Research Article

$\text{RuCl}_3 \cdot n\text{H}_2\text{O}$ as Catalyst for Diastereoselective Direct Aldol Reaction: An Efficient Route to Hormone Steroid Derivatives

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Ru^{III} -catalyzed regio- and diastereoselective direct aldol reaction of progesterone with aromatic aldehydes has been developed in good yields (58–78%). Advantages of this method include catalytic efficiency, short reaction times, and ease of operation and workup.

1. Introduction

The aldol reaction is one of the most powerful and useful tools for the construction of carbon-carbon bonds, creating the β -hydroxy carbonyl derivatives [1–7]. It provides an atom-economic approach to β -hydroxy carbonyls [8–16], which make up a large family of intermediates for the synthesis of biologically active substances and natural products. It is extensively applied in the synthesis of carbohydrates, amino sugars, steroids, and other valuable organic compounds. However, the most classical and conventional aldol reaction which involves the mixed aldol reaction between a ketone containing α -hydrogen with an aldehyde in the presence of base or acid has not been well exploited due to the following reasons: (1) side reactions such as self-condensation of the ketone or/and dimerization of the aldehyde can be a problem; (2) the harsh reaction conditions employed which usually require a strong acid or base make it unattractive for the complex molecule synthesis which contains acid or base sensitive functional groups; (3) the desired aldol product is usually accompanied by dehydrated products, dimmers, and polymers; (4) low regioselectivity is observed in most of the cases. Therefore, mild reaction conditions are much sought after to overcome some, if not all, the above problems.

Ru^{III} salts are well known to catalyze a variety of organic transformations, including Michael reactions [17], oxidation reactions of alkanes [18], oxidative cyanation of amines

[19], and many others [20–22]. We have recently reported the organic reactions using catalytic amounts of Ru^{III} [23–27], and the investigation of the chemistry of ruthenium continues to be one of the most active areas of organometallic chemistry.

On the other hand, progesterone [28] is one of the most important hormones of the steroidal pregnane series, secreted by corpus luteum and placenta, which can be regarded as a hormonal balancer, particularly of estrogens. It also helps to create a balance of all other steroids and has intrinsic calming and diuretic properties. Thus, due to the importance of progesterone to the human body and in continuation of our previous works [29–32] on aldol reactions, we would like to report the formation of β -hydroxy ketone derivatives derived from progesterone.

2. Results and Discussion

In our investigation, we found that $\text{RuCl}_3 \cdot n\text{H}_2\text{O}$ can effectively promote the direct aldol reaction of hormone steroid, that is, progesterone with different aromatic aldehydes. Treatment of progesterone (0.24 mmol) with aldehydes (0.24 mmol) in the presence of $\text{RuCl}_3 \cdot n\text{H}_2\text{O}$ (5.8 mol%) and KOH in dioxane at room temperature gave the corresponding aldol adducts in good yields with complete diastereoselectivities.

TABLE 1: Ruthenium-catalyzed direct aldol reaction of aldehydes with progesterone at room temperature.

Entry ^a	Aldehyde	Yield (%) ^b	Time (h)	Dr (syn/anti) ^c
1	2a 4-ClC ₆ H ₄	71	4.5	>99/<1
2	2b 3-ClC ₆ H ₄	73	5	>99/<1
3	2c 4-BrC ₆ H ₄	69	5	>99/<1
4	2d 4-MeSC ₆ H ₄	78	5.5	>99/<1
5	2e 4-FC ₆ H ₄	65	6	>99/<1
6	2f 4-NO ₂ C ₆ H ₄	58	6	>99/<1
7	2g 4-MeC ₆ H ₄	69	6	>99/<1
8	2h 3-NO ₂ C ₆ H ₄	58	6	>99/<1

^aAll products were characterized by ¹H NMR, ¹³C NMR, and IR.^bYields after purification by chromatography.^cDetermined by ¹H NMR analysis.

SCHEME 1

Having established suitable reaction conditions, a series of aldehydes were employed to investigate the reaction scope. The results are shown in Table 1. The table demonstrates that this catalytic reaction can be extended to a wide range of aromatic aldehydes with good yields (Scheme 1).

As shown in Table 1, compounds **3a–h** have been successfully synthesized and characterized by typical spectroscopic techniques, namely IR, ¹H, and ¹³C magnetic resonance (NMR). The infrared spectra of these compounds showed three significant stretching vibrations of (O–H), (C=O), and (C=C) at 3400–3500, 1640–1720, and 1600–1650 cm^{−1}, respectively. Also, in a ¹H NMR study, we observed the existence of the olefinic moiety along with the presence of three singlets corresponding to the three methyl protons of the progesterone skeleton showed the aldehydes react with progesterone from side **a**. Additionally, from the NMR spectrum, it was evident that only one diastereomer has been formed. It is a well-understood phenomenon that the lower values of the ¹H NMR coupling constants of the carbinol protons than 1 Hz clearly indicate the relative stereochemistry of the aldol adducts in favor of the *syn* geometry [33].

The most plausible mechanism for the aldol reaction of progesterone with aldehydes, which rationalizes the formation of products, is presented in Scheme 2. The mechanism involves ruthenium enolates which can be trapped by an aldehyde to give the aldol products (Scheme 3).

In summary, this paper describes a successful cross-coupling reaction between progesterone and various aromatic aldehydes in the presence of ruthenium catalyst. The results presented herein show the catalytic role of RuCl₃ · nH₂O at room temperature in the regio- and

SCHEME 2

diastereoselective aldol reaction between progesterone and aldehydes. From an operational viewpoint, the reactions do not require an inert atmosphere and can be performed over a short period of time without preactivation of the donor substrates (direct aldol reaction). The scope (such as asymmetric synthesis) and synthetic applications of this reaction are currently under investigation in our laboratory.

3. Experimental

All reactions were followed by TLC with detection by UV light. IR spectra were recorded on Shimadzu FTIR-8400S spectrometer. ¹H NMR spectra were obtained on a Bruker DRX-500 Avance spectrometer and ¹³C NMR were obtained on a Bruker DRX-125 Avance spectrometer. Samples were analyzed in CDCl₃, and the chemical shift values are reported in ppm relative to TMS as the internal reference. Elemental analyses were made by a Carlo-Erba EA1110 CHNO-S analyzer and agreed with the calculated values. The isolation of pure products was carried out via preparative thin layer chromatography (silica gel 60 GF₂₅₄, Merck). Excess of solvent was evaporated under reduced

SCHEME 3

pressure at a bath temperature of 50 and 60°C. All solvents, organic and inorganic compounds, were purchased from Merck and Fluka and used without further purification.

3.1. Typical Procedure for Synthesis of Aldol Products. A catalytic amount of $\text{RuCl}_3 \cdot n\text{H}_2\text{O}$ (3 mg, 0.014 mmol) was added to a vial containing aldehyde (0.24 mmol), progesterone (75.5 mg, 0.24 mmol), KOH (14 mg, 0.25 mmol), dioxane (1 mL) and stirred at room temperature (monitored by TLC). After the indicated reaction time, the reaction mixture was purified by thin layer chromatography (silica gel, EtOAc-petroleum ether, 6:12) providing the aldol adduct.

3.1.1. Product (3a). $[\alpha]_D^{36} + 170$ (c 1.0, CHCl_3), yellow oil; IR (neat) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3450, 3100, 2980, 1700, 1680, 1650, 1460. ^1H NMR (500 MHz, CDCl_3): δ_{H} (ppm) 0.71 (s, 3H), 1.30 (s, 3H), 2.20 (s, 3H), 0.70–2.82 (m, 19H), 3.14 (s, OH), 5.78 (s, 1H), 6.24 (d, $J = 0.8$ Hz, 1H), 7.31 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3): δ_{C} (ppm) 14.4, 14.5, 25.1, 25.3, 27.5, 28.2, 28.2, 43.0, 47.7, 48.1, 55.2, 57.5, 58.2, 68.6, 129.7, 130.2, 130.7, 132.9, 134.7, 135.6, 143.6, 191.3, 215.1. (Found: C, 73.94; H, 7.72. Calc. for $\text{C}_{28}\text{H}_{35}\text{O}_3\text{Cl}$: C, 73.92; H, 7.70%).

3.1.2. Product (3b). $[\alpha]_D^{36} + 165$ (c 1.0, CHCl_3), yellow oil; IR (neat) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3450, 2910, 1700, 1680, 1645, 1590, 1575. ^1H NMR (500 MHz, CDCl_3): δ_{H} (ppm) 0.70 (s, 3H), 1.20 (s, 3H), 2.20 (s, 3H), 0.87–2.75 (m, 19H), 5.96 (s, 1H), 6.21 (d, $J = 0.5$ Hz, 1H), 7.37–7.46 (m, 2H), 7.54–7.56 (m, 1H), 7.71–7.73 (m, 1H). ^{13}C NMR (125 MHz, CDCl_3): δ_{C} (ppm) 12.4, 16.2, 25.1, 25.2, 27.4, 28.2, 31.3, 47.6, 47.7, 48.0, 48.1, 67.5, 70.8, 70.9, 73.3, 73.5, 128.9, 129.2, 129.3, 129.7, 129.9, 134.0, 134.2, 135.7, 192.2, 205.1. (Found: C, 73.93; H, 7.73. Calc. for $\text{C}_{28}\text{H}_{35}\text{O}_3\text{Cl}$: C, 73.92; H, 7.70%).

3.1.3. Product (3c). $[\alpha]_D^{36} + 168$ (c 1.0, CHCl_3), yellow oil; IR (neat) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3420, 2905, 1680, 1662, 1642, 1540, 1420. ^1H NMR (500 MHz, CDCl_3): δ_{H} (ppm) 0.73 (s, 3H), 1.22 (s, 3H), 2.18 (s, 3H), 0.87–2.72 (m, 19H), 5.84 (s, 1H), 6.24 (d, $J = 0.7$ Hz, 1H), 7.27–7.35 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3): δ_{C} (ppm) 14.1, 14.5, 25.0, 26.3, 27.5, 28.2, 28.3, 43.1, 47.7, 48.1, 55.2, 57.5, 59.2, 68.6, 129.5, 130.1,

130.7, 132.8, 134.7, 135.5, 143.6, 191.2, 215.0. (Found: C, 67.35; H, 7.05. Calc. for $\text{C}_{28}\text{H}_{35}\text{O}_3\text{Br}$: C, 67.33; H, 7.01%).

3.1.4. Product (3d). $[\alpha]_D^{36} + 155$ (c 1.0, CHCl_3), yellow oil; IR (neat) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3450, 3100, 2940, 1680, 1645, 1600, 1510. ^1H NMR (500 MHz, CDCl_3): δ_{H} (ppm) 0.71 (s, 3H), 1.23 (s, 3H), 2.17 (s, 3H), 2.19 (s, 3H), 0.72–2.82 (m, 19H), 3.14 (s, OH), 5.78 (s, 1H), 6.24 (d, $J = 0.8$ Hz, 1H), 7.25–7.31 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3): δ_{C} (ppm) 13.8, 14.4, 15.1, 25.2, 25.3, 27.5, 28.2, 28.2, 43.5, 47.7, 48.5, 55.2, 57.5, 58.1, 68.6, 129.7, 130.2, 130.4, 132.7, 134.7, 135.6, 143.6, 191.0, 215.1. (Found: C, 74.69; H, 8.12. Calc. for $\text{C}_{29}\text{H}_{38}\text{O}_3\text{S}$: C, 74.67; H, 8.15%).

3.1.5. Product (3e). $[\alpha]_D^{36} + 170$ (c 1.0, CHCl_3), yellow oil; IR (neat) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3450, 3100, 1720, 1640, 1390. ^1H NMR (500 MHz, CDCl_3): δ_{H} (ppm) 0.73 (s, 3H), 1.21 (s, 3H), 2.18 (s, 3H), 0.70–2.76 (m, 19H), 5.75 (s, 1H), 6.24 (s, 1H), 7.39–7.42 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3): δ_{C} (ppm) 12.5, 16.0, 25.1, 25.2, 27.5, 28.2, 31.2, 47.6, 47.8, 48.1, 48.4, 67.5, 70.7, 70.9, 73.3, 73.5, 128.9, 129.1, 129.3, 129.7, 129.9, 134.0, 134.2, 135.7, 192.1, 205.0. (Found: C, 76.73; H, 7.98. Calc. for $\text{C}_{28}\text{H}_{35}\text{O}_3\text{F}$: C, 76.71; H, 7.99%).

3.1.6. Product (3f). $[\alpha]_D^{36} + 175$ (c 1.0, CHCl_3), yellow oil; IR (neat) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3455, 3070, 2985, 1700, 1680, 1650, 1460. ^1H NMR (500 MHz, CDCl_3): δ_{H} (ppm) 0.71 (s, 3H), 1.30 (s, 3H), 2.20 (s, 3H), 0.69–2.80 (m, 19H), 3.15 (s, OH), 5.75 (s, 1H), 6.24 (d, $J = 0.8$ Hz, 1H), 7.34 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3): δ_{C} (ppm) 14.2, 14.5, 25.0, 25.3, 27.4, 28.1, 28.2, 43.0, 47.5, 48.0, 55.2, 57.5, 58.1, 68.5, 129.8, 130.2, 130.7, 132.9, 134.7, 135.6, 143.6, 191.2, 215.1. (Found: C, 75.9; H, 7.85. Calc. for $\text{C}_{28}\text{H}_{35}\text{O}_5\text{N}$: C, 75.5; H, 7.86%).

3.1.7. Product (3g). $[\alpha]_D^{36} + 160$ (c 1.0, CHCl_3), yellow oil; IR (neat) ($\nu_{\text{max}}/\text{cm}^{-1}$): 3450, 3090, 2950, 1680, 1645, 1600, 1525. ^1H NMR (500 MHz, CDCl_3): δ_{H} (ppm) 0.71 (s, 3H), 1.23 (s, 3H), 1.45 (s, 3H), 2.19 (s, 3H), 0.70–2.82 (m, 19H), 3.12 (s, OH), 5.77 (s, 1H), 6.22 (d, $J = 0.8$ Hz, 1H), 7.22–7.30 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3): δ_{C} (ppm) 13.9, 14.04, 15.1, 25.2, 25.4, 27.5, 28.2, 29.2, 43.5, 48.7, 48.9, 55.2, 58.5, 59.1, 71.6, 129.5, 130.1, 130.4, 132.5, 134.5, 135.6, 143.6,

191.1, 215.5. (Found: C, 80.21; H, 8.77. Calc. for $C_{29}H_{38}O_3$: C, 80.18; H, 8.75%).

3.1.8. Product (3h). $[\alpha]_D^{36} + 162$ (c 1.0, $CHCl_3$), yellow oil; IR (neat) (ν_{max}/cm^{-1}): 3450, 2910, 1700, 1685, 1645, 1590, 1555. ^1H NMR (500 MHz, $CDCl_3$): δ_H (ppm) 0.71 (s, 3H), 1.22 (s, 3H), 2.21 (s, 3H), 0.85–2.75 (m, 19H), 3.17 (s, OH), 5.79 (s, 1H), 6.22 (d, $J = 0.8$ Hz, 1H), 7.35–7.46 (m, 2H), 7.53–7.56 (m, 1H), 7.70–7.72 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ_C (ppm) 12.4, 16.2, 25.0, 25.2, 27.4, 30.2, 31.3, 45.6, 47.7, 48.2, 48.5, 67.5, 70.7, 70.9, 73.3, 73.6, 128.9, 129.2, 129.3, 129.7, 129.8, 134.1, 134.2, 135.8, 192.2, 205.2. (Found: C, 75.6; H, 7.87. Calc. for $C_{28}H_{35}O_5N$: C, 75.5; H, 7.86%).

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