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A new and effective method to synthesize carbazolones by rhodium (I)-catalyzed annulation of 2-aminobenzaldehyde with cyclohexane-1,3-diones

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In this research, substituted carbazolones have been synthesized by treating 2-aminobenzaldehyde and cyclohexane-1,3-diones using chlorobis(cyclooctene)rhodium(I)-catalyzed $[Rh_{(}coe)_2Cl]_2$ in one-pot reactions and are found to have applications in organic synthesis and the pharmaceutical industry. The reaction proceeds smoothly under mild conditions, affording a range of carbazolone derivatives, and the product isolated in good to excellent yields (up to 93%).

Keywords: Carbazolones, $[Rh_{(}coe)_2Cl]_2$-catalyzed, Annulation reaction, Biological activities, Pharmaceutical properties

The formation of carbazolones is a privileged building block with wide applications in natural products and the pharmaceutical industry[1-4], and they have been of considerable interest to chemists because of their relationship to indole and their properties. Such products include ondensetron, an HIV-integrase inhibitor, and murrayaquinone (Fig. 1)[5,6].

They display potent biological properties, including antimicrobial, antitumor, antiepileptic, antihistaminic, antioxidative, anti-inflammatory, antidiarrhoeal, analgesic, neuroprotective, and pancreatic lipase inhibition properties[7]. This is an active and ongoing area of method development in organic synthesis. Accordingly, a variety of methods have been devoted to obtain efficient methods for the construction of these structures[8-11]. One of the classical synthetic routes is Fischer indole synthesis[12,13]. Additionally, The synthesis of carbazolene scaffolds has been achieved *via* alternative strategies. These strategies involved different catalyst reactions, Palladium-catalyzed cyclization of aryl enaminones[14], rhodium (III) catalyzed cylization of salicylamides with cyclic 2-diazo-1,3-ketones[15], [1+2+3] annulation of *o*-alkenyl arylisocyanides with α, β-unsaturated ketones under metal-, base-, and acid-free conditions reactions[16], CuI/L-prolien-catalyzed intramolecular arylation reaction of 2-iodobenzenamine with 1,3-cyclohexanedione[17], acid-catalyzed cyclization of 1*H*-indole-2-butanoic acid[18], and PIFA-mediated annulation of 2-aryl enaminones[19]. We have previously developed many catalysts for cyclic compounds containing natural products[20-24].

As an alternative, the development of new methods for the synthesis of specifically functionalized carbazoles attracts organic chemists because many carbazole alkaloids with different pharmacological properties have been discovered. Therefore, it is desirable to develop more general and effective methods for the preparation of carbazolones. Herein, we report the first Chlorobis(cyclooctene)rhodium(I)-catalyzed ($(Rh_2Cl_2(coe)_4$) annulation of 2-aminobenzaldehyde with cyclohexane-1,3-diones, which provides a simple and efficient method for the synthesis of pharmacetically classes of carbazolone skeletons (Scheme 1)[25-27].

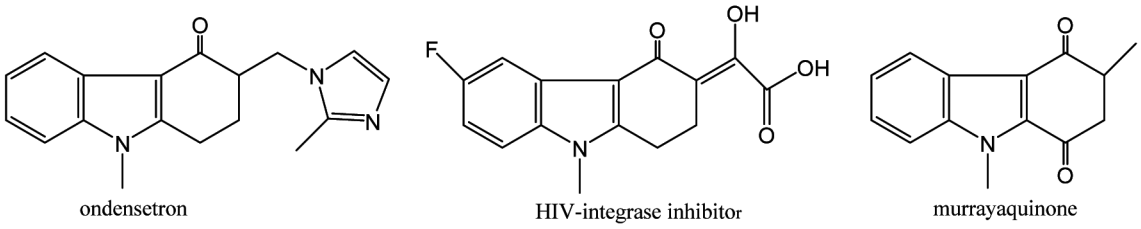


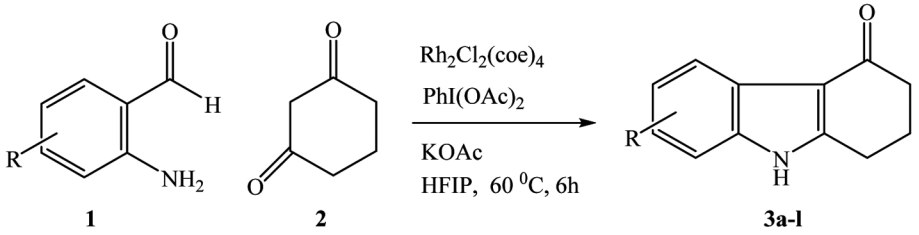
Fig. 1 — Bioactive representatives of carbazolene derivatives

The reactions represent a very attractive route to the chemistry of tetrahydrocarbazole because of the straightforward processes that were established, the mild reaction conditions that were employed, and the shorter reaction periods of substrates bearing electron-donating and electron-withdrawing groups. Herein, we envisaged developing an alternative and different strategy to reach carbazolone derivatives 3 a-l. Further , the product yields are very high, ranging between 68-93% (Table 1).

Experimental Details

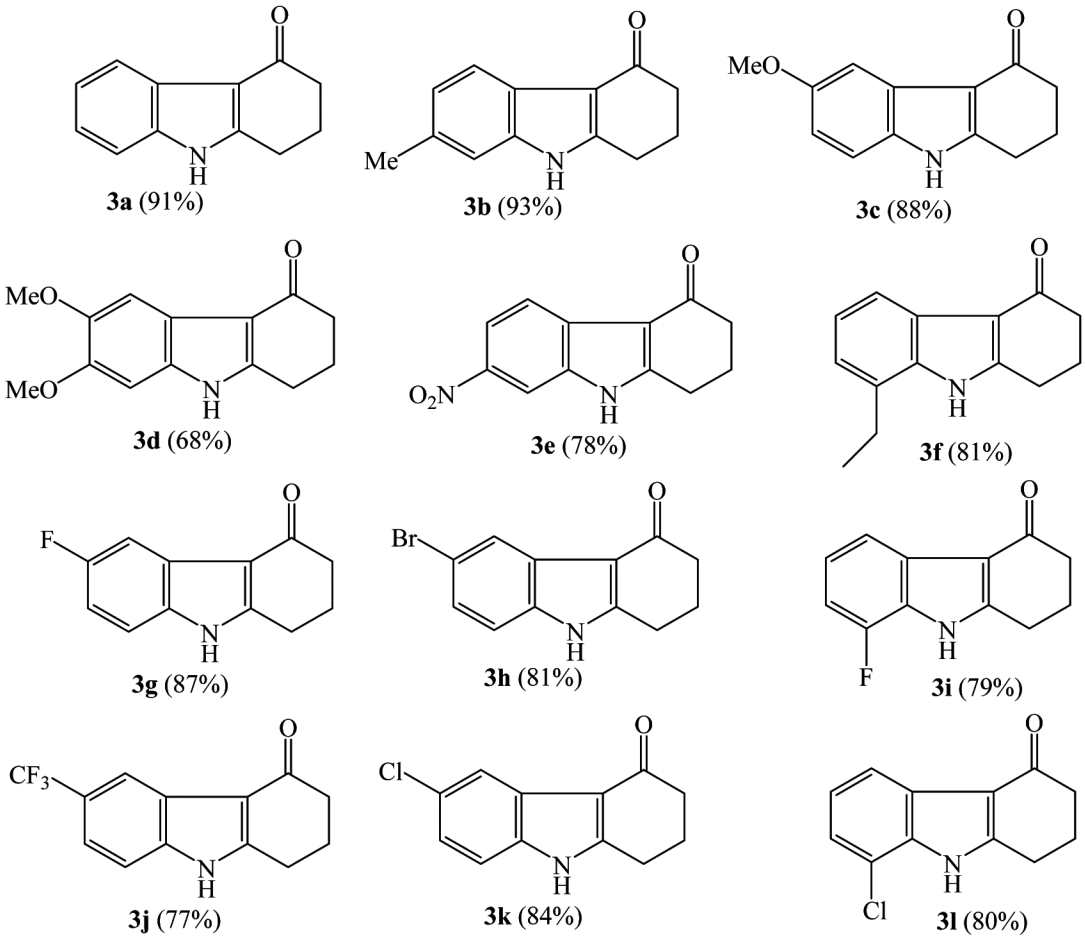
Materials and methods

Proton (^{1}H NMR, 400 MHz) and Carbon (^{13}C NMR, 100 MHz) spectra were performed on a Bruker instrument DPX-400 MHz High-Performance Digital FT-NMR spectrometer $CDCl_3$, DMSO-d_6 and tetramethylsilane (TMS) as the international standard at 25°C. Chemical shifts were reported in parts per million (ppm). Thin-layer chromatography was performed using commercially prepared 100-mesh



Scheme 1 — Synthesis of the carbazolones derivation

Table 1 — Synthesis of carbazolone derivation from 2-aminobenzaldehyde and cyclohexane-1,3-dione



silica gel plates (silica gel60 F254). All solvents were dried and distilled according to the standard methods before use. IR spectra were recorded as KBr pellets using a Mattson 1000 FT-IR Spectrometer. Melting points were determined in capillary tubes on a Gallenkamp apparatus and are uncorrected. Elemental analyses were performed on a vario MACRO cube CHNS elemental analyzer.

General procedure for synthesis of 1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3a-l

General procedure for the synthesis of ethyl 3-(1-oxo-3-phenyl-1*H*-isochromen-4-yl)propanoate (**3a-3l**). Into a solution of the 2-aminobenzaldehyde **1a-l** (1.0 mmol), cyclohexane-1,3-dione **2** (2.0 mol), Chlorobis(cyclooctene)rhodium(I) (2.5 mol), $Ph(OAc)_2$ (0.5 mmol), KOAc (0.5 mmol) , and HFIP (20 mL) under nitrogen atmosphere, and the solution was heated 60°C (6 h) until the total disappearance of the starting material as determined by TLC under a nitrogen atmosphere.

After allowing it to reach RT, and treated in one portion with 50 mL of 10% Na_2CO_3. The resulting solution was extracted using ethyl acetate. The combined organic extracts were dried over anhydrous $MgSO_4$ and concentrated under vacuum. The crude product was purified by column chromatography (EtOAc/CH_2Cl_2, 8:1) to afford pure compounds **3** (a-l).

1,2,3,9-Tetrahydro-4*H*-carbazol-4-one, 3a: The product was obtained as a white solid. Yield 91%. m.p.224-225°C. R_f (EtOAc): 0.73; IR (KBr): 3351, 3052, 3030, 2958, 2943, 1611, 1451, 1241, 1173 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 8.93 (s, 1H, N-H indole), 8.07 (d, J = 7.6 Hz, 1H, H_{Ar}), 7.97 (d, J = 8.1 Hz, 1H, H_{Ar}), 7.88-7.81 (m, 1H, H_{Ar}), 7.63-7.56 (m, 1H, H_{Ar}), 3.34 (t, J = 6.4 Hz, 2H, CH_2CH_2 CH_2), 2.83 (t, J = 6.4 Hz, 2H, $CH_2CH_2CH_2$), 2.36-2.28 (m, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.8, 162.8, 138.2, 131.3, 129.6, 127.5, 126.9, 126.5, 126.1, 38.0, 32.4, 21.9. Anal. Found: C 77.89; H 5.64; N 8.58. Calcd for $C_{12}H_{11}NO$: C 77.81; H 5.99; N 8.64%.

7-Methyl-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3b: The product was obtained as a white solid. Yield 93%. m.p.249-251°C. R_f (EtOAc): 0.58; IR (KBr): 3180, 2931, 2848, 1611, 1462, 1223, 1179 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 11.74 (s, 1H, N-H indole), 7.82 (d, J = 7.6 Hz, 1H, H_{Ar}), 7.24 (s, 1H, H_{Ar}), 6.93 (d, J = 7.9 Hz, 1H, H_{Ar}), 2.97 (t, J = 5.4 Hz, 2H, $CH_2CH_2CH_2$), 2.41 (t, J = 6.1 Hz, 5H, $CH_2CH_2CH_2$, CH_3), 2.18 (t, J = 5.8 Hz, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 192.1, 151.3, 135.7, 131.0, 124.4, 123.7, 121.3, 112.8, 37.2, 23.6, 23.2, 22.5. Anal. Found: C 78.29; H 6.63; N 7.11. Calcd for $C_{13}H_{13}NO$: C 78.36; H 6.58; N 7.03%.

6-Methoxy-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3c: The product was obtained as a pale solid. Yield 88%. m.p.221-223°C. R_f (EtOAc): 0.71; IR (KBr): 3222, 2945, 2888, 1622, 1572, 1480, 1455, 1251, 1223, 1171, 1038, 791 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 11.63 (s, 1H, NH- indole), 7.45 (d, J = 2.5 Hz, 1H, H_{Ar}), 7.28 (d, J = 8.6 Hz, 1H, H_{Ar}), 6.83 (dd, J = 8.6, 2.4 Hz, 1H, H_{Ar}), 3.73 (s, 3H, CH_3O-), 2.3 (t, J = 6.2 Hz, 2H, $CH_2CH_2CH_2$), 2.43 – 2.32 (m, 2H, $CH_2CH_2CH_2$), 2.17 – 2.06 (m, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 192.1, 154.3, 151.8, 132.0, 124.7, 113.7, 113.2, 111.1, 104.0, 56.7, 37.3, 22.9, 22.3. Anal. Found: C 72.68; H 6.01; N 6.59. Calcd for $C_{13}H_{13}NO_2$: C 72.54; H 6.09; N 6.51%.

6,7-Dimethoxy-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3d: The product was obtained as a white solid. Yield 68%. m.p.231-233°C. R_f (EtOAc): 0.68; IR (KBr): 3181, 2966, 2889, 1638 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 11.51 (br, 1H, NH- indole), 7.42 (s, 1H, H_{Ar}), 6.91 (s, 1H, H_{Ar}), 3.75 (s, 3H, OCH_3), 3.71 (s, 3H, OCH_3), 2.93 (t, J = 6.1 Hz, 2H, $CH_2CH_2CH_2$), 2.37 (t, J = 6.1 Hz, 2H, $CH_2CH_2CH_2$), 2.11 (dd, J = 12.1, 5.8 Hz, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 192.2, 151.8, 148.1, 147.3, 131.3, 116.6, 113.3, 104.1, 97.1, 57.2, 57.2, 39.2, 25.1, 24.2. Anal. Found: C 68.47; H 6.09; N 5.79. Calcd for $C_{14}H_{15}NO_3$: C 68.56; H 6.16; N 5.71%.

7-Nitro-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3e: The product was obtained as a solid. Yield 78%. m.p.330-332°C. R_f (EtOAc): 0.56; IR (KBr): 3234, 2955, 2878, 1638 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 12.45 (br, 1H, NH- indole), 8.21 (d, J = 1.4 Hz, 1H, H_{Ar}), 8.33 (d, J = 1.3 Hz, 1H, H_{Ar}), 8.04 (m, 2H, H_{Ar}), 3.01 (t, J = 6.2 Hz, 2H, $CH_2CH_2CH_2$), 2.44 (t, J = 6.5 Hz, 2H, $CH_2CH_2CH_2$), 2.11 (m, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 192.8, 156.3, 143.3, 134.6, 128.8, 118.6, 117.1, 112.4, 107.7, 37.3, 23.1, 22.4. Anal. Found: C 62.69; H 4.41; N 12.24. Calcd for $C_{12}H_{10}N_2O_3$: C 62.61; H 4.38; N 12.17%.

8-Ethyl-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3f: The product was obtained as a brown solid. Yield 81%. m.p.214-216°C. R_f (EtOAc): 0.61; IR (KBr): cm^{-1}:

3168, 2946, 2873, 1643; ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.73 (s, 1H, N-H indole), 7.77 (d, 1H, H_{Ar}), 6.94 (d, 1H, H_{Ar}), 7.09-7.02 (m, 1H, H_{Ar}), 2.94 (t, J = 6.1 Hz, 2H, CH_2CH_2CH), 2.81 (q, J = 15.1 and 7.5 Hz, 2H, $CH_2CH_2CH_2$), 2.44 (t, 2H, J = 7.3 Hz CH_2CH_3), 2.10-2.14 (m, 2H, $CH_2CH_2CH_2$), 1.21 (t, 3H, H = 7. 3Hz, CH_2CH_3); ^{13}C NMR (100 MHz, DMSO-d_6): δ 195.3, 134.6, 134.1, 126.2, 125.3, 123.6, 122.2, 119.5, 109.2, 37.4, 27.4, 24.6, 22.3, 13.7. Anal. Found: C 78.77; H 7.15; N 6.64. Calcd for $C_{14}H_{15}NO$: C 78.84; H 7.09; N 6.57%.

6-Fluoro-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3g: The product was obtained as a brown solid. Yield 87%. m.p.246-247°C. R_f (EtOAc): 0.55; IR (KBr): 3221, 2942, 2928, 2861, 1620, 1583, 1454, 1121. cm^{-1} ; ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.93 (brs, N-H indole), 7.63 (dd, J = 9.4, 23 Hz, H_{Ar}), 7.43-7.38 (m, 1H, H_{Ar}), 7.05-6.97 (m, 1H, H_{Ar}), 2.93 (t, J = 6.2 Hz, 2H, $CH_2CH_2CH_2$),), 2.41 (t, J = 6.5 Hz, 2H, $CH_2CH_2CH_2$), 2.17-2.07 (m, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 193.3, 157.3 (J_{C-F} = 231.8 Hz), 154.4, 133.3, 124.1 (J_{C-F} = 10.8 Hz), 112.3 (J_{C-F} = 9.5 Hz), 111.3 (J_{C-F} = 4.2 Hz), 110.3 (JCF = 25.3 Hz), 104.2 (J_{C-F} = 23 Hz), 36.3, 21.9. Anal. Found: C 70.87; H 4.91; N 6.79. Calcd for $C_{12}H_{10}FNO$: C 70.93; H 4.96; N 6.89%.

6-Bromo-2,3-dihydro-1*H*-carbazol-4(9*H*)-one, 3h: The product was obtained as a brown solid. Yield 81%. m.p.291-293°C. R_f (EtOAc): 0.63 ; IR (KBr): 3167, 2955, 2931, 1617, 1572, 1436, 1361, 1282, 1193, 1176 cm^{-1}: ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.01 (brs, N-H indole), 8.02 (d, J = 1.5 Hz, 1H, H_{Ar}), 7.34 (d, J = 8.6 Hz, 1H, H_{Ar}), 7.32 (dd, J = 8.6 and 1.91 Hz, 1H, H_{Ar}), 2.94 (t, J = 6.2 Hz, 2H, $CH_2CH_2CH_2$), 2.42 (t, J = 6.6 Hz, 2H, $CH_2CH_2CH_2$), 2.13-2.07 (m, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 193.4, 154.3, 135.5, 133.4, 127.1, 123.5, 121.4, 115.1, 112.5, 110.1, 38.5, , 24.1, 21.6. Anal. Found: C 54.62; H 3.91; N 5.38. Calcd for $C_{12}H_{10}BrNO$: C 54.57; H 3.83; N 5.30%.

8-Fluoro-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3i: The product was obtained as a white solid . Yield 79%. m.p.220-222°C. R_f (EtOAc): 0.69; IR (KBr): 3226, 2977, 2888, 1634, 1120 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.18 (br, 1H, N-H indole), 8.22 (d, J = 7.7 Hz, 1H, H_{Ar}), 7.51 (d, J = 7.7 Hz, 1H, H_{Ar}), 7.33 (t, J = 7.7, 7.7 Hz, 1H, H_{Ar}), 3.11 (t, J = 6.3 Hz, 2H, $CH_2CH_2CH_2$), 2.41 (t, J = 6.8 Hz, 2H, $CH_2CH_2CH_2$), 2.17-2.02 (m, 2H, $CH_2CH_2CH_2$). ^{13}C NMR (100 MHz, DMSO-d_6): δ 194.2, 157.8 (d, J_{C-F} = 231 Hz), 153.2, 133.9, 124.4, 114.1 (d, J_{C-F} = 9.5 Hz), 111.6 (d, J_{C-F} = 25.3 Hz), 107.7, 104.5, 36.2, 24.7, 23.23. Anal. Found: C 70.87; H 4.88; N 6.94. Calcd for $C_{12}H_{10}FNO$: C 70.93; H 4.96; N 6.89%.

6-(Trifluoromethyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3j: The product was obtained as a white solid. Yield 77%. m.p.208-210°C. R_f (EtOAc): 0.61; IR (KBr): 3178, 2934, 2888, 1620, 1121 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.91 (br, 1H, N-H indole), 7.61 (m, 1H, H_{Ar}), 7.44 (m, 1H, H_{Ar}), 7.07 (m, 1H, H_{Ar}), 2.93 (t, J = 6.1 Hz, 2H, $CH_2CH_2CH_2$), 2.41 (t, J = 6.1 Hz, 2H, $CH_2CH_2CH_2$), 2.11 (m, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 194.2, 157.8 (d, J_{C-F} = 233 Hz), 153.2, 131.9, 124.4, 112.1 (d, J_{C-F} = 9.6 Hz), 111.6 (d, J_{C-F} = 25.2 Hz), 104.7, 104.5, 38.2, 24.7, 24.2. Anal. Found: C 61.58; H 3.89; N 5.61. Calcd for $C_{13}H_{10}F_3NO$: C 61.66; H 3.98; N 5.53%.

6-Chloro-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3k: The product was obtained as a solid. Yield 84%. m.p.218-220°C. R_f (EtOAc): 0.61; IR (KBr): 3159, 2957, 2928, 2833, 1629, 1461, 1277, 1171 cm^{-1}: ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.11 (brs, N-H indole), 7.88 (d, J = 1.7 Hz, 1H, H_{Ar}), 7.41 (d, J = 8.4 Hz, 1H, H_{Ar}), 7.22 (dd, J = 8.4, 1.7 Hz, 1H, H_{Ar}), 2.90 (t, J = 6.3 Hz, 2H, $CH_2CH_2CH_2$), 2.41 (t, J = 6.6 Hz, 2H, $CH_2CH_2CH_2$), 2.15-2.09(m, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, DMSO-d_6): δ 193.9, 154.7, 133.4, 127.1, 126.6, 121.3, 118.1, 112.1, 110.3, 36.6, 24.2, 21.6. Anal. Found: C 65.68; H 4.49; N 6.40. Calcd for $C_{12}H_1ClNO$: C 65.61; H 4.59; N 6.38%.

8-Chloro-1,2,3,9-tetrahydro-4*H*-carbazol-4-one, 3l: The product was obtained as a white solid. Yield 80%. m.p.224-226°C. R_f (EtOAc): 0.55; IR (KBr): 314, 2933, 2928, 1627, 1468, 1283, 1173 cm^{-1}: ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.78 (s, 1H, N-H indole), 7.93-7.86 (m, 2H, H_{Ar}), 7.54 (t, J = 7.5 Hz, 1H, H_{Ar}), 3.43 (s, 2H, $CH_2CH_2CH_2$), 2.81 (s, 2H, $CH_2CH_2CH_2$), 2.37 (t, J = 5.6 Hz, 2H, $CH_2CH_2CH_2$); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.6, 161.9, 144.7, 138.4, 133.9, 133.1, 127.8, 127.1, 125.9, 125.5, 39.3, 32.6, 22.6. Anal. Found: C 65.70; H 4.46; N 6.43. Calcd for $C_{12}H_1ClNO$: C 65.61; H 4.59; N 6.38%.

Results and Discussion

We commenced our investigation with 2-aminobenzaldehyde **1** and cyclohexane-1,3-dione 2

in the presence of chlorobis(cyclooctene)rhodium(I) and $Ph(OAc)_2$ as model substrate[28,29]. When this compound was carried out at RT, the desired product **3a** could be obtained with a 37% yield. Encouraged by this promising result, we applied the same reaction conditions under reflux at 60°C conditions, and the expected product **3a** was significantly improved to 91% and we also studied the reaction of 2-aminobenzaldehyde with electron-donating groups and electron-withdrawing groups, which could be efficiently transformed into the desired products (**3a-3l**) in good yields. Functional groups such as CH_3 at the C7 position were increased due to the formation of an inductive effect. However, the position caused a decrease in strong electron-donating compounds such as CH_3O at the C6 position. It is worth mentioning that the reaction also proceeded smoothly with electron-withdrawing groups (NO_2, F, Br, CF_3 and Cl) to provide the corresponding products **3e, 3g, 3h, 3i, 3j, 3k** and **3l** in the position of C6, C7, and C8. The alkyl group such as ethyl in the position of C8 position did not influence the reaction and desired products **3f** can be reached in good yield over short reaction times, as determined by thin layer chromatography, as well as reaction yield. Based on the above experimental results in Scheme 1, this protocol also provides a straightforward method to access the synthesis of carbazolone derivatives by chlorobis (cyclooctene) rhodium(I)-catalyzed decarbonylative annulation from readily available starting materials[30]. The method was reliable and tolerated several substrate types. There did not appear to be major substituent effects on reaction times and yields. Moreover, this approach can be applied to the synthesis of an analogue of naturally occurring natural products with a different method for further functionalization of such compounds[31-33].

Conclusion

As a result of this work, we have presented here an efficient method for the straightforward synthesis of carbazolone derivatives with electron-donating and electron-deficient as a novel catalysis of chlorobis (cyclooctene) rhodium (I). Moreover, the high reaction yields were achieved due to the application of chlorobis(cyclooctene)rhodium(I)-catalyzed reaction. This reaction proceeds smoothly under simple condition, good functional group tolerance and a powerful route to construct carbazolone derivatives in one step. We also briefly examined the reactivity of the effect of substituents, which represents an example for other similar studies.

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