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MANGANESE(III)-BASED REACTION OF 1,1-DIARYLETHENES AND 2,3-PYRROLIDINEDIONES. A SIMPLE ROUTE TO 4-ETHENYL- AND 4- ETHYL-2,3-PYRROLIDINEDIONE DERIVATIVES

Nguyen Van Ha*, Le Thi My Trinh

Department of Chemistry, Dalat University, 1 Phu Dong Thien Vuong, Dalat, Vietnam

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Abstract

2,3-Pyrrolidinediones reacted with manganese(III) acetate dihydrate in the presence of 1,1-diarylethenes to give 4-(2,2-diaryl)ethenyl-2,3-pyrolidinediones and/or 4-(2-acetoxy-2,2-diaryl)ethyl-2,3-pyrrolidinediones in good yields. The distribution of reaction products depended on the nature of 4-substituent on benzene ring of the 1,1-diarylethene used. The reaction mechanism is discussed.

Keywords. Manganese(III) oxidation, 2,3-Pyrrolidinediones, 1,1-Diarylethenes, new 2,3-Pyrrolidinedione derivatives.

1. INTRODUCTION

The synthesis of pyrrolidinediones has been, and continues to be, a topic of interest [1] since these types of compounds have been found to be potent and specific inhibitors of aldose reductase [2] and endothelin receptor antagonists [3]. Pyrrolidinediones were also used as versatile reagents for the preparation of β-lactams [4]. It was found that manganese(III) oxidation of alkenes was a simple and straightforward route to the introduction of a substituted ethenyl- or ethyl- group to β-keto ester systems [5] and previously, we showed an efficient insertion of such groups into ethyl-1-benzyl-3-hydroxy-3-pyrrolin-2-one-4-carboxylate to provide the corresponding 2,3-pyrrolidinedione derivatives [6]. In the present work, we described the application of this approach for the synthesis of other new 2,3-pyrrolidinediones.

2. EXPERIMENTAL SECTION

2.1. Measurements

All of the ^{1}H and ^{13}C NMR spectra were recorded with a JNM-AL 300 FT NMR spectrometer at 300 MHz for ^{1}H and 75 MHz for ^{13}C, respectively, with tetramethylsilane as the internal standard. The chemical shifts are shown in δ values (ppm) and coupling constants in Hz. The IR spectra were measured on a Paragon 1000 FT IR spectrometer and the IR spectral data are expressed in cm^{-1} (ν). All of the melting points were determined with a Yanaco micromelting-point apparatus MP-J3 and were uncorrected.

2.2. Materials

Manganese(III) acetate dihydrate, $Mn(OAc)_3 \cdot 2H_2O$, was prepared according to the literature method [7]. 1,1-Diarylethenes **1a-d** were prepared by dehydration of the corresponding alcohols, which were synthesized from substituted acetophenones and arylmagnesium bromides [8]. 2,3-Pyrrolidinediones **2a-d** were prepared according to the method described in the literature [6b,9]. Manganese(II) acetate tetrahydrate, and glacial acetic acid were purchased from Wako Pure Chemical Ind., Ltd., and were used as received.

2.3. Reaction procedure

A general procedure is as follows. 1,1-diarylethene (1 mmol) was weighed into a 50 mL flask equipped with a magnetic stirrer. Glacial acetic acid (15 mL) and 2,3-pyrrolidinedione (1.5-2 mmol) were added. The flask was placed in an oil bath and fitted with a reflux condenser. The mixture was stirred and heated and manganese (III) acetate dihydrate (3 mmol) was added just before refluxing. The reaction was allowed to proceed until the reaction mixture turned colorless or yellow

(normally for 2 min). The solvent was removed *in vacuo*, and the residue was quenched with water. The aqueous mixture was extracted with chloroform. The extract was dried over anhydrous sodium sulfate, filtered and concentrated to dryness. The products were separated on silica gel TLC (Wakogel B-10 or Merck Kieselgel 60 F_{254}) with methanol/dichloromethane (1:99 v/v) as the developing solvent. Solid products were further recrystallized by indicated solvent. Specific details are given below.

2.4. Product data

1-Benzyl-4-methoxycarbonyl-4-(2,2-diphenyl)-ethenyl-2,3-pyrrolidinedione (3aa): colorless needles (from dichloromethane/ *n*-hexane); mp 160-161 ^oC; IR ($CHCl_3$) 1772.5 (–COO–), 1742.2 (–CO–), 1716.5 (–CON–); ^1H NMR ($CDCl_3$, δ, ppm) 3.20 (1H, d, *J* = 11.37, $\underline{H}^a$–CHN<), 3.55 (1H, d, *J* = 11.37, $\underline{H}^b$–CHN<), 3.62 (3H, s, $–OCH_3$), 4.33 (1H, d, *J* = 14.41, $\underline{H}^a$–CHPh), 4.51 (1H, d, *J* = 14.41, $\underline{H}^b$–CHPh), 6.57 (1H, s, =CH–), 6.97-7.33 (15H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.57 (>C=O), 167.20 (–COO–), 156.84 (–CON–), 146.22 (>C=), 140.67, 138.31, 133.53 (arom C), 129.43 (2C), 129.13 (2C), 128.66 (2C), 128.45 (2C), 128.20, 128.12, 128.08 (2C), 127.89, 127.16 (2C) (arom CH), 122.55 (=CH–), 56.77 (>C<), 53.61 (OCH_3), 50.63, and 48.27 (CH_2).

1-Benzyl-4-methoxycarbonyl-4-[2,2-bis(4-methylphenyl)]ethenyl-2,3-pyrrolidinedione (3ba): colorless microcrystals (from dichloromethane/ *n*-hexane); mp 171-171.5 ^oC; IR ($CHCl_3$) 1770.5 (–COO–), 1740.2 (–CO–), 1716.5 (–CON–); ^1H NMR ($CDCl_3$, δ, ppm) 2.26 (3H, s, $–CH_3$), 2.36 (3H, s, $–CH_3$), 3.23 (1H, d, *J*=11.19, $\underline{H}^a$–CHN<), 3.56 (1H, d, *J* = 11.19, $\underline{H}^b$–CHN<), 3.60 (3H, s, $–OCH_3$), 4.33 (1H, d, *J* = 14.41, $\underline{H}^a$–CHPh), 4.53 (1H, d, *J* = 14.41, $\underline{H}^b$–CHPh), 6.51 (1H, s, =CH–), 6.82-7.28 (13H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.64 (>C=O), 167.12 (–COO–), 156.75 (–CON–), 145.86 (>C=), 137.91, 137.68 (2C), 135.33, 133.41 (arom C), 128.85 (4C), 128.54 (2C), 128.39 (2C), 128.17 (2C), 127.88, 126.88 (2C) (arom CH), 121.42 (=CH–), 56.65 (>C<), 53.32 (OCH_3), 50.56, 48.00 (CH_2), 20.89, and 20.67 (CH_3).

1-Benzyl-4-methoxycarbonyl-4-[2,2-bis(4-methoxyphenyl)]ethenyl-2,3-pyrrolidinedione (3ca): colorless microcrystals (from dichloromethane/*n*-hexane); mp 168-169 ^oC; IR ($CHCl_3$) 1772.5 (–COO–), 1742.2 (–CO–), 1716.5 (–CON–); ^1H NMR ($CDCl_3$, δ, ppm) 3.23 (1H, d, *J* = 11.19, $\underline{H}^a$–CHN<), 3.59 (1H, d, *J* = 11.19, $\underline{H}^b$–CHN<), 3.63 (3H, s, $–OCH_3$), 3.73 (3H, s, $–OCH_3$), 3.81 (3H, s, $–OCH_3$), 4.37 (1H, d, *J* = 14.41, $\underline{H}^a$–CH_2Ph), 4.54 (1H, d, *J* = 14.41, $\underline{H}^b$–CHPh), 6.43 (1H, s, =CH–), 6.74-7.29 (13H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.82 (>C=O), 167.29 (–COO–), 156.81 (–CON–), 145.26 (>C=), 159.38, 159.15, 133.49 (2C), 130.51, (arom C), 130.27 (2C), 128.47 (2C), 128.31 (2C), 128.20 (2C), 127.92, 113.63 (2C), 113.23 (2C) (arom CH), 120.47 (=CH–), 56.77 (>C<), 54.95, 54.88, 53.40 (OCH_3), 50.65, and 48.09 (CH_2).

4-Butoxycarbonyl-4-[2,2-bis(4-methylphenyl)]ethenyl-1-methyl-2,3-pyrrolidinedione (3bb): colorless microcrystals (from dichloromethane/ *n*-hexane); mp 118-118.5 ^oC; IR ($CHCl_3$) 1772.5 (–COO–), 1740.2 (–CO–), 1716.5 (–CON–); ^1H NMR ($CDCl_3$, δ, ppm) 0.89 (3H, t, *J* = 7.34, $–CH_2CH_2CH_2C\underline{H}_3$), 1.32 (2H, br sex, $–CH_2CH_2C\underline{H}_2CH_3$), 1.58 (2H, br quin, $–CH_2C\underline{H}_2CH_2CH_3$) , 2.32 (3H, s, $–CH_3$), 2.39 (3H, s, $–CH_3$), 2.87 (3H, s, $>NCH_3$), 3.33 (1H, d, *J* = 11.20, $\underline{H}^a$–CHN<), 3.67 (1H, d, *J* = 11.20, $\underline{H}^b$–CHN<), 4.10-4.18 (2H, m, $–C\underline{H}_2CH_2CH_2CH_3$), 6.56 (1H, s, =CH–), 6.92-7.20 (8H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.37 (>C=O), 166.69 (–COO–), 156.99 (–CON–), 145.50 (>C=), 138.07, 137.69, 137.52, 135.34 (arom C), 128.81 (4C), 128.46 (2C), 126.81 (2C) (arom CH), 121.81 (=CH–), 66.35, 52.98, 29.80, 18.41 (CH_2), 56.52 (>C<), 30.91, 20.77, 20.57, and 13.10 (CH_3).

4-Ethoxycarbonyl-1-ethyl-4-[2,2-bis(4-methylphenyl)]ethenyl-2,3-pyrrolidinedione (3bc): colorless liquid; IR ($CHCl_3$) 1772.5 (–COO–), 1740.2 (–CO–), 1716.5 (–CON–); ^1H NMR ($CDCl_3$, δ, ppm) 1.02 (3H, t, *J*=7.34 Hz, $–CH_2C\underline{H}_3$), 1.24 (3H, t, *J* = 7.16, $–OCH_2C\underline{H}_3$), 2.32 (3H, s, $–CH_3$), 2.38 (3H, s, $–CH_3$), 2.90-3.51 (2H, m, $>NC\underline{H}_2CH_3$), 3.33 (1H, d, *J* = 11.01, $\underline{H}^a$–CHN<), 3.67 (1H, d, *J* = 11.01, $\underline{H}^b$–CHN<), 4.10-4.21 (2H, m, $–OC\underline{H}_2CH_3$), 6.58 (1H, s, =CH–), 6.92-7.19 (8H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.90 (>C=O), 166.68 (–COO–), 156.67 (–CON–), 145.64 (>C=), 138.12, 137.78, 137.62, 135.49 (arom C), 128.91 (4C), 128.51 (2C), 126.89 (2C) (arom CH), 121.78 (=CH–), 62.69, 50.37, 38.85 (CH_2), 56.68 (>C<), 20.82, 20.63, 13.44, and 11.29 (CH_3).

4-Butoxycarbonyl-1-butyl-4-[2,2-bis(4-methylphenyl)]ethenyl-2,3-pyrrolidinedione (3bd): colorless liquid; IR ($CHCl_3$) 1770.5 (–COO–), 1740.2 (–CO–), 1716.5 (–CON–); ^1H NMR ($CDCl_3$, δ, ppm) 0.89 (6H, t, *J* = 7.34, $–OCH_2CH_2CH_2C\underline{H}_3$ and $–NCH_2CH_2CH_2C\underline{H}_3$), 1.28-1.37 (6H, m, $–OCH_2CH_2C\underline{H}_2CH_3$ and $–NCH_2C\underline{H}_2C\underline{H}_2CH_3$), 1.59 (2H, br quin, $–OCH_2C\underline{H}_2CH_2CH_3$), 2.32 (3H, s,

$–CH_3$), 2.39 (3H, s, $–CH_3$), 3.14-3.36 (2H, m, $–NC\underline{H}_2CH_2CH_2CH_3$), 3.32 (1H, d, *J* = 11.01, $\underline{H}^a–CHN<$), 3.68 (1H, d, *J* = 11.01, $\underline{H}^b–CHN<$), 4.10-4.18 (2H, m, $–OC\underline{H}_2CH_2CH_2CH_3$), 6.58 (1H, s, =CH–), 6.92-7.19 (8H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.83 (>C=O), 166.82 (–COO–), 156.93 (–CON–), 145.60 (>C=), 138.20, 137.73, 137.58, 135.54 (arom C), 128.91 (4C), 128.51 (2C), 126.91 (2C) (arom CH), 121.88 (=CH–), 66.41, 50.90, 43.80, 29.88, 28.23, 19.42, 18.49 (CH_2), 56.68 (>C<), 20.82, 20.63, and 13.16 (2C) (CH_3).

4-(2-Acetoxy-2,2-diphenyl)ethyl-1-benzyl-4-methoxycarbonyl-2,3-pyrrolidinedione (4aa): colorless liquid; IR ($CHCl_3$) 1772.5, 1760.2 (–COO–), 1745.0 (–CO–), 1716.5 (–CON–); ^{1}H NMR ($CDCl_3$, δ, ppm) 1.94 (3H, s, $–COCH_3$), 2.85 (1H, d, *J* = 11.20, $\underline{H}^a–CHN<$), 3.39 (3H, s, $–OCH_3$), 3.49 (1H, d, *J* = 15.14, $\underline{H}^a–CHC(OAc)Ph_2$), 3.60 (1H, d, *J* = 11.20, $\underline{H}^b–CHN<$), 3.60 (1H, d, *J* = 15.14, $\underline{H}^b–CHC(OAc)Ph_2$), 4.36 (1H, d, *J* = 14.31, $\underline{H}^a–CHPh$), 4.55 (1H, d, *J* = 14.31, $\underline{H}^b–CHPh$), 7.13-7.33 (15H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.93 (>C=O), 168.58, 167.19 (–COO–), 157.48 (–CON–), 143.78, 142.65, 133.84 (arom C), 128.93 (2C), 128.71 (2C), 128.41, 128.35 (2C), 128.26 (2C), 127.63, 127.58, 126.20 (2C), 125.92 (2C) (arom CH), 83.32 (COAc), 56.34 (>C<), 53.50 (OCH_3), 48.56, 47.43, 40.36 (CH_2), and 22.09 ($–COCH_3$).

4-[2-Acetoxy-2,2-bis(4-chlorophenyl)]ethyl-1-benzyl-4-methoxycarbonyl-2,3-pyrrolidinedione (4da): colorless liquid; IR ($CHCl_3$) 1772.5, 1760.2 (–COO–), 1745.0 (–CO), 1716.5 (–CON–); ^{1}H NMR ($CDCl_3$, δ, ppm) 1.94 (3H, s, $–COCH_3$), 2.84 (1H, d, *J* = 11.10, $\underline{H}^a–CHN<$), 3.32 (1H, d, *J* = 15.05, $\underline{H}^a–CHC(OAc)Ar_2$), 3.44 (3H, s, $–OCH_3$), 3.64 (1H, d, *J* = 11.10, $\underline{H}^b–CHN<$), 3.73 (1H, d, *J* = 15.05, $\underline{H}^b–CHC(OAc)Ar_2$), 4.46 (1H, d, *J* = 14.16, $\underline{H}^a–CHPh$), 4.52 (1H, d, *J* = 14.16, $\underline{H}^b–CHPh$), 7.09-7.34 (13H, m, arom H); ^{13}C NMR ($CDCl_3$, δ, ppm) 193.44 (>C=O), 168.05, 166.30 (–COO–), 156.97 (–CON–), 141.71, 140.73, 133.41 (2C), 133.32 (arom C), 128.65 (2C), 128.45 (2C), 128.71 (2C), 128.46, 128.31 (2C), 127.39 (2C), 127.12 (2C) (arom CH), 83.10 (COAc), 54.23 (>C<), 53.26 (OCH_3), 48.24, 47.23, 39.59 (CH_2), and 21.71 ($–COCH_3$).

3. RESULTS AND DISCUSSION

3.1. Manganese(III)-Based Reaction of 1,1-Diarylethenes and Pyrrolidinediones

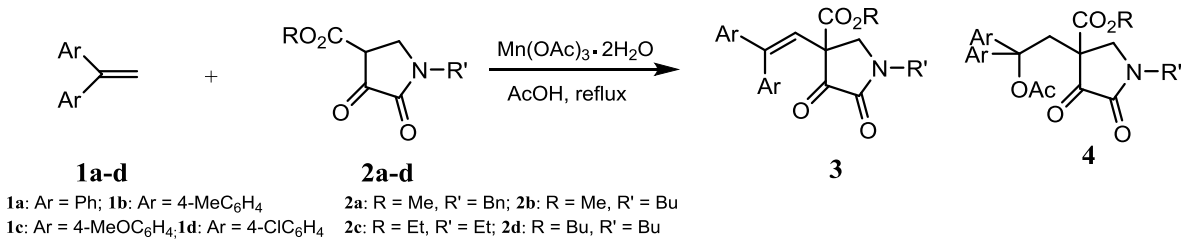
Reaction of 1,1-diphenylethene (**1a**), methyl 1-benzyl-3-hydroxy-3-pyrrolin-2-one-4-carboxylate (**2a**), and manganese(III) acetate dihydrate in glacial acetic acid at reflux provided 1-benzyl-4-methoxycarbonyl-4-(2,2-diphenyl)ethenyl-2,3-pyrrolidinedione (**3aa**) and 4-(2-acetoxy-2,2-diphenyl)ethyl-1-benzyl-4-methoxycarbonyl-2,3-pyrrolidinedione (**4aa**) in 46 % and 17 % isolated yields, respectively (Scheme 1 and Table 1, Entry 1). The structures of the products were established by spectroscopic methods. The ^{1}H NMR spectrum of **3a** showed one vinyl proton at 6.57 ppm and two pairs of doublet (4.33 and 4.53 ppm, 3.23 and 3.56 ppm). One pair of doublet was assigned to the benzyl methylene group and the other to the methylene group of pyrrolidinedione ring. In the ^{1}H NMR spectrum of **4a**, signal of vinyl proton disappeared and the presence of methyl group of acetate moiety was confirmed by a singlet at 1.94 ppm. An increase in the amount of **2a** used resulted in an improvement on the product yields (table 1, Entry 2). Interestingly, in the reaction of 1,1-bis(4-methylphenyl)ethene (**1b**), **3ba** was obtained in high yield without the formation of any **4ba** (Table 1, Entry 3). When a similar reaction was performed with 1,1-bis(4-methoxyphenyl)ethene (**1c**), product **3ca** was also obtained in 73% isolated yield without a trace amount of product **4** (Table 1, Entry 4). These results revealed that an electron-releasing substituent at the 4-position of benzene ring of 1,1-diarylethene favored the formation of product **3**. To further investigate the substituent effect on product formation, we next turned our attention to a similar reaction of 1,1-bis(4-chlorophenyl)ethene (**1d**). Surprisingly, this reaction gave **4da** in 54% yield as a sole isolated product (Table 1, Entry 5).

It was then of interest to investigate the applicability of this approach for the ethenylation of other pyrrolidinediones. Thus, several pyrrolidinediones bearing different alkyl groups were subjected to oxidation by manganese(III) acetate dihydrate in the presence of **1b**. These reactions afforded the corresponding **3** in good yields (table 1, Entry 6-8).

3.2. Reaction mechanism

At a glance, product **3** seems to be formed by an electrophilic substitution at the C_{sp2} of alkene. In fact, the reaction mechanism could involve two oxidative steps by manganese (III) complexes. Fristad reported that enolization of acetic acid ligand was the key step in the formation of radicals from acetic acid derivatives by a manganese(III) acetate-based oxidation system [10]. In a similar oxidation system of 2- substituted 1,3-dicarbonyl esters, Snider demonstrated that the key step was also an enolization [11]. Thus, in this present reaction, the

formation of products **3** and **4** could be explained through the sequence shown in scheme 2. In the first step, it seems likely that pyrrolidinedione forms the corresponding manganese(III) enolate complex **A** with manganese(III) acetate dihydrate *via* a ligand exchange process. In the presence of an alkene, the alkene and 1,3-dicarbonyl ligand would generate an electron donor-acceptor-like complex at this stage [12] and a subsequent one-electron transfer oxidation step could occur to give radical **B** [13]. Products **3** and **4** should be yielded from the intermediate carbocation **C** *via* a further oxidation of radical **B**. The former could be formed by an elimination of β-proton while the latter could be produced by an attack of acetate ion. It could be reasoned that, at this stage, an electron-withdrawing substituent on benzene ring of the 1,1-diarylethene could make the attack of an acetate ion more easily. Thus, the reaction of 1,1-bis(4-chlorophenyl)ethene produced only **4** as an isolated product.

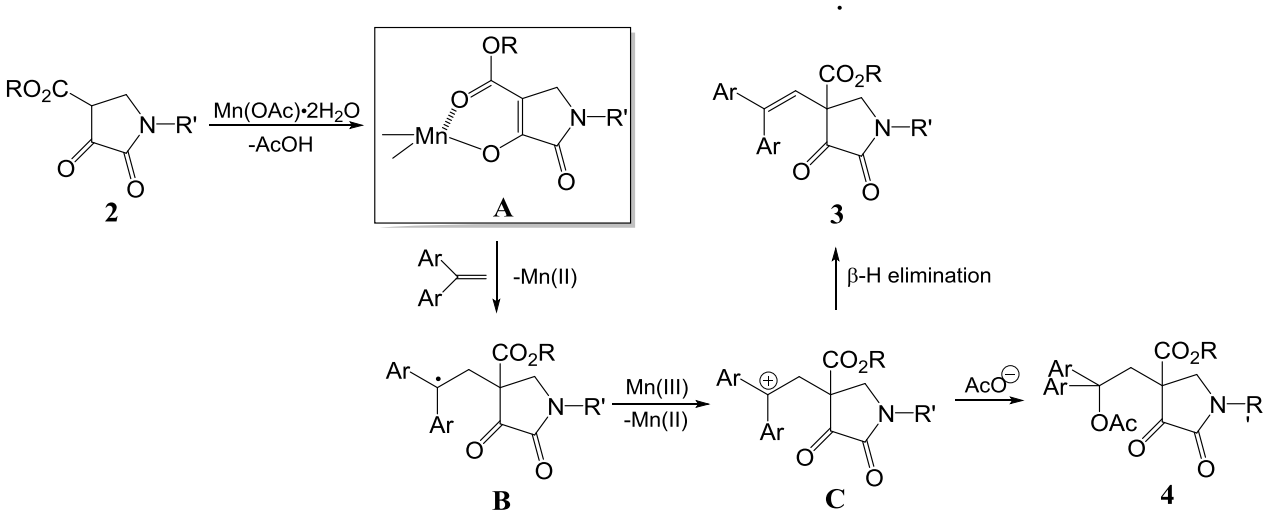


Scheme 1: Reaction of 1,1-diarylethenes **1a-d** and pyrrolidinediones **2a-d** in the presence of manganese triacetate dihydrate

Table 1: Reaction of 1,1-diarylethenes **1a-d** with 2,3-pyrrolidine-diones **2a-d** in the presence of Manganese(III) acetate dihydrate[a]

Entry	Alkene	Pyrrolidinedione	Product (yield %)[b]	
1[c]	**1a**	**2a**	**3aa** (46)	**4aa** (17)
2	**1a**	**2a**	**3aa** (48)	**4aa** (22)
3	**1b**	**2a**	**3ba** (72)	
4	**1c**	**2a**	**3ca** (73)	
5	**1d**	**2a**		**4da** (54)
6	**1b**	**2b**	**3bb** (87)	
7	**1b**	**2c**	**3bc** (74)	
8	**1b**	**2d**	**3bd** (85)	

[a] The reaction was caried out in glacial acetic acid under reflux at the molar ratio of **1**:**2**::$Mn(OAc)_3$ $2H_2O$ = 1:2:3 unless otherwise stated. [b] Isolated yield based on the amount of 1,1-diarylethene used. [c] The reaction was carried out at the molar ratio of **1**:**2**:$Mn(OAc)_3$ $2H_2O$ = 1:1.5:3.



Scheme 2: Proposed formation pathways of **3** and **4**

4. CONCLUSION

Manganese(III)-based oxidation system was a useful means to introduce a 2- substituted ethenyl- or ethyl- group to the 4-position of alkyl 2,3-pyrrolidinedione-4-carboxylates. This new approach has been applied successfully for the synthesis of some pyrrolidinedione derivatives. The product formation was greatly influenced by substituents on benzene ring of the 1,1-diarylethene used.

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Corresponding author: **Nguyen Van Ha**
Department of Chemistry, Dalat University
01 Phu Dong Thien Vuong, Da Lat, Lam Dong, Vietnam
E-mail: hanv@dlu.edu.vn.