

Diastereoselective three-component Mannich reaction catalyzed by silica-supported ferric hydrogensulfate

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Abstract A highly *anti*-diastereoselective three-component Mannich reaction of aromatic amines and aromatic aldehydes with cyclohexanone in the presence of silica-supported ferric hydrogensulfate has been developed. The best selectivity was obtained where there were electron-donating groups on both aldehyde and amine. Selectivity decreases when electron-withdrawing groups are present on the aldehyde; in these cases selectivity is improved if an electron-donating group is present on the amine.

Keywords Three-component ·
Diastereoselective *anti*-Mannich reaction ·
Silica-supported ferric hydrogensulfate ·
Iron heterogeneous catalysis

Introduction

The Mannich reaction is one of the most important methods for construction of carbon–carbon bonds to build β -aminocarbonyl compounds [1–5]. These compounds are useful precursors for synthesis of β -lactams [3, 6, 7], α [8–11] and γ -aminoalcohols [13], α and β -amino acid derivatives [13], peroxy acetylenic alcohols/ethers [4], and medicinally important materials [1].

Several strategies are available for diastereoselective synthesis of β -aminocarbonyl compounds, including organocatalysis [6, 7, 12–21], transition-metal catalysis [8–11, 22–25], Bronsted and Lewis acid catalysis [26–38], phase-transfer catalysis [39–41], HPA catalysis [42, 43], biocatalysis [44], and ionic-liquid catalysis [45, 46]. Organocatalytic asymmetric Mannich reactions are the most important approach to the direct *anti*-enantioselective reaction of aldehydes and ketones [47, 48].

Iron is an important metal in living systems and is a sustainable metal catalyst for performing a wide range of different chemical transformations. Iron salts have often been used in organic synthesis, for example oxidation, reduction, coupling reactions, and cycloaddition, because they are inexpensive, nontoxic, readily available, easily recyclable, and environmentally benign [49, 50].

Therefore, to achieve diastereoselective synthesis of β -aminoketones via a three-component Mannich reaction, we chose ferric hydrogensulfate (FHS) as catalyst. Recently we have successfully used FHS for nucleophilic addition of nucleophiles to aldehydes [51–53]. In this work, we performed nucleophilic addition of enols to aldimines. Herein we report, for the first time, the iron-salt-catalyzed three-component Mannich reaction of aromatic aldehydes and aromatic amines with cyclohexanone to afford Mannich products with high *anti*-diastereoselectivity.

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Results and discussion

We selected the three-component Mannich reaction of aniline (1 eq), benzaldehyde (1 eq), and cyclohexanone (1.2 eq) as model reaction to optimize the reaction conditions. When we used FHS (10 mol %) as catalyst in ethanol, moderate diastereoselectivity (*anti:syn* = 67:33) was obtained.

Furthermore reduction of catalyst molar ratio did not change the diastereoselectivity of the reaction significantly (Table 1, entries 2 and 3). Other catalysts, for example FeCl_2 and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (Table 1, entries 10 and 14) also gave moderate diastereoselectivity. With $\text{Mn}(\text{HSO}_4)_2$ as catalyst the percentage of the *anti* isomer improved to 75 % (Table 1, entry 9). When we used FHS supported on silica (1:9) as catalyst better diastereoselectivity was observed. The best result (>99 % *anti*) was obtained when the molar ratio of catalyst to starting material was approximately 10 mol % (Table 1, entry 6).

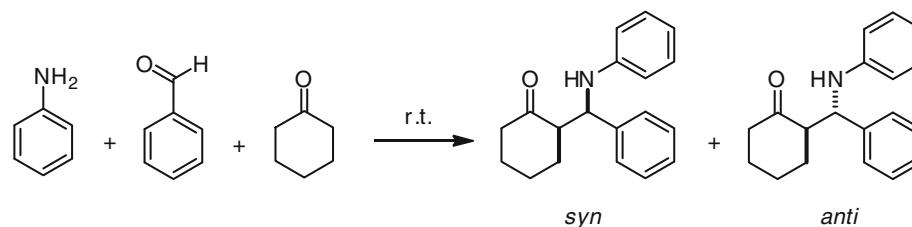
As the results in of Table 1 show, silica alone improves the selectivity of the reaction to 84 % *anti* isomer compared with the catalyst free and solid state reactions (Table 1, entries 4, 5, 12). However when FHS is supported on silica using the same conditions the reaction time drops from 2 h to 30 min and diastereoselectivity increases from 84 % to more than 99 %. According to Table 1, when the reaction is carried out under catalyst-free conditions in

ethanol, diastereoselectivity decreases to 60:40 ratio (Table 1, entry 4).

We believe diastereoselectivity depends on different factors, for example solvent, silica, and iron salt. When the ratio of ferric hydrogensulfate to silica gel was 9:1, catalytic activity was highest. However, when NaHSO_4 was used instead of $\text{Fe}(\text{HSO}_4)_3$, with the same molar ratio, diastereoselectivity decreased to 72:28. This observation shows that besides the solvent and silica gel, the iron cation has a significant effect on the diastereoselectivity of the reaction.

When the reaction conditions had been optimized for the model reaction, we screened aromatic aldehydes and aromatic amines in reactions with cyclohexanone. As the results in Table 2 show, with benzaldehyde itself the only stereoisomer obtained is *anti*, except with 4-Cl aniline which gives 80 % *anti* isomer (Table 2, entries 1–5). Weak electron-withdrawing or electron donating groups on benzaldehyde, for example 4-chloro and 4-methyl,

Table 1 Catalytic *anti*-diastereoselective three-component Mannich reaction of aniline and benzaldehyde with cyclohexanone

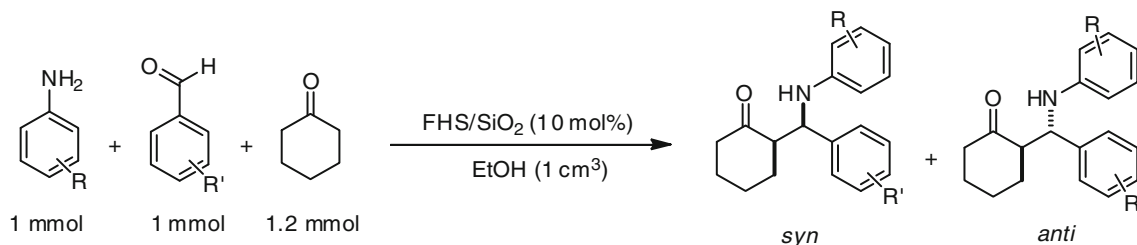


Entry	Cat (mol %)	Solvent	Yield/% ^a	<i>anti</i> : <i>syn</i> ^b	Time
1	$\text{Fe}(\text{HSO}_4)_3$ (10 mol %)	EtOH	76	67:33	30 min
2	$\text{Fe}(\text{HSO}_4)_3$ (5 mol %)	EtOH	72	68:32	30 min
3	$\text{Fe}(\text{HSO}_4)_3$ (1 mol %)	EtOH	72	68:32	30 min
4	Catalyst-free	EtOH	72	60:40	30 min
5	SiO_2 (0.9 equiv)	EtOH	–	84:16	2 h
6	$\text{Fe}(\text{HSO}_4)_3/\text{SiO}_2$ (10 mol %) ^c	EtOH	72	>99:<1	30 min
7	$\text{Fe}(\text{HSO}_4)_3/\text{SiO}_2$ (5 mol %)	EtOH	72	67:33	30 min
8	$\text{Fe}(\text{HSO}_4)_3/\text{SiO}_2$ (2.5 mol %)	EtOH	77	68:32	2 h
9	$\text{Mn}(\text{HSO}_4)_2$ (7.5 mol %)	EtOH	87	75:25	30 min
10	FeCl_2 (10 mol %)	EtOH	82	67:33	30 min
11	$\text{Fe}(\text{HSO}_4)_3/\text{SiO}_2$ (10 mol %)	CH_3CN	71	71:29	30 min
12	$\text{Fe}(\text{HSO}_4)_3/\text{SiO}_2$ (10 mol %)	Solid state	–	50:50	15 min
13	$\text{Fe}(\text{HSO}_4)_3$ (10 mol %)	MeOH	–	54:46	30 min
14	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (10 mol %)	MeOH	70	67:33	30 min
15	$\text{FeCl}_3/\text{SiO}_2$ (10 mol %)	EtOH	–	72:28	1 h
16	$\text{NaHSO}_4/\text{SiO}_2$ (30 mol %)	EtOH	–	74:26	2 h

^a Isolated yield

^b Determined by ^1H NMR spectroscopy

^c Ferric hydrogensulfate (10 mmol) and silica for column chromatography (90 mmol, 230 mesh) were mixed

Table 2 Scope of the *anti*-diastereoselective three-component Mannich reaction of aromatic amines and aldehydes with cyclohexanone

Compound	R	R'	Yield/% ^a	<i>anti:syn</i> ^b	Time	M.p. (lit. m.p.)/°C
1	H	H	72	>99:<1	30 min	117–118 (116–118 [45])
2	2-Me	H	65	>99:<1	24 h	137–138 (not reported [62])
3	3-Me	H	76	>99:<1	1 h	115–116 (123–124 [54])
4	4-Me	H	82	>99:<1	4 h	113–114 (117–118 [45])
5	4-Cl	H	68	80:20	4 h	118–119 (137–138 [55])
6	2-Me	4-Cl	54	>99:<1	24 h	106–107
7	3-Me	4-Cl	77	>99:<1	3 h	127–128 (not reported [37])
8	4-Me	4-Cl	86	>99:<1	4 h	125–126 (119–121 [60])
9	4-Cl	4-Cl	70	>99:<1	3 h	137–138 (98–99 [45])
10	H	4-Me	70	>99:<1	6 h	118–119 (115–118 [56])
11	2-Me	4-Me	60	>99:<1	24 h	114–115
12	3-Me	4-Me	72	>99:<1	4 h	124–125
13	4-Cl	4-Me	68	>99:<1	6 h	138–139 (105.3–105.9 [61])
14	H	4-NO ₂	56	64:36	6 h	100–101 (123–125 [58])
15	3-Me	4-NO ₂	85	58:42	10 h	161–162 (not reported [63])
16	4-Me	4-NO ₂	76	>96:<4	6 h	145–146 (137–138 [45])
17	4-Cl	4-NO ₂	76	47:53	8 h	121–122 (169–171 [40])
18	H	3-NO ₂	84	58:42	8 h	123–124 (163–165 [40])
19	4-Me	3-NO ₂	85	70:30	4 h	136–137
20	4-Cl	3-NO ₂	74	48:52	12 h	139–140 (127–128 [45])

^a Isolated yield^b Determined by ¹H NMR spectroscopy

respectively, do not change the diastereoselectivity of the reaction. However, the presence of a strong electron-withdrawing group (EWG), for example NO₂, at the *para* or *meta* positions of the benzaldehyde destroys the diastereoselectivity of the reaction. Interestingly, with these EWGs on the aldehyde, some diastereoselectivity is observed when an electron-donating group (EDG) is present on the aromatic amine (Table 2, compounds 16 and 19). As expected, the presence of substitution in the *ortho* position of aniline reduces the nucleophilicity of the compound and thus reaction times increase substantially (Table 2, compounds 2, 6, and 11).

The efficiency of our catalyst was then tested with other ketones as substrates (Table 3). In an initial series of experiments, a representative set of ketones, including both cyclic and acyclic substrates, were reacted with benzaldehyde and aniline in the presence of 10 mol % catalyst.

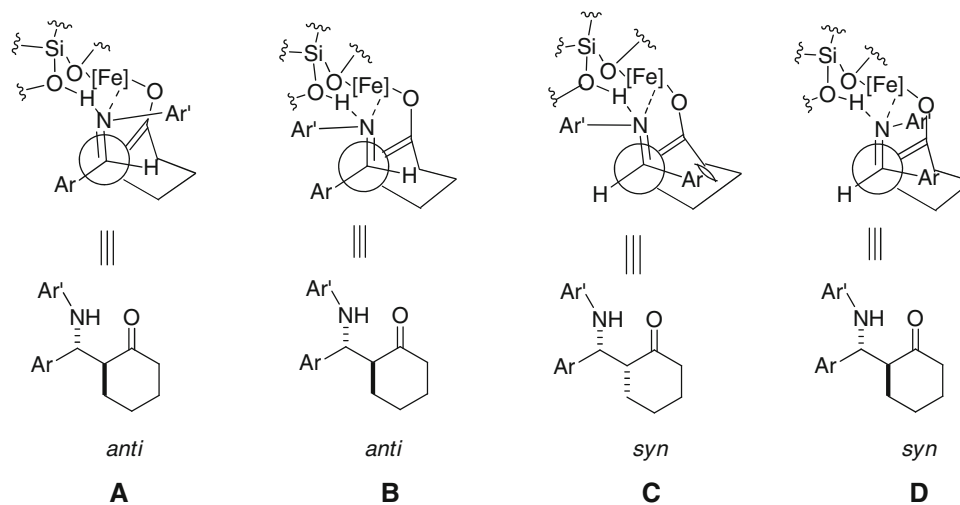
Acyclic ketones required longer reaction times for complete conversion, but regioselectivity was very high. Cyclopentanone behaved similar to cyclohexanone and gave exclusively the *anti* product.

The most plausible mechanism is imine formation between amine and aldehyde followed by nucleophilic addition to the imine of the enol formed by the catalyst. The most probable transition states, which explain the diastereoselectivity of the reaction, are shown in Fig. 1.

We believe the catalyst is mostly involved in the second step of the mechanism. Although it can assist enol formation and nucleophilic addition of enol to aldimine, it mostly controls the diastereoselectivity of the reaction by controlling the stereochemistry of the transition state. As shown in Fig. 1, transition states A and B are more favorable sterically. These transition states will give the *anti* isomer. Transition states C and D are highly hindered

Table 3 Regioselective Mannich reactions of some ketones with aniline and benzaldehyde, catalyzed by silica ferric hydrogensulfate

Entry	Ketone	Product	Yield/% ^a	<i>anti:syn</i> ^b	Time/h	M.p. (lit. m.p.)/ ^o C
1			88	–	6	168–169 (168–169 [53])
2			85	–	6	88–89 (87–88 [57])
3			74	>99:<1	10	169–170 (168–170 [58])
4			83	>99:<1	5	165–166 (164–166 [59])

^a Isolated yield^b Determined by ¹H NMR spectroscopy**Fig. 1** Probable transition state structures

and so unlikely to be formed in the presence of catalyst. This highly energetic transition state will lead to the *syn* isomer. According to the proposed mechanism the *anti* product is expected from the *cis* imine (Fig. 1, **B**), and,

because of the steric effect, the *anti* product is more likely to be formed from the *trans* imine (Fig. 1, **A** and **C**).

Electronic effects of substituents on the aldehyde and amine affect the stability of these transition states. Whereas

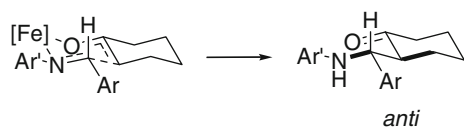


Fig. 2 Electronic effect of the transition state

EWGs on the aldehyde reduce the stability of the transition state and thus destroy the diastereoselectivity of the reaction, EDGs on the amine compensate for the EWGs and increase the diastereoselectivity of the reaction.

We propose that the transition state is chair like and both oxygen and nitrogen coordinate to the iron center (Fig. 2). When the nitro group is located on benzaldehyde the nitrogen of the imine will not coordinate well with the iron center, and thus the steric effect will not determine the diastereoselectivity of the products.

The modified transition state illustrated in Fig. 2 shows that the electronic effect is a major factor affecting the diastereoselectivity of the product. When a methyl group is present at the *para* position of the aniline diastereoselectivity increases. In fact, with imines containing 4-methyl substitution on the aniline ring, the electron density of transition state depicted in Fig. 2 improves and chelation of nitrogen to the iron center will occur more efficiently and diastereoselectivity proceeds toward *anti* (Table 2, compounds **16** and **19**).

In conclusion, we present in this paper a completely diastereoselective three-component Mannich reaction catalyzed by ferric hydrogensulfate supported on silica gel. The advantages of our method are the high yield and excellent selectivity of the reaction, and the inexpensive and heterogeneous catalyst.

Experimental

All solvents and reagents were purchased from Merck and Fluka. The silica for preparation of the supported catalyst was 230 mesh for column chromatography and was purchased from Merck. NMR spectra were recorded on Bruker Aspect 3000 (100 MHz) and Bruker Avance (400 MHz) spectrometers. All chemical shifts are reported as ppm and were referenced to residual solvent signals. IR spectra were recorded on a ThermoNicolet Avatar-370-FTIR spectrometer.

Preparation of silica ferric hydrogensulfate (10 mol %)

Ferric hydrogensulfate (5 mmol) and silica gel 230 mesh for column chromatography (45 mmol) were placed in a mortar and the mixture was ground for 5 min. The mixture was then placed in a 50 cm³ flask, 25 cm³ absolute ethanol

was added, and the mixture was stirred at room temperature for 10 h. The mixture was then filtered and the residue was dried at 100 °C for 2 h. A white homogeneous powder was obtained which was stored in a desiccator.

Typical procedure for synthesis of β -aminocyclohexanones

To a mixture of aromatic amine (1 mmol), aromatic aldehyde (1 mmol), and cyclohexanone (1.2 mmol) in 1 cm³ ethanol, 88.7 mg silica ferric hydrogensulfate (10 mol %) was added. The reaction mixture was stirred at room temperature and the progress of the reaction monitored by TLC. After completion of the reaction, 2 cm³ methanol was added, followed by dropwise addition of water until the product began to precipitate. The mixture was then filtered by suction and the residue was washed with 0.5 cm³ methanol and 0.5 cm³ petroleum ether. The crude product was extracted from the precipitate by washing with CHCl₃. The solution was dried over Na₂SO₄ then the solvent was removed. The solid product obtained was suitable for spectroscopic application. Further purification was performed by crystallization from aqueous ethanol.

2-[(4-Chlorophenyl)][(2-methylphenyl)amino]methyl]-cyclohexanone (**6**, C₂₀H₂₂ClNO)

Yield 54 %; m.p.: 106–107 °C; ¹H NMR (400 MHz, CDCl₃): δ = 1.70–1.90 (m, 3H), 1.90–2.10 (m, 3H), 2.27 (s, 3H), 2.30–2.50 (m, 2H), 2.80–2.90 (m, 1H), 4.67 (d, 1H, *J* = 6 Hz, *anti*), 6.37 (d, 1H, *J* = 8 Hz), 6.65 (t, 1H, *J* = 7.5 Hz), 6.98 (t, 1H, *J* = 7.2 Hz), 7.08 (d, 1H, *J* = 5.6 Hz), 7.34 (AB-q, 4H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ = 17.68, 23.90, 27.92, 31.63, 42.06, 57.50, 57.57, 110.65, 117.29, 126.90, 128.64, 128.69, 128.74, 130.14, 131.60, 140.51, 144.92, 212.95 ppm; IR (KBr): $\bar{\nu}$ = 3,374 (s, NH), 3,029 (w), 2,945 (s), 1,702 (s, C=O), 1,605 (m), 1,518 (s), 1,449 (m), 1,314 (m), 826 (m), 744 (m) cm⁻¹.

2-[(4-Methylphenyl)][(2-methylphenyl)amino]methyl]-cyclohexanone (**11**, C₂₁H₂₅NO)

Yield 60 %; m.p.: 114–115 °C; ¹H NMR (400 MHz, CDCl₃): δ = 1.65–1.85 (m, 2H), 1.86–2.00 (m, 4H), 2.23 (s, 3H), 2.33 (s, 3H), 2.30–2.50 (m, 2H), 2.75–2.85 (m, 1H), 4.65 (d, 1H, *J* = 7.2 Hz, *anti*), 4.60–4.80 (br, NH), 6.41 (d, 1H, *J* = 7.6 Hz), 6.60 (t, 1H, *J* = 7.2 Hz), 6.95 (t, 1H, *J* = 7.2 Hz), 7.05 (d, 1H, *J* = 7.2 Hz), 7.13 (d, 2H, *J* = 7.6 Hz), 7.28 (d, 2H, *J* = 8 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃): δ = 17.67, 21.13, 23.42, 27.92, 31.21, 41.68, 57.69, 57.73, 110.65, 116.93, 122.55, 126.85, 127.08, 129.24, 129.99, 136.78, 138.76, 145.16, 213.53 ppm; IR (KBr): $\bar{\nu}$ = 3,402 (m, NH), 3,382 (m, NH), 3,014 (w), 2,945 (m), 1,701 (s, C=O), 1,604 (m), 1,518 (s), 1,450 (m), 823 (m), 745 (m) cm⁻¹.

2-[(4-Methylphenyl)[(3-methylphenyl)amino]methyl]-cyclohexanone (**12**, C₂₁H₂₅NO)

Yield 72 %; m.p.: 124–125 °C; ¹H NMR (400 MHz, CDCl₃): δ = 1.60–1.82 (m, 2H), 1.82–2.10 (m, 4H), 2.25 (s, 3H), 2.35 (s, 3H), 2.35–2.55 (m, 2H), 2.70–2.80 (m, 1H), 4.65 (d, 1H, *J* = 7.2 Hz, *anti*), 6.39 (d, 1H, *J* = 7.6 Hz), 6.44 (s, 1H), 6.50 (d, 1H, *J* = 7.6 Hz), 7.00 (t, 1H, *J* = 7.6 Hz), 7.16 (d, 2H, *J* = 7.6 Hz), 7.31 (d, 2H, *J* = 8 Hz) ppm; ¹³C NMR (100 MHz, CDCl₃): δ = 21.13, 21.48, 23.53, 27.96, 31.20, 41.70, 57.58, 57.62, 110.50, 114.57, 118.44, 127.15, 128.98, 129.21, 130.55, 136.70, 138.77, 147.33, 213.12 ppm; IR (KBr): $\bar{\nu}$ = 3,359 (s, NH), 3,051 (w), 2,942 (m), 1,702 (s, C = O), 1,605 (s), 1,533 (m), 1,305 (m), 821 (m), 782 (m) cm⁻¹.

2-[[[(4-Methylphenyl)amino](3-nitrophenyl)methyl]-cyclohexanone (**19**, C₂₀H₂₂N₂O₃)

Yield 85 %; m.p.: 136–137 °C; ¹H NMR (400 MHz, CDCl₃): δ = 1.50–1.90 (m, 3H), 1.90–2.20 (m, 3H), 2.21 (s, 3H), 2.30–2.50 (m, 2H), 2.85–2.95 (m, 1H), 4.73 (d, 0.7H, *J* = 5.2 Hz, *anti*), 4.86 (d, 0.3H, *J* = 4.4 Hz, *syn*), 6.48 (d, 2H, *J* = 8.4 Hz), 6.93 (d, 2H, *J* = 8 Hz), 7.49 (t, 1H, *J* = 8 Hz), 7.80 (d, 1H, *J* = 7.2 Hz), 8.09 (d, 1H, *J* = 8 Hz), 8.28 (s, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ = 20.38, 20.40, 24.46, 24.99, 27.21, 27.85, 29.23, 31.98, 42.41, 42.53, 56.37, 57.12, 57.33, 57.91, 113.69, 114.21, 122.23, 122.36, 122.54, 127.31, 127.55, 129.32, 129.38, 129.72, 129.80, 133.72, 134.25, 144.36, 144.66, 148.41, 210.90 (*syn*), 212.03 (*anti*) ppm; IR (KBr): $\bar{\nu}$ = 3,383 (s, NH), 2,932 (m), 1,704 (C=O, *syn*), 1,697 (C=O, *anti*), 1,616 (m), 1,518 (s), 1,346 (s), 811 (s), 711 (m) cm⁻¹.

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