DNDMH-Mediated Direct Nitration of Aryl Alkenes

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TABLE OF CONTENTS

1. General informationS	3
2. Explorations on optimization of the direct nitration of 6a with DNDMH	4
3. General procedure for preparation alkenes from corresponding aldehydes o	r
ketonesS	7
4. General procedure for the direct nitration of aryl alkenes 6 with DNDMH	0
5. Copies of NMR specture	5
References	5

1. General information

For Solvent and Reagent: Anhydrous tetrahydrofuran (THF) was dried and distilled from sodium and benzophenone. Other solvents and reagents were purchased commercially and used without further purification.

For Reaction Operation: Reactions were generally performed in round-bottom flask unless other specific illuminations, and monitored by thin-layer chromatography (TLC, 254 nm silica gel 60-F plates) with fluorescence upon 254 nm irradiation, potassium permanganate (KMnO4) stain, and phosphomolybdic acid (PMA) stain. Flash chromatographies were applied for the purification of reaction products with silica gel 200-300 mesh.

For NMR Spectroscopy: All NMR spectra were obtained at ambient temperature using Bruker AVANCE III-400MHz, JEOL-ZETA 400MHz or JEOL-ZETA 600MHz spectrometers. ¹H NMR and ¹³C NMR spectra were recorded with CDCl₃ unless other specific illuminations. Spectra were referenced internally to the residual proton resonance of CDCl₃ (δ 7.26 ppm ¹H NMR, δ 77.16 ppm ¹³C NMR) with tetramethylsilane (TMS, δ 0.00 ppm) as the standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ¹H NMR data were recorded as follows: multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, septet or unresolved), coupling constant (Hz), integration. ¹³C NMR spectra were recorded with complete ¹H decoupling.

For Mass Spectrometry: High resolution mass spectrometry (HRMS) data were obtained on a MicrOTOF-Q II (hybrid quadrupolar/time-of-flight) API US system by electrospray ionization (ESI) in the positive or negative ion mode from Bruker Corporation.

For IR Spectroscopy: Infrared spectra were recorded on an INVENIO spectrometer from Bruker Corporation as thin films. Absorptions are given in wavenumbers (cm⁻¹).

2. Explorations on optimization of the direct nitration of 6a with

DNDMH

2.1 Examinations of other Lewis Acids in direct nitration of 6a with DNDMH

Table S1: Examinations different Lewis/Acid in direct nitration of 6a with DNDMH

	i) 2.5 eq DNDMH ii) 0.5 eq LewisAcid	NO ₂
6a	iii) 3mL HFIP iv) 60 °C, 6h	7a
Entry	LewisAcid	Yield/%
1	Mg(ClO ₄) ₂	trace
2	Cu(OTf) ₂	3%
3	Sn(OTf) ₂	No reaction
4	Ni(OTf) ₂	trace
5	Zn(OTf) ₂	trace
6	Yb(OTf) ₂	No reaction
7	AgOTf	No reaction
8	Sc(OTf) ₃	No reaction

2.2 Examinations of DNDMH mediated direct nitration of **6a** with TEMPO in various solvent

 Table S2: Examinations of DNDMH mediated direct nitration of 6a with TEMPO in various solvent

	i) 2.eq DNDMH ii) 0.5eq TEMPO	NO ₂
63	iii) 3ml solvent, 80 °C, 6h	7a
Entry	solvent	Yield/%
1	DCE	40
2	HFIP	No reaction
3	Acetone	No reaction
4	THF	trace
5	MTBE	38
6	CCl ₄	31
7	1,4-Dioxane	No reaction
8	EtOAc	trace
9	MeNO ₂	No reaction
10	MeCN	trace
	S4	

11	DMF	No reaction
12	TFE	No reaction

2.3 Optimization of the amount of Cu(OAc)₂ in direct nitration of 6a with DNDMHTable S3: Optimization of the amount of Cu(OAc)₂ in direct nitration of 6a with DNDMH

	i) 2.5 eq DNDMH ii) 0.5 eq TEMPO	NO ₂
6a	iii) x eq Cu(OAc)₂ iv) 3mL DCE, 80 °C, 6h	7a
Entry	Cu(OAc) ₂	Yield/%
1	0.7 eq	73
2	1 eq	84
3	13 eq	81
4	1.7 eq	94
5	2 eq	84

2.4 Optimization of the amount of TEMPO in direct nitration of 6a with DNDMHTable S4: Optimization of the amount of TEMPO in direct nitration of 6a with DNDMH

	i) 2.5 eq DNDMH ii) x eq TEMPO	NO ₂
6a	iii) 1.7 eq Cu(OAc) ₂ iv) 3mL DCE, 80 °C, 6h	7a
Entry	TEMPO	Yield/%
1	0.1 eq	46
2	0.2 eq	96
3	0.3 eq	87
4	0.5 eq	94
5	0.7 eq	81
6	1 eq	78

2.5 Optimization of the amount of DNDMH in direct nitration of 6a with DNDMHTable S5: Optimization of the amount of DNDMH in direct nitration of 6a with DNDMH

	i) x eq DNDMH ii) 0.2 eq TEMPO	NO ₂
6a	iii) 1.7 eq Cu(OAc) ₂ iv) 3mL DCE, 80 °C, 6h	7a
Entry	DNDMH	Yield/%
1	1.1 eq	73
2	1.5 eq	77
3	2 eq	93
4	2.5 eq	96
5	3.5 eq	90
6	4.5 eq	91

2.6 Examinations of direct nitration of alkenes with alkyl substitueuts

 Table S6: Examinations of direct nitration of alkenes with alkyl substituents (no

 expected product observed in every case)



3. General procedure for preparation alkenes from corresponding



aldehydes or ketones

3.1. Substrates 6a, 6b, 6c, 6d, 6e, 6f, 6g, 6h, 6i, 6k, 6l, 6m, 6n, 6o, 6p, 6q, 6r, 6s, 6t, 6u, 6y, 6aa, 6ab, 6ac are commercially available.

3.2. Substrates **6v**, **6x**, **6z**, **6ad**, **6af**, **6ag** are known compounds and prepared according to the general procedure as below.

To a stirred suspension of (methyl)triphenylphosphonium bromine (4.57 mmol) in anhydrous THF, potassium tert-butoxide (3.96 mmol) was added under argon at 0 °C and stirred for 2 h. Then the solution of aromatic aldehyde or ketone (3.05 mmol) in anhydrous THF was added slowly and stirred for 30 minute. The mixture was mixed with water and the aqueous layer was extracted with ethyl acetate (3 x 15 mL). The combined organic extracts were dried over anhydrous Na₂SO₄, concentrated and purified by silica gel column chromatography using petroleum ether and ethyl acetate as the diluent to afford expected product.

Characteristic Data of Synthesized 6:

1-Methoxy-2-(1-methylethenyl)benzene (6v)



¹**H NMR** (400 MHz, CDCl₃): δ 7.31 – 7.21 (m, 2H), 7.00 – 6.89 (m, 2H), 5.25 – 5.06 (m, 2H), 3.87 (s, 3H), 2.19 – 2.14 (m, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 156.4, 144.1, 132.6, 129.2, 128.1, 120.3, 114.9,

110.6, 55.2, 23.0 ppm.

6v is a known compound.^[S1]

2-Bromo-5-ethenylthiophene (6x)



¹**H NMR** (400 MHz, CDCl₃): δ 6.90 (d, J = 3.7 Hz, 1H), 6.78 – 6.63 (m, 2H), 5.46 (d,

J = 17.2 Hz, 1H), 5.13 (d, *J* = 11.0 Hz, 1H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 144.8, 130.3, 129.5, 126.2, 113.9, 111.4 ppm.

6x is a known compound.^[S2]

5-Ethenyl-2,3-dihydrobenzofuran (6z)



¹**H NMR** (400 MHz, CDCl₃): δ 7.30 (d, *J* = 1.9 Hz, 1H), 7.16 (dd, *J* = 8.2, 1.8 Hz, 1H), 6.77 – 6.61 (m, 2H), 5.58 (dt, *J* = 17.6, 1.0 Hz, 1H), 5.13 – 5.05 (m, 1H), 4.58 (t, *J* = 8.7 Hz, 2H), 3.20 (t, *J* = 8.7 Hz, 2H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 160.1, 136.7, 130.7, 127.5, 126.8, 122.5, 111.0, 109.3, 71.6, 29.7 ppm.

6z is a known compound.^[S3]

5-(1-Methylethenyl)-1,3-benzodioxole (6ad)



¹H NMR (400 MHz, CDCl₃): δ 7.02 – 6.92 (m, 2H), 6.78 (d, J = 8.1 Hz, 1H), 5.95 (s, 2H), 5.26 (dd, J = 1.6, 0.8 Hz, 1H), 5.00 (t, J = 1.5 Hz, 1H), 2.17 – 2.08 (m, 3H) ppm;
¹³C NMR (100 MHz, CDCl₃): δ 147.6, 146.8, 142.5, 135.4, 118.9, 111.1, 107.7, 105.8, 100.8, 21.9 ppm.

6ad is a known compound.^[S4]

2-Chloro-1-ethenyl-3,4-dimethoxybenzene (6af)



¹**H NMR** (400 MHz, CDCl₃): δ 7.29 (d, *J* = 8.7 Hz, 1H), 7.03 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.82 (d, *J* = 8.7 Hz, 1H), 5.62 (dd, *J* = 17.5, 1.2 Hz, 1H), 5.27 (dd, *J* = 10.9, 1.2 Hz, 1H), 3.87 (d, *J* = 7.0 Hz, 6H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 153.3, 145.5, 133.1, 129.6, 128.0, 121.5, 114.9,

110.8, 60.7, 56.2 ppm.

6af is a known compound.^[S5]

1-Ethenyl-4-methoxy-2-methylbenzene (6ag)



¹**H NMR** (400 MHz, CDCl₃): δ 7.44 (d, *J* = 8.5 Hz, 1H), 6.89 (dd, *J* = 17.4, 11.0 Hz, 1H), 6.79 – 6.69 (m, 2H), 5.55 (dd, *J* = 17.5, 1.5 Hz, 1H), 5.19 (dd, *J* = 11.0, 1.5 Hz, 1H), 3.81 (s, 3H), 2.35 (s, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 158.9, 136.7, 134.0, 129.4, 126.3, 115.2, 112.9, 111.4, 55.0, 19.7 ppm.

6ag is a known compound.^[S6]

3.3 Substrate 6j was prepared as following

tert-Butyl (4-vinylphenyl)carbamate (6j)



4-Vinylaniline (1.0 equiv., 12.8 mmol, 1.5 mL) was added to a solution of di-tert-butyl dicarbonate (1.1 equiv., 14 mmol, 3.06 g) and DMAP (0.01 eq, 0.13 mmol, 15.67 mg) in MeCN (16 mL). The reaction mixture was stirred for 23 h at ambient temperature. After that time ethyl acetate (30 mL) was added and the layers separated. The aqueous layer was extracted with ethyl acetate (3x10 mL). The combined organic layers were washed with brine and dried over anhydrous Na₂SO₄. After filtration the solvent was removed under reduced pressure. The crude product was purified by column chromatography through a silica gel column using petroleum ether/EtOAc (10:1) as eluent to afford **6j** (12 mmol, 2,70 g, 97%) as a white solid.

¹H NMR (400 MHz, CDCl₃): δ 7.33 (d, J = 2.6 Hz, 4H), 6.65 (dd, J = 17.6, 10.9 Hz, 1H), 6.48 (s, 1H), 5.65 (dd, J = 17.6, 0.9 Hz, 1H), 5.16 (dd, J = 10.8, 0.9 Hz, 1H), 1.52 (s, 9H) ppm;
¹³C NMR (100 MHz, CDCl₃): δ 152.77, 138.06, 136.33, 132.65, 126.99, 118.54, 112.53, 28.47 ppm.
6j is a known compound.^[S7]

4. General procedure for the direct nitration of aryl alkenes 6 with **DNDMH**

Ar
$$H$$
 $\frac{0.2 \text{ eq. TEMPO, } 1.7 \text{ eq. Cu}(OAc)_2}{2.5 \text{ eq. DNDMH (4), DCE, } 80 ^{\circ}\text{C}}$ Ar 7

To an oven-dried reaction glass tube charged with a magnetic stir-bar was added alkenes 5 (0.48 mmol), TEMPO (0.096 mmol), DNDMH (0.96 mmol) and Cu(OAc)₂ (0.82 mmol). Under nitrogen atmosphere, 2 mL DCE was added with a laboratory syringe. The mixture was stirred at 80 °C into an oil bath for 6 h. The reaction was monitored by TLC analysis. Until the completion the solvent was evaporated under reduced pressure. The residue was purified with silica gel column chromatography with petroleum ether/EtOAc as the eluent.

Characteristic Data of Synthesized Nitralknes 7:

(E)-(2-nitrovinyl)benzene (7a)



Yield of **7a** 96%, 74 mg; yellow solid; **mp** 57-58 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): 7.44 – 7.48 (m, 2H), 7.49 – 7.52 (m, 1H), 7.54 – 7.57 (m, 2H), 7.57 – 7.61 (dd, *J* = 13.7, 1.1 Hz, 1H), 7.99 – 8.02 (dd, *J* = 13.7, 1.8 Hz, 1H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ139.2, 137.2, 132.3, 130.2, 129.5, 129.3 ppm;

HRMS (ESI) m/z: [M+Na]⁺ calculated for C₈H₇NNaO₂⁺ 172.0369, found 172.0317;

IR (film) vmax = 3332, 2953, 2917, 2850, 1736, 1694, 1595, 1457, 1376, 1262, 1044, 1020, 890, 875, 807, 770, 742.

7a is a known compound.^[S8]

(E)-1-fluoro-4-(2-nitrovinyl)benzene (7b)



Yield of **7b** 80%, 50 mg; yellow solid; **mp** 102-103 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.98 (d, *J* = 13.7 Hz, 1H), 7.60 – 7.50 (m, 3H), 7.15 (t, *J* = 8.5 Hz, 2H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 165.4 (d, J = 177.52 Hz), 138.0, 137.0, 131.4 (d, J = 9.0 Hz), 126.5, 116.9 (d, J = 22.3 Hz) ppm;

HRMS (ESI) m/z: [M+Na]⁺ calculated for C₈H₆FNNaO₂⁺ 190.0274, found 190.0274; **IR** (film) vmax = 3116, 2964, 2919, 2851, 1638, 1593, 1503, 1345, 1232, 1170, 978, 882.

7b is a known compound.^[S8]

(E)-1-chloro-4-(2-nitrovinyl)benzene (7c)



Yield of **7c** 64%, 42 mg; yellow solid; **mp** 113-114 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.95 (dd, *J* = 13.7, 7.1 Hz, 1H), 7.64 – 7.38 (m, 5H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 137.9, 137.6, 132.9, 130.5, 130.4, 129.9 ppm;

IR (film) vmax = 3365, 3184, 2925, 2845, 2361, 1723, 1638, 1458, 1424, 1368, 1086,

1018, 956, 810, 724, 640.

7c is a known compound.^[S8]

(E)-1-bromo-4-(2-nitrovinyl)benzene (7d)



Yield of **7d** 48%, 30 mg; yellow solid; **mp** 141-144 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.94 (d, J = 13.7 Hz, 1H), 7.60 – 7.56 (m, 3H), 7.42 –

7.40 (m, 2H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 137.9, 137.6, 132.9, 130.5, 129.1, 126.9 ppm;

IR (film) vmax = 3743, 3111, 2919, 2857, 2366, 2343, 1633, 1520, 1351, 1250, 1187, 1080, 968, 815, 668.

7**d** is a known compound.^[S9]

(E)-1-methyl-4-(2-nitrovinyl)benzene (7e)



Yield of **7e** 98%, 42 mg; yellow solid; **mp** 106-107 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.97 (d, *J* = 13.6 Hz, 1H), 7.56 (d, *J* = 13.7 Hz, 1H), 7.45 – 7.43 (m, 2H), 7.24 (s, 1H), 2.40 (s, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 143.2, 139.3, 136.3, 130.2, 129.3, 127.3, 21.7 ppm; IR (film) vmax = 3111, 2925, 2863, 1644, 1599, 1520, 1503, 1424, 1340, 1272, 1182, 973, 815, 730.

7e is a known compound.^[S8]

(E)-(2-nitrovinyl)benzene (7f)



Yield of **7f** 92%, 62 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H** NMR (400 MHz, CDCl₃): δ 8.00 (d, J = 13.6 Hz, 1H), 7.58 (d, J = 13.7 Hz, 1H),

7.51 – 7.46 (m, 4H), 1.34 (s, 9H) ppm;

¹³**C NMR** (100 MHz, CDCl₃): δ 156.3, 139.2, 136.6, 129.2, 127.4, 126.6, 35.3, 31.2 ppm;

HRMS (ESI) m/z: [M+Na]⁺ calculated for C₁₂H₁₅NNaO₂⁺ 228.0995, found 228.0989; **IR** (film) vmax = 3105, 2969, 2930, 2372, 2338, 1644, 1526, 1345, 1278, 1182, 1114, 973, 820, 674.

7f is a known compound.^[S10]

(E)-1-(chloromethyl)-4-(2-nitrovinyl)benzene (7g)



Yield of **7g** 94%, 66 mg; yellow solid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.00 (d, J = 13.7 Hz, 1H), 7.60 – 7.54 (m, 3H), 7.48

(d, *J* = 8.2 Hz, 2H), 4.61 (s, 2H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 142.0, 138.4, 137.6, 130.2, 129.7, 129.6, 45.4 ppm;

HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{10}H_5ClNNaO_2^+$ 198.0316, found 198.0355;

IR (film) vmax = 3122, 3049, 2969, 2930, 2845, 2372, 1633, 1526, 1509, 1413, 1345,

1272, 1204, 973, 820, 798, 730, 674.

7g is a known compound.^[S9]

(E)-4-(2-nitrovinyl)-1,1'-biphenyl (7h)



Yield of **7h** 82%, 70 mg; yellow solid; **mp** 190-192 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.05 (d, *J* = 13.6 Hz, 1H), 7.70 – 7.61 (m, 7H), 7.50 – 7.39 (m, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 145.1, 139.6, 138.9, 137.0, 129.8, 129.2, 129.0, 128.5, 128.1, 127.2 ppm;

IR (film) vmax = 3404, 3179, 3111, 2964, 2919, 2857, 1633, 1605, 1560, 1498, 1407, 1340, 1255, 1187, 978, 826, 770, 730, 691.

7h is a known compound.^[S10]

(E)-1-(2-nitrovinyl)-4-(trifluoromethyl)benzene (7i)



Yield of 7i 92%, 58 mg; yellow solid; **mp** 90-92 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.02 (d, *J* = 13.8 Hz, 1H), 7.70 (q, *J* = 8.4 Hz, 4H), 7.63 (d, *J* = 13.7 Hz, 1H) ppm;

¹³**C NMR** (100 MHz, CDCl₃): δ 139.0, 137.3, 133.8-133 (q, *J* = 32.76, 30.24 Hz), 129.4, 126.31-126.31 (q, *J* = 16.38, 3.78 Hz), 124.6, 122.4 ppm;

IR (film) vmax = 3116, 2925, 2845, 1661, 1526, 1413, 1340, 1317, 1170, 1120, 1074, 968, 838, 748.

7i is a known compound.^[S11]

tert-butyl (E)-(4-(2-nitrovinyl)phenyl)carbamate (7j)



Yield of **7j** 83%, 50 mg; yellow solid; **mp** 143-145 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.93 (d, *J* = 13.6 Hz, 1H), 7.51 (d, *J* = 13.6 Hz, 1H), 7.45 (s, 4H), 1.50 (s, 9H) ppm;

¹³**C NMR** (100 MHz, CDCl₃): δ 152.3, 142.4, 139.0, 135.6, 130.6, 124.4, 118.6, 81.6, 28.4 ppm;

IR (film) vmax = 3337, 2925, 2851, 2366, 1734, 1599, 1531, 1424, 1345, 1317, 1232, 1154, 1052, 978, 820, 776.

methyl (E)-4-(2-nitrovinyl)benzoate (7k)



Yield of **7k** 94%, 60 mg; yellow solid; **mp** 176-178 °C; $R_f = 0.5$ (silica, PE:EtOAc = 6:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.14 – 8.08 (m, 2H), 8.01 (d, J = 13.8 Hz, 1H), 7.67 –

7.59 (m, 3H), 3.95 (s, 3H) ppm;

¹³**C NMR** (100 MHz, CDCl₃): δ 166.1, 138.8, 137.7, 134.3, 133.2, 130.6, 129.1, 52.7 ppm;

IR (film) vmax = 3393, 3105, 2953, 2919, 2857, 2366, 1718, 1644, 1571, 1526, 1464,

1390, 1345, 1289, 1092, 1018, 962, 764.

7k is a known compound.^[S8]

(E)-1-methoxy-4-(2-nitrovinyl)benzene (7l)



Yield of **7l** 93%, 57 mg; yellow solid; **mp** 87-90 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.97 (d, *J* = 13.6 Hz, 1H), 7.57 – 7.46 (m, 3H), 6.95 (d, *J* = 8.9 Hz, 2H), 3.86 (s, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 163.1, 139.2, 135.1, 131.3, 122.6, 115.0, 55.6 ppm; HRMS (ESI) m/z: [M+Na]⁺ calculated for C₉H₉NNaO₃⁺ 202.0474, found 202.0453; IR (film) vmax = 3737, 3404, 3116, 2969, 2930, 2845, 2366, 2332, 1610, 1498, 1430, 1351, 1250, 1176, 1030, 973, 843, 798, 668. **7l** is a known compound.^[S8]

(E)-4-(2-nitrovinyl)benzonitrile (7m)



Yield of **7m** 96%, 65 mg; yellow solid; **mp** 186-187 °C; $R_f = 0.5$ (silica, PE:EtOAc = 10:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.99 (d, *J* = 13.7 Hz, 1H), 7.79 – 7.72 (m, 2H), 7.71 – 7.59 (m, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 139.6, 136.7, 134.5, 133.1, 129.5, 117.9, 115.3 ppm;

IR (film) vmax = 3099, 2953, 2913, 2851, 2231, 1734, 1644, 1565, 1531, 1480, 1464,

1379, 1345, 1260, 968, 832.

7m is a known compound.^[S8]

(E)-4-(2-nitrovinyl)phenyl acetate (7n)



Yield of **7n** 86%, 55 mg; yellow solid; **mp** 176-178 °C; $R_f = 0.5$ (silica, PE:EtOAc = 6:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.04 (d, *J* = 13.7 Hz, 1H), 7.66 – 7.58 (m, 3H), 7.25 (d, *J* = 8.6 Hz, 2H), 2.38 (s, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 169.0, 153.6, 138.2, 137.2, 130.5, 127.8, 122.9, 21.3 ppm;

HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{10}H_{10}NO_4^+$ 208.0604, found 208.0632;

IR (film) vmax = 3404, 2959, 2925, 2851, 1751, 1565, 1509, 1374, 1204, 1164, 1012, 916, 860.

7n is a known compound.^[S10]

(E)-1-fluoro-3-(2-nitrovinyl)benzene (70)



Yield of **70** 64%, 46 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹H NMR (400 MHz, CDCl₃): δ 7.95 (d, J = 13.7 Hz, 1H), 7.56 (d, J = 13.7 Hz, 1H), 7.44 (td, J = 7.9, 5.7 Hz, 1H), 7.34 (dt, J = 7.7, 1.4 Hz, 1H), 7.27 – 7.17 (m, 2H) ppm;
¹³C NMR (100 MHz, CDCl₃): δ 163.0 (d, J = 247.03 Hz), 138.2, 137.8 (d, J = 2.71 Hz), 132.2, 131.2, 125.3 (d, J = 3 Hz), 119.1 (d, J = 21.3 Hz), 115.5 (d, J = 22.4 Hz) ppm;
IR (film) vmax = 3111, 2947, 2925, 2845, 2355, 2338, 1627, 1576, 1514, 1486, 1447, 1340, 1266, 1216, 1154, 962, 849, 786, 724, 668.

70 is a known compound.^[S12]

(E)-1-bromo-3-(2-nitrovinyl)benzene (7p)



Yield of **7p** 74%, 46 mg; yellow solid; **mp** 59-61 °C; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.91 (d, *J* = 13.8 Hz, 1H), 7.68 (t, *J* = 1.8 Hz, 1H), 7.61 (ddd, *J* = 8.0, 1.9, 1.0 Hz, 1H), 7.55 (d, *J* = 13.7 Hz, 1H), 7.48 (dt, *J* = 7.8, 1.5 Hz, 1H), 7.33 (t, *J* = 7.9 Hz, 1H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 138.1, 137.5, 135.0, 132.1, 131.8, 131.0, 127.8, 123.5 ppm;

IR (film) vmax = 3105, 2959, 2925, 2857, 2372, 1638, 1582, 1526, 1480, 1452, 1345, 1272, 1221, 1142, 962, 838, 781, 724, 668.

7p is a known compound.^[S13]

(E)-1-methyl-3-(2-nitrovinyl)benzene (7q)



Yield of **7q** 75%, 54 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H** NMR (400 MHz, CDCl₃): δ 7.98 (d, J = 13.6 Hz, 1H), 7.58 (d, J = 13.7 Hz, 1H),

7.38 – 7.28 (m, 4H), 2.40 (s, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 139.4, 139.3, 137.0, 133.2, 130.1, 129.8, 129.4, 126.5, 21.4 ppm;

IR (film) vmax = 3737, 3635, 2959, 2919, 2845, 2355, 2332, 1638, 1514, 1351, 968,

668.

7q is a known compound.^[S13]

(E)-1-chloro-2-(2-nitrovinyl)benzene (7r)



Yield of **7r** 79%, 53 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.40 (d, *J* = 13.7 Hz, 1H), 7.62 – 7.56 (m, 2H), 7.49 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.43 (td, *J* = 7.7, 1.7 Hz, 1H), 7.34 (td, *J* = 7.5, 1.4 Hz, 1H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 138.9, 136.2, 135.2, 133.0, 130.9, 128.7, 128.6, 127.6 ppm;

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_8H_6ClNNaO_2^+$ 205.9979, found 205.9914;

IR (film) vmax = 3399, 3122, 2930, 2834, 1633, 1593, 1509, 1469, 1441, 1334, 1289, 1052, 962, 849, 758, 691.

7r is a known compound.^[S9]

(1-nitroprop-1-en-2-yl)benzene (7s)



Yield of **7s** 83%, 83 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.45 (s, 5H), 7.31 (d, *J* = 1.5 Hz, 1H), 2.65 (d, *J* = 1.5 Hz, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 150.1, 138.4, 136.4, 130.5, 129.1, 126.9, 18.7 ppm;

HRMS (ESI) m/z: [M+K]⁺ calculated for C₉H₉NKO₂⁺ 202.0264, found 202.0286;

IR (film) vmax = 3111, 2953, 2925, 2857, 2366, 2327, 1622, 1520, 1340, 781, 708.

7s is a known compound.^[S14]

1-fluoro-4-(1-nitroprop-1-en-2-yl)benzene (7t)



Yield of **7t** 60%, 40 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.49 – 7.42 (m, 2H), 7.28 (d, J = 1.5 Hz, 1H), 7.12 (t,

J = 8.6 Hz, 2H), 2.62 (d, *J* = 1.5 Hz, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 164.0 (d, *J* = 249.75 Hz), 148.8, 136.3, 134.3 (d, *J* =

3.51 Hz), 129.0 (d, *J* = 8.67 Hz), 116.2 (d, *J* = 21.79 Hz), 18.6 ppm;

HRMS (ESI) m/z: [M+Na]⁺ calculated for C₉H₈FNNaO₂⁺ 204.0431, found 204.0430;

IR (film) vmax = 3105, 2919, 2851, 2372, 1610, 1509, 1340, 1238, 1164, 1108, 1018, 916, 838, 719.

7t is a known compound.^[S2]

1-chloro-4-(1-nitroprop-1-en-2-yl)benzene (7u)



Yield of 7u 92%, 60 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.45 – 7.36 (m, 4H), 7.28 (d, J = 1.5 Hz, 1H), 2.62 (d,

J = 1.5 Hz, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 148.6, 136.7, 136.7, 136.5, 129.4, 128.3, 18.5 ppm;

HRMS (ESI) m/z: $[M+Na]^+$ calculated for C₉H₈ClNNaO₂⁺ 220.0135, found 220.0132;

IR (film) vmax = 3099, 2919, 2857, 2361, 1622, 1588, 1554, 1509, 1492, 1340, 1097,

1012, 922, 820, 753.

7**u** is a known compound.^[S3]

1-methoxy-2-(1-nitroprop-1-en-2-yl)benzene (7v)



Yield of 7v 61%, 39 mg; yellow liquid; $R_f = 0.5$ (silica, PE:EtOAc = 20:1);

¹H NMR (400 MHz, CDCl₃): δ 7.38 (ddd, J = 8.3, 7.5, 1.8 Hz, 1H), 7.15 (dq, J = 4.2, 1.9 Hz, 2H), 7.01 – 6.93 (m, 2H), 3.85 (s, 3H), 2.56 (d, J = 1.6 Hz, 3H).
¹³C NMR (100 M;z, CDCl₃): δ 156.7, 150.3, 137.6, 131.0, 128.9, 128.2, 120.8, 111.4, 55.6, 20.1;

HRMS (ESI) m/z: $[M+Na]^+$ calculated for $C_{10}H_{12}NO_3^+$ 194.0811, found 194.0878;

IR (film) vmax = 3111, 2953, 2930, 2845, 2361, 1605, 1554, 1520, 1492, 1464, 1436, 1340, 1245, 1182, 1131, 1030, 753.

7v is a known compound.^[S3]

(E)-2-(2-nitrovinyl)thiophene (7w)



Yield of **7w** 43%, 30 mg; yellow solid; **mp** 82-84 °C; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H** NMR (400 MHz, CDCl₃): δ 8.15 (d, *J* = 13.3 Hz, 1H), 7.57 (dd, *J* = 5.0, 1.0 Hz,

1H), 7.52 – 7.43 (m, 2H), 7.15 (dd, *J* = 5.1, 3.7 Hz, 1H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 135.5, 134.8, 133.9, 132.2, 131.8, 129.0 ppm;

IR (film) vmax = 3105, 3093, 3031, 2925, 2851, 2361, 1622, 1526, 1492, 1362, 1334,

1232, 1193, 1051, 973, 950, 854, 815, 730.

7w is a known compound.^[S15]

(E)-2-bromo-5-(2-nitrovinyl)thiophene (7x)



Yield of 7x 92%, 57 mg; yellow solid; **mp** 82-84 °C; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.02 (d, *J* = 13.5 Hz, 1H), 7.38 (d, *J* = 13.4 Hz, 1H), 7.21 (d, *J* = 3.9 Hz, 1H), 7.12 (d, *J* = 3.9 Hz, 1H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 135.6, 135.4, 135.2, 132.0, 131.2, 119.8 ppm;

IR (film) vmax = 3399, 3189, 3093, 2959, 2919, 2851, 2361, 1729, 1638, 1616, 1531, 1458, 1424, 1322, 1131, 810, 719.

7x is a known compound.^[S8]

(E)-3-(2-nitrovinyl)bicyclo[4.2.0]octa-1,3,5-triene (7y)



Yield of **7y** 74%, 50 mg; yellow solid; **mp** 124-126 °C; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.01 (d, *J* = 13.6 Hz, 1H), 7.57 (d, *J* = 13.6 Hz, 1H), 7.39 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.26 (s, 1H), 7.16 (d, *J* = 13.6 Hz, 1H), 3.25 (s, 4H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 151.5, 147.2, 140.6, 136.0, 129.4, 128.9, 123.7, 122.4, 30.1, 29.5 ppm;

IR (film) vmax = 3111, 2919, 2851, 1627, 1605, 1503, 1436, 1351, 1272, 1204, 968, 826.

(E)-5-(2-nitrovinyl)-2,3-dihydrobenzofuran (7z)



Yield of **7Z** 55%, 37 mg; yellow liquid; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.96 (d, *J* = 13.5 Hz, 1H), 7.50 (d, *J* = 13.5 Hz, 1H), 7.44 – 7.31 (m, 2H), 6.83 (d, *J* = 8.3 Hz, 1H), 4.67 (t, *J* = 8.7 Hz, 2H), 3.26 (t, *J* = 8.7 Hz, 2H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 164.2, 139.7, 134.6, 131.5, 129.1, 125.8, 122.7, 110.5, 72.4, 29.2 ppm;

IR (film) vmax = 3365, 3184, 2925, 2857, 2186, 1638, 1498, 1475, 1334, 1250, 1102, 984, 815, 714.

(E)-1,3,5-trimethyl-2-(2-nitrovinyl)benzene (7aa)



Yield of **7aa** 57%, 37 mg; yellow solid; **mp** 123-124 °C; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.27 (d, *J* = 13.9 Hz, 1H), 7.30 (d, *J* = 13.9 Hz, 1H), 6.95 (s, 2H), 2.39 (s, 6H), 2.32 (s, 3H) ppm;

¹³**C NMR** (100 MHz, CDCl₃): δ 141.0, 139.7, 138.6, 136.6, 130.0, 125.8, 21.6, 21.3 ppm;

IR (film) vmax = 3133, 2964, 2930, 2851, 1633, 1605, 1503, 1334, 1148, 1046, 973, 922, 849, 730.

7aa is a known compound.^[S8]

(2-nitroethene-1,1-diyl)dibenzene (7ab)



Yield of **7aa** 64%, 40 mg; yellow liquid; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.52 – 7.39 (m, 7H), 7.33 – 7.23 (m, 4H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 150.6, 137.2, 135.7, 134.5, 131.0, 129.4, 129.1,

129.0, 128.9, 128.6 ppm;

IR (film) vmax = 3111, 2959, 2925, 2857, 1638, 1560, 1520, 1469, 1334, 1255, 1198, 968, 900, 838, 786, 668.

7ab is a known compound.^[S8]

2-(1-nitroprop-1-en-2-yl)naphthalene (7ac)



Yield of **7ac** 79%, 50 mg; yellow liquid; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.96 – 7.84 (m, 4H), 7.61 – 7.41 (m, 4H), 2.73 (d, *J* = 1.5 Hz, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 149.9, 136.6, 135.4, 134.0, 133.0, 129.0, 128.7, 127.8, 127.6, 127.1, 123.7, 18.6 ppm;

HRMS (ESI) m/z: [M+H]⁺ calculated for C₁₃H₁₂NNaO₄⁺ 214.0862, found 214.0805;

IR (film) vmax = 3111, 3049, 2925, 2851, 2361, 1605, 1514, 1334, 1272, 956, 815,

742, 708.

7ac is a known compound.^[S2]

5-(1-nitroprop-1-en-2-yl)benzo[d][1,3]dioxole (7ad)



Yield of **7ad** 78%, 50 mg; yellow solid; **mp** 114-115 °C; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 7.29 (d, *J* = 1.4 Hz, 1H), 7.00 (dd, *J* = 8.2, 2.0 Hz, 1H), 6.93 (d, *J* = 1.9 Hz, 1H), 6.85 (d, *J* = 8.1 Hz, 1H), 6.03 (s, 2H), 2.61 (d, *J* = 1.4 Hz, 3H) ppm;

¹³**C NMR** (100 MHz, CDCl₃): δ 149.7, 148.4, 135.5, 132.1, 121.5, 108.7, 107.0, 101.8, 29.7, 18.6 ppm;

IR (film) vmax = 3359, 3184, 2964, 2925, 2851, 1729, 1644, 1469, 1374, 1244, 1040, 820.

7ac is a known compound.^[S16]

1,2-dimethoxy-4-(2-nitroprop-1-en-1-yl)benzene (7ae)



Yield of **7ae** 50%, 33 mg; yellow liquid; $R_f = 0.6$ (silica, PE:EtOAc = 10:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.07 (s, 1H), 7.09 (dd, J = 8.3, 2.1 Hz, 1H), 6.97 –

6.95 (m, 1H), 6.82 (s, 1H), 3.96 – 3.83 (m, 9H), 2.49 (d, J = 1.0 Hz, 3H) ppm;

¹³**C NMR** (100 MHz, CDCl₃): δ 134.0, 125.1, 124.1, 122.1, 113.2, 111.4, 111.1, 56.2, 56.1, 56.0, 14.3 ppm;

HRMS (ESI) m/z: [M+Na]⁺ calculated for C₁₁H₁₃NNaO₄⁺ 246.0736, found 246.0729;
IR (film) vmax = 2964, 2925, 2837, 1734, 1599, 1520, 1464, 1379, 1278, 1154, 1030.
7ae is a known compound.^[S5]

(E)-2-chloro-3,4-dimethoxy-1-(2-nitrovinyl)benzene (7af)



Yield of **7af** 81%, 50 mg; yellow liquid; $R_f = 0.6$ (silica, PE:EtOAc = 10:1);

¹**H** NMR (400 MHz, CDCl₃): δ 8.35 (d, J = 13.6 Hz, 1H), 7.56 (d, J = 13.6 Hz, 1H),

7.36 (d, *J* = 8.8 Hz, 1H), 6.89 (d, *J* = 8.8 Hz, 1H), 3.94 (s, 3H), 3.88 (s, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 156.8, 146.5, 137.2, 135.6, 131.1, 124.6, 121.8,

111.0, 60.8, 56.4 ppm;

HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{10}H_{10}NO_3^+$ 244.0371, found 244.0332;

IR (film) vmax = 3111, 2925, 2851, 1723, 1635, 1588, 1498, 1402, 1351, 1272, 1046, 968, 810, 657.

7af is a known compound.^[S1]

(E)-4-methoxy-2-methyl-1-(2-nitrovinyl)benzene (7ag)



Yield of **7ag** 40%, 26 mg; yellow solid; **mp** 221-223 °C; $R_f = 0.6$ (silica, PE:EtOAc = 20:1);

¹**H NMR** (400 MHz, CDCl₃): δ 8.25 (d, *J* = 13.5 Hz, 1H), 7.53 – 7.44 (m, 2H), 6.78 (dd, *J* = 4.6, 2.1 Hz, 2H), 3.84 (s, 3H), 2.46 (s, 3H) ppm;

¹³C NMR (100 MHz, CDCl₃): δ 162.8, 141.9, 136.7, 135.5, 129.5, 121.5, 116.6, 112.9, 55.5, 20.4 ppm;

HRMS (ESI) m/z: $[M+H]^+$ calculated for $C_{10}H_{12}NO_3^+$ 194.0811, found 194.0809.

7ag is a known compound.^[S10]

5. Copies of NMR specture







¹H NMR of 6x (400 MHz, CDCl₃)



¹³C NMR of 6x (100 MHz, CDCl₃)











S29

¹H NMR of 6af (400 MHz, CDCl₃)



¹³C NMR of 6af (100 MHz, CDCl₃)



¹H NMR of 6ag (400 MHz, CDCl₃)



¹³C NMR of 6ag (100 MHz, CDCl₃)



¹H NMR of 7a (400 MHz, CDCl₃)



¹³C NMR of 7a (100 MHz, CDCl₃)



¹H NMR of 7b (400 MHz, CDCl₃)



¹³C NMR of 7b(100 MHz, CDCl₃)



¹H NMR of 7c (400 MHz, CDCl₃)

fl (ppm)


S34

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¹H NMR of 7d (400 MHz, CDCl₃)



¹³C NMR of 7d (100 MHz, CDCl₃)



¹H NMR of 7e (400 MHz, CDCl₃)



¹H NMR of 7f (400 MHz, CDCl₃)



¹³C NMR of 7f (100 MHz, CDCl₃)





S38

¹H NMR of 7h (400 MHz, CDCl₃)





¹³C NMR of 7i (100 MHz, CDCl₃)



¹H NMR of 7j (400 MHz, CDCl₃)



¹H NMR of 7k (400 MHz, CDCl₃)



¹³C NMR of 7k (100 MHz, CDCl₃)



¹H NMR of 7l (400 MHz, CDCl₃)



¹H NMR of 7m (400 MHz, CDCl₃)



¹³C NMR of 7m (100 MHz, CDCl₃)



¹H NMR of 7n (400 MHz, CDCl₃)







¹H NMR of 70 (400 MHz, CDCl₃)



¹³C NMR of 70 (100 MHz, CDCl₃)



¹H NMR of 7p (400 MHz, CDCl₃)



¹³C NMR of 7p (100 MHz, CDCl₃)





S48

¹H NMR of 7r (400 MHz, CDCl₃)



¹³C NMR of 7r (100 MHz, CDCl₃)



¹H NMR of 7s (400 MHz, CDCl₃)





S51

¹H NMR of 7u (400 MHz, CDCl₃)



¹³C NMR of 7u (100 MHz, CDCl₃)









¹H NMR of 7w (400 MHz, CDCl₃)



¹³C NMR of 7w (100 MHz, CDCl₃)





S55

¹H NMR of 7y (400 MHz, CDCl₃)



¹H NMR of 7z (400 MHz, CDCl₃)





¹H NMR of 7aa (400 MHz, CDCl₃)



¹³C NMR of 7aa (100 MHz, CDCl₃)





¹³C NMR of 7ab (100 MHz, CDCl₃)







¹³C NMR of 7ad (100 MHz, CDCl₃)



¹H NMR of 7ae (400 MHz, CDCl₃)



¹³C NMR of 7ae (100 MHz, CDCl₃)







S63

¹H NMR of 7ag (400 MHz, CDCl₃)



¹³C NMR of 7ag (100 MHz, CDCl₃)



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