SUPPLEMENTARY MATERIALS

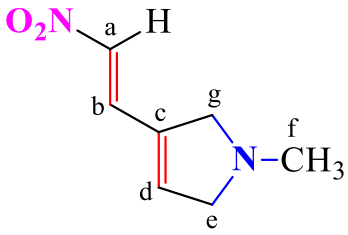
Unexpected course of reaction between (1E,3E)-1,4-dinitro-1,3-butadiene and N-methyl azomethine ylide — A comprehensive experimental and quantum-chemical study

Mikołaj Sadowski and Karolina Kula *

Department of Organic Chemistry and Technology, Cracow University of Technology, Warszawska 24, 31-155 Cracow, Poland;

*Correspondence adress: karolina.kula@pk.edu.pl (K.K)

PHYSICAL CHARACTERISTICS



(**3a**)

$C_7H_{10}N_2O_2$

1-methyl-3-(*trans*-2-nitrovinyl)-Δ^3-pyrroline (**3a**):
Yellow crystal solid, m.p. 91.6 °C (diethyl ether)

EA:
Calculated for brutto formula $C_7H_{10}N_2O_2$ [%]: C 54.55, H 6.49, N 18.18; Found [%]: C 54.52, H 6.51, N 18.20.

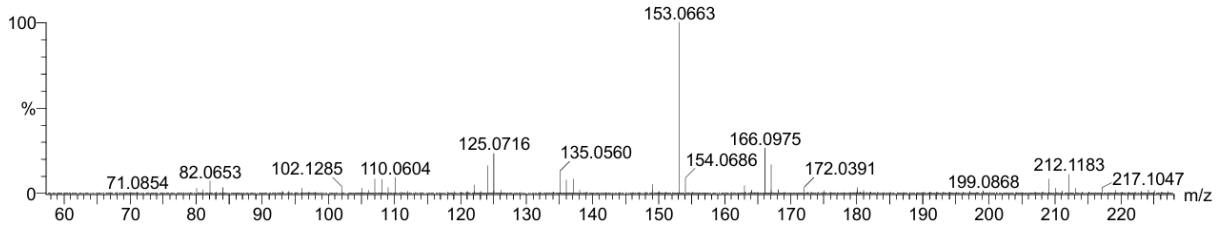
HR-MS: (−APCI): m/z
Calculated for formula $C_7H_9N_2O_2$: 153.0664 $[M-H]^-$;
Found 153.0663

FT-IR (ATR): υ [cm^{-1}]
3114 (~C–H stretch, alkene, medium),
2917 (~C–H stretch, alkane, medium),
1727 (>C=C< stretch, *trans* alkene, weak),
1614 (C=C< stretch, conjugated alkene, strong),
1537 (~C–H bend, alkene, medium),
1476 (~N–O stretch, asymmetrical, nitro group, strong),
1420 (~CH_3 rock, medium),
1376 (~CH_3 rock, medium),
1322 (~N–O stretch, symmetrical, nitro group, strong),
1232 (–N< stretch, pyrroline ring, medium),
1154 (–N< stretch, pyrroline ring, medium),
955 (>C=C< bend, *trans* alkene, strong),
717 (=C–H bend, alkene, strong).

^{1}H NMR (400 MHz, $CDCl_3$): δ [ppm]
7.96 (d, 1H, C**H**$_a$–NO_2, J = 13.3 Hz),
7.39 (d, 1H, =C**H**$_b$–ring, J = 13.4 Hz),
7.03 – 7.01 (m, 1H, =C**H**$_d$–ring),
6.67 – 6.65 (m, 2H, =C**H**$_g$–ring),
6.38 – 6.36 (m, 2H, =C**H**$_e$–ring).

^{13}C NMR (100 MHz, $CDCl_3$): δ [ppm]
134.08 (**C**$_a$), 132.82 (**C**$_b$), 128.06 (**C**$_d$), 125.08 (**C**$_g$),
115.92 (**C**$_c$), 107.71 (**C**$_e$), 36.65 (**C**$_f$).

HR-MS



Minimum:				-1.5				
Maximum:		5.0	5.0	90.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
153.0663	153.0664	-0.1	-0.7	4.5	1253.1	n/a	n/a	C7 H9 N2 O2

Figure S1. HR-MS spectrum of 1-methyl-3-(*trans*-2-nitrovinyl)-Δ³-pyrroline (**3a**).

FT-IR

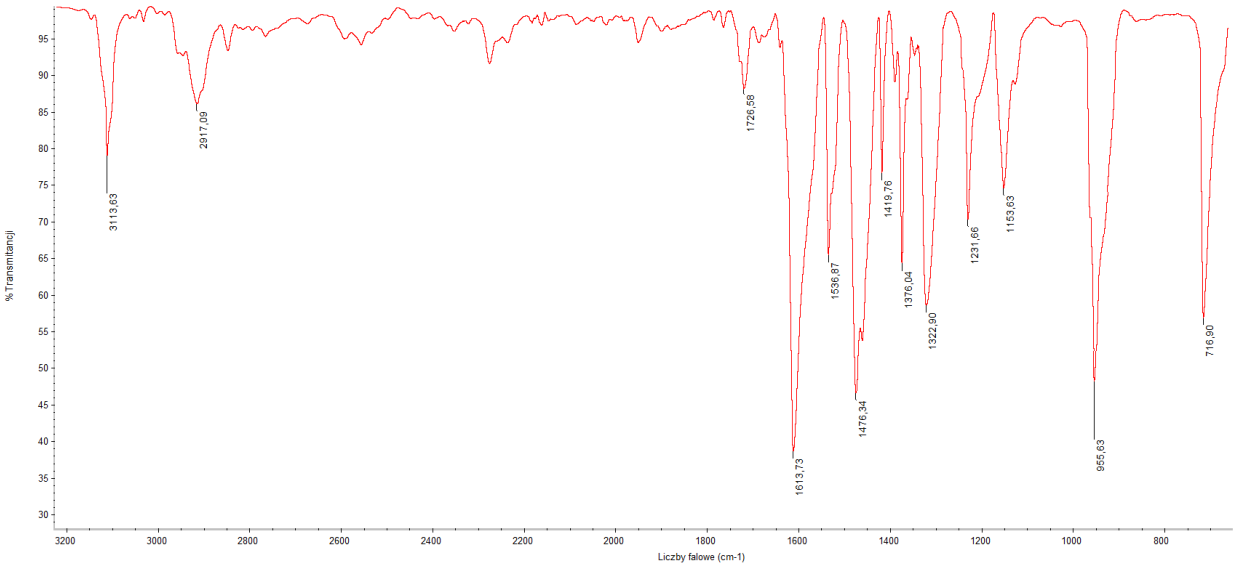


Figure S2. FT-IR spectrum of 1-methyl-3-(*trans*-2-nitrovinyl)-Δ³-pyrroline (**3a**).

^{1}H NMR

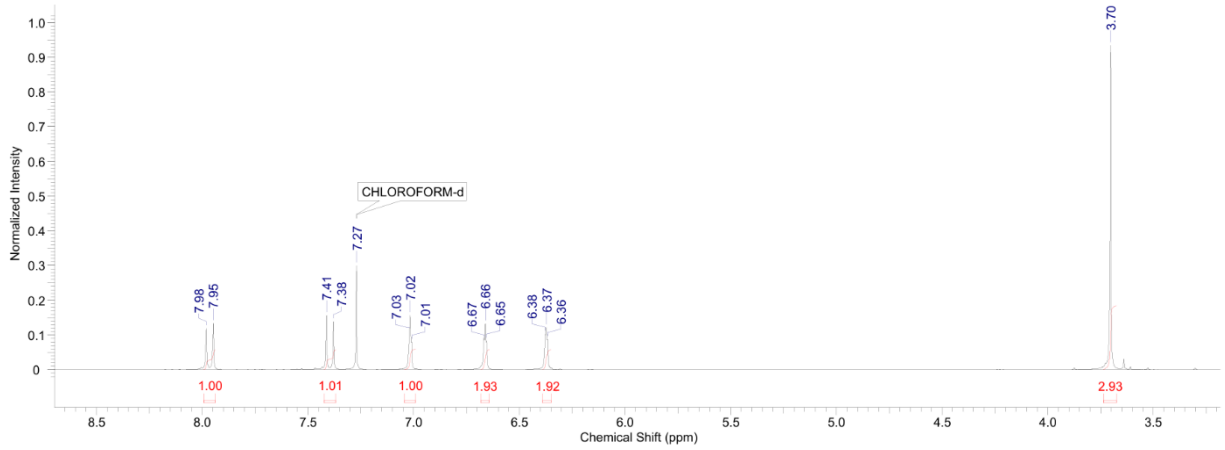


Figure S3. ^{1}H NMR spectrum of 1-methyl-3-(*trans*-2-nitrovinyl)-Δ^{3}-pyrroline (**3a**).

^{13}C NMR

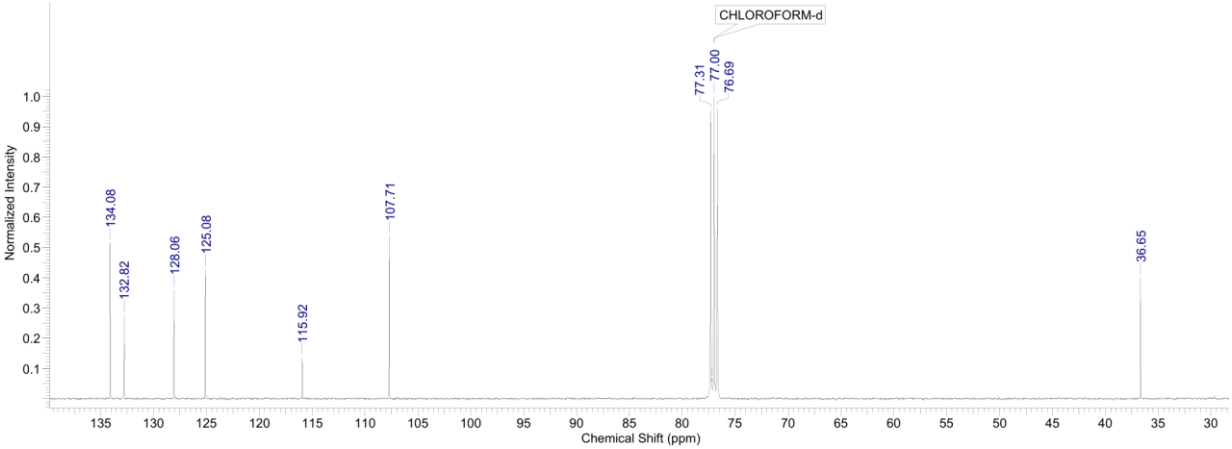


Figure S4. ^{13}C NMR spectrum of 1-methyl-3-(*trans*-2-nitrovinyl)-Δ^{3}-pyrroline (**3a**).

HMQC

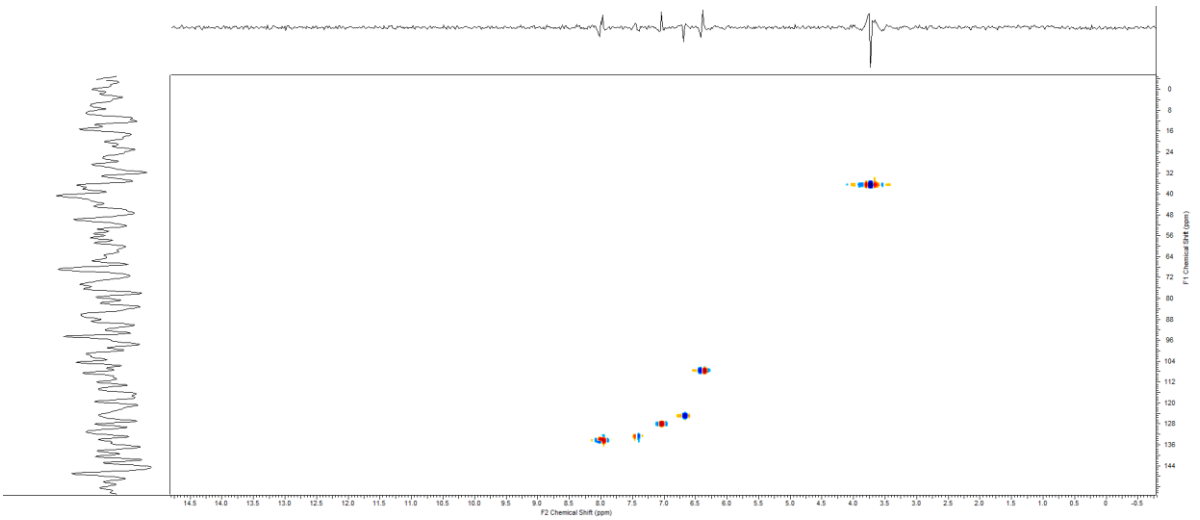


Figure S5. 2D ^{1}H-^{13}C HMQC NMR spectrum of 1-methyl-3-(*trans*-2-nitrovinyl)-Δ^{3}-pyrroline (**3a**).

Table S1. Cartesian coordinates of (1*E*,3*E*)-1,4-dinitro-1,3-butadiene (**1**) (B3LYP/6-31G(d), gas phase).

Center	Coordinates (Angstroms)		
	X	Y	Z
C	-0.67478800	0.26019800	-0.00001300
C	-1.74894700	-0.54117400	0.00007900
H	-0.83429000	1.33511200	-0.00008100
N	-3.09583400	0.01451300	0.00011100
O	-3.23204000	1.23800400	0.00005000
O	-4.01099700	-0.80958200	0.00019300
C	0.67483900	-0.26016800	-0.00001600
H	0.83434300	-1.33508500	0.00005400
C	1.74899200	0.54120400	-0.00011100
N	3.09584100	-0.01452300	-0.00012600
O	4.01101300	0.80956700	-0.00019400
O	3.23192900	-1.23803100	0.00001700
H	-1.74652700	-1.62326800	0.00015900
H	1.74660400	1.62329900	-0.00019200

Table S2. Cartesian coordinates of N-methyl azomethine ylide (**2**) (B3LYP/6-31G(d), gas phase).

Center	Coordinates (Angstroms)		
	X	Y	Z
C	-0.710539	-1.207047	-0.00295
C	-0.709039	1.207894	-0.002882
H	-1.788147	-1.268999	0.047181
H	-0.086684	-2.085684	-0.011042
H	-0.084075	2.085743	-0.011332
H	-1.786566	1.271202	0.047231
N	-0.13311	0.000066	-0.019536
C	1.346378	-0.000805	0.00999
H	1.713952	0.887717	-0.50449
H	1.68955	-0.000041	1.047141
H	1.712938	-0.890653	-0.502896