, but Morris complex **C1** afforded the β-CF_3 dihydrochalcone **2a** in 84% ee and 89% es (Table 4). These results demonstrate that the iron(II)-catalyzed isomerization could proceed enantiospecifically through *syn*-specific 1,3-hydride shift. In addition, we attempted the enantioselective isomerization from racemic allylic alcohol **1a** and a chiral Morris-type catalyst featuring an enantiopure diamine (*R*,*R*)-1,2-diphenylethylenediamine. Unfortunately however, the result was a very poor ee value

Table 2
Screening of reaction parameters.

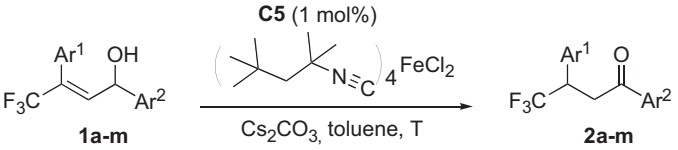


Entry	Solvent	Base	**C5** (*x* mol%)	Time (h)	**2** (Yield %)[a]
1	Toluene	Cs_2CO_3	1	22	72
2	CH_2Cl_2	Cs_2CO_3	1	47	60
3	$CHCl_3$	Cs_2CO_3	1	28	–
4	THF	Cs_2CO_3	1	52	42
5	MeOH	Cs_2CO_3	1	25	–
6	MeCN	Cs_2CO_3	1	25	60
7	Toluene	Without	1	24	–
8	Toluene	K_2CO_3	1	22	16[b]
9	Toluene	*t*-BuOK	1	22	58[b]
10	Toluene	Cs_2CO_3	0.1	18	56

[a] Yield of isolated product by column chromatography.
[b] Conversion determined by ^{19}F NMR.

Please cite this article in press as: D. Cahard, et al., J. Fluorine Chem. (2013), http://dx.doi.org/10.1016/j.jfluchem.2013.05.028

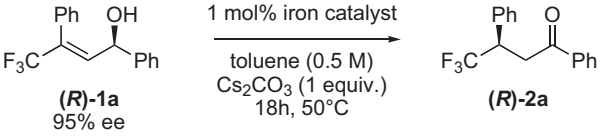
Table 3
β-CF_3 Dihydrochalcone syntheses through iron(II)-catalyzed isomerization.



Entry	Ar^1	Ar^2	*T* (°C)	Time (h)	**2**	**2** (Yield %)[a]
1	Ph	Ph	25	22	**2a**	72
2	4-$OMeC_6H_4$	Ph	40	22	**2b**	76
3	4-BrC_6H_4	Ph	40	21	**2c**	72
4	4-MeC_6H_4	Ph	40	22	**2d**	75
5	3,4-$Me_2C_6H_3$	Ph	40	23	**2e**	69
6	4-$CF_3C_6H_4$	Ph	40	13	**2f**	65
7	4-ClC_6H_4	Ph	40	23	**2g**	74
8	Ph	4-BrC_6H_4	40	23	**2h**	85
9	Ph	4-ClC_6H_4	40	22	**2i**	69
10	Ph	2-$OMeC_6H_4$	100	120	**2j**	28
11	Ph	3-$OMeC_6H_4$	40	22	**2k**	70
12	Ph	4-$NO_2C_6H_4$	40–80	48	**2l**	–
13	4-ClC_6H_4	4-$OMeC_6H_4$	40	42	**2m**	49

[a] Yield of isolated product by column chromatography.

Table 4
Isomerization of enantioenriched allylic alcohol **1a**.



Entry	Catalyst	Yield (%)[a]	Ee (%)[b]	Es (%)[c]
1	**C4** or **C5**	75	34	36
2	**C1**	86	84	89

[a] Yield of isolated product.
[b] Enantiomeric excess measured by HPLC using OD-H column.
[c] Enantiospecificity: Es = 100 × (ee product)/(ee reactant).

(<10% ee). This outcome may indicate that a similar mechanism underpins both ruthenium and iron catalysts. Further investigations are required in order to gain mechanistic insights.

3. Conclusion

We have demonstrated for the first time the potential of iron(II)-catalysts, in particular dichlorotetra(isonitrile) iron(II) in the isomerization of a series of trifluoromethylated allylic alcohols. Indeed, iron(II) catalysts appear to represent a cost-effective replacement of platinum metal catalysts and an environmentally friendly substitute for toxic iron(0) complexes. A series of β-CF_3 dihydrochalcones diversely decorated on each aromatic rings have been synthesized in yields ranging from 28 to 85%. The mechanism of this transformation in the presence of iron catalyst remains elusive, but we have demonstrated a high enantiospecific process from enantioenriched allylic alcohol leading to optically enriched β-CF_3 dihydrochalcone in up to 84% ee. Further applications and mechanistic investigations are in progress in our laboratories.

4. Experimental

4.1. General remarks

^1H (300 MHz), ^{13}C (75.5 MHz) and ^{19}F (282 MHz) NMR spectra were recorded on Bruker AVANCE 300. Chemical shifts in NMR spectra are reported in parts per million from TMS or $CFCl_3$ resonance as the internal standard. IR spectra were recorded on a Perkin-Elmer IRFT 1650 spectrometer. The conversions were determined by ^{19}F NMR. Unless otherwise noted, all reagents were purchased from commercial sources and were used without further purification. Toluene was distilled from sodium benzophenone under a positive pressure of nitrogen and degassed before use. The allylic alcohols were prepared using literature methods [12a].

4.2. Representative procedure for the isomerization

In a Schlenk tube under inert atmosphere, were added the (*E*)-4,4,4-trifluoro-1,3-diphenylbut-2-en-1-ol **1a** (278.27 mg, 1 mmol), degassed toluene (2 mL), caesium carbonate (325.8 mg, 1 mmol), and iron catalyst **C5** (6.84 mg, 1 mol%). The reaction was conducted at 25 °C for 22 h until the signal of starting allylic alcohol disappeared by ^{19}F NMR analysis. Then, the reaction mixture was filtered through a pad of celite, concentrated under reduced pressure and purified by column chromatography on silica gel (petroleum ether/ethyl acetate: 99/1) to give the desired 4,4,4-trifluoro-1,3-diphenylbutan-1-one **2a**. Yield: 72%; white solid (mp = 66 °C). ^1H NMR ($CDCl_3$) δ 3.52 (dd, 1H, J = 17.8 Hz, J = 4.3 Hz), 3.64 (dd, 1H, J = 17.8 Hz, J = 8.8 Hz), 4.11–4.25 (m, 1H), 7.18–7.87 (m, 10H); ^{13}C NMR ($CDCl_3$) δ 38.4 (q, J = 2.0 Hz), 44.9 (q, J = 27.4 Hz), 127.1 (q, J = 279.5 Hz), 128.2, 128.4, 128.8,

128.9, 129.2, 133.7, 134.7 (q, J = 1.9 Hz), 136.4, 195.4; ^{19}F NMR ($CDCl_3$) δ -70.2 (d, J = 9.7 Hz); HRMS Calcd for $C_{16}H_{13}F_3O$ (M+), 278.0918, Found 278.0920; IR (neat) ν 3068, 1680, 1300, 1250, 1187, 1153, 1103 cm^{-1}.

Acknowledgments

This work is promoted by the Interregional CRUNCh Network. V.B. thanks the Région Haute-Normandie for a PhD fellowship. X.D. thanks the China Scholarship Council for a PhD fellowship.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jfluchem.2013.05.028.

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