

Tricarbonylchromium complexes of 4-aryl-1,4-dihydropyridine derivatives: Regioselective reaction

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ABSTRACT

Tricarbonylchromium complexes of 4-aryl-1,4-dihydropyridines have been synthesized. Selective complexation of the $\text{Cr}(\text{CO})_3$ on the aryl ring in the presence of a 1,4-dihydropyridine ring using the standard thermodynamic conditions ($\text{Cr}(\text{CO})_6$ in refluxing dibutyl ether/THF) was achieved.

KEYWORDS: 1,4-dihydropyridines, tricarbonylchromium complexes, Hantzsch synthesis, calcium channel blockers, regioselectivity

INTRODUCTION

1,4-Dihydropyridines (4-aryl-1,4-dihydro-2,6 dimethyl-3,5-pyridinedicarboxylates) are known to have potent antihypertensive and vasodilative actions through calcium antagonism [1, 2]. Various 1,4-dihydropyridine derivatives have been developed for clinical purposes and are used as drugs against hypertension and ischemic heart disease. Nifedipine (Fig. 1), with symmetrical substituents on its dihydropyridine ring, is achiral; while second-generation derivatives, such as nimodipine, amlodipine, and nicardipine (Fig. 1), with unsymmetrical substitution, are chiral, and demonstrate moderate to significant enantioselectivity in their pharmacological effects. Although the two enantiomers possessing an asymmetric carbon at the position 4 have been reported to have different biological activities [3, 4, 5], most 1,4-dihydropyridines are provided as racemates. Because of importance of C-4 chirality in the

pharmacological activity of 1,4-dihydropyridines, the availability of asymmetric synthesis or resolution of racemate forms are desirable.

On the other hand (arene) chromium tricarbonyl derivatives have found wide application in synthesis [6] and in biological applications as probes of drug-receptor binding [7]. Since unsymmetrically 1,2- and 1,3-disubstituted arenechromium tricarbonyl complexes are chiral and are enantiomeric on the basis of which face of the arene the chromium tricarbonyl fragment occupies [8], our attention has focused on the preparation of tricarbonylchromium complexes of Hantzsch esters. To our knowledge, there is just one report on the synthesis of tricarbonylchromium complexes of Hantzsch esters [9]. Herein, we report our preliminary results on the synthesis of some tricarbonyl(η^6 -arene)chromium complexes of 4-aryl-1,4-dihydropyridines. Further studies in this field are being actively pursued in our laboratory.

EXPERIMENTAL

All manipulations involving chromium complexes were performed under atmosphere of purified argon and using gas/vacuum double manifold and standard Schlenk technique [17]. THF was distilled from sodium/benzophenone ketyl immediately prior to use. Dibutyl ether was dried over sodium and distilled under an atmosphere of argon prior to use. $\text{Cr}(\text{CO})_6$ was purchased from Aldrich and sublimed prior to use. Elemental analyses: Elementer Model Vario EL III; FT-IR: Bruker PS-15; ^1H and ^{13}C NMR: Bruker SP-400 AVANC; melting points: Electrothermal-9100.

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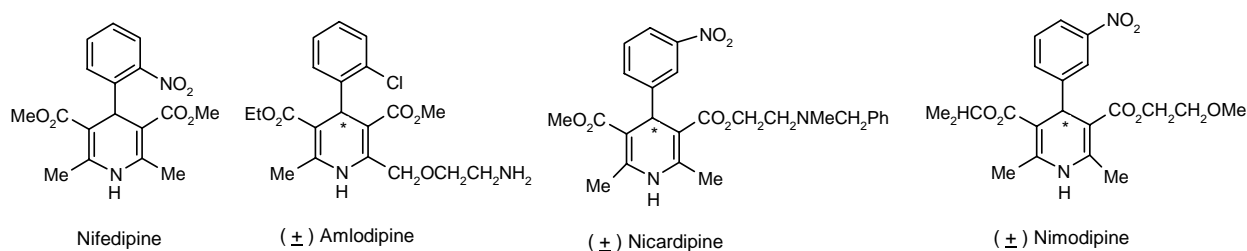


Figure 1. Biologically important 1,4-dihydropyridines.

General procedure for preparation of tricarbonyl(η^6 -arene)chromium(0) complexes

A mixture of 4-aryl-1,4-dihydropyridine (0.83 mmol) and freshly sublimed $\text{Cr}(\text{CO})_6$ (0.22 g, 1 mmol) in dibutyl ether (27ml) and THF (3ml) was heated under reflux (bath temperature: 140 °C) for 20-68 h. Volatiles were removed in vacuo. The resulting yellow oil or solid was then purified by column chromatography [Al_2O_3 Grade III] or recrystallization to give the chromium complexes.

[3,5-dicarboethoxy-2,6-dimethyl-4-(η^6 -2-methoxyphenyl)-1,4-dihydropyridine] tricarbonylchromium(0) **2**

The general procedure was followed using 4-aryl-1,4-dihydropyridine **1a** for 48 h and crystallization of crude yellow solid from dichloromethane / hexane gave **2** as yellow solid (0.34 g, 80%).

m.p. 164 °C (Dec.); IR (KBr) ν_{max} . 1961 and 1883 cm^{-1} (CO); ^1H NMR (400 MHz, CDCl_3): δ =1.11 (t, J =7.09 Hz, 3H, CH_3 ester), 1.21 (t, J =7.11 Hz, 3H, CH_3 ester), 2.24 (s, 3H, CH_3 -2), 2.32 (s, 3H, CH_3 -6), 3.69 (s, 3H, O- CH_3), 3.94-4.10 (m, 4H, $2\times\text{CH}_2$ ester), 4.96 (t, J =6.28 Hz, 1H, Ar- H_a), 5.00 (s, 1H, C(4)-H), 5.39 (d, J =6.79 Hz, 1H, Ar- H_c), 5.63 (dd, J_1 =5.30 Hz, J_2 =1.23 Hz, 1H, Ar- H_d), 5.86 (dt, J_1 =6.53 Hz, J_2 =1.22 Hz, 1H, Ar- H_b), 8.96 (s, 1H, NH) ppm; Anal. Calcd for $\text{C}_{23}\text{H}_{25}\text{N}\text{Cr}\text{O}_8$: C, 55.75; H, 5.08; N, 2.82. Found: C, 55.46; H, 4.75; N, 2.79.

[3,5-dicarboethoxy-2,6-dimethyl-4-(η^6 -2-chlorophenyl)-1,4-dihydropyridine] tricarbonylchromium(0) **3**

The general procedure was followed using 4-aryl-1,4-dihydropyridine **1b** for 48 h and Crystallization of crude yellow solid from toluene/ hexane gave **3** as yellow crystalline solid (51%).

m.p. 160 °C (Dec.); IR (KBr) ν_{max} . 1961 and 1883 cm^{-1} (CO); ^1H NMR (400 MHz, CDCl_3): δ =1.18-1.27(m, 6H, $2\times\text{CH}_3$ ester), 2.32(s, 3H, CH_3 -2), 2.39(s, 3H, CH_3 -6), 4.08-4.28(m, 4H, $2\times\text{CH}_2$ ester), 4.6(s, 1H, C(4)-H), 5.08-5.80 (m, 4H, Ar-H), 7.19(br.s, 1H, NH) ppm.

[2,10-dimethyl-1,8b-dihydro- η^6 -benzo-2-pyrone[3,4c]pyridine-9-carboxylic acid ethyl ester] tricarbonylchromium(0) **4**

The general procedure was followed using 4-aryl-1,4-dihydropyridine **1c** or **1d** for 68 h and crystallization of crude yellow solid from dichloromethane / hexane gave **4** as yellow crystalline solid (62%).

m.p. 180 °C(Dec.); IR (KBr) ν_{max} . 1959 and 1875 cm^{-1} (CO); ^1H NMR (400 MHz, DMSO-d_6): δ =1.17(t, J =7.08 Hz, 3H, CH_3 ester), 2.07 (s, 3H, CH_3 - 2), 2.27(s, 3H, CH_3 - 6), 4.05- 4.25(m, 2H, CH_2 ester), 4.68(s, 1H, C(4)-H), 5.24(t, J =6.19Hz, 1H, Ar- H_b), 5.59 (d, J =6.35 Hz, 1H, Ar- H_d), 5.83- 5.91(m, 2H, Ar- H_a and Ar- H_c), 9.29(br.s, 1H, NH) ppm.

[2-morpholinomethyl-3,5-dicarboethoxy-6-dimethyl-4-(η^6 -2-methoxyphenyl)-1,4-dihydropyridine] tricarbonylchromium(0) **9**

The general procedure was followed using 4-aryl-1,4-dihydropyridine **6** for 68 h and purification of yellow oil by column chromatography [Al_2O_3 Grade III, EtOAc/Hex 1:2] and then crystallization from ethanol gave **9** as yellow crystalline solid (47%).

m.p. 180 °C(Dec.); IR (KBr) ν_{max} . 1947 and 1858 cm^{-1} (CO); ^1H NMR (400 MHz, DMSO-d_6): δ =1.13(t, J =6.95 Hz, 3H, CH_3 ester), 1.22 (t, J =7.01 Hz 3H, CH_3 ester), 2.29(s, 3H, CH_3 -6), 2.39-2.42(m, 4H, $2\times\text{CH}_2$ -N), 3.57-3.66

(m, 6H, $2\times\text{CH}_2\text{-O}$ and $\text{CH}_2\text{-2}$), 3.69 (s, 3H, O- CH_3), 3.96-4.12(m, 4H, $2\times\text{CH}_2$ ester), 4.96 (t, $J=6.23\text{Hz}$, 1H, Ar- H_b), 5.05(s, 1H, C(4)-H), 5.41(d, $J=6.87\text{ Hz}$, 1H, Ar- H_d), 5.60-5.65(m, 1H, and Ar- H_a), 5.87(t, $J=6.33\text{Hz}$, 1H, Ar- H_c), 8.60(br.s, 1H, NH) ppm.

[2-(Methylthio)-methyl-3,5-dicarboethoxy-6-methyl-4-($\eta^6\text{-2-methoxyphenyl}$)-1,4-dihydropyridine] tricarboylchromium(0) 10

The general procedure was followed using 4-aryl-1,4-dihydropyridine **7** for 20 h and purification of yellow oil by column chromatography [Al_2O_3 Grade III, EtOAc/Hex 1:3] gave mixture of **10** and **1a** (1:2) as yellow crystalline. The ratio of products was obtained by analyzing of the ^1H NMR spectrum. When the reaction was carried out at 160 °C, compound **1a** was the sole product of the reaction.

Compound **10**: ^1H NMR (400 MHz, DMSO- d_6): $\delta=1.09$ (t, $J=7.04\text{ Hz}$, 6H, $2\times\text{CH}_3$ ester), 2.03(s, 3H, S- CH_3), 2.36(s, 3H, $\text{CH}_3\text{-6}$), 3.70 (s, 3H, O- CH_3), 3.76- 4.09(m, 6H, $\text{CH}_2\text{-S}$ and $2\times\text{CH}_2$ ester), 4.98(t, $J=6.14\text{ Hz}$, 1H, Ar-H), 5.05 (s, 1H, C4-H), 5.41(m, 1H, Ar-H), 5.64 (m, 1H, Ar-H), 5.88(m, 1H, Ar-H), 9.10(br.s, 1H, NH) ppm.

Compound **1a**: ^1H NMR (400 MHz, DMSO- d_6): $\delta=1.09$ (t, $J=7.04\text{ Hz}$, 6H, $2\times\text{CH}_3$ ester), 2.19 (s, 6H, $\text{CH}_3\text{-2}$ and $\text{CH}_3\text{-6}$), 3.70(s, 3H, O- CH_3), 3.93(q, $J=7.03\text{Hz}$, 4H, $2\times\text{CH}_2$ ester), 5.15(s, 1H, C4-H), 6.77(t, $J=7.35\text{ Hz}$, 1H, Ar- H_b), 6.83 (d, $J=8.08\text{ Hz}$, 1H, Ar- H_a), 7.03- 7.08(m, 2H, Ar- H_c and Ar- H_d), 8.36(br.s, 1H, NH) ppm.

Preparation of 2-morpholinomethyl-3,5-dicarboethoxy-6-dimethyl-4-(2-methoxyphenyl)-1,4-dihydropyridine 6

A solution of 2-bromomethyl-3,5-dicarboethoxy-6-methyl-4-(2-methoxyphenyl)-1,4-dihydropyridine **5** (obtained from 1.39 mmol of **1a**) in THF (20 ml) was added *via* a cannula, to a magnetically stirred solution of sodium salt of morpholine in THF at 0°C. Sodium salt of morpholine was prepared *in situ* from morpholine (0.24 ml, 2.18 mmol) and NaH 60% (0.096 g, 2.4 mmol) in THF (15 ml) at room temperature under argon atmosphere for 1 h and then cooled to 0°C. The mixture was stirred by warming to room

temperature for 3 h and then evaporated. The residue was partitioned between CH_2Cl_2 and HCl 2M, the organic layer washed with saturated Na_2CO_3 solution and water, dried over Na_2SO_4 and evaporated. The resulting oil was purified by column chromatography [silicagel 60, 70-230 mesh, EtOAc/Hex (5:2)] and then recrystallization of obtained yellow oil from ethanol to give **6** (0.34 g, 55%) as yellow crystals.

m.p. 132.5-134 °C; IR (KBr) $\bar{\nu} = 3283$ (m), 2981-2850(m), 1682(s), 1648(m), 1606(m), 1471(s), 1280(s), 1200(s), 1103(s), 749(s) cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6): $\delta=1.07$ -1.14(m, 6H, $2\times\text{CH}_3$ ester), 2.27(s, 3H, $\text{CH}_3\text{-6}$), 2.39(t, $J=4.14\text{Hz}$, 4H, $2\times\text{CH}_2\text{-N}$), 3.52(AB quartet, $J=14.44\text{ Hz}$, 2H, $\text{CH}_2\text{-2}$), 3.60(t, $J=4.33\text{Hz}$, 4H, $2\times\text{CH}_2\text{-O}$), 3.69(s, 3H, O- CH_3), 3.91-3.98(m, 4H, $2\times\text{CH}_2$ ester), 5.18(s, 1H, C(4)-H), 6.77(t, $J=7.75\text{ Hz}$, 1H, Ar- H_b), 6.85(d, $J=7.75\text{Hz}$, 1H, Ar- H_d), 7.02-7.10(m, 2H, Ar- H_a and Ar- H_c), 8.35(br.s, 1H, NH) ppm.

Anal. Calcd for $\text{C}_{24}\text{H}_{32}\text{N}_2\text{O}_6$: C, 64.85; H, 7.25; N, 6.30. Found: C, 64.72; H, 7.24; N, 6.57.

S-[(6-methyl-3,5-dicarboethoxy-4-(2-methoxyphenyl)1,4-dihydropyridin-2-yl)-methyl]-isothiuronium bromide 7

A mixture of 2-bromomethyl-3,5-dicarboethoxy-4-(2-methoxyphenyl)-6-methyl-1,4-dihydropyridine (obtained from 1.39 mmol of **1a**), thiourea (0.12 g, 1.53 mmol) and ethanol (35 ml) was heated to reflux for 1.5 h and then evaporated. Recrystallization of crude product from acetonitrile furnished **7** (0.54 g, 75%) as yellow crystals.

m.p. 152 °C (Dec.); IR (KBr) $\bar{\nu} = 3300$ 2750(br.s), 1672(s), 1646(s), 1507(s), 1306(s), 1289(s), 1217(s), 1097(s), 757(m) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): $\delta=1.22$ (m, 6H, $2\times\text{CH}_3$ ester), 2.37(s, 3H, $\text{CH}_3\text{-6}$), 3.77(s, 3H, O- CH_3), 3.98-4.13(m, 4H, $2\times\text{CH}_2$ ester), 4.44(AX, $J=15.20\text{Hz}$, 1H, $\text{CH}_2\text{-2}$), 4.73(AX, $J=15.20\text{Hz}$, 1H, $\text{CH}_2\text{-2}$), 5.17(s, 1H, C(4)-H), 6.78-6.82 (m, 2H, Ar- H_a and Ar- H_c), 7.09-7.16(m, 2H, Ar- H_b and Ar- H_d), 8.26(br.s, 1H, NH), 8.79(br.s, 2H, NH_2), 9.18(br.s, 2H, NH_2) ppm; Anal. Calcd for $\text{C}_{20}\text{H}_{28}\text{BrN}_3\text{O}_5\text{S}$: C, 47.81; H, 5.62; N, 8.36. Found: C, 48.01; H, 5.49; N, 8.57.

2-(Methylthio)-methyl-3,5-dicarboethoxy-6-methyl-4-(2-methoxyphenyl)-1,4-dihydropyridine **8**

An aqueous solution of NaOH (32%, 0.53 ml) was added to a stirred solution of **7** (0.5g, 0.97 mmol) and methyl iodide (0.27 ml, 2.33 mmol) in ethanol/water (1:1, 20 ml), under an argon atmosphere. After 1 h stirring at room temperature, the mixture was filtered. Recrystallization of the crude product from ethanol/water furnished compound **8** (0.37 g, 66%).

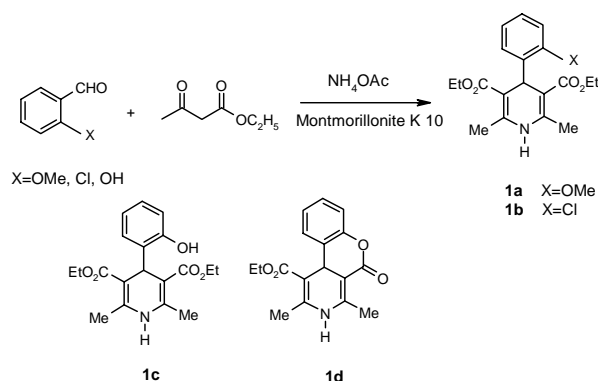
m.p. 124-126 °C; IR (KBr) $\bar{\nu}$ = 3328(s), 3091(w), 2977-2919(m), 1673(s), 1638(m), 1617(m), 1492(s), 1286(s), 1214(s), 1103(s), 746(s) cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ =1.17-1.23(m, 6H, 2 \times CH₃ ester), 2.03(s, 3H, S-CH₃), 2.34(s, 3H, CH₃-6), 3.81(s, 3H, O-CH₃), 3.81- 4.09(m, 6H, CH₂-S and 2 \times CH₂ ester), 5.30(s, 1H, C4-H), 6.65(br.s, 1H, NH), 6.78- 6.83(m, 2H, Ar-H_a and Ar-H_c), 7.11 (dt, $J_1=7.60$ Hz, $J_2=1.73$ Hz, 1H, Ar-H_b), 7.21 (dd, $J_1=7.64$ Hz, $J_2=1.73$ Hz, 1H, Ar-H_d) ppm; Anal. Calcd for C₂₁H₂₇NO₅S: C, 62.20; H, 6.71; N, 3.45. Found: C, 61.93; H, 6.58; N, 3.66.

RESULTS AND DISCUSSION

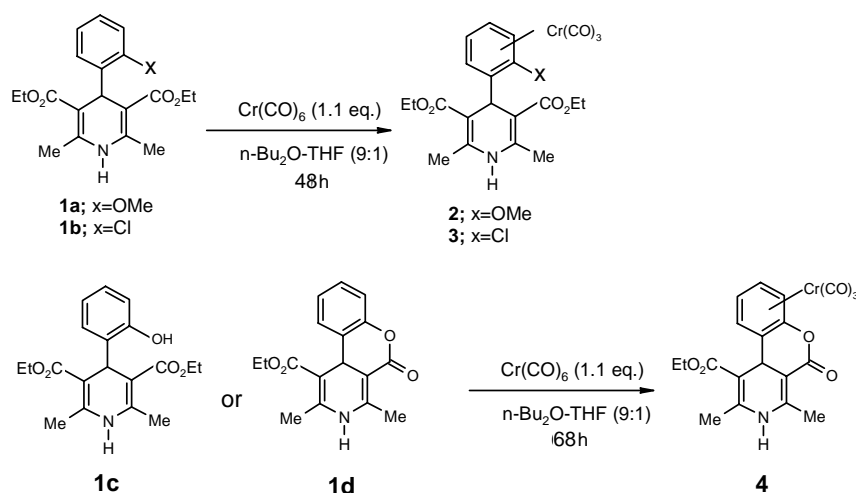
Synthesis was started by Hantzsch reaction of ethyl acetoacetate with appropriate aldehyde and ammonium acetate in refluxing ethanol over the montmorillonite K10 catalyst for 20 minutes,

which afforded the 1,4-dihydropyridines **1a-d** (Scheme 1) [10]. In the reaction with salicylaldehyde compounds **1c** and **1d** were obtained in 23% and 77% respectively, the ratio of products was determined by the ^1H NMR spectrum.

Tricarbonylchromium complexes **2** and **3** were synthesized by treating **1a** and **1b** with hexacarbonylchromium in di-n-butyl ether and tetrahydrofuran (THF) (9:1 v/v) at reflux temperature. Treating of both **1c** and **1d** with $\text{Cr}(\text{CO})_6$ under this condition gave complex **4** as the sole product of the reaction (Scheme 2). This result demonstrates the intramolecular transesterification



Scheme 1. Synthesis of 4-aryl-1,4-dihydropyridine derivatives.

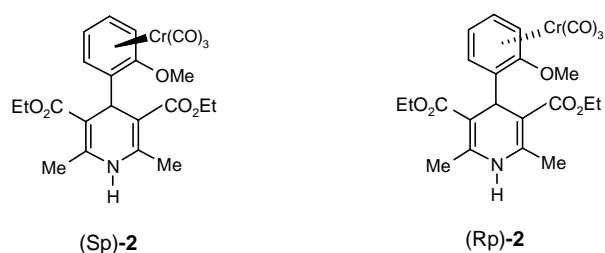


Scheme 2. Synthesis of tricarbonylchromium complexes of 4-aryl-1,4-dihydropyridines.

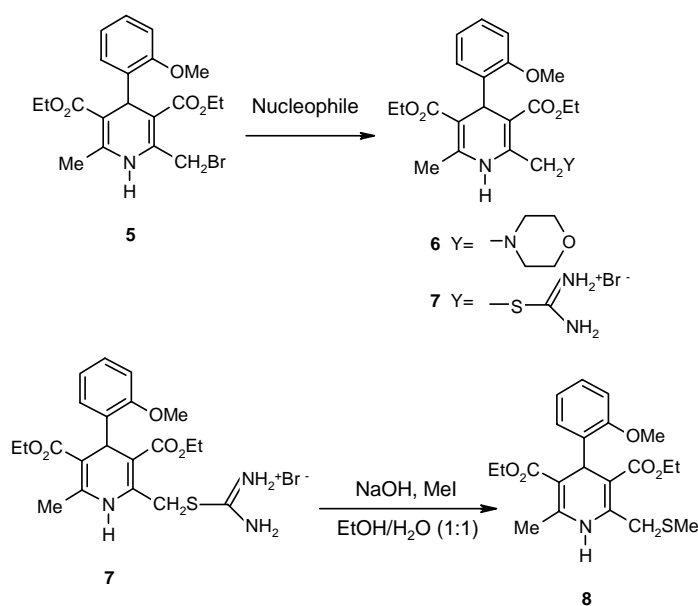
between hydroxyl substituent of 4-aryl ring and the ester group of dihydropyridine ring. In all these compounds complexation was achieved selectively on the aryl ring in the presence of a 1,4-dihydropyridine ring. Regioselectivity was clearly established as η^6 to the 4-aryl substituent by the ^1H chemical shifts. For example the complexation of **1a** at the benzo ring with the $\text{Cr}(\text{CO})_3$ group causes a large up field shift of these aromatic proton resonances (ca. 1.5 ppm) and a smaller up field shift of the benzylic proton (C(4)-H) resonance (0.27 ppm). These effects are commonly observed when aromatic substrates are complexed with tricarbonylchromium [11].

Unsymmetrically 1,2- and 1,3-disubstituted arenechromium tricarbonyl complexes are planar chiral compounds and are enantiomeric on the basis of which face of the arene the chromium tricarbonyl fragment occupies [8]. The stereochemical descriptor for a planar chirality is usually determined following the rules introduced by Schögl: [12, 13] the arene ring is monitored from that side which is not coordinated to the chromium moiety. The priority of the substituents is then determined employing the Cahn-Ingold-Prelog (CIP) rules. If the shortest path from the substituent displaying highest priority to the

following one is clockwise, the absolute configuration is denoted as Rp, and the apposite case is referred to as Sp (e.g. (Sp)-**2** and (Rp)-**2**). There exists also a different procedure for the stereochemical assignment consisting of an extension of the CIP system which results in opposite planar chiral descriptors [14].



Then, we prepared the unsymmetrical derivatives of **1a** for the preparation of diastereomeric tricarbonyl chromium complexes of 1,4-DHPs. The Reaction of 1,4-dihydropyridine **1a** with 1.1 equivalents of pyridinium bromide perbromide in dichloromethane/pyridine at $-20\text{ }^\circ\text{C}$ for 45 minutes afforded the crude product **5** as a yellow gum. We have published before the synthesis of **5** [2-bromomethyl-3,5-dicarboethoxy-6-methyl-4-(2-



Scheme 3. Preparation of unsymmetrical 1,4-dihydropyridine derivatives.

methoxyphenyl)-1,4-dihydropyridine] in high yield [15] by modifying the literature methods [16]. Without further purification this brominated adduct was coupled with sodium salt of morpholine and thiourea as nucleophiles at different conditions to give 2-substituted 1,4-dihydropyridines **6** and **7** respectively. In the reaction of **5** with thiourea in refluxing ethanol for 5h, evaporation of solvent and recrystallization from EtOAc/Hex, isothiuronium salt **7** is formed. On the other hand, reaction of isothiuronium salt **7** with methyl iodide in the presence of base produced S-alkylated derivative **8** (Scheme 3). The C-4 carbon atom of 1,4-dihydropyridines is a prochiral atom. When at least one of the substituents, bound to the C-2 and C-3 carbon atoms, is different from those on the symmetric C-6 and C-5 positions of ring, the C-4 carbon atom is chiral and the compounds are racemates. Meanwhile compounds **6-8** with different substituents at C-2 and C-6 are racemic mixtures.

Complexation of racemate **6** [2-morpholinomethyl-3,5-dicarboethoxy-6-methyl-4-(3-methoxyphenyl)-1,4-dihydropyridine] with hexacarbonyl chromium, under thermal condition, gave complex **9**. The reaction of **8** [2-(methylthio)-methyl-3,5-dicarboethoxy-6-methyl-4-(3-methoxyphenyl)-1,4-dihydropyridine] with hexacarbonyl chromium, under thermal condition, gave 1:2 mixtures of tricarbonyl chromium complex **10** and desulfurized compound **1a** [2,6-dimethyl-3,5-dicarboethoxy-4-(2-methoxyphenyl)-1,4-dihydropyridine]. The ratio of products was determined by ¹H NMR spectrum, unfortunately we couldn't separate them by recrystallization and chromatography methods.

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