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A One-Pot Synthesis of Functionalized Tetrahydro-4H-Chromenes and Tetrahydro-4H-Thiopyrans Derivatives

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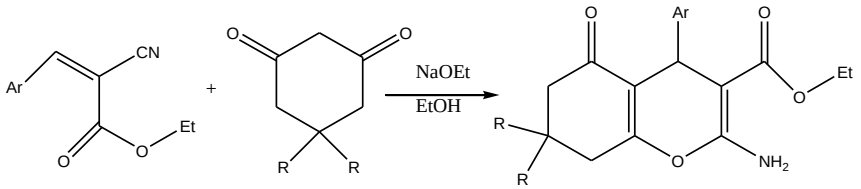
Abstract: *An efficient approach for the synthesis of functionalized tetrahydro-4H-chromenes **3a-c** and tetrahydro-4H-thiopyran **3d-e** derivatives moderate to high yields have been achieved via a addition-cyclization reaction of α, β-Unsaturated cyanoesters **1a-c** with dimedone **2p** or 1, 3-cyclohexandione **2q** in presence of sodium ethoxide and Benzaldehyde with 2-propanol in presence of ammonium sulphide respectively. This methodology differs from the previous classical methods in its simplicity and ready availability of the catalyst. The structures of the compounds **3a-e** were confirmed by their ultraviolet, infrared, ^{1}H NMR, ^{13}C NMR and elemental analyses.*

Keywords: *4H- chromenes, 4H-thiopyran, Active methylene, Knoevenagel adducts, Sodium ethoxide, Ammonium sulphide, 2-propanol, Michal-cyclization.*

1. INTRODUCTION

The synthesis of 4H-chromenes and 4H-thiopyrans derivatives have attracted great interest to their biological and pharmalogical activities. Chromene derivatives have attracted increasing attention from synthetic chemists due to their diverse biological activities, including antitumor [1], antibacterial [2], antiviral [3], antioxidative [4], antidepressant [5], antihypertensive [6], antidiabetic [7], fungicidal [8], and insecticidal properties [9]. Tetrahydro-4H-thiopyrans are a class of important heterocycles that have been used as analgesics and anti-inflamatory [10], insecticides, herbicides [11], sensitizers [12], fire-resistant polymers [13]. Thus, several methods have been reported for the synthesis of these compounds [14-26]. However, many of these methods were associated with use of hazardous organic solvents, poor yields of products, long reaction times and lack of general applicability; particularly, synthesis of substituted 4H-chromene and 4H-thiopyran were rarely addressed. Therefore, the introduction of milder, faster and more ecofriendly methods, accompanied with higher yields is needed. This prompted us to develop a convenient method for the synthesis of 4-aryl-4H-chromene derivatives, and we report herein the synthesis of 4-aryl-4H-chromene derivatives via a tandem Knoevenagel and cyclocondensation reaction using sodium ethoxide as catalyst and for the synthesis of benzylidene-2,6-diphenyl-tetra-hydro-thiopyran we carried out the reaction of benzaldehyde with 2-propanol in presence of ammonium sulphides. α,β-Unsaturated cyanoesters **1a–c** were prepared via Knoevenagel condensation of the corresponding aldehydes with ethyl cyanoacetate in the presence of a base catalyst as reported in the literature[27]. Compounds **1a–c** were reacted with dimedone or 1,3-cyclohexanedione 2p–q in the presence of sodium ethoxide in ethanol to give tetrahydro-4H-chromenes **3a–c (Scheme 1)**. The formation of the Tetrahydro-4H-thiopyrans derivative **3d-e** prepared by a different mode of cyclization**(Scheme-2)**. In addition, the synthesized compounds' structures **(3a – e)** were characterized and confirmed with the help of their ultraviolet (UV), Infrared (IR), ^{1}H NMR, ^{13}C NMR and elemental analyses.

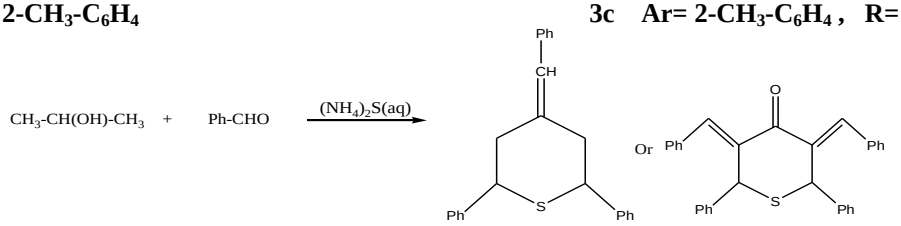


Scheme1.

1a-1c
1a Ar= 4-CH_3O-C_6H_4
1b Ar= 2-CH_3-C_6H_4
1c Ar= 2-CH_3-C_6H_4

2p-2q
2p R= CH_3
2q R= H

3a-3c
3a Ar= 4-CH_3O-C_6H_4, R= CH_3
3b Ar= 2-CH_3-C_6H_4 , R= H
3c Ar= 2-CH_3-C_6H_4 , R= H



3d **3e**

Scheme2.

2. EXPERIMENTAL

Melting points were determined on an Electrothermal micromelting-point apparatus and uncorrected. The Ultraviolet-Visible spectra of the samples were recorded on a SHIMADZU-UV-160A ultraviolet spectrometer with a scanning range of 800-200 nm. IR spectra were recorded with FT-IR 8400S Shimadzu spectrometer in the range 4000-400 cm^{-1}. The ^1H NMR and ^{13}C NMR spectra of the samples were recorded on a JEOL ECA-600 operating at 400.17 MHz spectrometer using $CDCl_3$ as solvent with Tetramethylsilane (TMS) as an internal standard. High resolution MS was obtained by JEOL JMS-AX505HF for electron impact ionization (EI) and a JEOL JMS-T100LC for electron spray ionization (ESI). All the solvents used were dried and distilled using standard methods. Super dry ethanol was used for the reactions.

2.1. General Procedure for the Preparation of 4H-Chromenes

A mixture of α,β-unsaturated cyanoester (5 mmol), 1,3-cyclohexanedione or dimedone (5 mmol), 5% sodium ethoxide in dry ethanol (1.5 mmol), and dry ethanol (25 mL) was refluxed for 8-10 hrs. The progress of the reaction was followed by thin-layer chromatography (TLC) on SiO_2 plate using an appropriate eluting solvent. After completion of the reaction the mixture was cooled to room temperature and the volume was reduced to one-fourth by evaporation. It was then neutralized with 0.1 M HCl solution, extracted with ether (3×30 mL) and dried over anhydrous Na_2SO_4. The extracted organic layer was evaporated in a rotary vacuum evaporator. A solid mass was obtained which was recrystallized from absolute alcohol.

2.2. General Procedure for the Preparation of 4H-Thiopyran

Benzaldehyde (2.15 g, 0.02 mol) and 2-propanol (50.0 cm^3) was taken in a three-necked round bottomed flask fitted with a reflux condenser, a pressure equalizing dropping funnel and a magnetic stirrer. The whole setup was placed in an oil bath. An aqueous solution of Ammonium sulphide (20 cm^3, 20%) was added dropwise from the dropping funnel to the above mixture over a period of about 15 min at room temperature. During the addition of Ammonium sulphide solution, the system was stirred constantly with a magnetic stirrer, when turbidity appeared. The solution was then heated at 45^0-50^0C temperature. The stirring was continued for 6 hours till no further precipitate formed. The precipitates then separated by filtration, washed with water and recrystallized by petroleum ether and few drops of chloroform and an off- white solid was obtained. The remaining filtrate after the separation of the above precipitates,was extracted with chloroform, on concentration by vacuum rotary evaporator resulted a pale yellow solid.

2.3. 2-Amino-7,7-Dimethyl-4-(4/-Methoxy-Phenyl)-5-Oxo-5,6,7,8-Tetrahydro-4H-Chromene-3-Carboxylic Acid Ethyl Ester

Yield 85%; white crystalline solid; mp 120^0C-122^oC; R_f value in TLC 0.51 (Chloroform:Pet. Ether 4:1); UV (λ_{max}) at 301.0 nm (n → π* transition of C=O); IR (KBr) (γ max, cm^{-1}): 3414, 3350 (N-H), 2958, 2838 (C-H stretching of saturated aliphatic protons), 1689 (C=O), 1527, 1509 (C=C stretching of phenyl), 1367 (C-N stretching), 1288, 1200 (C-O stretching), 996, 909 (C-H bending of phenyl) ; ^1H NMR δ (in ppm): 7.15 (d, J=7.8 Hz, ArH, 2H), 6.72 ((d, J=6.9 Hz, ArH, 2H), 6.20 (br s, NH_2, 2H), 4.63 (s, C_4-H, 1H), 2.18 (q, J=16.1 Hz, -CH_2CH_3, 2H), 3.71 (s, OCH_3, 3H), 4.01 (s, C_6-H, 2H), 2.38 (s, C_8-H, 2H), 1.14 (t, J=7.80 Hz, -CH_2CH_3, 3H), 1.06 (s, CH_3 at C-7, 3H), 0.95 (s, another CH_3 at C-

7, 3H); ^{13}C NMR δ (in ppm): 196.551, (C=O), 169.109(C-2), 162.732(–COOCH$_2$CH$_3$), 158.223(C-9), 154.345, 138.346, 133.663,133.596, 129.125 ,124.366 (6C-aromatic), 116.170 (C-10), 80.964 (C-3), 59.593 (-COOCH$_2$CH$_3$), 55.572 (-OCH$_3$), 36.863 (C-6), 32.879 (C-4), 27.231 (CH$_3$ at C-7), 27.121 (another CH$_3$ at C-7), 26.914 (C-8), 20.212 (C-7) 14.199 (-COOCH$_2$CH$_3$); MS: m/z 371.20 (M$^+$). Anal. calcd. for $C_{21}H_{25}NO_5$: C, 67.92; H, 6.79; N, 3.77. Found: C, 67.73; H, 6.74; N, 3.75.

2.4. 2-Amino-5-Oxo-4-O-Tolyl-5,6,7,8-Tetrahydro-4H-Chromene-3-Carboxylic Acid Ethyl Ester

Yield 92%; white crystalline solid; mp 182^{0}C-184^{0}C; R$_f$ value in TLC 0.70 (Chloroform:Pet. Ether 4:1); UV (λ_{max}) at 305.0 nm (n → π* transition of C=O); IR (KBr) (γ max, cm_1): 3380, 3264 (N-H stretching), 2976 (C-H stretching of saturated aliphatic protons), 1686, (C=O), 1532 (C=C stretching of phenyl), 1368 (C-N stretching), 1282, 1182 (C-O stretching), 993 (C-H bending of phenyl); ^{1}H NMR δ (in ppm): 7.02 (m, ArH, 4H), 6.15 (br s, NH$_2$, 2H), 4.79 (s, C4-H, 1H), 4.04 (q, J=7.20 Hz, -CH$_2$CH$_3$, 2H), 2.68 (s, ArCH$_3$, 3H), 2.62–2.47 (m, methylene protons at C-6, 2H), 2.31–2.25 (m, methylene protons at C-8, 2H), 2.02–1.85 (m, methylene protons at C-7, 2H), 1.13 (t, J=7.10 Hz, -CH$_2$CH$_3$, 3H) ; MS: m/z 327.20 (M$^+$). Anal. calcd. for $C_{19}H_{21}NO_4$: C, 69.72; H, 6.42; N, 4.28. Found: C, 69.62; H, 6.50; N, 4.15.

2.5. 2-Amino-7,7-Dimethyl-5-Oxo-4-O-Tolyl-5,6,7,8-Tetrahydro-4h-Chromene-3-Carboxylic Acid Ethyl Ester

Yield 88%; white crystalline solid; mp 166^{0}C-169^{o}C; Rf value in TLC 0.70 (Chloroform:Pet. Ether 4:1); UV (λ_{max}) at 301.0 nm (n → π* transition of C=O); IR (KBr) (γ_{max} in cm^{-1}): 3420, 3300 (N-H stretching), 2960 (C-H stretching of saturated aliphatic protons), 1690 (C=O), 1510 (C=C stretching of phenyl), 1352 (C-N stretching), 1285, 1175 (C-O stretching), 854 (C-H bending of phenyl); ^{1}H NMR δ (in ppm): 6.98 (m, ArH, 4H),, 6.18 (br s, NH$_2$, 2H), 4.81 (s, C4-H, 1H), 4.03 (q, J=16.1 Hz, -CH$_2$CH$_3$, 2H), 2.68 (s, ArCH$_3$, 3H), 2.41 (s, C$_6$-H, 2H), 2.15 (s, C$_8$-H, 1H), 1.12 (t, J=7.8 Hz, -CH$_2$CH$_3$, 3H), 1.08 (s, CH$_3$ at C-7, 3H), 0.93 (s, another CH$_3$ at C-7, 3H); ^{13}C NMR δ (in ppm): 196.8 (C=O), 169.3 (C-2), 163.1 (–COOCH$_2$CH$_3$), 159.6 (C-9), 139.6 (2C), 129.1 (2C), 127.4 (2C) (6C-aromatic), 113.2 (C-10), 81.4 (C-3), 59.9 (COOCH$_2$CH$_3$), 53.8 (C-6), 45.1 (C-8), 34.3 (C-4), 31.7 (C-7), 27.1 (CH$_3$ at C-7), 27.5 (another CH$_3$ at C-7), 16.2 (ArCH$_3$), 13.6 (COOCH$_2$CH$_3$); MS: m/z 355.11 (M$^+$). Anal. calcd. for $C_{21}H_{25}NO_4$: C, 70.98; H, 7.04; N, 3.94. Found: C, 70.82; H, 7.06; N, 3.92.

2.6. 4-Benzylidene-2,6-Diphenyl-Tetra-Hydro-Thiopyran

Yield 84%; off-white crystalline solid; mp 62^{0}C-65°C; R$_f$ Value in TLC 0.6 (chloroform:pet. Ether,1:4); UV (λ_{max}) at 288 nm (π → π* transition of C=C); IR (KBr)(ν_{max}, cm^{-1}): 3340,3000 (C-H stretching of aromatic and olefinic system), 2330 (C-H stretching of aliphatic system), 2850 (Aliphatic C-H stretching), 1570,1530 (>C=C< skeletal vibration for aromatic ring), 1490, 1215 (-CH$_2$- bending), 890, 760, 600 (Aromatic out of plane C-H bending); ^{1}H NMR δ (in ppm): (a) 3.64 (s, 5H, C-2 or C-6 methine (-S-CH-Ph) & C-3 and C-5) (b) 4.06 (s, 1H, =CH-Ph) (c) 4.19 – 4.23 (t,1H, J=18Hz, C-2 or C-6) (d) 7.2–7.39 (m,15H, C-2, C-6 & C-4 Phenyl of benzylidene, (=CH-C$_6$H$_5$)); ^{13}C NMR δ (in ppm): 43.0(C-3), 43.2 (C-5), 43.5 (C-2), 44.2 (C-6), 76.5 (C-4), 77.4 (=CH-C$_6$H$_5$), 127.4-137.3(all aromatic carbons of the phenyl ring); MS: m/z 342.67 (M$^+$). Anal. calcd. for $C_{24}H_{22}S$.

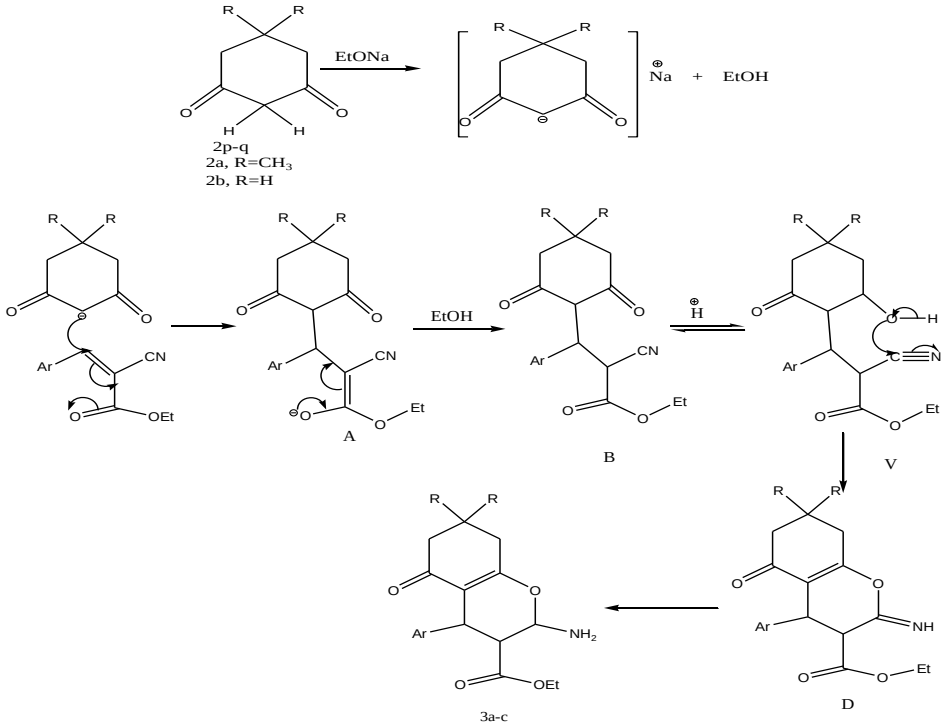
2.7. 3, 5-Dibenzylidene-2, 6- Diphenyltetrahydrothiopyran-4-One

Yield 80%; Pale yellow solid; mp 105^{0}C-107°C; R$_f$ Value in TLC 0.65 (chloroform:pet. Ether,1:1); UV (λ_{max}) at 301.0 nm (n → π* transition of C=O), 221.0 nm (π → π* transition of C=C); IR (KBr)(ν_{max}, cm^{-1}): 3350,3220 (C-H stretching of aromatic and olefinic system), 2340 (C-H stretching of aliphatic system), 2850 (Aliphatic C-H stretching), 1615 (conjugation of C=O with >C=C<), 1440, 1390 (Medium to strong absorptions of aromatic ring), 880, 770, 690 (Aromatic out of plane C-H bending); ^{1}H NMR δ (in ppm): 0.995-1.291 (d, 2H, C-2 and C-6 methine (-S-CH-Ph) 7.245-7.906 (22H, m, phenyl groups at C-2 and C-6, and two benzylidene phenyl groups at C-3 and C-5, and two methine protons of benzylidene groups (=CH-Ph) at C-3 and C-5).; ^{13}C NMR δ (in ppm): 29.61(2C,C-2 and C-6), 126.801, 128.417, 131.932 (C-2 and C-6 phenyl and C-3 and C-5 benzylidene aromatic ring carbons), 202.813 (>C=O); ^{13}C- DEPT (δ/ppm): no inverse signal (absence -CH$_2$-); MS: m/z 444.11 (M$^+$). Anal. calcd. for $C_{31}H_{24}SO$.

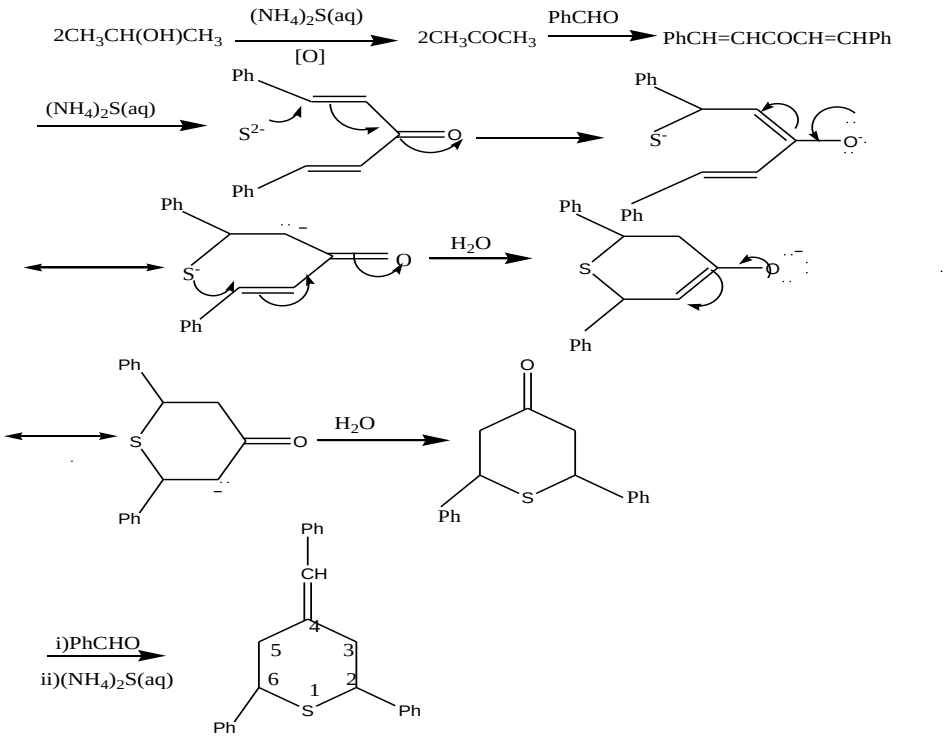
3. RESULT AND DISCUSSION

α, β-Unsaturated cyanoesters **1a-c** were prepared via Knoevenagel condensation of the corresponding aldehydes with ethyl cyanoacetate in presence of a base catalyst as reported in the literature[28]. Compounds **1a-c** were reacted with dimedone **2p** or 1, 3-cyclohexandione **2q** in presence of sodium ethoxide in ethanol to give substituted tetrahydro-4*H*-chromenes **3a-c** and Tetrahydro-4H-thiopyrans was prepared Latif reaction. The structures of **3a-e** were confirmed on the basis of their IR, ^{1}H NMR, ^{13}C NMR and HRMS data.

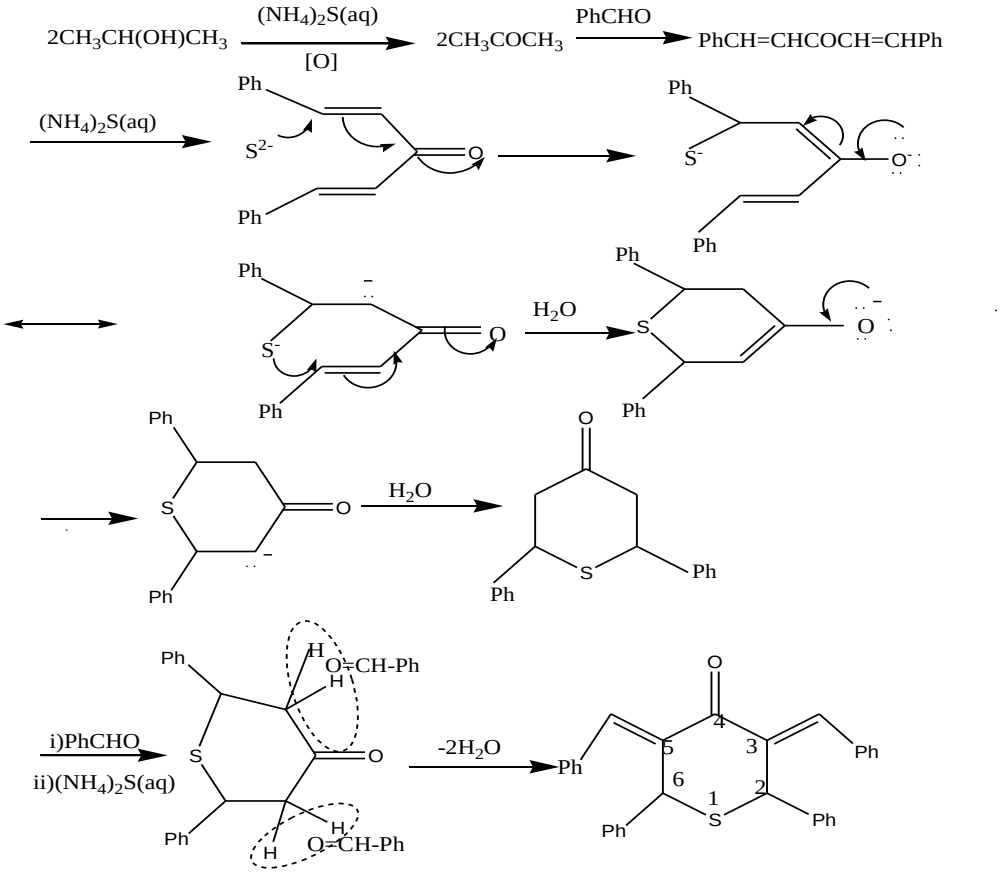
The formation of 4*H*-chromenes **3a -c** may be explained by the initial formation of a 1:1 adduct which subsequently underwent cyclization **(Scheme 3)**. The formation of the Tetrahydro-4H-thiopyrans derivative **3d-e** may be explained by a different mode of cyclization (**Scheme 4 & Scheme 5**).



Scheme3. *Formation of tetrahydro-4H-chromenes **3a-c***



Scheme4. *Formation of tetrohydro-4H-thiopyran **3d***



Scheme5.*Formation of tetrohydro-4H-thiopyran* ***3e***

4. CONCLUSION

We have developed an efficient procedure for the synthesis of 4H-chromenes and 4H-thiopyrans derivatives. This method offers several advantages such as inexpensive catalysts, easy synthetic procedure, high yields, simple work-up procedure and easy product isolation.

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