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Nitroalkanes as a New Source of 2-Alkylidene-1,4-diols, in Two Steps

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Abstract: A variety of 2-alkylidene-1,4-diols have been conveniently prepared, in two steps, by conjugate addition of a nitroalkane to the appropriate enedione derivatives under basic conditions (DBU), followed by chemoselective reduction ($LiAlH_4/Et_2O$) of the carbonyl functionalities of the Michael adduct, obtained after elimination of nitrous acid.

Keywords: Nitro compounds, Diols, Nitrous acid, Elimination, Michael reaction.

Diols are important intermediates in organic synthesis,[1] in particular 1,4-diols are widely used for the preparation of important heterocycles such as γ-lactones,[2] pyrroles and tetrahydrofurans,[3] Various methods have been proposed for the synthesis of diols,[3,4] however many of these suffer from drawbacks such as employment of expensive chemicals, the need for several steps, low yields and/or tedious procedures. In view of the synthetic potential of this class of compounds it is desirable to produce them efficiently, and this prompted us to search for a more convenient synthetic method.

Nitroalkanes are useful synthetic intermediates in organic synthesis because of their excellent ability to form a carbon-carbon bond.[5] Moreover, both the activating effect of the nitro group and its easy transformation to a variety of functional groups have extended the importance of nitrocompounds in the preparation of complex molecules.[6,7]

Now we wish to report the first, two step, synthesis of 1,4-diols starting from nitroalkanes. Our procedure (Scheme 1) involves the conjugate addition of the nitroalkane **1** to dimethyl maleate **2**, in acetonitrile and with DBU as base; after the elimination of nitrous acid, this yields (75-95%) the adduct **3**. Chemoselective reduction of the enone derivative **3** with $LiAlH_4$ in Et_2O, at room temperature, affords (70-91%) the 1,4-diol **4**.

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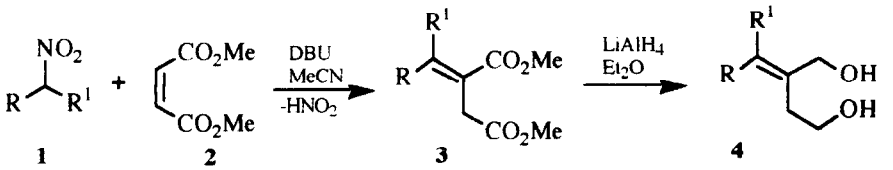
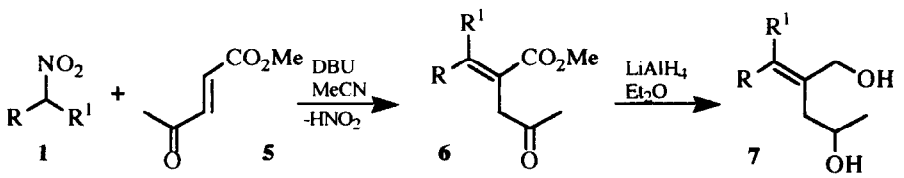


3,4	R	R^1	Yield(%)[a] of 3	Yield(%)[a] of 4
a	$CH_3(CH_2)_2$	H	94	82
b	CH_3	CH_3	95	75
c	$-(CH_2)_5-$	H	85	78
d	$(CH_3)_2CHCH_2$	H	93	80
e	$HO(CH_2)_4CH_2$	H	75	70

[a]Yield of pure isolated product.

Scheme 1

We also achieved the preparation of diols with the concomitant presence of a primary and a secondary hydroxyl group. In fact, the regiospecific addition of the nitroalkane **1** (Scheme 2) to *trans*-4-oxo-2-pentenoate **5**, with DBU/MeCN, produces (68-93%) compounds **6** which, following the reduction conditions as for the conversion of **3** to **4**, are transformed into the diols **7** in good yields (70-88%).



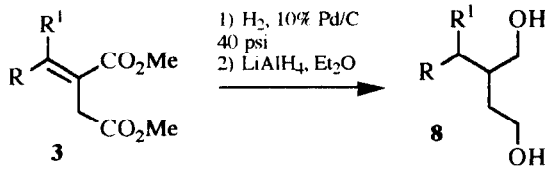
1,6,7	R	R^1	Yield(%)[a] of 6	Yield(%)[a] of 7
a	$CH_3(CH_2)_2$	H	68	75
b	CH_3	CH_3	88	70
c	$-(CH_2)_5-$	H	93	88
d	$(CH_3)_2CHCH_2$	H	87	82
e	$HO(CH_2)_4CH_2$	H	85	80

[a]Yield of pure isolated product.

Scheme 2

The conjugate addition of nitroalkane **1** to the enedione **2** (Scheme 1) or **4** (Scheme 2), affords the *E*-isomer as the predominant form (up to 93%, determined by NMR analysis of the crude reaction mixture). In fact, it is well documented that a β-alkyl substituent *syn* to a carbonyl moiety in α,β-conjugated enones resonates downfield relative to the *anti* alkyl substituent in the NMR spectrum.[8] Consequently, the NMR chemical shifts provide a reliable guide for the assignment of the olefin configuration.

We also examined the possibility of converting the enones **3** into saturated diols, and we found that hydrogenation (10% Pd/C as catalyst) of the C-C double bond (Scheme 3), followed by treatment with $LiAlH_4$ in Et_2O, produces the 2-alkylated-1,4-diols **8** in good yields (70-91%).



8	R	R^1	Yield(%)[a] of **8**
a	$CH_3(CH_2)_2$	H	70
b	CH_3	CH_3	75
c	$-(CH_2)_5-$	H	91
d	$(CH_3)_2CHCH_2$	H	87
e	$HO(CH_2)_4CH_2$	H	90

[a]Yield of pure isolated product.

Scheme 3

With our procedure, which requires inexpensive chemicals and mild reaction conditions, both primary and secondary nitroalkanes can be successfully employed, so that by the appropriate choice of the starting nitrocompounds many different 2-alkylated 1,4-diols can be prepared in only two steps. It is important to point out that the concomitant presence of an allylic and a homoallylic hydroxyl (compounds **4** and **7**) and their different reactivity offers the opportunity to carry out the selective conversion of both the alcohol functionalities. Moreover, because even hydroxylated nitroalkanes can be used, one more alcohol functionality can be introduced, then, trihydroxylated derivatives (**4e**, **7e** and **8e**) are also accessible.

Experimental

General. All ^1H NMR spectra were recorded in $CDCl_3$ at 200 MHz on a Varian Gemini instrument; *J* values are given in Hz. IR spectra were recorded with a Perkin Elmer 257 spectrophotometer. The reactions were monitored by TLC or GC performed on a Carlo Erba Fractovap 4160 using a capillary column of Duran Glass, stationary phase OV1. Microanalyses were performed using a Fisons model EA 1108. The products were

purified by flash chromatography on Merck silica gel with EtOAc/cyclohexane as eluent. Nitroalkanes **1a-c**, dimethyl maleate **2** and *trans*-4-oxo-2-pentenoate **5** are commercially available, while the nitrocompounds **1d**[9] and **1e**[10] were obtained as previously reported.[9,10]

Conjugate Addition of Nitroalkanes (1) to the Enediones (2 and 5). DBU (3.04 g, 20 mmol) was added at room temperature to a solution of nitroalkane **1** (20 mmol) and the enedione **2** or **5** (20 mmol) in CH_3CN (100 ml). The solution was stirred for 7 hours at room temperature, then silica gel (Merck 0.04-0.063 mm, 5-6 g) was added and the solution was evaporated. The residue was flash chromatographed, using a suitable ratio of EtOAc/cyclohexane as eluent, affording the pure compounds **3** and **6**.

(**3a**): Oil. IR (film): ν = 1715 and 1654 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.95 (t, 3H, *J* = 7.3Hz), 1.4-1.6 (m, 2H), 2.1-2.2 (m, 2H), 3.35 (s, 2H), 3.7 (s, 3H), 3.75 (s, 3H), 6.95 (t, 1H, *J* = 7.3Hz). Anal. Calcd. for $C_{10}H_{16}O_4$: C, 59.98; H, 8.05. Found: C, 60.11; H, 7.98.

(**3b**): Oil. IR (film): ν = 1730 and 1640 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.87 (s, 3H), 1.92 (s, 3H), 3.22 (s, 2H), 3.63 (s, 3H), 3.7 (s, 3H). Anal. Calcd. for $C_9H_{14}O_4$: C, 58.05; H, 7.58. Found: C, 57.95; H, 8.06.

(**3c**): Oil. IR (film): ν = 1740 and 1636 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.43-1.66 (m, 6H), 2.1 (s, 3H), 2.59-2.71 (m, 4H), 3.43 (s, 2H), 3.78 (s, 3H). Anal. Calcd. for $C_{12}H_{18}O_4$: C, 63.70; H, 8.02. Found: C, 63.83; H, 7.91.

(**3d**): Oil. IR (film): ν = 1720 and 1638 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.95 (d, 6H, *J* = 6.9Hz), 1.6-1.7 (m, 1H), 2.05-2.1 (m, 2H), 3.42 (s, 2H), 3.65 (s, 3H), 3.69 (s, 3H), 6.35 (t, 1H, *J* = 7.2Hz). Anal. Calcd. for $C_{11}H_{18}O_4$: C, 61.66; H, 8.47. Found: C, 61.83; H, 8.38.

(**3e**): Oil. IR (film): ν = 3409, 1714 and 1631 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.36-1.42 (m, 6H), 2.21-2.35 (m, 2H), 3.45 (s, 2H), 3.62 (t, 2H, *J* = 6.5Hz), 3.66 (s, 3H), 3.76 (s, 3H), 6.65 (t, 1H, *J* = 7.0Hz). Anal. Calcd. for $C_{12}H_{20}O_5$: C, 59.00; H, 8.25. Found: C, 59.14; H, 8.13.

(**6a**): Oil. IR (film): ν = 1718 and 1654 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.95 (t, 3H, *J* = 7.2Hz), 1.4-1.55 (m, 2H), 2.1-2.18 (m, 2H), 2.2 (s, 3H), 3.45 (s, 2H), 3.75 (s, 3H), 7.00 (t, 1H, *J* = 7.0Hz). Anal. Calcd. for $C_{10}H_{16}O_3$: C, 65.19; H, 8.75. Found: C, 65.08; H, 8.88.

(**6b**): Oil. IR (film): ν = 1717 and 1638 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.8 (s, 3H), 2.12 (s, 3H), 2.15 (s, 3H), 3.45 (s, 2H), 3.7 (s, 3H). Anal. Calcd. for $C_9H_{14}O_3$: C, 63.15; H, 8.29. Found: C, 63.23; H, 8.34.

(**6c**): Oil. IR (film): ν = 1731 and 1625 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.43-1.66 (m, 6H), 2.1 (s, 3H), 2.59-2.71 (m, 4H), 3.43 (s, 2H), 3.78 (s, 3H). Anal. Calcd. for $C_{12}H_{18}O_3$: C, 68.55; H, 8.63. Found: C, 68.65; H, 8.67.

(**6d**): Oil. IR (film): ν = 1715 and 1654 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.91 (d, 6H, *J* = 6.3Hz), 1.55-1.62 (m, 1H), 1.9-1.95 (m, 2H), 2.12 (s, 3H), 3.42 (s, 2H), 3.69 (s, 3H), 6.41 (t, 1H, *J* = 7.1Hz). Anal. Calcd. for $C_{11}H_{18}O_3$: C, 66.64; H, 9.15. Found: C, 66.51; H, 9.08.

(**6e**): Waxy solid. IR (film): ν = 3350, 1728 and 1641 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.3-1.45 (m, 6H), 2.09 (s, 3H), 2.16-2.24 (m, 2H), 3.43 (s, 2H), 3.64 (t, 2H, *J* = 6.5Hz), 3.73 (s, 3H), 6.54 (t, 1H, *J* = 7.0Hz). Anal. Calcd. for $C_{12}H_{20}O_4$: C, 63.13; H, 8.83. Found: C, 63.04; H, 8.94.

Reduction of the Enones (3) and (6) to 2-Alkylidene-1,4-Diols (4) and (7). To a stirred solution of lithium aluminium hydride (0.68 g, 16 mmol) in dry Et_2O (60 ml), under nitrogen, the appropriate enone **3** or **6** (4 mmol) in dry Et_2O (20 mmol) was added dropwise. The mixture was stirred at room temperature for 5-

11 h (TLC and/or GC), then diluted with water (50 ml), acidified with 2 N HCl to pH = 2-3, and extracted with EtOAc (3 x 30 ml). The organic layer was dried ($MgSO_4$), the solvent removed by distillation, and the crude product was purified by flash chromatography (EtOAc/cyclohexane), affording the pure diol **4** or **7**.

(**4a**): Oil. IR (film): ν = 3326 and 1663 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.9 (t, 3H, *J* = 7.3Hz), 1.3-1.5 (m, 2H), 1.95-2.1 (m, 2H), 2.4 (t, 2H, *J* = 5.9Hz), 3.7 (t, 2H, *J* = 5.9Hz), 4.0 (s, 2H), 5.55 (t, 1H, *J* = 7.3Hz). Anal. Calcd. for $C_8H_{16}O_2$: C, 66.63; H, 11.18. Found: C, 66.54; H, 11.21.

(**4b**): Oil. IR (film): ν = 3339 and 1640 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.7 (s, 3H), 1.8 (s, 3H), 2.45 (t, 2H, *J* = 5.8Hz), 3.7 (t, 2H, *J* =5.8Hz), 4.15 (s, 2H). Anal. Calcd. for $C_7H_{14}O_2$: C, 64.58; H, 10.84. Found: C, 64.67; H, 10.77.

(**4c**): Oil. IR (film): ν = 3306 and 1651 cm^{-1}; ^1H NMR ($CDCl_3$): δ =1.5-1.6 (m, 6H), 2.15-2.3 (m, 4H), 2.45 (t, 2H, *J* = 5.9Hz), 3.7 (t, 2H, *J* = 5.9Hz), 4.15 (s, 2H). Anal. Calcd. for $C_{10}H_{18}O_2$: C, 70.55; H, 10.66. Found: C, 70.46; H, 10.72.

(**4d**): Oil. IR (film): ν = 3320 and 1653 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.91 (d, 6H, *J* = 5.3Hz), 1.2-1.45 (m, 1H), 1.8-1.9 (m, 2H), 2.45 (t, 2H, *J* = 6.0Hz), 3.67 (t, 2H, J = 6.0Hz), 4.0-4.05 (m, 2H), 5.35 (t, 1H, *J* = 7.0Hz). Anal. Calcd. for $C_9H_{18}O_2$: C,68.31; H, 11.46. Found: C, 68.44; H, 11.51.

(**4e**): Waxy solid. IR (film): ν = 3386 and 1651 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.3-1.65 (m, 6H), 2.05-2.15 (m, 2H), 2.43 (t, 2H, *J* = 7.3Hz), 3.6-3.75 (m, 4H), 4.05 (s, 2H), 5.55 (t, 1H, *J* = 7.2Hz). Anal. Calcd. for $C_{10}H_{20}O_3$: C, 63.80; H, 10.71. Found: C, 63.96; H, 10.82.

(**7a**): Oil. IR (film): ν = 3326 and 1659 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.9 (t, 3H, *J* = 7.3Hz), 1.23 (d, 3H, *J* = 6.4Hz), 1.3-1.5 (m, 2H), 1.95-2.25 (m, 4H), 3.85-3.95 (m, 1H), 4.05 (s, 2H), 5.55 (t, 1H, *J* = 7.3Hz). Anal. Calcd. for $C_9H_{18}O_2$: C, 68;31; H, 11.46. Found: C, 68;23; H, 11.56.

(**7b**): Oil. IR (film): ν = 3306 and 1660 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.23 (d, 3H, *J* = 6.4Hz), 1.7 (s, 3H), 1.8 (s, 3H), 2.1-2.2 (m, 2H), 3.85-4.0 (m, 1H), 4.35 (s, 2H). Anal. Calcd. for $C_8H_{16}O_2$: C, 66.63; H, 11.18. Found: C, 66.50; H, 11.27.

(**7c**): Oil. IR (film): ν = 3326 and 1663 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.23 (d, 3H, *J* = 6.3Hz), 1.5-1.6 (m, 6H), 2.1-2.4 (m, 6H), 3.8-3.95 (m, 1H). Anal. Calcd. for $C_{11}H_{22}O_2$: C, 71.69; H, 10.94. Found: C, 71.77; H, 11.08.

(**7d**): Oil. IR (film): ν = 3330 and 1655 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.9 (d, 6H, *J* = 6.5Hz), 1.21 (d, 3H, *J* = 6.3Hz), 1.3-1.4 (m, 1H), 1.8-1.9 (m, 2H), 3.8-3.9 (m, 1H), 4.0-4.05 (m, 1H), 5.11 (t, 1H, *J* = 7.1Hz). Anal. Calcd. for $C_{10}H_{20}O_2$: C, 69.72; H, 11.70. Found: C, 69.83; H, 11.78.

(**7e**): Waxy solid. IR (film): ν = 3401 and 1665 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.2-1.3 (m, 5H), 1.3-1.6 (m, 4H), 2.0-2.2 (m, 2H), 2.4 (dd, 2H, *J* = 8.7 and 15.4 Hz), 3.6-3.8 (m, 2H), 3.85-4.0 (m, 1H), 4.05 (s, 2H), 5.55 (t, 1H, *J* = 7.1Hz). Anal. Calcd. for $C_{11}H_{22}O_3$: C, 65.31; H, 10.96. Found: C, 65.20; H, 11.05.

Conversion of the Enones (3) to Saturated 1,4-Diols (8). The enone **3** (16 mmol) was dissolved in EtOAc (150 ml) and 10% Pd/C (0.3 g) was added. The suspension was hydrogenated (40 psi) at room temperature for 5 hours. The catalyst was removed by filtration through celite pad and washed with EtOAc (3 x 15 ml). After evaporation of the solvent the crude product was dissolved in dry Et_2O (20 ml) and added, dropwise (under nitrogen), to a stirred solution of lithium aluminum hydride (0.68 g, 16 mmol) in dry Et_2O (60 ml). The mixture was stirred at room temperature for 6-12 h (GC), then diluted with water (50 ml), acidified with 2 N HCl to pH = 2-3, and extracted with EtOAc (3 x 30 ml). The organic layer was dried

($MgSO_4$), the solvent removed by distillation, and the crude product was purified by flash chromatography (EtOAc/cyclohexane), affording the pure diol **8**.

(**8a**): IR (film): $\nu = 3338\ cm^{-1}$; ^1H NMR ($CDCl_3$): $\delta = 0.9$ (t, 3H, $J = 6.6Hz$), 1.2-1.35 (m, 6H), 1.5-1.75 (m, 3H), 3.35-3.8 (m, 4H). Anal. Calcd. for $C_8H_{18}O_2$: C, 65.71; H, 12.41. Found: C, 65.83; H, 12.50.

(**8b**): IR (film): $\nu = 3340\ cm^{-1}$; ^1H NMR ($CDCl_3$): $\delta = 0.8$ (d, 3H, $J = 7.0Hz$), 0.9 (d, 3H, $J = 7.0Hz$), 1.3-1.65 (m, 4H), 3.45 (dd, 1H, $J = 8.9$ and 10.3 Hz), 3.65 (dd, 1H, $J = 4.5$ and 10.3Hz), 3.68 (t, 2H, $J = 6.5Hz$). Anal. Calcd. for $C_7H_{16}O_2$: C, 63.60; H, 12.20. Found: C, 63.53; H, 12.31.

(**8c**): IR (film): $\nu = 3323\ cm^{-1}$; ^1H NMR ($CDCl_3$): $\delta = 1.0$-1.8 (m, 14H), 3.55-3.65 (m, 2H), 3.7 (t, 2H, $J = 6.5Hz$). Anal. Calcd. for $C_{10}H_{20}O_2$: C, 69.72; H, 11.70. Found: C, 69.81; H, 11.60.

(**8d**): IR (film): $\nu = 3340\ cm^{-1}$; ^1H NMR ($CDCl_3$): $\delta = 0.8$ (d, 3H, $J = 7.0Hz$), 0.9 (d, 3H, $J = 7.0Hz$), 1.3-1.65 (m, 4H), 3.45 (dd, 1H, $J = 8.9$ and 10.3 Hz), 3.65 (dd, 1H, $J = 4.5$ and 10.3Hz), 3.68 (t, 2H, $J = 6.5Hz$). Anal. Calcd. for $C_9H_{20}O_2$: C, 67.45; H, 12.58. Found: C, 67.53; H, 12.71.

(**8e**): IR (film): $\nu = 3350\ cm^{-1}$; ^1H NMR ($CDCl_3$): $\delta = 1.25$-2.2 (m, 13H), 3.6-3.8 (m, 4H), 4.15 (t, 2H, $J = 6.9Hz$). Anal. Calcd. for $C_{10}H_{22}O_3$: C, 63.12; H, 11.65. Found: C, 63.05; H, 11.74.

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