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General Synthesis of γ-Functionalized β-Aryl-Substituted Primary Nitro Compounds*

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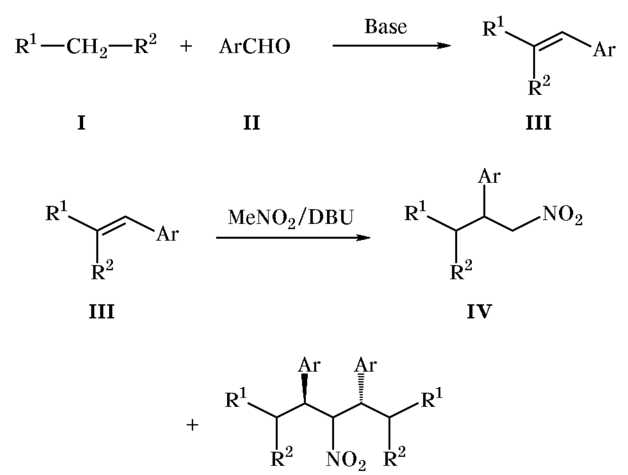
Abstract—A simple and general procedure was developed for synthesis of γ-functionalized β-aryl-substituted primary nitro compounds from aromatic aldehydes, carbonyl compounds with an activated methylene group, and nitromethane.

We recently found that silylation of γ-functionalized nitroalkanes proceeds in an unusual manner and yields various products, such as *N*,*N*-divinylhydroxylamines [1, 2], functionalized unsaturated oximes [3, 4], and some other derivatives [1, 5]. γ-Functionalized β-aryl-substituted primary nitro compounds (β-arylnitroalkanes) attract specific interest, for their silylation could give rise to quite unexpected cyclizations [6]. Detailed study of such reactions requires a wide series of β-arylnitroalkanes to be available, which can readily be prepared from simple starting compounds. With the above in mind, we have developed a convenient two-step procedure for synthesis of nitro compounds **IV** from accessible carbonyl derivatives and nitromethane (Scheme 1, Table 1). The first step is the well-known Knoevenagel [7] (catalyzed by CH_3COOH or $C_5H_{12}COOH$/piperidine, azeotropic distillation of water with benzene, yield 64–85%) or aldol condensation (crotonization) [8] (catalyzed by NaOH in aqueous ethanol, yield 80–95%). The second step is Michael reaction, i.e., conjugate addition of nitromethane to activated alkene **III**. This process was well studied for various nitroalkanes and Michael substrates. Usually, the reaction requires the presence of a base catalyst. Numerous examples of successful application of various catalysts were reported: Al_2O_3 and inorganic fluorides on Al_2O_3 [9], tetramethylguanidine [10], triphenylphosphine [11], tetrabutylammonium fluoride [12], 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) [13], etc. [14].

Scheme 1.



However, the main process can be accompanied by side reactions including formation of bis-adducts **V** (which is especially typical of nitromethane [15]) and elimination of HNO_2 (in the case of polyfunctionalized alkenes) [16]. Therefore, in each particular case thorough selection of the reaction conditions is necessary to ensure successful Michael addition. It should be noted that syntheses of many compounds **IV** were reported previously (except for compounds **IVb**, **IVf**, and **IVk**–**IVm**); however, each of these compounds was obtained under specific conditions

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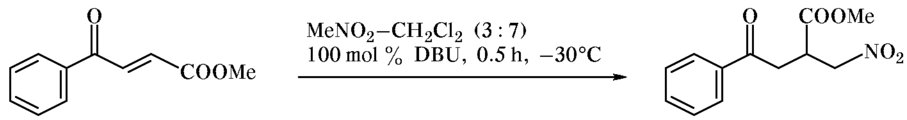
Scheme 2.



IIIo **IVo** (51%)

using a specific catalyst. We now propose a general procedure for synthesis of β-arylnitroalkanes **IVa**–**IVn** (Table 1); in all cases DBU was used as catalyst.

It is very important to keep the specified temperature conditions and amount of the catalyst (Table 1) in order to minimize bis-alkylation of nitromethane, leading to products **V**. For example, addition of nitromethane to alkene **IIIc** in the presence of 10 mol % of DBU in 4 h at 20°C results in 90% conversion of the substrate, and a mixture of products **IVc** and **Vc** is formed at a ratio of 4 : 1. The same reaction performed at –40°C in 1 h gives better results (Table 1).

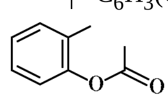
The proposed conditions for Michael addition make it possible to obtain not only β-arylnitroalkanes **IV** but also other γ-functsionalized β-substituted primary nitro compounds. In particular, we thus synthesized 1-benzoyl-2-metoxycarbonyl-3-nitropropane (**IVo**) in 51% yield (after recrystallization, Scheme 2). The result of the present study is that we have obtained a large series of γ-functionalized nitro compounds. We are now able to perform a detailed study of their silylation and utilization of this reaction in organic synthesis.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker AM-300 spectrometer (300 MHz) in $CDCl_3$; the chemical shifts were measured relative to tetramethylsilane. Dry reagents and solvents were used for Michael addition reactions; DBU, CH_2Cl_2, and $MeNO_2$ were distilled over CaH_2.

Methylene-active compounds **III** were synthesized by known methods (see references given above for compounds **IIIa**–**IIIn** and [17] for **IIIo**) and had the following melting points, °C (published data are given in parentheses): **IIIa**, 43–44 (42–43 [18]); **IIIb**, 45–50 (39–41 [19]); **IIIc**, 131–132 (133–134 [20]); **IIId**, 64–65 (60–62 [21]); **IIIe**, 54–60 (50 [22]); **IIIf**, 84–85

Table 1. Conditions of synthesis and yields of γ-functionalized β-aryl-substituted primary nitro compounds **IVa**–**IVn**[a]

Comp. no.	R^1	R^2	Ar	Temperature, °C	Time, h	Yield,[b] %
IVa	$COOCH_3$	$COOCH_3$	C_6H_5	0	2	60
IVb	$COOCH_3$	$COOCH_3$	C_6H_4Cl-4	0	0.7	57[c]
IVc	$COOCH_3$	$COOCH_3$	$C_6H_4NO_2$-4	–40	1	55
IVd	$COOCH_3$	$COOCH_3$	$C_6H_4OCH_3$-4	0	0.7	63
IVe	$COOCH_3$	$COOCH_3$	$C_6H_4CH_3$-4	0	1	63
IVf	$COOCH_3$	$COOCH_3$	$C_6H_4OCH_3$-3	0	1	43
IVg	$COOCH_3$	$COOCH_3$	$C_6H_4OCH_3$-2	0	1	47
IVh	$COOCH_3$	CN	C_6H_5	–40	1	95[c]
IVi	$C(O)C_6H_5$	H	C_6H_5	–30	1.5	89
IVj	$C(O)CH_3$	H	C_6H_5	0	2.5	53
IVk	$C(O)C_3H_5$-*cyclo*	H	$C_6H_4OCH_3$-4	20	3.7	65
IVl	$C(O)C_6H_4CH_3$-4	H	$C_6H_4OCH_3$-4	0	2	67
IVm	$C(O)C_6H_4CH_3$-4	H	$C_6H_3(OCH_3)_2$-2,4	20	1	79
IVn	$COOCH_3$			0	3	50

[a] Amounts of reactants: compound **III**, 2.5 mmol; 7 : 3 CH_2Cl_2–$MeNO_2$ mixture, 10 ml; DBU, 100 mol % with respect to **III**.
[b] After recrystallization from MeOH.
[c] Purified by flash chromatography.

Table 2. Melting points and ^{1}H NMR spectra of β-arylnitroalkanes **IVa–IVo**

Comp. no.	mp, °C	^{1}H NMR spectrum, δ, ppm
IVa	67–68 (63 [17])	3.56 s (3H, CH_3O), 3.76 s (3H, CH_3O), 3.87 d [1H, $C\mathbf{H}(COOCH_3)_2$, ^3J = 8.6 Hz], 4.25 m (1H, $C\mathbf{H}C_6H_5$), 4.87 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 13.1, ^3J = 8.2 Hz), 4.93 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 5.5 Hz), 7.20–7.36 m (5H, C_6H_5)
IVb	70–76	3.58 s (3H, CH_3O), 3.76 s (3H, CH_3O), 3.83 d [1H, $C\mathbf{H}(COOCH_3)_2$, ^3J = 8.7 Hz], 4.22 m (1H, $C\mathbf{H}C_6H_4Cl$-4), 4.83 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 13.4, ^3J = 8.7 Hz), 4.91 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 5.4 Hz), 7.18 d (2H, C_6H_4Cl-4, ^3J 8.7 Hz), 7.30 d (2H, C_6H_4Cl-4)
IVc	90–92 (91 [28])	3.61 s (3H, CH_3O), 3.79 s (3H, CH_3O), 3.89 d [1H, $C\mathbf{H}(COOCH_3)_2$, ^3J = 8.5 Hz), 4.38 m (1H, $C\mathbf{H}C_6H_4NO_2$-4), 4.92 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 13.4, ^3J = 8.6 Hz), 4.99 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 5.5 Hz), 7.46 d (2H, $C_6H_4NO_2$-4, ^3J = 8.6 Hz), 8.21 d (2H, $C_6H_4NO_2$-4)
IVd	103–104 (99 [28])	3.56 s (3H, CH_3O), 3.75 s (3H, CH_3O), 3.77 s (3H, CH_3O), 3.83 d [1H, $C\mathbf{H}(COOCH_3)_2$, ^3J = 9.4 Hz], 4.19 m (1H, $C\mathbf{H}C_6H_4OCH_3$-4), 4.82 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 13.4, ^3J = 9.4 Hz), 4.89 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 5.4 Hz), 6.83 d (2H, $C_6H_4OCH_3$-4, ^3J = 8.7 Hz), 7.14 d (2H, $C_6H_4OCH_3$-4)
IVe	77–80 (70 [28])	2.30 s (3H, CH_3), 3.57 s (3H, CH_3O), 3.75 s (3H, CH_3O), 3.85 d [1H, $C\mathbf{H}(COOCH_3)_2$, ^3J = 8.5 Hz], 4.20 m (1H, $C\mathbf{H}C_6H_4CH_3$), 4.84 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 13.1, ^3J = 8.5 Hz), 4.91 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 5.3 Hz), 7.11 br.s (4H, $C_6H_4CH_3$)
IVf	86–87	3.60 s (3H, CH_3O), 3.76 s (3H, CH_3O), 3.77 s (3H, CH_3O), 3.86 d [1H, $C\mathbf{H}(COOCH_3)_2$, ^3J = 8.8 Hz], 4.22 m (1H, $C\mathbf{H}C_6H_4OCH_3$-3), 4.86 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 13.2, ^3J = 8.8 Hz), 4.92 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 5.9 Hz), 6.74–6.85 m (3H, $C_6H_4OCH_3$-3), 7.21–7.28 m (1H, $C_6H_4OCH_3$-3)
IVg	93–95 (45 [29])	3.49 s (3H, CH_3O), 3.74 s (3H, CH_3O), 3.85 s (3H, CH_3O), 4.17 d [1H, $C\mathbf{H}(COOCH_3)_2$, ^3J = 9.6 Hz], 4.39 m (1H, $C\mathbf{H}C_6H_4OCH_3$-2), 4.87 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 13.2, ^3J = 5.1 Hz), 5.03 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 8.8 Hz), 6.84–6.89 m (2H, $C_6H_4OCH_3$-2), 7.10–7.15 m (1H, $C_6H_4OCH_3$-2), 7.21–7.27 m (1H, $C_6H_4OCH_3$-2)
IVh	Oily substance	Two diastereoisomers, 1 : 1.3: 3.65 s (3H, CH_3O, major), 3.73 s (3H, CH_3O, minor), 3.96 d [1H, $C\mathbf{H}(CH)COOCH_3$, minor, ^3J = 5.5 Hz], 4.15 d [1H, $C\mathbf{H}(CH)COOCH_3$, major, ^3J = 8.8 Hz], 4.23 m ($C\mathbf{H}C_6H_5$), 4.77–5.05 m ($C\mathbf{H}_AH_BNO_2$), 7.25–7.40 m (C_6H_5)
IVi	100–102 (98 [30])	See [30]
IVj	105–110 (99–100 [31])	2.11 s (3H, CH_3CO), 2.92 d (2H, CH_2CO), 4.01 m (1H, $C\mathbf{H}C_6H_5$), 4.59 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 12.7, ^3J = 8.1 Hz), 4.70 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 6.7 Hz), 7.19–7.37 m (5H, C_6H_5)
IVk	53–54	0.79–1.06 m (4H, *cyclo*-C_3H_5), 1.89 m (1H, CHCO), 3.00 d (2H, CH_2CO), 3.77 s (3H, CH_3O), 3.98 m (1H, $C\mathbf{H}C_6H_4OCH_3$-4), 4.56 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 12.2, ^3J = 8.1 Hz), 4.68 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 6.6 Hz), 6.85 d (2H, $C_6H_4OCH_3$-4, ^3J = 8.7 Hz), 7.14 d (2H, $S_6H_4OCH_3$-4)
IVl	60–61	2.41 s (3H, CH_3), 3.34 d.d (1H, $C\mathbf{H}_AH_9BCO$, ^2J = 17.2, ^3J = 7.1 Hz), 3.43 d.d (1H, $CH_A\mathbf{H}_BCO$, ^3J = 6.6 Hz), 3.76 s (3H, CH_3O), 4.17 m (1H, $CHC_6H_4OCH_3$-4), 4.63 d.d (1H, $C\mathbf{H}_AH_BNO_2$, ^2J = 12.4, ^3J = 8.2 Hz), 4.80 d.d (1H, $CH_A\mathbf{H}_BNO_2$, ^3J = 6.5 Hz), 6.85 d (2H, $C_6H_4OCH_3$-4, ^3J = 8.4 Hz), 7.20 d (2H, $C_6H_4OCH_3$-4), 7.25 d (2H, $C_6H_4CH_3$-4, ^3J = 8.2 Hz), 7.82 d (2H, $C_6H_4CH_3$-4)
IVm	77–78	2.42 s (3H, CH_3), 3.45 d (2H, CH_2CO), 3.78 s (3H, CH_3O), 3.85 s (3H, CH_3O), 4.31 m [1H, $C\mathbf{H}C_6H_3(OCH_3)_2$-2,4], 4.80 d (2H, CH_2NO_2), 6.38–6.49 m [2H, $C_6H_3(OCH_3)_2$-2,4], 7.08 d [1H, $C_6H_3(OCH_3)_2$-2,4, ^3J = 8.4 Hz], 7.24 d (2H, $C_6H_4CH_3$-4, ^3J = 8.3 Hz), 7.82 d (2H, $C_6H_4CH_3$-4)

Table 2. (Contd.)

Comp. no.	mp, °C	^{1}H NMR spectrum, δ, ppm
IVn	75–82	3.69 s (3H, CH_3O), 4.00 d (1H, CHCO, ^3J = 3.0 Hz), 4.31 d.d.d (1H, C**H**CH_2NO_2, ^3J = 6.6, ^3J = 8.0 Hz), 4.54 d.d (1H, C$\mathbf{H}_A H_B NO_2$, ^2J = 13.5 Hz), 4.62 d.d (1H, $CH_A\mathbf{H}_B NO_2$), 7.10–7.42 m (4H, C_6H_4)
IVo	58–60 (57 [27])	3.38 d.d (1H, C$\mathbf{H}_A H_B$CO, ^2J = 18.1, ^3J = 8.1 Hz), 3.61 d.d (1H, $CH_A\mathbf{H}_B$CO, ^3J = 4.7 Hz), 3.76 s (3H, CH_3O), 3.77 m (1H, C**H**$COOCH_3$), 4.77 d.d (1H, C$\mathbf{H}_A H_B NO_2$, ^2J = 14.8, ^3J = 5.4 Hz), 4.87 d.d (1H, $CH_A\mathbf{H}_B NO_2$, ^3J = 6.7 Hz), 7.46–7.51 m (2H, C_6H_5), 7.57–7.64 m (1H, C_6H_5), 7.93–7.97 m (2H, C_6H_5)

Table 3. Elemental analyses of β-arylnitroalkanes **IVb**, **IVf**, **IVh**, and **IVk**–**IVn**

Compound no.	Found, %			Formula	Calculated, %		
	C	H	N		C	H	N
IVb[a]	49.45	4.50	4.35	$C_{13}H_{14}ClNO_6$	49.46	4.47	4.44
IVf	54.11	5.35	4.47	$C_{14}H_{17}NO_7$	54.02	5.50	4.50
IVh	57.96	4.92	11.25	$C_{12}H_{12}N_2O_4$	58.06	4.87	11.28
IVk	63.95	6.45	5.39	$C_{14}H_{17}NO_4$	63.87	6.51	5.32
IVl	69.03	6.05	4.52	$C_{18}H_{19}NO_4$	68.99	6.11	4.47
IVm	66.52	6.21	4.05	$C_{19}H_{21}NO_5$	66.46	6.16	4.08
IVn	54.42	4.09	5.32	$C_{12}H_{11}NO_6$	54.34	4.18	5.28

[a] Found, %: Cl 11.24. Calculated, %: Cl 11.23.

(87–88 [21]); **IIIg**, 50–51 (57–59 [21]); **IIIh**, 86–88 (86 [23]); **IIIi**, 52–54 (52 [24]); **IIIj**, 39–40 (40 [25]); **IIIk**, 73–74 (70–72 [26]); **IIIl**, 98–100 (91 [24]); **IIIm**, 90–91; **IIIo**, 115–120 (111–113 [27]). Compound **IIIm**. Found, %: C 76.68; H 6.32. $C_{18}H_{18}O_3$. Calculated, %: C 76.57; H 6.43.

Synthesis of γ-functionalized β-aryl-substituted primary nitro compounds IV (*general procedure*). 1,8-Diazabicyclo[5.4.0]undec-7-ene, 0.38 ml, was added to a mixture of 2.5 mmol of compound **III** in 10 ml of 3:7 nitromethane–methylene chloride at a temperature specified in Table 1. The progress of the reaction was monitored by TLC (for reaction time, see Table 1). When the reaction was complete, the mixture was poured into a mixture of 10 ml of $CHCl_3$ and 10 ml of a solution of 0.45 g of $NaHSO_4 \cdot H_2O$. The organic phase was washed with water and with a saturated solution of NaCl, dried over $MgSO_4$, and evaporated. The residue was recrystallized from methanol or was purified by column chromatography (see notes to Table 1). The melting points, ^{1}H NMR spectra, and analytical data of compounds **IV** are given in Tables 2 and 3.

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