## Supporting Information for:

# Photoaffinity Labeling via Nitrenium Ion Chemistry: Protonation of the Nitrene Derived from a 4-Amino-3-nitrophenylazides to Afford Reactive Nitrenium Ion Pairs.

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**CONTENTS:**

General information S5

Synthesis of Starting Materials

Synthesis of 1-Fluoro-2-nitro-4-azidobenzene (**4**) S7

Synthesis of 4-*N*,*N*-Diethylamino-3-nitrophenylazide (**5**) S7

General procedure for Photolysis of 4-*N*,*N*-Diethylamino-3-Nitrophenylazide (**5**) in Various Solvents S8

a) Irradiation in MeOH S9

b) Irradiation in *n*-BuOH S10

c) Irradiation in i-PrOH S10

d) Irradiation in *t*-BuOH S11

e) Irradiation in Acetonitrile S12

f ) Irradiation in Toluene S12

General Procedure for the Photolysis of

4-Azido-2-nitro-*N*,*N*-Diethylaniline (5) in the presence

of Targeted Functional groups S13

a) Irradiation in Presence of Phenol S14

b) Irradiation in the Presence of Dimethylamine Hydrochloride S14

Acetylation of Adducts **7** and **8** S15

X-Ray Crystallographic Structure Determination of **9** S17

**Table SM1**. Crystal data and structure refinement for **9** S19

**Table SM2**. Atomic coordinates and equivalent isotropic

displacement parameters for **9** S20

**Table SM3**. Bond lengths and angles for **9** S22

**Table SM4**. Anisotropic displacement parameters for **9** S23

**Table SM5**. Torsion angles for **9** S24

**Table SM6**. Observed and calculated structure factors for **9** S25

Laser Flash Photolysis S39

General Procedure for Nanosecond Experiments S39

Nanosecond Spectra/Nucleophile S40

a) Transient absorption spectra in ethanol S40

b) Transient absorption spectra in *n*-butyl alcohol S40

c) Transient absorption spectra in *t*-butyl alcohol S41

d) Transient absorption spectra in ethyl glycolate S41

General Procedure for Ultrafast Transient

Absorption Measurements S42

**Fig. SM1.** Transient absorption spectra for azide 5

in i-PrOH irradiated at 305 nm S44

**Fig. SM2.** Transient absorption spectra of azide **5**

in acetonitrile irradiated at 350 nm S45

Theoretical Calculations for

Open- and Closed-Shell Singlet Nitrenes

**Table SM7** S46

**Fig. SM3.** Calculated geometry of :

A) the closed-shell nitrene S48

B) the opened-shell nitrene S49

Azide **5**

**Table SM8.** Optimized Coordinates of Azide **5** S50

**Table SM9.** Optimized Coordinates of Methyl Analog

of Azide **5** S51

Triplet Nitrene

**Table SM10.** Optimized Coordinates of Methyl Analog

of the Triplet Nitrene S53

Nitrogen Radical

**Table SM11.** Optimized Coordinates of Methyl Analog

of Nitrogen Radical S53

Nitrenium Ion

**Table SM12.** Optimized Coordinates of Methyl Analog of the Nitrenium Ion **10** S54

Nucleophile Adducts

**Table SM13.** Optimized Coordinates of Methyl Analog of Adduct **14** S54

**Table SM14.** Optimized Coordinates of Methyl Analog of Adduct **13** S55

**Alternative Scenarios**

**Figure SM4.** Alternative scenarios for closed-shell singlet nitrene reaction S57

**Formation of Adducts 13 and 14 and Their Relative Stability** S58

**Figure SM5.** Attack profiles for protonation of nitrene S59

**Figure SM6.** Reaction profiles for nucleophile attack and adduct interconversion S60

**Spectroscopic Data** S61-S70

**General Information.**

Reagents and anhydrous solvents were purchased from Aldrich, EMD, and were used without further purification. The 4-fluoro-3-nitroaniline was purchased from Alfa. All reactions were conducted using oven-dried glassware under an atmosphere of nitrogen or argon. Preparative TLC was performed on glass plates (Merck Kieselgel 60 F254; layer thickness, 0.25 and 0.2 mm). Products were purified via flash chromatography using 60 μm silica gel.[1] $^{1}$H-NMR and $^{13}$C-NMR spectra were recorded on a 300 MHz Bruker spectrometer using $CD_3CN$, $CDCl_3$ as solvents. The chemical shifts (δ) are reported in parts per million (ppm) relative to the residual $CHCl_3$ peak (7.26 ppm for $^{1}$H-NMR and 77.0 ppm for $^{13}$C-NMR), and coupling constants (*J*) are reported in Hertz (Hz). UV-visible absorption spectra were measured with an Agilent 8453 spectrophotometer, IR spectra were measured with a ThermoNicolet IR 200 spectrometer. EI mass spectra (70 eV) were measured in-house using a direct insertion probe in a Shimadzu QP5050A spectrometer. Exact mass and $MS_n$ determinations were done in the mass spectrometry facility in the Chemistry Department of the University of Cincinnati using a ThermoFinnigan LTQ Linear Ion-Trap FTMS pESI instrument. Transition state calculations for the formation of adducts 13 and 14 were conducted using Spartan '06.[2] Open-shell and closed-shell nitrenes were optimized at the CASSCF(4,4)/6-31G(d) level using the Gaussian 03 program.[3]

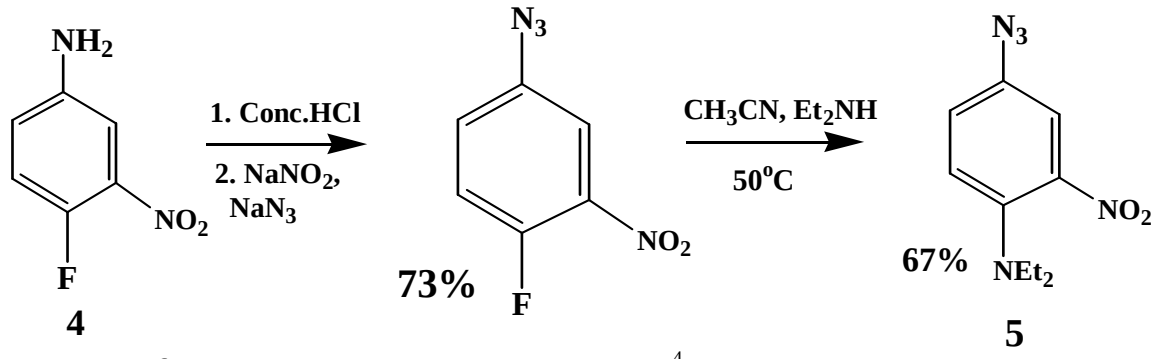
[1] Still, W. C.: Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923-2925.

[2] Spartan '06, Wavefunction, Inc., Irvine CA, Shao, Y.; Molnar, L. F.; Jung, Y.; Kussmann, J.; Ochsenfeld, C.; Brown, S. T.; Gilbert, A. T. B.; Slipchenko, L. V.; Levchenko, S. V.; O'Neill, D. P.; DiStasio Jr., R. A.; Lochan, R. C.; Wang, T.; Beran, G. J. O.; Besley, N. A.; Herbert, J. M.; Lin, C. Y.; Van Voorhis, T.; Chien, S. H.; Sodt, A.; Steele, R. P.; Rassolov, V. A.; Maslen, P. E.; Korambath, P. P.; Adamson, R. D.; Austin, B.; Baker, J.; Byrd, E. F. C.; Dachsel, H.;

Doerksen, R. J.; Dreuw, A.; Dunietz, B. D.; Dutio, A. D.; Furlani, T. R.; Gwaltney, S. R.; Heyden, A.; Hirata, S.; Hsu, C.-P.; Kedziora, G.; Khalliulin, R. Z.; Klunzinger, P.; Lee, A. M.; Lee, M. S.; Liang, W. Z.; Lotan, I.; Nair, N.; Peters, B.; Proynov, E. I.; Pieniazek, P. A.; Rhee, Y. M.; Ritchie, J.; Rosta, E.; Sherrill, C. D.; Simmonett, A. C.; Subotnik, J. E.; Woodcock III, H. L.; Zhang, W.; Bell, A. T.; Chakraborty, A. K.; Chipman, D. M.; Keil, F. J.; Warshel, A.; Hehre, W. J.; Schaefer, H. F.; Kong, J.; Krylov, A. I.; Gill, P. M. W., and Head-Gordon, M. *Phys Chem. Chem. Phys*, **8**, 3172 (2006).

[3] Gaussian 03, Revision C.02, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Montgomery, Jr., J. A.; Vreven, T. ; Kudin, K. N.; Burant, J. C.; Millam, J. M.; Iyengar, S. S.; Tomasi, J.; Barone, V.; Mennucci, B.; Cossi, M.; Scalmani, G.; Rega, N.; Petersson, G. A.; Nakatsuji, H.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Klene, M.; Li, X.; Knox, J. E.; Hratchian, H. P.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J\.; Gomperts, R.; Stratmann, R. E.; Yazvev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Ayala, P. Y.; Morokuma, K.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Zakrzewski, V. G.; Dapprich, S.; Daniels, A. D.; Strain, m. C.; Farkas, O.; Malick, D. K.; Rabuck, A. D.; Raghavachari, K.; Foresman, J. B.; Ortiz, J. V.; Cui, Q.; Baboul, A. G.; Clifford, S.; Cioslowdki, J.; Stefanov, B. B.; Liu, G.; Liashenko, A.; Piskorz, P.; Komaronmi, I.; Martin, R. L.; Fox, D. J.; Keith, T.; Al-Laham, M. A.; Peng, C. Y.; Nanayakkara, A.; Challacombe, M.; Gill, P.; M. W.; Johnson, B.; Chen, W.; Wong, M. W.; Gonzalez, C.; and Pople, J. A. Gaussian, Inc., Wallingford CT, 2004.

**Synthesis of Starting Materials.**




**Synthesis of 1-Fluoro-2-nitro-4-azidobenzene (4).**[4]

The diazonium salt was prepared from 4-fluoro-3-nitroaniline (0.95 g, 6 mmol) dissolved in warm (40-50° C) concentrated hydrochloric acid (6 mL). The amine solution was cooled to 5° C and a solution of sodium nitrite (0.5 mg, 7.5 mmol) in 4 mL of water added. The solution was stirred for 30 min. at ice-water bath temperature, and added dropwise to a cold solution (0° C) of sodium azide (0.51 g, 7.5 mmol) in 10 mL of water. The light orange crystals were formed immediately. The yield of 1-Fluoro-2-nitro-4-azidobenzene (**4**) was 73 % (mp = 54°C (lit.[5] 53–55 °C). $^{1}$H NMR (300 MHz, $CD_3CN$): δ 7.72 (dd, *J* = 2.7 Hz, *J* = 6 Hz, 1H), 7.26 (m, 2H); $^{13}$C NMR (75.5 MHz, $CD_3CN$): δ 137.25, 128.5 (d, *J* = 240 Hz), 125.35 (d, *J* = 30 Hz), 123.13, 118 (d, *J* = 90 Hz), 112.97. IR (KBr): 2121 (m) $cm^{-1}$.

[4] Leyva, E.; Munoz, D.; Platz, M.S., *J. Org. Chem.* **1989**, *54*, 5938-5945

[5] Hagedorn, M.; Sauers, R. R.; Eichholz, A. *J. Org. Chem.* **1978**, *43*, 2070-2072

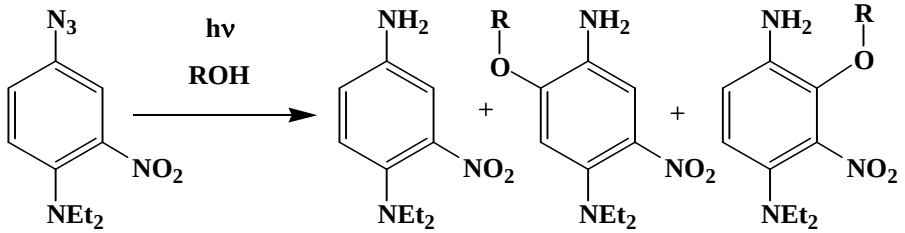
**Synthesis of 4-*N*,*N*-Diethylamino-3-nitrophenylazide (5).**[6]

A solution of 4-fluoro-3-nitrophenyl azide (**4**) (1.82 g, 0.01 moles) and diethylamine (7.3 g, 0.1 moles) was heated in acetonitrile (12 mL) at 40° C for 3 h. The crude product was purified by silica chromatography on a short column of silica gel

eluting with hexane to afford 4-*N*,*N*-Diethylamino-3-nitrophenylazide (**5**) as a red oil (1.57 g) in 67 %. $^{1}$H NMR (300 MHz, $CD_3CN$): δ 7.26 (d, *J* = 2.6 Hz,1H), 7.11 (d, *J* = 9 Hz, 1H), 7.03 (dd, *J* = 9 Hz, *J* = 2.6 Hz, 1H), 3.05 (q, *J* = 7.2 Hz, 4H), 0.96 (t, *J* = 7.2 Hz, 6H), $^{13}$C NMR (75.5 MHz, $CD_3CN$) δ 145.08, 141.57, 133.24, 124.59, 123.07, 115.54, 47.80, 12.59; UV-Vis ($CH_3CN$) $\lambda_{max}$ (nm) (ε, $M^{-1}$ $cm^{-1}$): 445 (1390); IR (KBr): 2117(m) $cm^{-1}$.

[6] Lormann, M. E. P.; Walker, C. H.; Es-Sayed, M.; Braese, S. Chem. Com 2002, 12, 1296-1297.

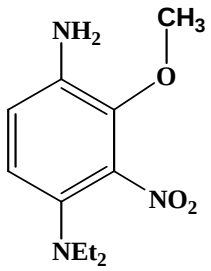
**General procedure for Photolysis of** 4-***N*,*N*-Diethylamino-3-nitrophenylazide (5) in Various Solvents.**



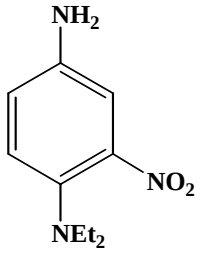

A solution of 20 mg of **5** in alcohol (5 mL) was flushed with nitrogen for 15 min and irradiated for 4 h using 350 nm light in a Rayonet Photochemical reactor. The crude photolysis mixture was concentrated to dryness *in vacuo*, leaving an oily residue, which was separated by preparative TLC (hexane) to afford the aniline **6** and addition products.

**Photolysis Products.**

**a) Irradiation in MeOH:**



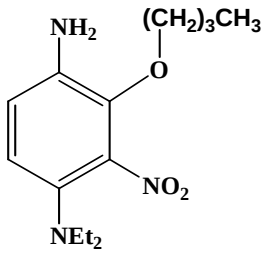

Irradiation in methanol afforded 4-*N*,*N*-diethylamino-2-methoxy-3-nitroaniline as the major addition product (98%): $^{1}$H NMR (300 MHz, $CDCl_3$) δ 6.92 (d, *J* = 9 Hz, 1H), 6.75 (d, *J* = 9 Hz, 1H), 3.5 (s, $NH_2$), 3.71(s, 3H), 2.9 (q, *J* = 7.2 Hz, 4H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CDCl_3$) δ 147.6, 137.9, 137.8, 134.3, 121.3, 116.7, 61.2, 49.6, 12.9; HRMS calcd for $C_{11}H_{18}N_3O_3$ (M + H) 240.13425, found 240.134817.




**6**

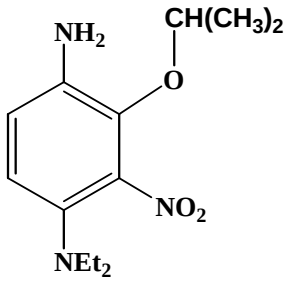
Trace amounts of 4-*N*,*N*-diethylamino-3-nitroaniline (**6**) (2%) were isolated: $^{1}$H NMR (300 MHz, $CDCl_3$): δ 7.4 (d, *J* = 9 Hz, 1H), 7.3 (d, *J* = 2.1 Hz, 1H), 7.2 (dd, *J* = 9 Hz, *J* = 2.1 Hz, 1H), 3.0 (q, *J* = 7.2 Hz, 4H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CDCl_3$): δ 145.08, 141.57, 133.24, 124.59, 123.07, 115.54; 47.13, 12.59; Mass spectrum: *m/z* (relative intensity): 209(40), 194(47), 174(15), 162(30), 147(20), 134(80), 119(75), 92(18), 65(100); HRMS calcd for $C_{10}H_{16}N_3O_2$ (M + H) 210.124252, found 210.12374.

**b) Irradiation in *n*-BuOH**




Irradiation in *n*-butanol afforded 4-*N*,*N*-diethylamino-2-*n*-butoxy-3-nitroaniline as the major addition product (95%): $^{1}$H NMR (300 MHz, $CDCl_3$): δ 6.9 (d, *J* = 9 Hz, 1H), 6.8 (d, *J* = 9 Hz, 1H), 4.2 (t, 2H), 2.9 (q, *J* = 7.2 Hz, 4H), 1.7 (m, 2H), 1.4 (m, 2H), 0.9 (t, *J* = 7.2 Hz, 6H), 0.8 (t, *J* = 7.2 Hz, 3H); $^{13}$C NMR (75.5 MHz, $CDCl_3$): δ 145.73, 137.12, 135.93, 133.26, 120.01, 115.63, 79.46, 48.75, 28.02, 17.91 (2C), 11.59; Mass spectrum: *m/z* (relative intensity): 281(70), 266(20), 264(18), 190(90), 178(17), 163(40), 150(30), 135(17), 107(10), 79(20) UV-vis 280-300 nm; HRMS calcd for $C_{14}H_{23}N_3O_3$ (M + H) 282.181767, found 282.18121.

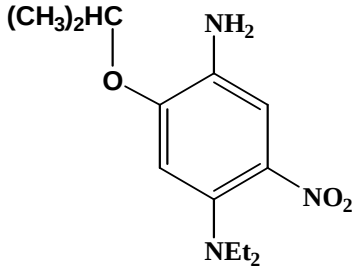
**c) Irradiation in *i*-PrOH:**




**8**

Irradiation in 2-propanol afforded 4-*N*,*N*-diethylamino-2-*i*-proproxy-3-nitroaniline (**8**) as the major addition product (92%): $^{1}$H NMR (300 MHz, $CD_3CN$): δ 6.96 (d, J = 9 Hz, 1H), 6.84 (d, J = 9 Hz, 1H), 4.4 (m, 1H), 4.25 (br.s, $NH_2$), 2.9 (q, *J* = 7.2 Hz, 4H), 1.2 (d,

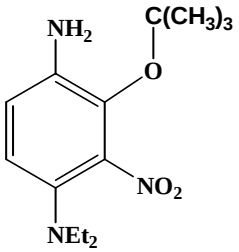
J = 6 Hz, 6H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CD_3CN$): δ 142.1, 140.16, 134.54, 132.82, 120.87, 116.63, 75.61, 49.74, 21.70, 12.36; Mass spectrum: *m/z* (relative intensity): 267(40), 225(10), 210(23), 190(100), 176(15), 163(30), 150(25), 148(15), 135(23), 121(20), 79(30).




**7**

The minor addition product was 4-*N*,*N*-diethylamino-6-*i*-proproxy-3-nitroaniline (**7**) (4%): $^1$H NMR (300 MHz, $CD_3CN$): δ 7.16 (s, 1H), 6.58 (s, 1H), 4.5 (m, 1H), 3.2 (q, *J* = 7.2 Hz, 4H), 1.26 (d, J = 6 Hz, 6H), 1.10 (t, *J* = 7.2 Hz, 6H); Mass spectrum: *m/z* (relative intensity): 267(40), 264(10), 250(12), 224(5), 210(22), 208(100), 193(5), 191(22), 178(25), 166(15), 164(30), 150(50), 136(23), 121(15), 108(20), 94(10), 80(20).

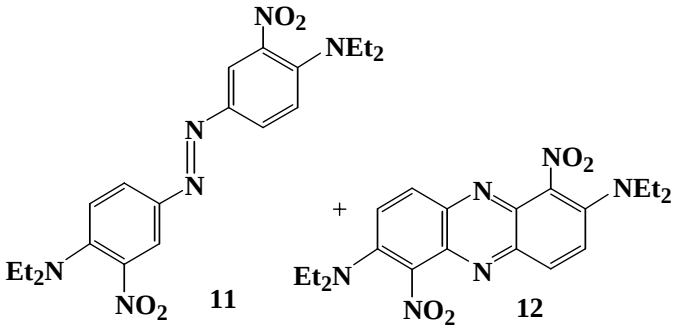
**d) Irradiation in *t*-BuOH:**




Irradiation in *t*-butanol afforded 4-*N*,*N*-diethylamino-2-*t*-butoxy-3-nitroaniline as the major addition product (86%): $^1$H NMR (300 MHz, $CD_3Cl$): δ 6.89 (d, *J* = 9 Hz, 1H), 6.76 (d, *J* = 9 Hz,1H), 2.85 (q, *J* = 7.2 Hz, 4H), 1.27 (s, 9H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C

NMR (75.5 MHz, $CDCl_3$): δ 145, 137, 133, 132, 123, 120, 79, 49, 28, 17 HRMS calcd for $C_{14}H_{23}N_3O_3$ (M + H) 282.181767, found 282.18119.

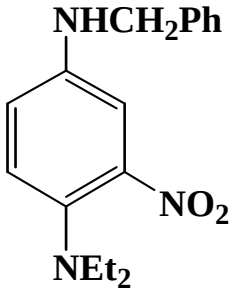
**e) Irradiation in Acetonitrile:**




Irradiation in Acetonitrile afforded two dimeric products **11** and **12**.

**11:** 65% yield, $^1$H NMR (300 MHz, $CDCl_3$) δ 8.69 (d, *J* = 2.4 Hz, 1H), 7.95 (dd, *J* = 9 Hz, *J* = 2.4 Hz 1H), 7.15 (d, *J* = 9 Hz, 1H), 3.4 (q, J = 7.2 Hz, 4H), 0.9 (t, *J* = 7.2 Hz, 6H) $^{13}$C NMR (75.5 MHz, $CDCl_3$) δ 160.73, 143.41, 141.8, 126.59, 121.95, 120.24, 46.12, 11.51 HRMS calcd for $C_{20}H_{27}N_6O_4$ (M + H) 415.209379, found 415.20915; **12**: 35% yield, $^1$H NMR (300 MHz, $CDCl_3$) δ 8.03 (d, *J* = 9 Hz, 1H), 7.59 (d, *J* = 9 Hz, 1H), 3.5 (q, J = 7.2 Hz, 4H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CDCl_3$) δ 162.73, 141.61, 138.48, 132.92, 131.26, 126.8, 46.03, 13.51; HRMS calcd for $C_{20}H_{25}N_6O_4$ (M + H) 413.193180, found 413.19317.

**f ) Irradiation in Toluene.**



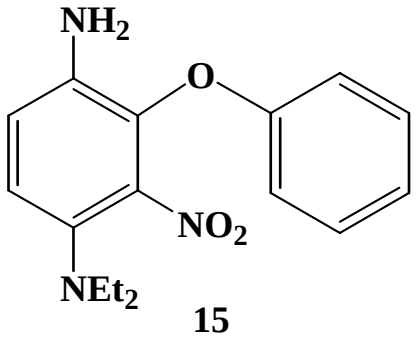
A solution of 20 mg of **5** in toluene (8 mL) was flushed with nitrogen for 15 min and irradiated for 4 h using 350 nm light in a Rayonet Photochemical reactor. The crude photolysis mixture was concentrated to dryness *in vacuo*, leaving an oily residue, which was separated by preparative TLC (hexane: dichloromethane in ratio 3:1) to afford the three major products (**6, 12** and corresponding benzyl amine in the ratio respectively 32:20:48).

$^1$H NMR (300 MHz, $CDCl_3$): δ 7.35-7.25 (m, 5H), 7.44 (d, *J* = 2.1 Hz, 1H), 7.23 (dd, *J* = 9 Hz, *J* = 2.1 Hz, 1H), 6.86(d, *J* = 9 Hz, Hz, 1H), 4.49 (broad NH), 4.38 (s, 2H), 3.0 (q, *J* = 7.2 Hz, 4H), 0.9 (t, *J* = 7.2 Hz, 6H); HRMS calcd for $C_{17}H_{21}N_3O_2$ ( M+H ) 300.170653; found 300.17064.

**General Procedure for the Photolysis of 4-*N*,*N*-diethylamino-3-nitrophenylazide (5) in the presence of Targeted Functional groups.**

A solution of 20 mg of **5** and 200 mg of the molecule containing the targeted functional group in acetonitrile (7 mL) was flushed with nitrogen for 15 min and photolyzed for 4 h using 350 nm light in a Rayonet Photochemical reactor. The crude photolysis mixture was concentrated to dryness *in vacuo*, extracted with water and $CH_2Cl_2$, dried over $Na_2SO_4$, leaving an oily residue, which was purified by preparative TLC (hexane/dichloromethane) to give aniline **6** and addition product as yellow oils.

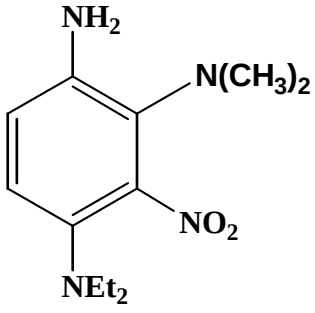
**a) Irradiation in Presence of Phenol.**




**15**

Irradiation in the presence of phenol afforded the phenol adduct in the 2-position **15** in 98% yield as an oil that had: $^{1}$H NMR (300 MHz, $CDCl_3$) δ 7.37-7.29 (m, 2H), 7.18 (d, *J* = 9 Hz, 1H), 7.09 (t, *J* = 7.2,  Hz, 1H), 6.8 (d, *J* = 9 Hz, 1H), 6.91 (dd, *J* = 7.2, 0.9 Hz, 2H), 4.3 (s, $NH_2$), 2.9 (q, *J* = 7.2 Hz, 4H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CDCl_3$) δ 171.4, 156.44, 139.1, 138.4, 132.6, 129.39, 122.87, 122.63, 114.66, 49.36, 12.93; HRMS calcd for $C_{16}H_{19}N_3O_3$ (M + H) 302.14989, found 302.150467.

**b) Irradiation in the Presence of Dimethylamine Hydrochloride.**



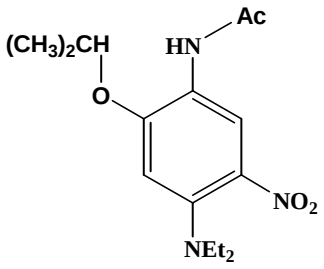

**16**

Irradiation in the presence of dimethylamine hydrochloride afforded the amine adduct in the 2-position **16** in 98% yield as an oil that had: $^{1}$H NMR (300 MHz, $CDCl_3$) δ 6.92 (d, *J*

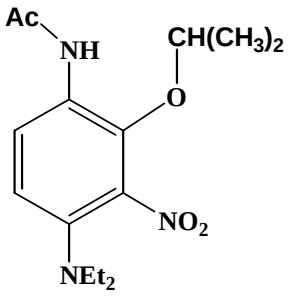
= 9 Hz, 1H), 6.75 (d, *J* = 9 Hz, 1H), 4.2 (s, $NH_2$), 2.9 (q, *J* = 7.2, 4H), 2.71 (s, 6 H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CDCl_3$) δ 147.6, 137.9, 137.8, 134.3, 121.3, 116.7, 61.2, 49.6, 12.9; HRMS calcd for $C_{12}H_{20}N_4O_2$ (M + H) 253.16590, found 253.166451.

**Acetylation of Adducts 7 and 8.**

The mixture of addition products **7** and **8**, isolated from the irradiation of **5** in *i*-PrOH, and acetic anhydride (150 ml) were dissolved in THF (10 mL) and heated at 60$^o$ C for 3 h. The crude product mixture was separated and purified by silica gel chromatography on a short column using hexane and dichloromethane as eluants. Two products were obtained: the acetamide of the minor addition product **7**, and the acetamide of the major addition product **8**, **9**.




Acetamide of **7** was isolated as a yellow solid in 2% yield: $^1$H NMR (300 MHz, $CDCl_3$) δ 9.84 (s, 1H), 6.58 (s, 1H), 4.62 (m, 1H), 3.19 (q, *J* = 7.2 Hz, 4H), 2.2 (s, 3H), 1.4 (d, *J* = 7.2, 6H), 1.2 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CDCl_3$) δ 171.3, 147.9, 143.22, 133.8, 124.3, 121.3, 116.7, 105,11, 71.1, 47.15, 21.98, 12.67; HRMS calcd for $C_{15}H_{24}N_3O_4$ (M + H) 310.176682, found 310.1765.




**9**

Acetamide of **8, 9** was isolated as a yellow solid in 96%: $^{1}$H NMR (300 MHz, $CDCl_3$) δ 8.44 (d, *J* = 9 Hz, 1H), 7.58 (s, NH), 7.02 (d, *J* = 9 Hz, 1H), 4.32 (m, 1H), 2.9 (q, *J* = 7.2 Hz, 4H), 2.2 (s, 3H), 1.29 (d, *J* = 7.2, 6H), 0.9 (t, *J* = 7.2 Hz, 6H); $^{13}$C NMR (75.5 MHz, $CDCl_3$) δ 168.01, 145.18, 139.81, 138.70, 129.43, 122.05, 120.33, 80.01, 48.99, 24.6, 22.78, 12.72; HRMS calcd for $C_{15}H_{24}N_3O_4$ (M + H) 310.176682, found 310.1767.

**X-Ray Crystallographic Structure Determination of 9.**

For X-ray examination and data collection, a suitable crystal, approximate dimensions 0.30 x 0.05 x 0.02 mm, was mounted in a loop with paratone-N and transferred immediately to the goniostat bathed in a cold stream.

Intensity data were collected at 150K on a standard Bruker SMART6000 CCD diffractometer using graphite-monochromated Cu Kα radiation, λ=1.54178Å. The detector was set at a distance of 5.165 cm from the crystal. A series of 10-s data frames measured at $0.3^o$ increments of ω were collected to calculate a unit cell. For data collection frames were measured for a duration of 8-s at $0.3^o$ intervals of ω with a maximum θ value of $\sim 135^o$. The data frames were processed using the program SAINT. The data were corrected for decay, Lorentz and polarization effects as well as absorption and beam corrections based on the multi-scan technique.

The structure was solved by a combination of direct methods SHELXTL v6.14 and the difference Fourier technique and refined by full-matrix least squares on $F^2$. Non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were located directly in the difference map and their positions refined. The isotropic displacement parameters for the H-atoms were defined as $a*U_{eq}$ of the adjacent atom, (a=1.5 for methyl and 1.2 for all others). The refinement converged with crystallographic agreement factors of R1=3.99%, wR2=10.00% for 2451 reflections with $I>2\sigma(I)$ (R1=5.44%, wR2=10.79% for all data) and 268 variable parameters.

**Acknowledgements, References and Notes:**

(1) Funding for the SMART6000 diffractometer was through NSF-MRI grant CHE-0215950.

(2) SMART v5.631 and SAINT v6.45A data collection and data processing programs, respectively. Bruker Analytical X-ray Instruments, Inc., Madison, WI; SADABS v2.10 for the application of semi-empirical absorption and beam corrections. G.M. Sheldrick, University of Göttingen, Germany; SHELXTL v6.14 for structure solution, figures and tables, neutral-atom scattering factors as stored in this package. G.M. Sheldrick, University of Göttingen, Germany and Bruker Analytical X-ray Instruments, Inc., Madison, WI.

**Table SM1**. Crystal data and structure refinement for $C_{15}H_{23}N_3O_4$.

| | | |
|---|---|---|
| Empirical formula | $C_{15}H_{23}N_3O_4$ | |
| Formula weight | 309.36 | |
| Temperature | 150(2) K | |
| Wavelength | 1.54178 Å | |
| Crystal system | Monoclinic | |
| Space group | $P2_1/c$ | |
| Unit cell dimensions | a = 12.2421(3) Å | α = 90° |
| | b = 14.8600(4) Å | β = 100.931(1)° |
| | c = 9.7185(2) Å | γ = 90° |
| Volume | 1735.89(7) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.184 $Mg/m^3$ | |
| Absorption coefficient | 0.714 $mm^{-1}$ | |
| F(000) | 664 | |
| Crystal size | 0.30 x 0.05 x 0.02 $mm^3$ | |
| θ range for data collection | 3.68 to 67.85° | |
| Index ranges | $-14 \leq h \leq 14$, $-16 \leq k \leq 17$, $-11 \leq l \leq 11$ | |
| Reflections collected | 14468 | |
| Independent reflections | 3122 [$R_{int}$ = 0.0353] | |
| Completeness to θ = 67.85° | 99.0 % | |
| Absorption correction | Multi-scan | |
| Max. and min. transmission | 0.9859 and 0.8143 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 3122 / 0 / 268 | |
| Goodness-of-fit on $F^2$ | 1.031 | |
| Final R indices [$I>2\sigma(I)$] | R1 = 0.0399, wR2 = 0.1000 | |
| R indices (all data) | R1 = 0.0544, wR2 = 0.1079 | |
| Largest diff. peak and hole | 0.195 and -0.161 $eÅ^{-3}$ | |

**Table SM2**. Atomic coordinates [x$10^4$] and equivalent isotropic displacement parameters [Å$^2$x$10^3$] for $C_{15}H_{23}N_3O_4$. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(1) | -1418(1) | 2505(1) | 5214(1) | 40(1) |
| O(2) | 279(1) | 876(1) | 2129(1) | 26(1) |
| O(3) | 2592(1) | -337(1) | 3793(2) | 57(1) |
| O(4) | 2737(1) | 577(1) | 2103(1) | 51(1) |
| N(1) | -755(1) | 2322(1) | 3210(1) | 26(1) |
| N(2) | 2453(1) | 391(1) | 3207(2) | 38(1) |
| N(3) | 3617(1) | 1212(1) | 5612(1) | 39(1) |
| C(1) | 343(1) | 2070(1) | 3834(2) | 26(1) |
| C(2) | 963(1) | 2545(1) | 4952(2) | 30(1) |
| C(3) | 2030(1) | 2286(1) | 5546(2) | 33(1) |
| C(4) | 2537(1) | 1550(1) | 5036(2) | 31(1) |
| C(5) | 1915(1) | 1110(1) | 3876(2) | 29(1) |
| C(6) | 826(1) | 1335(1) | 3279(1) | 25(1) |
| C(7) | -1569(1) | 2513(1) | 3931(2) | 28(1) |
| C(8) | -2681(2) | 2723(2) | 3051(2) | 40(1) |
| C(9) | -626(1) | 280(1) | 2385(2) | 30(1) |
| C(10) | -1370(2) | 154(2) | 975(2) | 44(1) |
| C(11) | -141(2) | -580(1) | 3050(2) | 46(1) |
| C(12) | 3550(2) | 540(2) | 6725(2) | 48(1) |
| C(13) | 4539(2) | -88(2) | 6990(4) | 81(1) |
| C(14) | 4450(2) | 1909(2) | 6103(2) | 49(1) |
| C(15) | 4637(2) | 2520(2) | 4934(3) | 60(1) |
| H(1) | -926(15) | 2307(12) | 2360(20) | 32 |
| H(2) | 609(14) | 3055(12) | 5302(18) | 36 |
| H(3) | 2453(15) | 2619(12) | 6320(20) | 39 |
| H(8A) | -2904(18) | 3296(15) | 3250(20) | 59 |
| H(8B) | -2680(18) | 2740(14) | 2070(20) | 59 |
| H(8C) | -3229(19) | 2307(15) | 3210(20) | 59 |
| H(9) | -1032(14) | 604(12) | 3005(18) | 36 |
| H(10A) | -1985(19) | -238(16) | 1050(20) | 66 |
| H(10B) | -953(19) | -98(15) | 330(20) | 66 |
| H(10C) | -1710(18) | 745(16) | 620(20) | 66 |
| H(11A) | -772(19) | -971(16) | 3210(20) | 69 |
| H(11B) | 372(19) | -466(15) | 4010(30) | 69 |
| H(11C) | 270(20) | -890(15) | 2400(30) | 69 |
| H(12A) | 3463(17) | 847(14) | 7640(20) | 58 |
| H(12B) | 2887(19) | 161(14) | 6420(20) | 58 |
| H(13A) | 4450(30) | -540(20) | 7730(40) | 122 |
| H(13B) | 5200(30) | 280(20) | 7380(30) | 122 |
| H(13C) | 4560(30) | -360(20) | 6070(40) | 122 |

| | | | | |
|---|---|---|---|---|
| H(14A) | 4280(18) | 2265(14) | 6910(20) | 59 |
| H(14B) | 5128(19) | 1554(14) | 6530(20) | 59 |
| H(15A) | 3990(20) | 2871(18) | 4560(30) | 90 |
| H(15B) | 5260(20) | 2950(18) | 5250(30) | 90 |
| H(15C) | 4830(20) | 2133(18) | 4190(30) | 90 |

**Table SM3**. Bond lengths [Å] and angles [°] for $C_{15}H_{23}N_3O_4$.

| | | | |
|---|---|---|---|
| O(1)-C(7) | 1.2256(18) | O(2)-C(6) | 1.3707(17) |
| O(2)-C(9) | 1.4750(18) | O(3)-N(2) | 1.2197(19) |
| O(4)-N(2) | 1.2203(19) | N(1)-C(7) | 1.352(2) |
| N(1)-C(1) | 1.4158(19) | N(2)-C(5) | 1.470(2) |
| N(3)-C(4) | 1.427(2) | N(3)-C(14) | 1.469(2) |
| N(3)-C(12) | 1.485(2) | C(1)-C(2) | 1.393(2) |
| C(1)-C(6) | 1.399(2) | C(2)-C(3) | 1.379(2) |
| C(3)-C(4) | 1.394(2) | C(4)-C(5) | 1.397(2) |
| C(5)-C(6) | 1.390(2) | C(7)-C(8) | 1.497(2) |
| CC(9)-C(11) | 1.504(2) | C(9)-C(10) | 1.506(2) |
| C(12)-C(13) | 1.511(3) | C(14)-C(15) | 1.505(3) |
| | | | |
| C(6)-O(2)-C(9) | 115.36(11) | C(7)-N(1)-C(1) | 124.47(13) |
| O(3)-N(2)-O(4) | 125.05(15) | O(3)-N(2)-C(5) | 118.18(14) |
| O(4)-N(2)-C(5) | 116.77(14) | C(4)-N(3)-C(14) | 114.41(15) |
| C(4)-N(3)-C(12) | 110.36(13) | C(14)-N(3)-C(12) | 111.64(14) |
| C(2)-C(1)-C(6) | 119.22(14) | C(2)-C(1)-N(1) | 122.22(14) |
| C(6)-C(1)-N(1) | 118.53(13) | C(3)-C(2)-C(1) | 121.17(15) |
| C(2)-C(3)-C(4) | 121.39(15) | C(3)-C(4)-C(5) | 116.28(14) |
| C(3)-C(4)-N(3) | 125.40(14) | C(5)-C(4)-N(3) | 118.31(15) |
| C(6)-C(5)-C(4) | 123.81(14) | C(6)-C(5)-N(2) | 117.71(13) |
| C(4)-C(5)-N(2) | 118.42(14) | O(2)-C(6)-C(5) | 120.15(13) |
| O(2)-C(6)-C(1) | 121.72(13) | C(5)-C(6)-C(1) | 118.01(13) |
| O(1)-C(7)-N(1) | 122.77(15) | O(1)-C(7)-C(8) | 121.91(15) |
| N(1)-C(7)-C(8) | 115.32(14) | O(2)-C(9)-C(11) | 109.59(14) |
| O(2)-C(9)-C(10) | 105.45(13) | C(11)-C(9)-C(10) | 113.96(16) |
| N(3)-C(12)-C(13) | 112.75(18) | N(3)-C(14)-C(15) | 112.14(17) |

**Table SM4**. Anisotropic displacement parameters [$Å^2x10^3$] for $C_{15}H_{23}N_3O_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+... +2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| O(1) | 41(1) | 56(1) | 22(1) | -3(1) | 8(1) | -3(1) |
| O(2) | 27(1) | 27(1) | 23(1) | -3(1) | 4(1) | -4(1) |
| O(3) | 58(1) | 36(1) | 75(1) | 1(1) | 11(1) | 13(1) |
| O(4) | 46(1) | 70(1) | 37(1) | -8(1) | 9(1) | 16(1) |
| N(1) | 31(1) | 29(1) | 18(1) | 0(1) | 3(1) | 4(1) |
| N(2) | 30(1) | 40(1) | 40(1) | -6(1) | 0(1) | 6(1) |
| N(3) | 27(1) | 52(1) | 34(1) | 5(1) | -1(1) | -1(1) |
| C(1) | 30(1) | 26(1) | 21(1) | 4(1) | 5(1) | -1(1) |
| C(2) | 36(1) | 26(1) | 27(1) | -3(1) | 7(1) | -3(1) |
| C(3) | 35(1) | 37(1) | 26(1) | -3(1) | 2(1) | -9(1) |
| C(4) | 28(1) | 38(1) | 27(1) | 3(1) | 2(1) | -4(1) |
| C(5) | 30(1) | 27(1) | 29(1) | 0(1) | 6(1) | 1(1) |
| C(6) | 29(1) | 25(1) | 21(1) | 1(1) | 4(1) | -4(1) |
| C(7) | 35(1) | 25(1) | 24(1) | -1(1) | 7(1) | -2(1) |
| C(8) | 34(1) | 54(1) | 32(1) | 2(1) | 8(1) | 6(1) |
| C(9) | 32(1) | 28(1) | 31(1) | -2(1) | 8(1) | -6(1) |
| C(10) | 41(1) | 52(1) | 37(1) | -2(1) | 2(1) | -18(1) |
| C(11) | 52(1) | 31(1) | 54(1) | 7(1) | 9(1) | -6(1) |
| C(12) | 35(1) | 63(1) | 45(1) | 14(1) | 4(1) | 3(1) |
| C(13) | 55(2) | 95(2) | 93(2) | 43(2) | 11(2) | 24(1) |
| C(14) | 34(1) | 75(1) | 36(1) | 4(1) | -1(1) | -14(1) |
| C(15) | 48(1) | 78(2) | 55(1) | 12(1) | 10(1) | -19(1) |

**Table SM5**. Torsion angles [°] for $C_{15}H_{23}N_3O_4$.

| | |
|---|---|
| C(7)-N(1)-C(1)-C(2) | -48.1(2) |
| C(7)-N(1)-C(1)-C(6) | 133.78(15) |
| C(6)-C(1)-C(2)-C(3) | -2.1(2) |
| N(1)-C(1)-C(2)-C(3) | 179.80(14) |
| C(1)-C(2)-C(3)-C(4) | 1.3(2) |
| C(2)-C(3)-C(4)-C(5) | 1.6(2) |
| C(2)-C(3)-C(4)-N(3) | -177.54(15) |
| C(14)-N(3)-C(4)-C(3) | -38.6(2) |
| C(12)-N(3)-C(4)-C(3) | 88.3(2) |
| C(14)-N(3)-C(4)-C(5) | 142.33(16) |
| C(12)-N(3)-C(4)-C(5) | -90.78(18) |
| C(3)-C(4)-C(5)-C(6) | -3.7(2) |
| N(3)-C(4)-C(5)-C(6) | 175.47(14) |
| C(3)-C(4)-C(5)-N(2) | 173.66(14) |
| N(3)-C(4)-C(5)-N(2) | -7.2(2) |
| O(3)-N(2)-C(5)-C(6) | -106.88(17) |
| O(4)-N(2)-C(5)-C(6) | 73.79(19) |
| O(3)-N(2)-C(5)-C(4) | 75.60(19) |
| O(4)-N(2)-C(5)-C(4) | -103.73(17) |
| C(9)-O(2)-C(6)-C(5) | 109.52(15) |
| C(9)-O(2)-C(6)-C(1) | -74.34(17) |
| C(4)-C(5)-C(6)-O(2) | 179.19(13) |
| N(2)-C(5)-C(6)-O(2) | 1.8(2) |
| C(4)-C(5)-C(6)-C(1) | 2.9(2) |
| N(2)-C(5)-C(6)-C(1) | -174.47(13) |
| C(2)-C(1)-C(6)-O(2) | -176.13(13) |
| N(1)-C(1)-C(6)-O(2) | 2.0(2) |
| C(2)-C(1)-C(6)-C(5) | 0.1(2) |
| N(1)-C(1)-C(6)-C(5) | 178.25(13) |
| C(1)-N(1)-C(7)-O(1) | 1.7(2) |
| C(1)-N(1)-C(7)-C(8) | -177.33(14) |
| C(6)-O(2)-C(9)-C(11) | -78.95(17) |
| C(6)-O(2)-C(9)-C(10) | 157.97(14) |
| C(4)-N(3)-C(12)-C(13) | 158.3(2) |
| C(14)-N(3)-C(12)-C(13) | -73.3(3) |
| C(4)-N(3)-C(14)-C(15) | -61.6(2) |
| C(12)-N(3)-C(14)-C(15) | 172.20(18) |

**Table SM6**. Observed and calculated structure factors for $C_{15}H_{23}N_3O_4$

| h | k | l | 10Fo | 10Fc | 10s | h | k | l | 10Fo | 10Fc | 10s | h | k | l | 10Fo | 10Fc | 10s | h | k | l | 10Fo | 10Fc | 10s | h | k | l | 10Fo | 10Fc | 10s |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0 | 0 | 1047 | 1093 | 4 | 2 | 6 | 0 | 70 | 67 | 1 | 4 | 13 | 0 | 70 | 72 | 3 | 13 | 2 | 1 | 25 | 12 | 11 | -11 | 6 | 1 | 52 | 53 | 4 |
| 2 | 0 | 0 | 0 | 9 | 1 | 3 | 6 | 0 | 33 | 33 | 2 | 5 | 13 | 0 | 44 | 40 | 3 | -14 | 3 | 1 | 28 | 31 | 7 | -10 | 6 | 1 | 54 | 52 | 4 |
| 3 | 0 | 0 | 77 | 75 | 1 | 4 | 6 | 0 | 136 | 123 | 1 | 6 | 13 | 0 | 0 | 2 | 1 | -13 | 3 | 1 | 0 | 5 | 1 | -9 | 6 | 1 | 30 | 37 | 6 |
| 4 | 0 | 0 | 605 | 605 | 2 | 5 | 6 | 0 | 98 | 89 | 2 | 7 | 13 | 0 | 23 | 18 | 7 | -12 | 3 | 1 | 32 | 25 | 7 | -8 | 6 | 1 | 65 | 69 | 2 |
| 5 | 0 | 0 | 240 | 240 | 1 | 6 | 6 | 0 | 233 | 239 | 2 | 8 | 13 | 0 | 111 | 112 | 2 | -11 | 3 | 1 | 38 | 36 | 6 | -7 | 6 | 1 | 103 | 104 | 1 |
| 6 | 0 | 0 | 390 | 372 | 3 | 7 | 6 | 0 | 46 | 42 | 2 | 9 | 13 | 0 | 79 | 71 | 2 | -10 | 3 | 1 | 44 | 32 | 5 | -6 | 6 | 1 | 159 | 144 | 2 |
| 7 | 0 | 0 | 41 | 58 | 11 | 8 | 6 | 0 | 21 | 9 | 8 | 0 | 14 | 0 | 168 | 167 | 3 | -9 | 3 | 1 | 48 | 45 | 5 | -5 | 6 | 1 | 149 | 143 | 2 |
| 8 | 0 | 0 | 171 | 181 | 3 | 9 | 6 | 0 | 54 | 58 | 3 | 1 | 14 | 0 | 21 | 17 | 13 | -8 | 3 | 1 | 32 | 28 | 4 | -4 | 6 | 1 | 148 | 152 | 1 |
| 9 | 0 | 0 | 105 | 104 | 4 | 10 | 6 | 0 | 81 | 82 | 2 | 2 | 14 | 0 | 116 | 122 | 2 | -7 | 3 | 1 | 329 | 329 | 2 | -3 | 6 | 1 | 48 | 48 | 1 |
| 10 | 0 | 0 | 398 | 395 | 4 | 11 | 6 | 0 | 83 | 72 | 2 | 3 | 14 | 0 | 153 | 154 | 2 | -6 | 3 | 1 | 56 | 44 | 2 | -2 | 6 | 1 | 118 | 115 | 1 |
| 11 | 0 | 0 | 283 | 280 | 3 | 12 | 6 | 0 | 20 | 9 | 15 | 4 | 14 | 0 | 58 | 58 | 3 | -5 | 3 | 1 | 108 | 113 | 1 | -1 | 6 | 1 | 119 | 107 | 1 |
| 12 | 0 | 0 | 0 | 10 | 1 | 13 | 6 | 0 | 26 | 24 | 12 | 5 | 14 | 0 | 0 | 10 | 1 | -4 | 3 | 1 | 309 | 314 | 1 | 0 | 6 | 1 | 12 | 23 | 7 |
| 13 | 0 | 0 | 17 | 38 | 16 | 1 | 7 | 0 | 27 | 37 | 3 | 6 | 14 | 0 | 12 | 6 | 11 | -3 | 3 | 1 | 745 | 730 | 2 | 1 | 6 | 1 | 279 | 271 | 1 |
| 14 | 0 | 0 | 39 | 26 | 7 | 2 | 7 | 0 | 11 | 18 | 11 | 7 | 14 | 0 | 6 | 4 | 6 | -2 | 3 | 1 | 272 | 265 | 1 | 2 | 6 | 1 | 10 | 6 | 10 |
| 1 | 1 | 0 | 409 | 416 | 1 | 3 | 7 | 0 | 206 | 203 | 1 | 8 | 14 | 0 | 0 | 5 | 1 | -1 | 3 | 1 | 185 | 186 | 1 | 3 | 6 | 1 | 13 | 13 | 7 |
| 2 | 1 | 0 | 488 | 497 | 2 | 4 | 7 | 0 | 28 | 25 | 4 | 1 | 15 | 0 | 53 | 53 | 4 | 0 | 3 | 1 | 534 | 527 | 2 | 4 | 6 | 1 | 99 | 108 | 2 |
| 3 | 1 | 0 | 254 | 252 | 1 | 5 | 7 | 0 | 82 | 92 | 3 | 2 | 15 | 0 | 69 | 62 | 3 | 1 | 3 | 1 | 173 | 172 | 1 | 5 | 6 | 1 | 165 | 151 | 2 |
| 4 | 1 | 0 | 71 | 71 | 1 | 6 | 7 | 0 | 171 | 178 | 1 | 3 | 15 | 0 | 31 | 34 | 6 | 2 | 3 | 1 | 343 | 343 | 1 | 6 | 6 | 1 | 121 | 131 | 2 |
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```

| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 2 | 2 | 8 | 179 | 176 | 2 | -9 | 7 | 8 | 0 | 0 | 1 | -4 | 1 | 9 | 131 | 130 | 2 | -2 | 6 | 9 | 115 | 123 | 2 | 4 | 1 | 10 | 15 | 21 | 15 |
| 3 | 2 | 8 | 69 | 73 | 5 | -8 | 7 | 8 | 73 | 77 | 2 | -3 | 1 | 9 | 154 | 161 | 2 | -1 | 6 | 9 | 27 | 20 | 7 | 5 | 1 | 10 | 0 | 21 | 1 |
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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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| 1 | 6 | 10 | 168 | 164 | 2 | -2 | 8 | 10 | 34 | 35 | 5 | -7 | 2 | 11 | 52 | 43 | 5 | 1 | 3 | 11 | 34 | 30 | 7 | | | | | | |

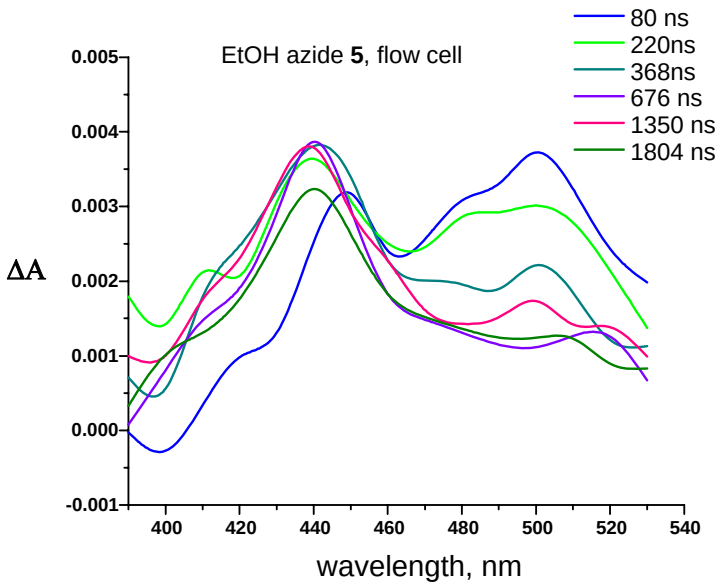
## Laser Flash Photolysis.

### General Procedure for Nanosecond Experiments.

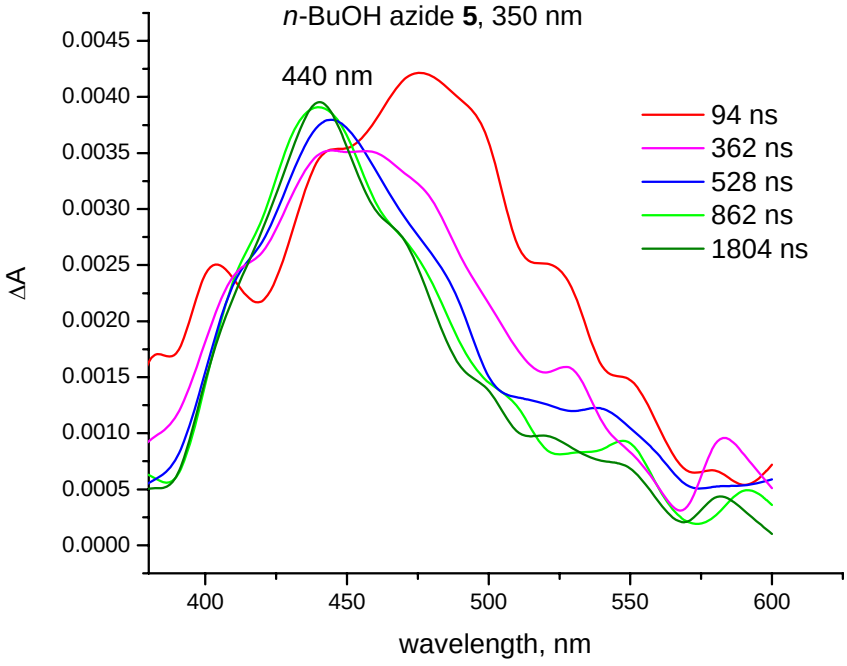
Nanosecond (ns) time-resolved laser flash photolysis was performed on a Proteus Nanosecond Transient Absorption spectrometer (Ultrafast Systems) equipped with a 150 W Xe-arc lamp (Newport), a Bruker Optics monochromator and photodiode detectors (DET 10A and DET 10C, Thorlabs). Excitation at 350 nm from a computer-controlled Nd:YAG laser/OPO system from Opotek (Vibrant LD 355 II) operating at 10 Hz was directed to the sample with an optical absorbance of 0.64 at the excitation wavelength. The data consisting of a 128-shot average were analyzed by Origin 7.1 software. The absorbance of the sample solutions is typically 0.6-0.7 at the excitation wavelength, and the sample volume was 25 mL. Pump pulse energy is about 5 mJ at the sample position; the number of spectra acquisitions was 16 to 32. All experiments are performed at room temperature and under nitrogen.

**Nanosecond Transient Spectra Following Irradiation of Azide 5 in Presence of Various Nucleophiles**

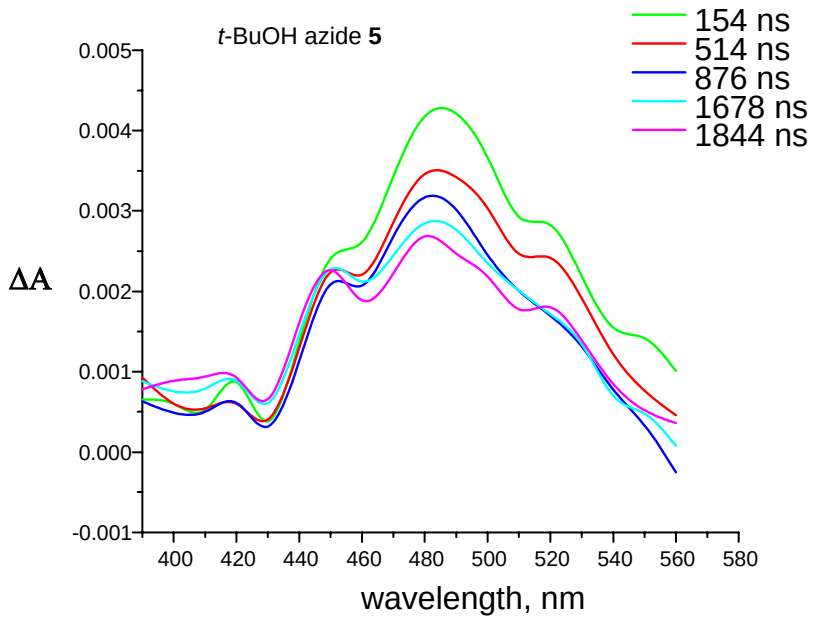
a) **Transient absorption spectra in ethanol**



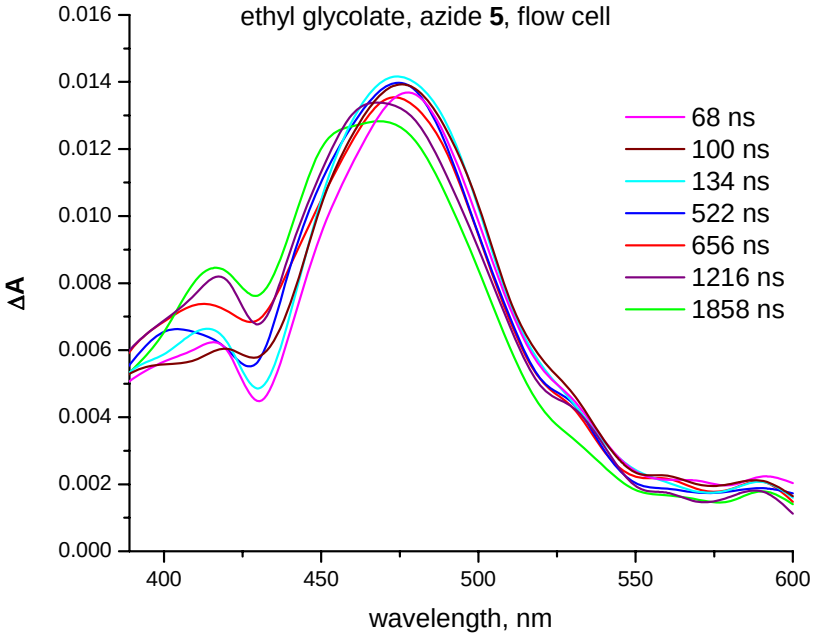

b) **Transient absorption spectra in *n*-butyl alcohol**



c) **Transient absorption spectra in *t*-butyl alcohol**




d) **Transient absorption spectra in ethyl glycolate**



**General Procedure for Ultrafast Transient Absorption Measurements.**

The femtosecond time-resolved transient absorption spectrometer used in this work is based on the combination of a Ti:Sapphire regenerative amplifier (Hurricane, Spectra Physics) and two interchangeable, computer-controlled pump and probe TOPAS-C optical parametrical amplifiers (Light Conversion Lt.). The amplified output is a train of 800-nm laser pulses with pulse widths of ~100 fs and pulse energy of 0.92 mJ with a repetition rate of 1 kHz. The amplified output is divided by a beam splitter into two beams. One beam (50%) pumps a TOPAS-C pump amplifier to generate 305, 350 and 420-nm light pulses used for sample excitation. The typical excitation energy is 3-4 μJ $pulse^{-1}$, which is focused into a 300 μm diameter spot at the sample position. The second beam is attenuated to ~4 μJ $pulse^{-1}$ and focused onto a 3-mm $CaF_2$ window to produce a white light continuum light. The white-light continuum beam is further split into reference and probe beams, the latter of which is focused to a 100 μm diameter spot and overlapped with the pump beam at an angle of $8^o$ at the sample position. Alternatively, for the 305 pump, the other half of the 800-nm amplified output was delivered to the TOPAS-C probe amplifier to produce UV-probe pulses tunable from 280 to 390 nm. After the sample, the reference and probe beams are sent to a monochromator/spectrograph (Spectra-Pro 2358, Acton Research) and registered on a 512-pixel dual diode array for simultaneous accumulation of kinetic traces within 274 nm spectral windows (white-light continuum) or two Si-photodiodes (TOPAS-C probe). The excitation beam is chopped (on/off) at 500 kHz repetition rate. Probe and reference diode array signals ($I_{pr}$, $I_{ref}$) are read after each laser shot for adjacent pairs of excitation on and off pulses and the transient absorption for each pair of pulses is obtained as follows: $\Delta A = \log (I_{pr}/I_{ref})_{on} - \log (I_{pr}/I_{ref})_{off}$. Per a kinetic trace, 300 pairs of excitation on/off ΔA points are collected at ~120 delay time positions between –10 ps and 1200 ps. Spectral data obtained in the complementary 274 nm ranges are averaged for about 10 successive scans of the delay line (total acquisition time, 45 min), and subsequently linked together to yield the resultant ΔA spectra from 345 to 765 nm. Time zero at different probe wavelengths is obtained by using the non-resonant or two-photon absorption pump-probe signals from neat solvents. The resultant group velocity dispersion curve (chirp rate, 2.0 ×

$10^{-5}$ $fs^{-2}$) is used to correct the ΔA spectra. A strong Gaussian-like emission feature (165 ± 15 fs fwhm) due to stimulated Raman scattering (Raman-active $CH_2$ symmetrical vibrational mode, v = 2853 $cm^{-1}$) observed in neat cyclohexane in the Stokes region with respect to the excitation wavelength delivers a cross-correlation signal between pump and probe pulses. The pump light polarization was set using a Berek compensator to be at 54.7$^o$ with respect to the probe light polarization, so all measurements are performed at magic angle polarization conditions. The samples were circulated through a Spectrosil quarz flow cell with a 0.2 or 0.5 mm path length (Starna) at a linear velocity of 0.6-1.6 m $s^{-1}$ to avoid secondary excitation. All samples were prepared in 25 or 50 mL of solvent with typical sample absorbance in the 0.4-0.9 range at the excitation wavelength per 0.2 or 0.5 mm thickness of the flow cell used.

The possibility that the solvent contributes to the measured transient absorption is checked by measuring the ΔA spectra from the neat solvent immediately prior or subsequent to the azide experiment under the same excitation conditions. The typical solvent (*i*-PrOH) contribution is illustrated in Fig. SM1 for excitation at 305 nm. We conclude that the short-time ΔA spectra (from -100 to 100 fs) are dominated (under our conditions: relatively low photon energy, $\lambda_{exc}$ = 420, 350, and 305 nm) by cross-phase modulation and impulsive stimulated Raman scattering, and, as the one pump/one probe photon process, vary approximately linearly with the pump intensity. The subsequent ΔA spectra (time delay, 200 fs and longer) for the neat solvent are due to the formation of product(s) via non-linear, typically, two-pump-photon absorption. As the result, when azide **5** is added to the solution, the excitation intensity is reduced by the solute absorption to the extent that the solvent contribution to the transient absorption measured after time delay of 200 fs becomes negligible.

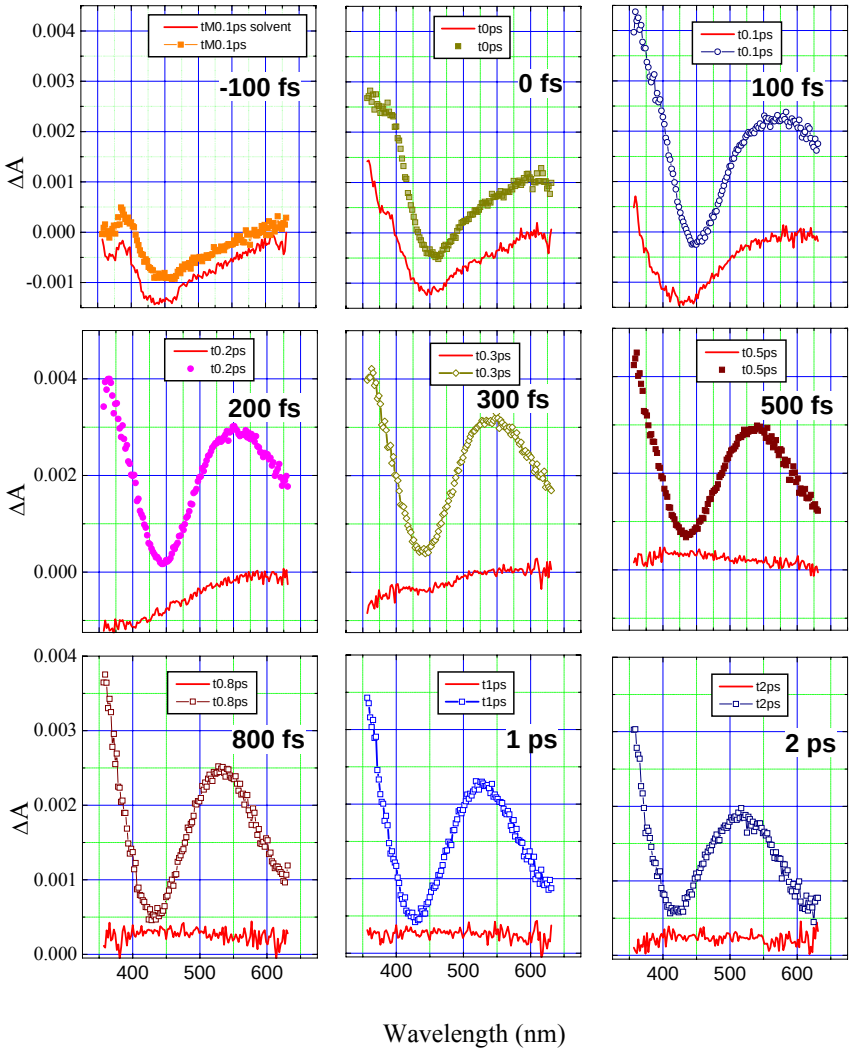



**Figure SM1.** Transient absorption (ΔA) spectra obtained after 305-nm excitation (3.8 μJ pulse$^{-1}$) of neat *i*-PrOH (lines) and the solution of 1.2 mM azide **5** in *i*-PrOH (lines and symbols) circulated through a 0.2 mm cell. The time delays (picoseconds) are shown inside each window. The *i*-PrOH contribution to the total ΔA spectra is minor at delay times equal or longer than 100 fs.

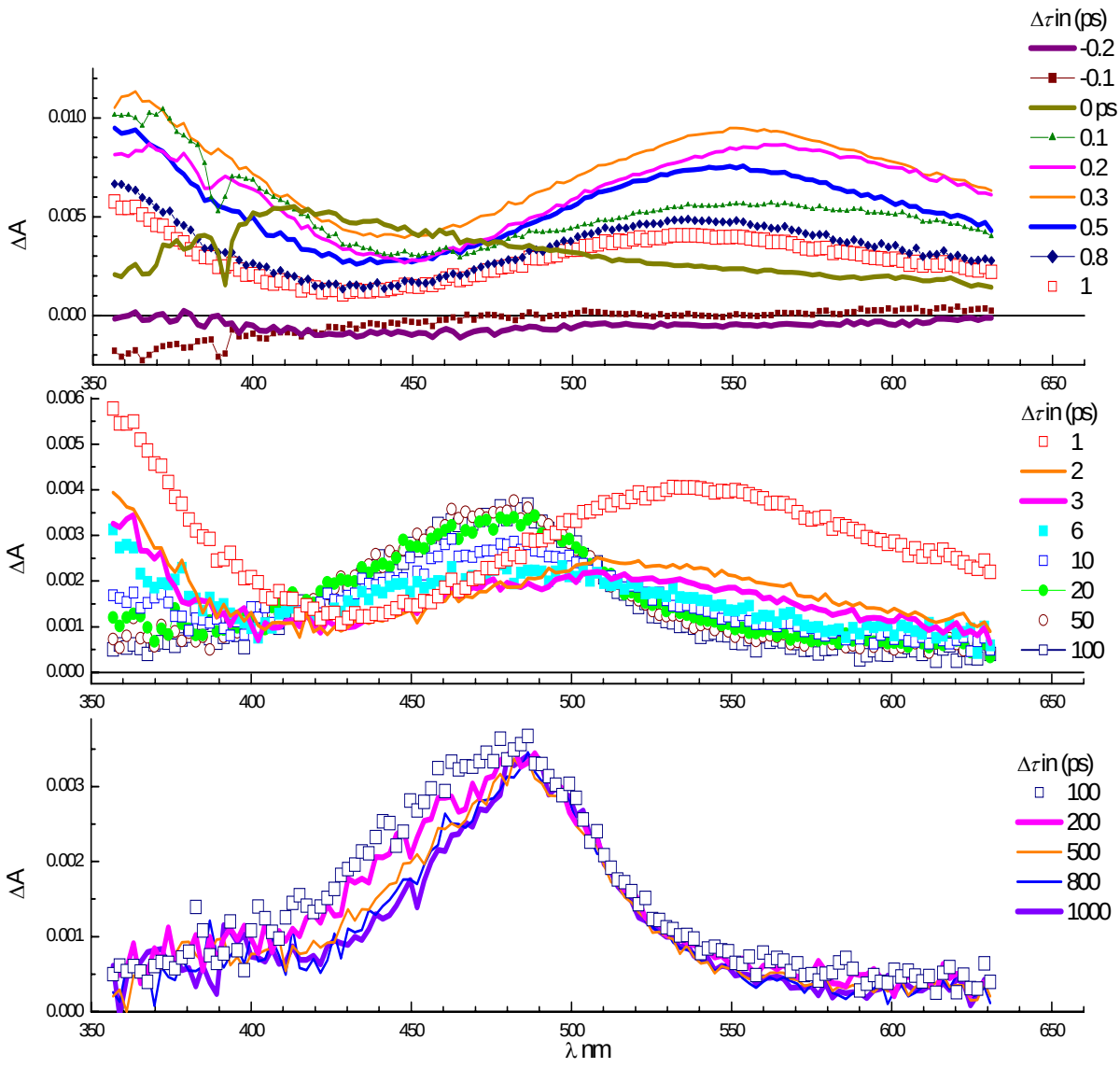
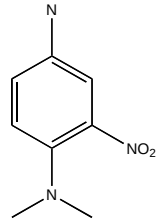



**Figure SM2.** Transient absorption (ΔA) spectra of azide **5** (9.3 mM) in acetonitrile upon 350-nm excitation. Delay times between the probe and pump pulses are shown in the legend. The solution was flowed through a 0.5 mm flow cell and excited with a pulse energy of 5.8 μJ-pulse$^{-1}$.

**Theoretical Calculations for:**

**Open- and Closed-Shell Singlet Nitrenes**

**Table SM7.** CASPT2(10,10)/pVDZ//CASSCF(10,10)/pVDZ calculations for the nitrenes




closed shell
root 2

CASPT2(10,10)/pVDZ for **closed-shell** nitrene (root 2).

XYZ Coordinates (Bohrs, left side; Angstroms right side)
Closed shell:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | C1 | 4.688166 | -0.558368 | 0.078600 | 2.480871 | -0.295476 | 0.041593 |
| 2 | C2 | -0.614314 | -1.127791 | 0.140920 | -0.325081 | -0.596801 | 0.074572 |
| 3 | C3 | 3.138530 | 1.490553 | -0.639245 | 1.660839 | 0.788767 | -0.338274 |
| 4 | C4 | 3.599195 | -2.879555 | 0.820107 | 1.904612 | -1.523795 | 0.433982 |
| 5 | C5 | 0.979221 | -3.143197 | 0.841267 | 0.518181 | -1.663309 | 0.445179 |
| 6 | C6 | 0.538823 | 1.153765 | -0.581126 | 0.285133 | 0.610546 | -0.307519 |
| 7 | C7 | -4.428506 | -1.034724 | 2.611209 | -2.343465 | -0.547553 | 1.381793 |
| 8 | C8 | -4.296636 | -3.401250 | -1.322612 | -2.273682 | -1.799864 | -0.699896 |
| 9 | H1 | 3.993350 | 3.252109 | -1.217715 | 2.113190 | 1.720942 | -0.644387 |
| 10 | H2 | 4.839102 | -4.410789 | 1.360836 | 2.560743 | -2.334089 | 0.720123 |
| 11 | H3 | 0.137505 | -4.909758 | 1.413220 | 0.072765 | -2.598132 | 0.747844 |
| 12 | H4 | -3.408062 | -3.462487 | -3.175055 | -1.803469 | -1.832270 | -1.680167 |
| 13 | H5 | -6.315752 | -3.109901 | -1.581103 | -3.342152 | -1.645689 | -0.836684 |
| 14 | H6 | -3.719073 | 0.660489 | 3.524833 | -1.968049 | 0.349516 | 1.865262 |
| 15 | H7 | -4.044936 | -2.666757 | 3.830108 | -2.140488 | -1.411187 | 2.026806 |
| 16 | H8 | -6.464502 | -0.848730 | 2.389437 | -3.420867 | -0.449129 | 1.264436 |
| 17 | H9 | -4.024922 | -5.231920 | -0.393059 | -2.129897 | -2.768613 | -0.207998 |
| 18 | O1 | -2.068405 | 4.454837 | 0.339755 | -1.094553 | 2.357398 | 0.179791 |
| 19 | O2 | -1.251401 | 3.719059 | -3.533707 | -0.662213 | 1.968042 | -1.869957 |
| 20 | N1 | 7.250891 | -0.283450 | 0.051996 | 3.837007 | -0.149995 | 0.027515 |
| 21 | N2 | -1.065673 | 3.276478 | -1.318038 | -0.563930 | 1.733838 | -0.697476 |
| 22 | N3 | -3.288119 | -1.289846 | 0.117617 | -1.739998 | -0.682557 | 0.062240 |

| Root (CASSCF) | E | ΔE (hart) CASPT2 | ΔE kcal/mol CASPT2 | nm CASPT2 | *f* CASSCF |
|---|---|---|---|---|---|
| 2 | -623.0004087 | | | | |
| 3 | -622.9522424 | 0.048167609 | 30.22565639 | 946 | 3.83E-06 |
| 4 | -622.957506 | 0.042903989 | 26.92268226 | 1062 | 1.97E-02 |
| 5 | -622.9362512 | 0.064158814 | 40.26029712 | 710 | 7.19E-06 |
| 6 | -622.9288808 | 0.071529152 | 44.88525805 | 637 | 2.15E-06 |
| 7 | -622.9122053 | 0.088204673 | 55.34931461 | 517 | 2.90E-07 |
| 8 | -622.8662468 | 0.134163227 | 84.18876632 | 340 | 6.46E-03 |
| 9 | -622.9173901 | 0.083019922 | 52.09583113 | 549 | 5.50E-05 |
| 10 | -622.8374619 | 0.162948077 | 102.2515475 | 280 | 1.90E-05 |

Notes:

-The roots by CASSCF are not in proper order. CASPT2 gives the roots the likely proper order.
-Solving for more roots than eleven we start to see intruder states corrupting some of the roots.
-Trying to increase the active space beyond 10,10 leads to resource allocation problems
- To get geometric convergence, we used a state-average calculation of the opened-shell and closed-shell roots to avoid convergence failures, following the closed-shell root.

Same method/active space/basis set as above. For ground-state **opened-shell** nitrene.

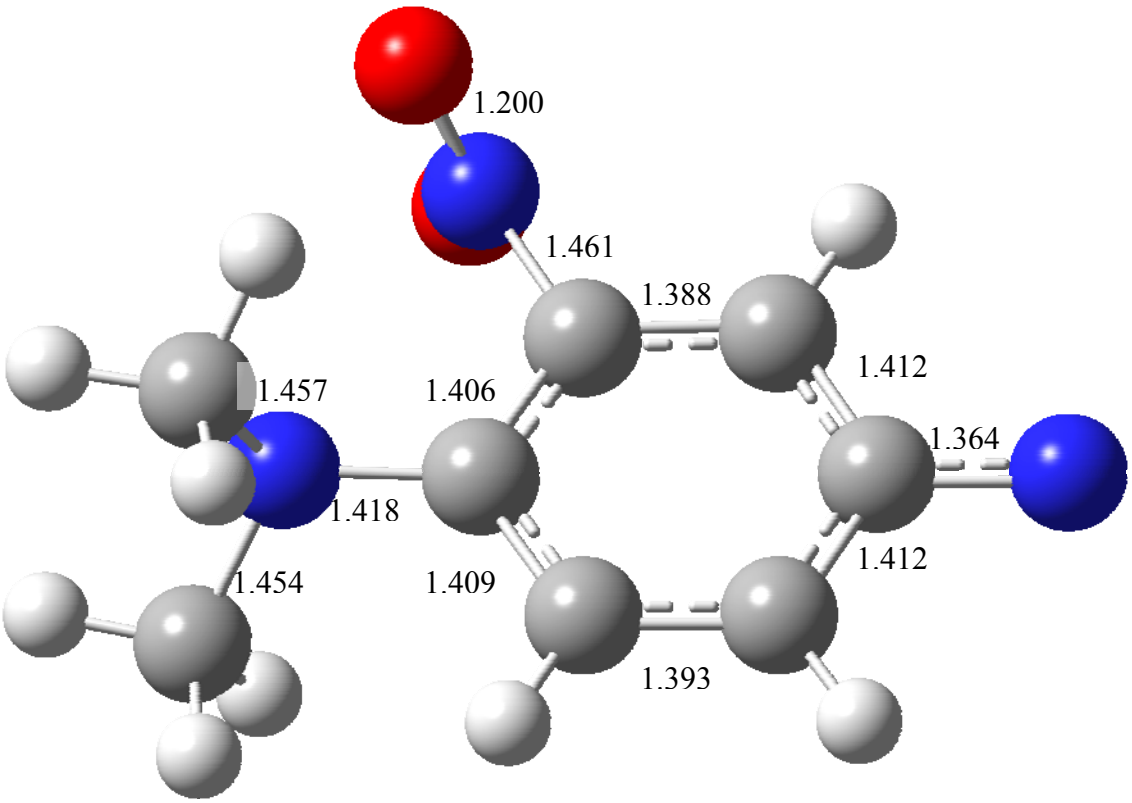
XYZ Coordinates (Bohrs, left side; Angstroms right side)

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | C1 | 4.707890 | -0.551230 | 0.075608 | 2.491308 | -0.291698 | 0.040010 |
| 2 | C2 | -0.645925 | -1.116928 | 0.142850 | -0.341809 | -0.591053 | 0.075593 |
| 3 | C3 | 3.144752 | 1.433397 | -0.634137 | 1.664131 | 0.758521 | -0.335571 |
| 4 | C4 | 3.585181 | -2.809216 | 0.807857 | 1.897196 | -1.486573 | 0.427499 |
| 5 | C5 | 0.949549 | -3.094659 | 0.846341 | 0.502479 | -1.637623 | 0.447864 |
| 6 | C6 | 0.522929 | 1.123665 | -0.587984 | 0.276722 | 0.594618 | -0.311148 |
| 7 | C7 | -4.444189 | -1.066241 | 2.629334 | -2.351764 | -0.564230 | 1.391384 |
| 8 | C8 | -4.317426 | -3.395658 | -1.316223 | -2.284683 | -1.796905 | -0.696515 |
| 9 | H1 | 3.943113 | 3.215070 | -1.228535 | 2.086605 | 1.701342 | -0.650113 |
| 10 | H2 | 4.775666 | -4.372112 | 1.359538 | 2.527174 | -2.313622 | 0.719437 |
| 11 | H3 | 0.152210 | -4.876398 | 1.422735 | 0.080546 | -2.580479 | 0.752879 |
| 12 | H4 | -3.444972 | -3.435515 | -3.176342 | -1.823001 | -1.817996 | -1.680848 |
| 13 | H5 | -6.340940 | -3.123054 | -1.554360 | -3.355481 | -1.652649 | -0.822532 |
| 14 | H6 | -3.735609 | 0.619063 | 3.560233 | -1.976799 | 0.327594 | 1.883994 |
| 15 | H7 | -4.046878 | -2.712103 | 3.823965 | -2.141516 | -1.435183 | 2.023555 |
| 16 | H8 | -6.481312 | -0.886625 | 2.420783 | -3.429763 | -0.469182 | 1.281023 |
| 17 | H9 | -4.018189 | -5.229036 | -0.401597 | -2.126334 | -2.767087 | -0.212516 |
| 18 | O1 | -2.106758 | 4.418986 | 0.292726 | -1.114849 | 2.338427 | 0.154904 |
| 19 | O2 | -1.103041 | 3.784047 | -3.554053 | -0.583704 | 2.002431 | -1.880724 |
| 20 | N1 | 7.426717 | -0.268030 | 0.045323 | 3.930050 | -0.141836 | 0.023984 |
| 21 | N2 | -1.030260 | 3.276733 | -1.344559 | -0.545190 | 1.733973 | -0.711510 |
| 22 | N3 | -3.318025 | -1.285392 | 0.128743 | -1.755823 | -0.680200 | 0.068128 |

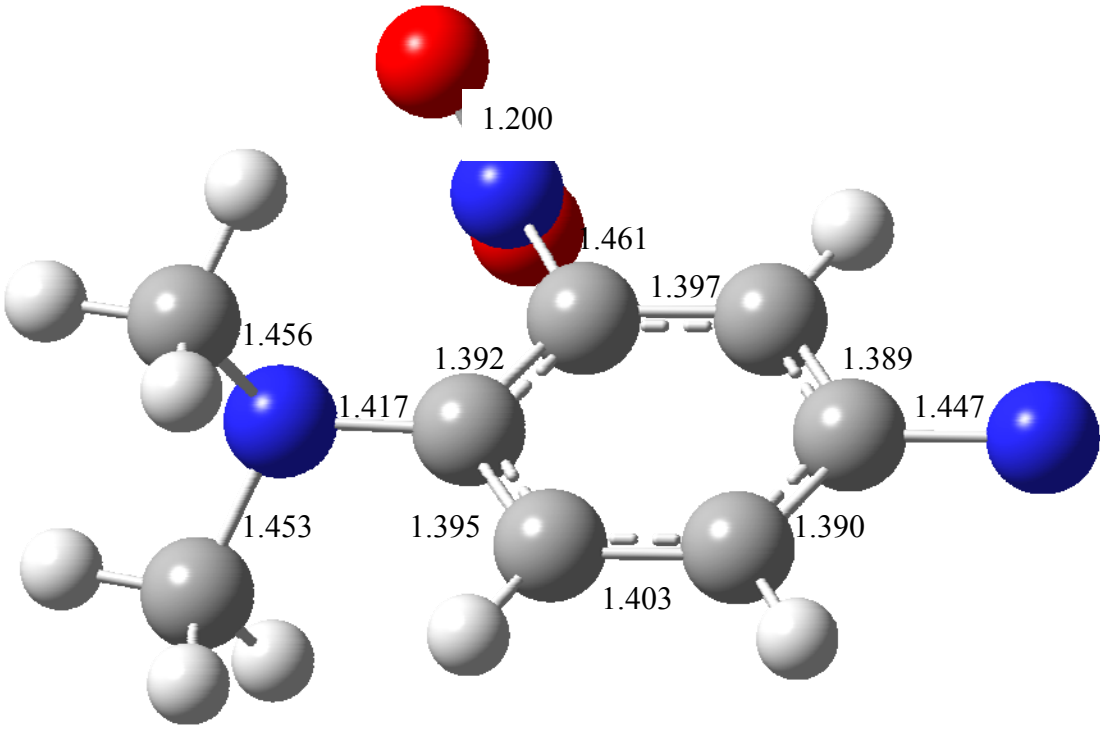
| Root CASSCF | E CASPT2 | ΔE (hart) CASPT2 | ΔE kcal/mol CASPT2 | nm CASPT2 | *f* CASSCF |
|---|---|---|---|---|---|
| 1 | -623.0077056223 | | | | |
| 2 | -622.9916134416 | 0.016096558 | 10.10075136 | 2831 | 0.47791931E-08 |
| 3 | -622.9484063380 | 0.059303662 | 37.21364094 | 769 | 0.10588284E-05 |
| 4 | -622.9277199544 | 0.079990046 | 50.19455351 | 570 | 0.28377127E-02 |
| 5 | -622.9382773664 | 0.069432634 | 43.56967191 | 656 | 0.37963539E-04 |
| 6 | -622.9212256357 | 0.086484364 | 54.26980344 | 526 | 0.49857005E-05 |
| 7 | -622.8956393032 | 0.112070697 | 70.32548295 | 407 | 0.13596426E-02 |
| 8 | Different ref. | 0.10345044 | 64.91618573 | 440 | 0.28299223E-02 |
| 9 | Different ref. | 0.094363394 | 59.21397318 | 483 | 0.32276268E-01 |
| 10 | Different ref. | 0.138693697 | 87.03168193 | 329 | 0.17899616E-04 |

--Solved using both 10 SA roots and 7 SA roots. The first 7 roots are from the first calculation (7 roots) since fewer roots give higher quality individual wavefunctions (although the values between the two calcs. did not change in any meaningful way). Roots 8-10 come from the 10 root SA calculation. In the 10 root SA calculation there was 1 root (7) that was corrupted by an intruder state. All the other roots were fine.
- To get geometric convergence, We used a state-average calculation of the open-shell and closed-shell roots to avoid convergence failures, following the open-shell root.

**Figure SM3**. A) Closed-Shell Nitrene optimized at CASSCF(10,10)/pVDZ level of theory using MOLCAS suite of programs



B) Opened-Shell Nitrene optimized at CASSCF(10,10)/pVDZ level of theory using MOLCAS suite of programs



**Geometry of Azide 5 and Its Methyl Analog**

**Table SM8. Optimized Coordinates of Azide 5**

RI-CC2/TZVP level of theory

| | | | |
|---|---|---|---|
| C | 1.0592499 | -1.9736836 | -0.2470072 |
| C | -0.3102988 | -1.7493012 | -0.1523434 |
| C | -0.8471279 | -0.4561988 | -0.0029944 |
| C | 0.0972976 | 0.5788899 | 0.1341844 |
| C | 1.4669672 | 0.3928764 | 0.0060254 |
| C | 1.9544519 | -0.9072213 | -0.1371428 |
| N | 3.3369659 | -1.2087978 | -0.2539377 |
| N | 4.1571727 | -0.3003500 | -0.0413188 |
| N | 5.0594913 | 0.4368427 | 0.1086128 |
| N | -0.3483068 | 1.9443897 | 0.4413937 |
| O | -1.0328555 | 2.0990815 | 1.4797803 |
| O | 0.0161820 | 2.8594161 | -0.3428841 |
| N | -2.2185009 | -0.1788876 | 0.0543191 |
| C | -2.7219302 | 0.6605569 | -1.0436375 |
| C | -3.0989177 | -1.2845365 | 0.4215932 |
| H | 1.4464988 | -2.9790828 | -0.3620598 |
| H | -0.9813186 | -2.5904473 | -0.2704808 |
| H | 2.1299082 | 1.2480586 | 0.0869487 |
| H | -2.1119654 | 1.5615211 | -1.1140540 |
| H | -3.7276241 | 0.9841301 | -0.7645461 |
| H | -4.1180363 | -0.9096648 | 0.3023767 |
| H | -3.0025002 | -2.1486291 | -0.2526495 |
| C | -2.8835204 | -1.7069363 | 1.8671559 |
| H | -3.5760863 | -2.5088872 | 2.1307108 |
| H | -1.8674224 | -2.0674554 | 2.0308927 |
| H | -3.0546759 | -0.8568814 | 2.5275092 |
| C | -2.7356901 | -0.0498690 | -2.3934961 |
| H | -3.3873139 | -0.9251827 | -2.3768434 |
| H | -3.0982180 | 0.6263526 | -3.1708491 |
| H | -1.7280569 | -0.3715388 | -2.6653756 |

TD-B3LYP/TZVP level of theory

| | | | |
|---|---|---|---|
| C | 1.1639118 | -1.9858388 | -0.2087298 |
| C | -0.2001637 | -1.7966073 | -0.1770996 |
| C | -0.7888386 | -0.5144937 | -0.0672427 |
| C | 0.1282775 | 0.5515030 | 0.0965474 |
| C | 1.5092892 | 0.3754533 | 0.0118210 |
| C | 2.0392859 | -0.8954031 | -0.1388766 |
| N | 3.4241988 | -1.1876012 | -0.2095173 |
| N | 4.2322066 | -0.2630322 | -0.1590766 |
| N | 5.0733287 | 0.4924740 | -0.1258164 |
| N | -0.2981882 | 1.9083954 | 0.4872031 |
| O | -1.2722441 | 2.0214338 | 1.2229581 |
| O | 0.3844422 | 2.8527958 | 0.0990499 |
| N | -2.1568791 | -0.3608365 | -0.1457176 |
| C | -2.7717423 | 0.7465488 | -0.8815588 |
| C | -3.0516372 | -1.4135933 | 0.3408282 |
| H | 1.5757620 | -2.9814871 | -0.3144861 |

| | | | |
|---|---|---|---|
| H | -0.8437373 | -2.6578006 | -0.2854195 |
| H | 2.1397395 | 1.2494183 | 0.1093635 |
| H | -1.9918947 | 1.4444409 | -1.1744348 |
| H | -3.4482772 | 1.2978137 | -0.2209015 |
| H | -4.0669640 | -1.0547854 | 0.1742834 |
| H | -2.9587629 | -2.3320590 | -0.2530752 |
| C | -2.8786427 | -1.7201950 | 1.8282806 |
| H | -3.6070117 | -2.4728717 | 2.1392523 |
| H | -1.8832127 | -2.1058071 | 2.0523999 |
| H | -3.0323897 | -0.8190659 | 2.4237796 |
| C | -3.5005608 | 0.2985475 | -2.1523610 |
| H | -4.3333633 | -0.3743144 | -1.9412956 |
| H | -3.9078897 | 1.1733850 | -2.6649838 |
| H | -2.8163946 | -0.2089251 | -2.8358667 |

**Table SM9. Optimized Coordinates of Methyl Analog of Azide 5**

RI-CC2/TZVP level of theory

| | | | |
|---|---|---|---|
| C | 0.9243118 | -2.0298696 | -0.0818867 |
| C | -0.4624294 | -1.9641624 | -0.0801889 |
| C | -1.1542076 | -0.7348545 | 0.0094465 |
| C | -0.3413492 | 0.4208205 | -0.0618201 |
| C | 1.0491274 | 0.3756659 | -0.0504141 |
| C | 1.6886110 | -0.8604153 | -0.0984167 |
| N | 3.1020402 | -1.0101485 | -0.1205497 |
| N | 3.8010818 | -0.0058332 | 0.0930678 |
| N | 4.6067960 | 0.8344326 | 0.2522283 |
| N | -0.9324724 | 1.7560937 | -0.2192781 |
| O | -1.8488202 | 1.8786512 | -1.0695708 |
| O | -0.4530332 | 2.6917139 | 0.4705593 |
| N | -2.5306239 | -0.6645567 | 0.0732747 |
| C | -3.3034963 | -1.8759576 | -0.1212863 |
| C | -3.1630194 | 0.3120595 | 0.9550049 |
| H | 1.4248461 | -2.9901138 | -0.1192900 |
| H | -1.0190663 | -2.8903409 | -0.0328981 |
| H | 1.6028932 | 1.3080374 | -0.0756080 |
| H | -2.9513923 | -2.4074079 | -1.0048391 |
| H | -3.2672365 | -2.5501838 | 0.7461741 |
| H | -4.3414543 | -1.5907012 | -0.2930106 |
| H | -3.7978169 | 1.0012096 | 0.3938077 |
| H | -3.7698649 | -0.2120750 | 1.7000514 |
| H | -2.4118399 | 0.8892431 | 1.4936182 |

TD-B3LYP/TZVP level of theory ($S_0$ State)

| | | | |
|---|---|---|---|
| C | -1.1740896 | 1.5035963 | 0.0686149 |
| C | -1.6430256 | 0.2056917 | 0.0784715 |
| C | -0.7842556 | -0.9162382 | 0.0017160 |
| C | 0.6011678 | -0.6100635 | -0.0126815 |
| C | 1.0758375 | 0.6961658 | -0.0743196 |
| C | 0.1976809 | 1.7658895 | -0.0315670 |
| N | 0.7676805 | 3.0630485 | -0.0746246 |
| N | 0.0253800 | 4.0399142 | -0.0234104 |
| N | -0.5448406 | 5.0168777 | 0.0157888 |

```
N     1.6411383   -1.6369285    0.1694452
O     1.3898964   -2.5855604    0.9068082
O     2.7252992   -1.4582322   -0.3779813
N    -1.3013608   -2.1852951   -0.0874176
C    -2.6786298   -2.4467826    0.2952791
C    -0.6636113   -3.2379382   -0.8673166
H    -1.8873699    2.3184837    0.1157416
H    -2.7114878    0.0481733    0.0985682
H     2.1419342    0.8675949   -0.1105008
H    -2.9418962   -1.8888438    1.1936909
H    -3.3978799   -2.2019163   -0.4989180
H    -2.7785555   -3.5076724    0.5260812
H    -0.1985329   -3.9969338   -0.2337614
H    -1.4161980   -3.7117905   -1.5035347
H     0.1013644   -2.8223119   -1.5221189
```

TD-B3LYP/TZVP level of theory ($S_1$ State)

```
C     1.0323745   -1.9898684    0.0855354
C    -0.3354952   -1.9624901    0.0880328
C    -1.0738700   -0.7380799    0.0550811
C    -0.3143674    0.4902375    0.0000512
C     1.0490420    0.4380798   -0.0282268
C     1.7511610   -0.7835694    0.0277449
N     3.1327633   -0.8928502    0.0208887
N     3.8319614    0.1319601   -0.0209329
N     4.6003766    0.9536115   -0.0557430
N    -0.9068330    1.7971829   -0.0942326
O    -1.5244611    2.0128173   -1.2195884
O    -1.2639351    2.2767012    1.0722852
N    -2.4262381   -0.7956653    0.0355650
C    -3.1117440   -2.0555345   -0.2546553
C    -3.3422644    0.3184225    0.3240819
H     1.5714995   -2.9258945    0.1399604
H    -0.8624110   -2.9011349    0.1603413
H     1.5763756    1.3807207   -0.1003848
H    -2.5940410   -2.6155980   -1.0304053
H    -3.2019145   -2.6735798    0.6445968
H    -4.1121122   -1.8185740   -0.6082192
H    -3.7300001    0.7181837   -0.6131015
H    -4.1609700   -0.0902032    0.9190431
H    -2.8406453    1.1109221    0.8699607
```

TD-B3LYP/TZVP level of theory ($S_2$ State)

```
C     1.0154213   -1.9841922    0.0650026
C    -0.3516014   -1.9709295    0.0811258
C    -1.1005280   -0.7581716    0.0621010
C    -0.3446598    0.4616907    0.0132942
C     1.0225985    0.4430491   -0.0166340
C     1.7478748   -0.7738916    0.0181684
N     3.1069876   -0.8975304    0.0070645
N     3.7630429    0.2433549    0.0033002
N     4.7848748    0.7552621   -0.0166327
N    -1.0015498    1.7482151   -0.1036202
O    -1.4361979    2.0560040   -1.2511930
```

| | | | |
|---|---|---|---|
| O | -1.1815087 | 2.3978588 | 0.9706727 |
| N | -2.4596281 | -0.8072460 | 0.0556532 |
| C | -3.1597606 | -2.0343253 | -0.3126014 |
| C | -3.3408794 | 0.2911591 | 0.4696906 |
| H | 1.5616294 | -2.9166525 | 0.1118401 |
| H | -0.8736558 | -2.9127552 | 0.1631981 |
| H | 1.5484066 | 1.3848554 | -0.0865928 |
| H | -2.6080485 | -2.5862533 | -1.0699502 |
| H | -3.3281975 | -2.6786410 | 0.5574964 |
| H | -4.1287540 | -1.7608482 | -0.7273392 |
| H | -3.6804253 | 0.8577761 | -0.3992680 |
| H | -4.2024870 | -0.1509058 | 0.9713208 |
| H | -2.8372630 | 0.9644926 | 1.1557087 |

**Table SM10. Optimized Coordinates of Methyl Analog of the Triplet Nitrene**

TD-B3LYP/TZVP level of theory

| | | | |
|---|---|---|---|
| C | 2.0066929 | -1.5444510 | -0.0781162 |
| C | 0.6645997 | -1.8025736 | -0.0938759 |
| C | -0.3194931 | -0.7765415 | 0.0021855 |
| C | 0.1895184 | 0.5537492 | 0.0187109 |
| C | 1.5370879 | 0.8383302 | 0.0804674 |
| C | 2.5067274 | -0.2043422 | 0.0383900 |
| N | 3.7988185 | 0.0526079 | 0.0835201 |
| N | -0.6876381 | 1.7272383 | -0.1745650 |
| O | -1.6161241 | 1.6166439 | -0.9689445 |
| O | -0.4039548 | 2.7594107 | 0.4213318 |
| N | -1.6426130 | -1.0985272 | 0.1172073 |
| C | -2.1283945 | -2.4129828 | -0.2781021 |
| C | -2.6017722 | -0.2831090 | 0.8530757 |
| H | 2.7197716 | -2.3572005 | -0.1273650 |
| H | 0.3396142 | -2.8328410 | -0.1225179 |
| H | 1.8615410 | 1.8688242 | 0.1051760 |
| H | -1.6027741 | -2.7690025 | -1.1627700 |
| H | -2.0280450 | -3.1566267 | 0.5227774 |
| H | -3.1849631 | -2.3277144 | -0.5329572 |
| H | -3.2707851 | 0.2654782 | 0.1863670 |
| H | -3.1930893 | -0.9385167 | 1.4974814 |
| H | -2.0880220 | 0.4303498 | 1.4951250 |

**Table SM11. Optimized Coordinates of Methyl Analog of Nitrogen Radical**

TD-B3LYP/TZVP level of theory

| | | | |
|---|---|---|---|
| C | 1.9764986 | -1.5472116 | -0.0725900 |
| C | 0.6334955 | -1.8081566 | -0.0861702 |
| C | -0.3466538 | -0.7795969 | 0.0052678 |
| C | 0.1695942 | 0.5505579 | 0.0202427 |
| C | 1.5153581 | 0.8253855 | 0.0733004 |
| C | 2.4935599 | -0.2131332 | 0.0345574 |
| N | 3.7768673 | 0.1237791 | 0.0790197 |
| N | -0.7070671 | 1.7252526 | -0.1741070 |
| O | -1.6162569 | 1.6236472 | -0.9920456 |

| | | | |
|---|---|---|---|
| O | -0.4442000 | 2.7471293 | 0.4471012 |
| N | -1.6726379 | -1.0938146 | 0.1153610 |
| C | -2.1607808 | -2.4091514 | -0.2732791 |
| C | -2.6318463 | -0.2709399 | 0.8430854 |
| H | 2.6792352 | -2.3721773 | -0.1195488 |
| H | 0.3046905 | -2.8375416 | -0.1079855 |
| H | 1.8546905 | 1.8515075 | 0.0897665 |
| H | -1.6457420 | -2.7640121 | -1.1648003 |
| H | -2.0478957 | -3.1529758 | 0.5259020 |
| H | -3.2208843 | -2.3264520 | -0.5133173 |
| H | -3.3021368 | 0.2682233 | 0.1700339 |
| H | -3.2222617 | -0.9188819 | 1.4960109 |
| H | -2.1191226 | 0.4504646 | 1.4763232 |
| H | 4.3574773 | -0.7163108 | 0.0283982 |

**Table SM12. Optimized Coordinates of Methyl Analog of the Nitrenium Ion 10**

TD-B3LYP/TZVP level of theory

| | | | |
|---|---|---|---|
| C | 2.0196162 | -1.4975087 | -0.2059825 |
| C | 0.7113613 | -1.7949322 | -0.2961461 |
| C | -0.3141765 | -0.7939456 | -0.0342818 |
| C | 0.1490170 | 0.5919836 | 0.0413821 |
| C | 1.4523348 | 0.8995662 | 0.1376371 |
| C | 2.4822895 | -0.1363658 | 0.0718874 |
| N | 3.6991812 | 0.2352834 | 0.2216331 |
| N | -0.7887049 | 1.7261326 | -0.2020457 |
| O | -1.6032304 | 1.5479949 | -1.0961522 |
| O | -0.6359076 | 2.7365386 | 0.4526865 |
| N | -1.5562723 | -1.1636347 | 0.1894973 |
| C | -2.0347812 | -2.5348400 | -0.0917252 |
| C | -2.5817173 | -0.3096062 | 0.8200526 |
| H | 2.7639164 | -2.2767990 | -0.3244858 |
| H | 0.4120917 | -2.8189048 | -0.4592775 |
| H | 1.7782985 | 1.9309323 | 0.1989793 |
| H | -1.5714070 | -2.9328311 | -0.9893479 |
| H | -1.8391975 | -3.1869574 | 0.7618938 |
| H | -3.1080051 | -2.4791340 | -0.2561472 |
| H | -3.2491592 | 0.1013393 | 0.0625994 |
| H | -3.1468566 | -0.9419435 | 1.5033536 |
| H | -2.1272598 | 0.4918021 | 1.3941274 |
| H | 4.3618173 | -0.5409525 | 0.1397717 |

**Table SM13. Optimized Coordinates of Methyl Analog of Adduct 14**

TD-B3LYP/TZVP level of theory

| | | | |
|---|---|---|---|
| C | -0.1035575 | -2.5218899 | 0.0161850 |
| C | -1.2412696 | -1.9511232 | -0.3974303 |
| C | -1.5642465 | -0.5352978 | -0.1366636 |
| C | -0.5395077 | 0.2787893 | 0.2876669 |
| C | 0.8435812 | -0.2407354 | 0.5620082 |
| C | 0.8880134 | -1.7454980 | 0.7617405 |
| N | 1.7905165 | -2.2352554 | 1.5207614 |
| N | -0.6218747 | 1.7186725 | 0.2803623 |

| | | | |
|---|---|---|---|
| O | -1.4384822 | 2.2728185 | -0.4641492 |
| O | 0.1777846 | 2.3430779 | 0.9830967 |
| N | -2.8846603 | -0.1950174 | -0.2199515 |
| C | -3.8110792 | -0.8567179 | -1.1335721 |
| C | -3.4867172 | 0.8387814 | 0.6068333 |
| H | 0.0660959 | -3.5843437 | -0.1250281 |
| H | -1.9992938 | -2.5630890 | -0.8655887 |
| H | -3.2840978 | -1.3282436 | -1.9591489 |
| H | -4.4318383 | -1.6022846 | -0.6236294 |
| H | -4.4703883 | -0.1005939 | -1.5641626 |
| H | -3.5741468 | 1.7927152 | 0.0824546 |
| H | -4.4783976 | 0.4938806 | 0.9113699 |
| H | -2.8857637 | 0.9945149 | 1.5018087 |
| H | 1.7696671 | -3.2585150 | 1.5038741 |
| H | 1.2551996 | 0.2431974 | 1.4480165 |
| O | 1.6429707 | 0.0718163 | -0.5958122 |
| C | 3.0672375 | 0.1959066 | -0.3693663 |
| H | 3.3712210 | -0.5567250 | 0.3656492 |
| C | 3.4119123 | 1.5876478 | 0.1508462 |
| H | 2.8806709 | 1.8154944 | 1.0756110 |
| H | 4.4846310 | 1.6590285 | 0.3490503 |
| H | 3.1409029 | 2.3475976 | -0.5851467 |
| C | 3.7307912 | -0.1113806 | -1.7033733 |
| H | 4.8164804 | -0.0242877 | -1.6189917 |
| H | 3.4880188 | -1.1232175 | -2.0323784 |
| H | 3.3872517 | 0.5896158 | -2.4676667 |

**Table SM14. Optimized Coordinates of Methyl Analog of Adduct 13**

TD-B3LYP/TZVP level of theory

| | | | |
|---|---|---|---|
| C | 0.2816120 | -1.3653129 | 0.2738914 |
| C | -0.8415021 | -0.4038627 | 0.0056434 |
| C | -0.6900688 | 0.9361732 | -0.0262221 |
| C | 0.6904034 | 1.4550950 | 0.1000358 |
| C | 1.7795190 | 0.6780683 | 0.1578722 |
| C | 1.6675541 | -0.7722552 | 0.0152403 |
| N | 2.6978378 | -1.4402768 | -0.3168249 |
| N | 0.9506064 | 2.9116361 | -0.0222541 |
| O | 0.5816599 | 3.4580444 | -1.0482169 |
| O | 1.5431267 | 3.4561146 | 0.9028000 |
| N | -1.7367561 | 1.8417738 | -0.2419843 |
| C | -2.9527402 | 1.3106994 | -0.8307568 |
| C | -2.0076992 | 2.8128876 | 0.8188111 |
| H | 0.2672431 | -1.5708267 | 1.3595587 |
| H | -1.8257700 | -0.8429812 | -0.0709445 |
| H | 2.7740123 | 1.1043926 | 0.1485043 |
| H | -2.7099320 | 0.7146782 | -1.7103888 |
| H | -3.5395516 | 0.6896725 | -0.1340463 |
| H | -3.5804375 | 2.1472055 | -1.1416432 |
| H | -2.5640192 | 3.6553306 | 0.4055530 |
| H | -2.6008555 | 2.3648322 | 1.6309239 |
| H | -1.0903231 | 3.1977119 | 1.2577276 |
| H | 2.4356457 | -2.4157414 | -0.4786140 |
| O | 0.1457912 | -2.5934612 | -0.4257609 |

| | | | |
|---|---|---|---|
| C | -0.1884931 | -3.7570015 | 0.3576879 |
| H | 0.4712956 | -3.7792845 | 1.2370015 |
| C | -1.6408451 | -3.7308249 | 0.8273956 |
| H | -1.8649033 | -4.6282775 | 1.4088157 |
| H | -1.8455674 | -2.8679860 | 1.4646679 |
| H | -2.3174722 | -3.6984176 | -0.0298379 |
| C | 0.1116493 | -4.9600146 | -0.5223896 |
| H | 1.1580428 | -4.9646919 | -0.8309297 |
| H | -0.0936741 | -5.8870329 | 0.0167886 |
| H | -0.5094995 | -4.9370115 | -1.4202998 |

**Alternative Scenarios**

Many alternative scenarios have been considered to in an effort to explain the cascade of transient intermediates formed in the photoreaction of azide 5. One example is shown in Fig. SM4 in which two scenarios are compared: Scenario I in which the closed-shell singlet nitrene initially undergoes protonation, and Scenario II in which the closed-shell singlet initially undergoes conversion to the opened-shell singlet nitrene before it becomes protonated. Even though the calculated spectra are in nearly perfect agreement with the observed spectra in scenario II, the protonation step occurs in the range of 20 ps not 560 ns. Therefore, scenario I is the more viable alternative.

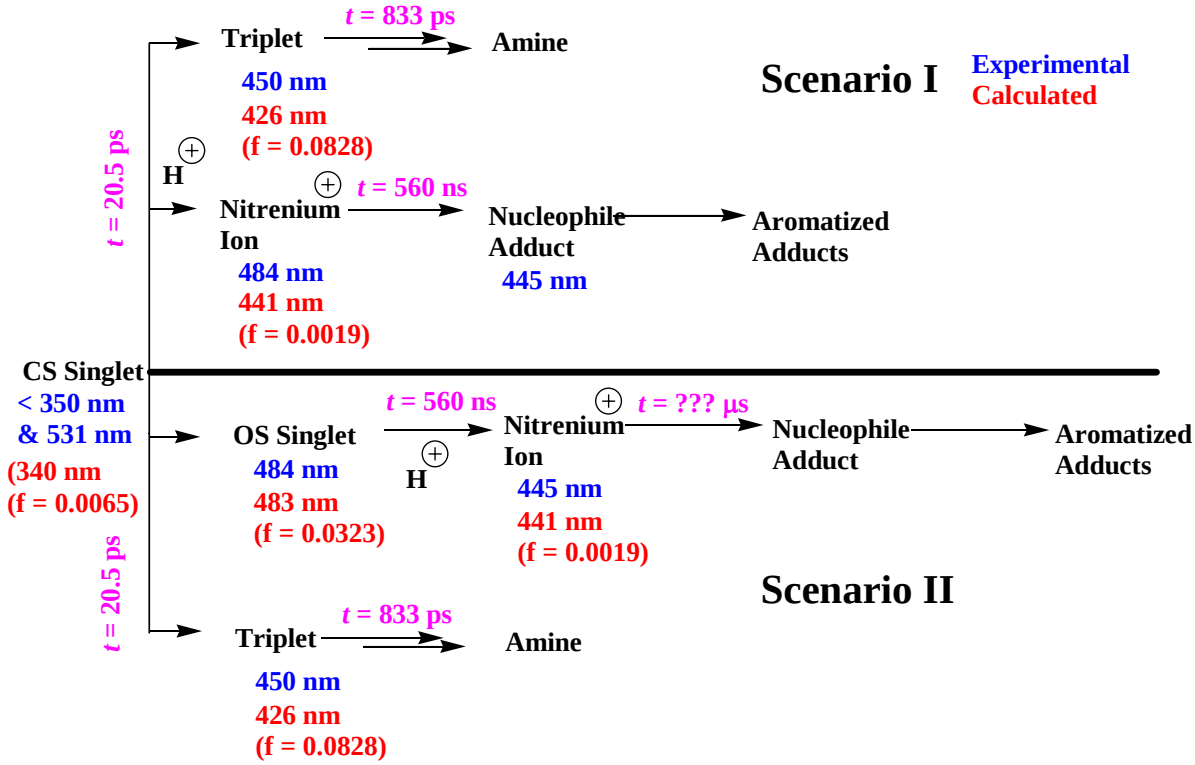



**Figure SM4. Alternative scenarios for closed-shell singlet nitrene reaction.**

**Theoretical Calculations of Formation of Adducts 13 and 14 and Their Relative Stability**

The formation of adducts **13** and **14** have been analyzed theoretically at the DFT B3LYP the reasons for the collapse of the ion pair with preferential bond formation at the 2-position between the nitrene nitrogen and the nitro group rather than at the less hindered 6-position (Fig. SM7). While these transition state imaginary frequencies are small, both initial attack transition states involve hydrogen bonding between the incoming methoxide ion and the =N-H hydrogen atom. The possibility of a 1-5 methoxy shift between the 2- and 6-positions has been considered, but was found to have very high activation energies in either direction, and thus, not to play an important role in the final product isomer distribution. On the other hand, the transition state for formation of the 2-methoxy adduct has a slightly lower energy, 2.05 kcal/mol, and higher intensity (probability) than that for the 6-methoxy adduct. Both of these transitions state occur with quite long O-C bond distances, 2.43 Å, and therefore, the attack at the 2-position is not particularly susceptible to steric interference from the nitro group, but is facilitated by the additional electron withdrawal of that group.

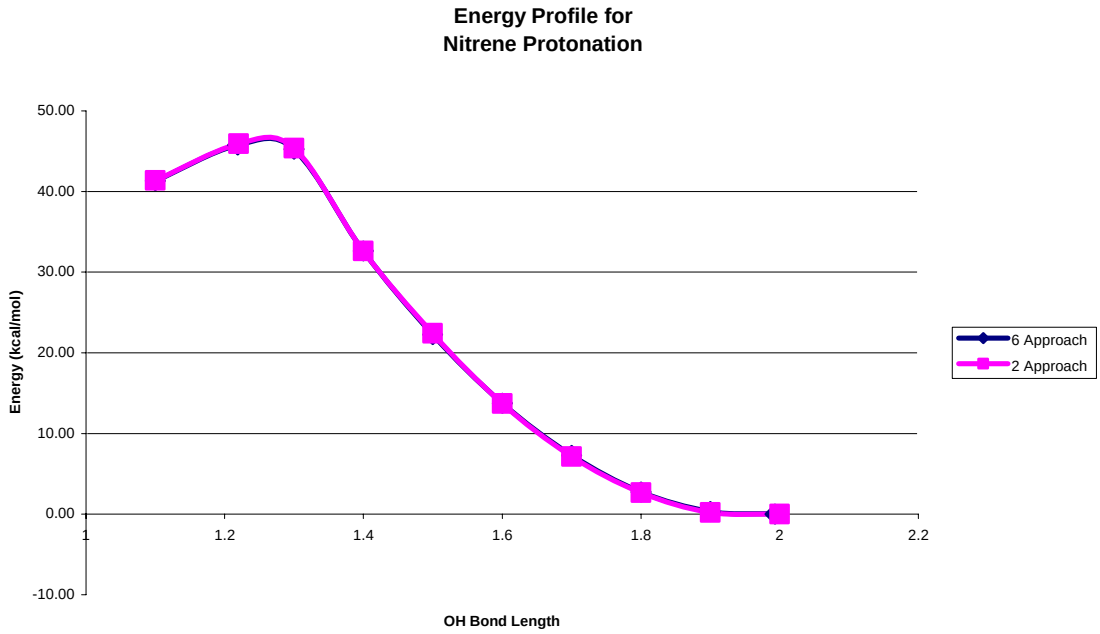



**Figure SM5.** Attack profile for protonation of nitrene with approach from the 2- or 6-side of the nitrene nitrogen (-O-H in Å) .

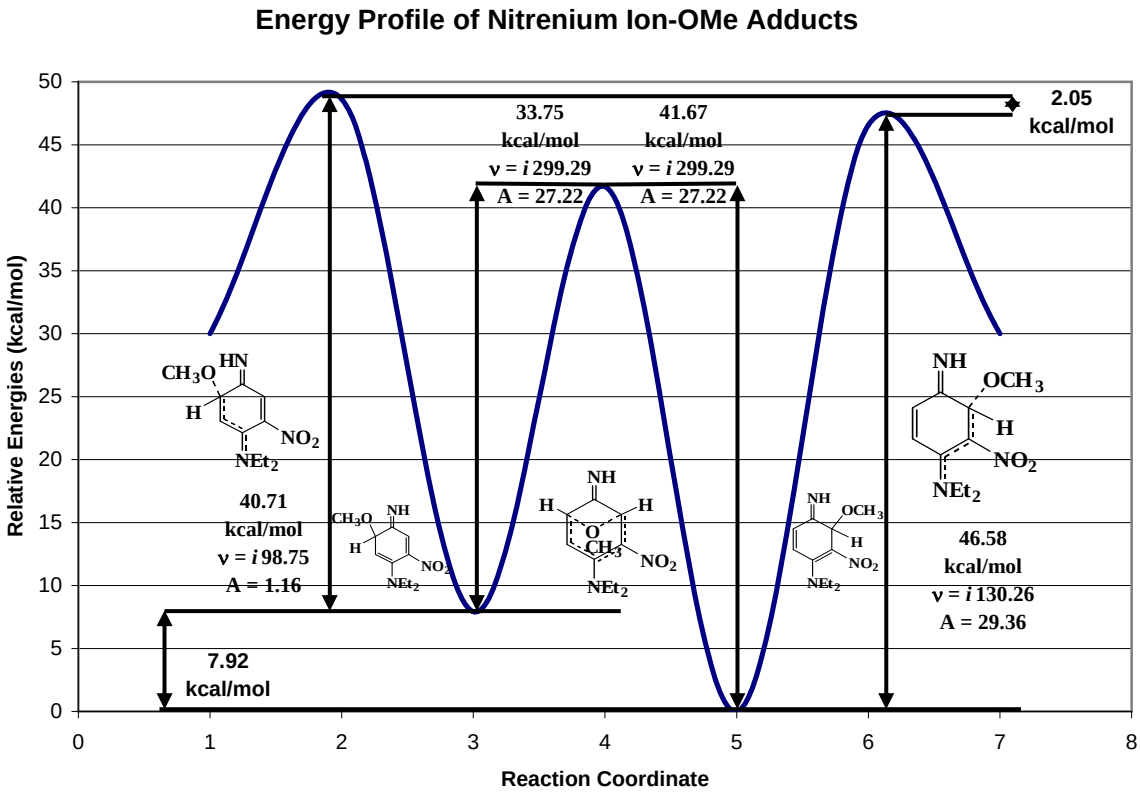
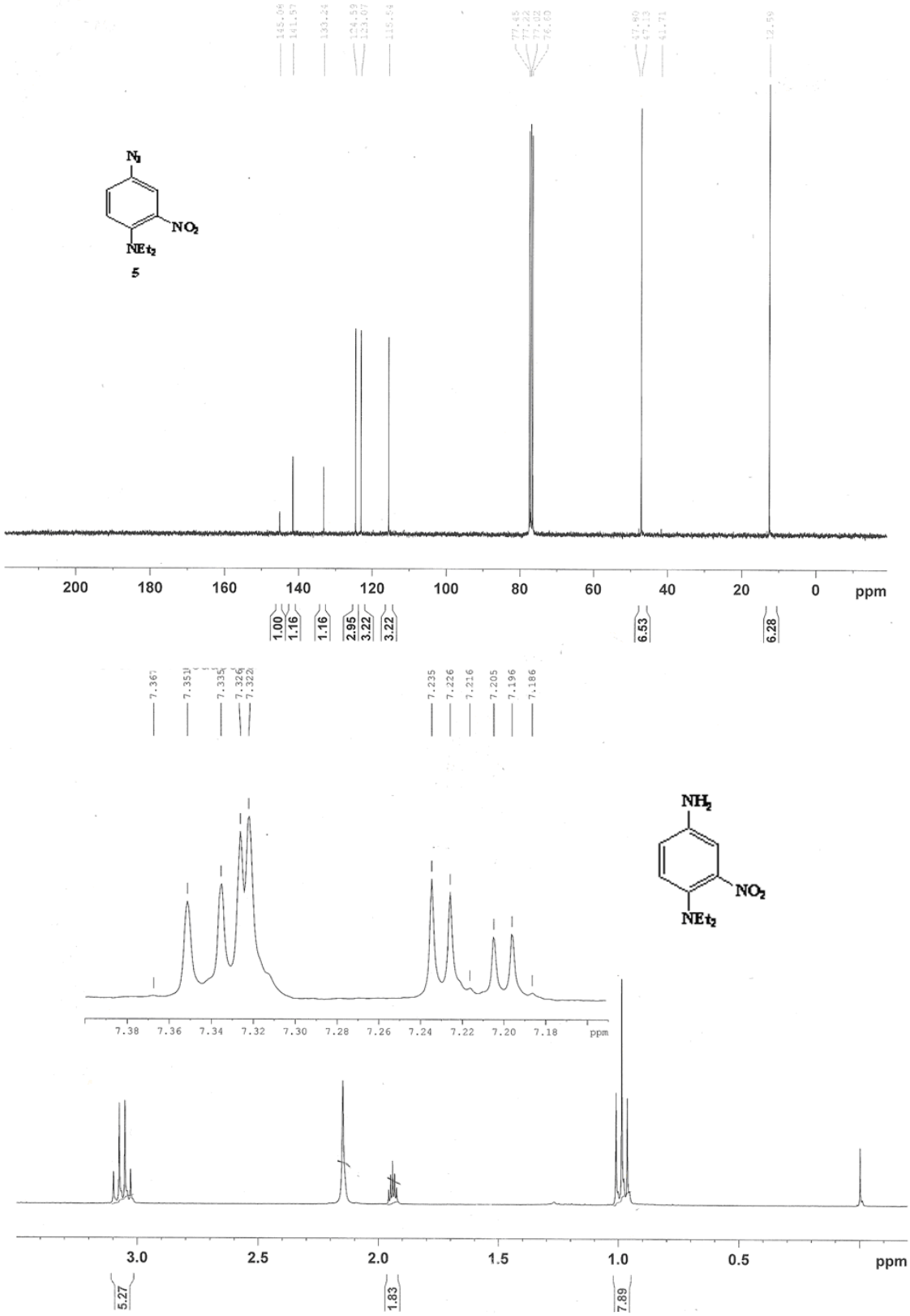
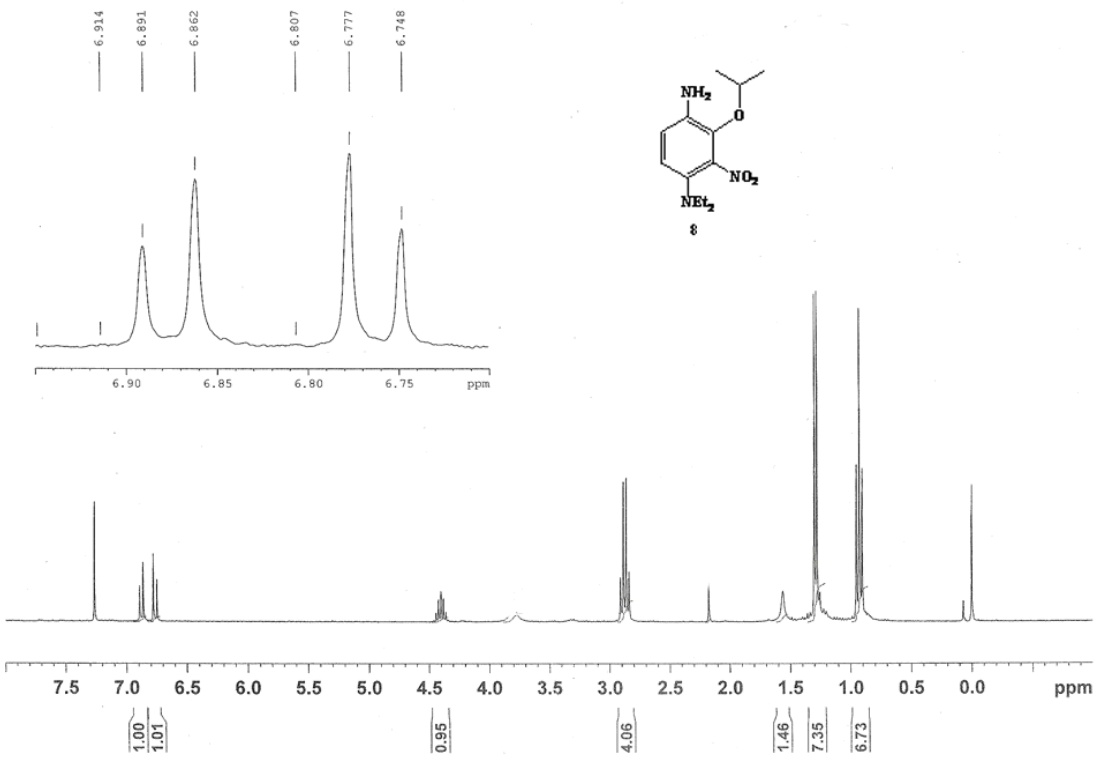


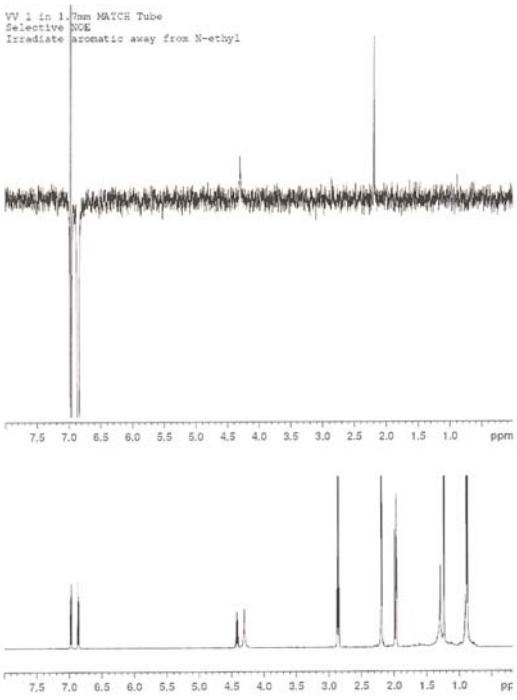

**Figure SM6.** Reaction profile for formation of 2- and 6-methanol adducts, interconversion of 2- and 6-adducts, **14** and **13**, respectively.

## Supplementary Material Spectra



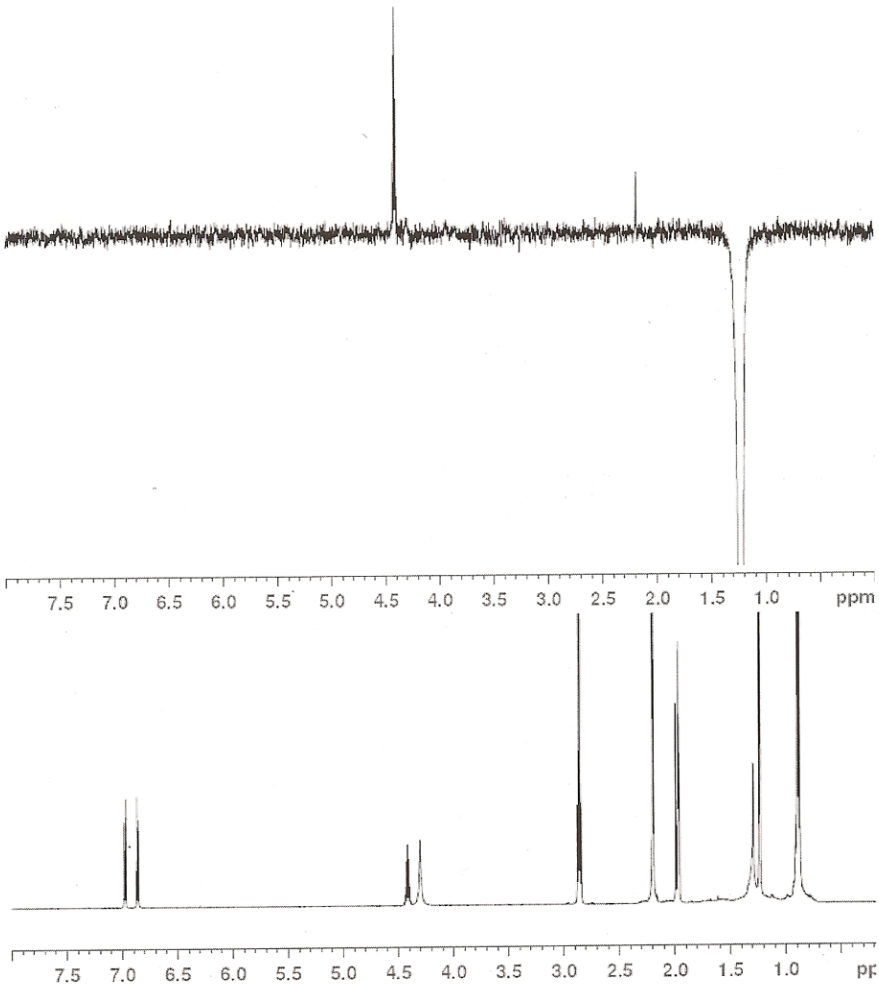

6.914
6.891
6.862
6.807
6.777
6.748
6.90
6.85
6.80
6.75
ppm
NH2
O
NO2
NEt2
8
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.01
0.95
4.06
1.46
7.35
6.73

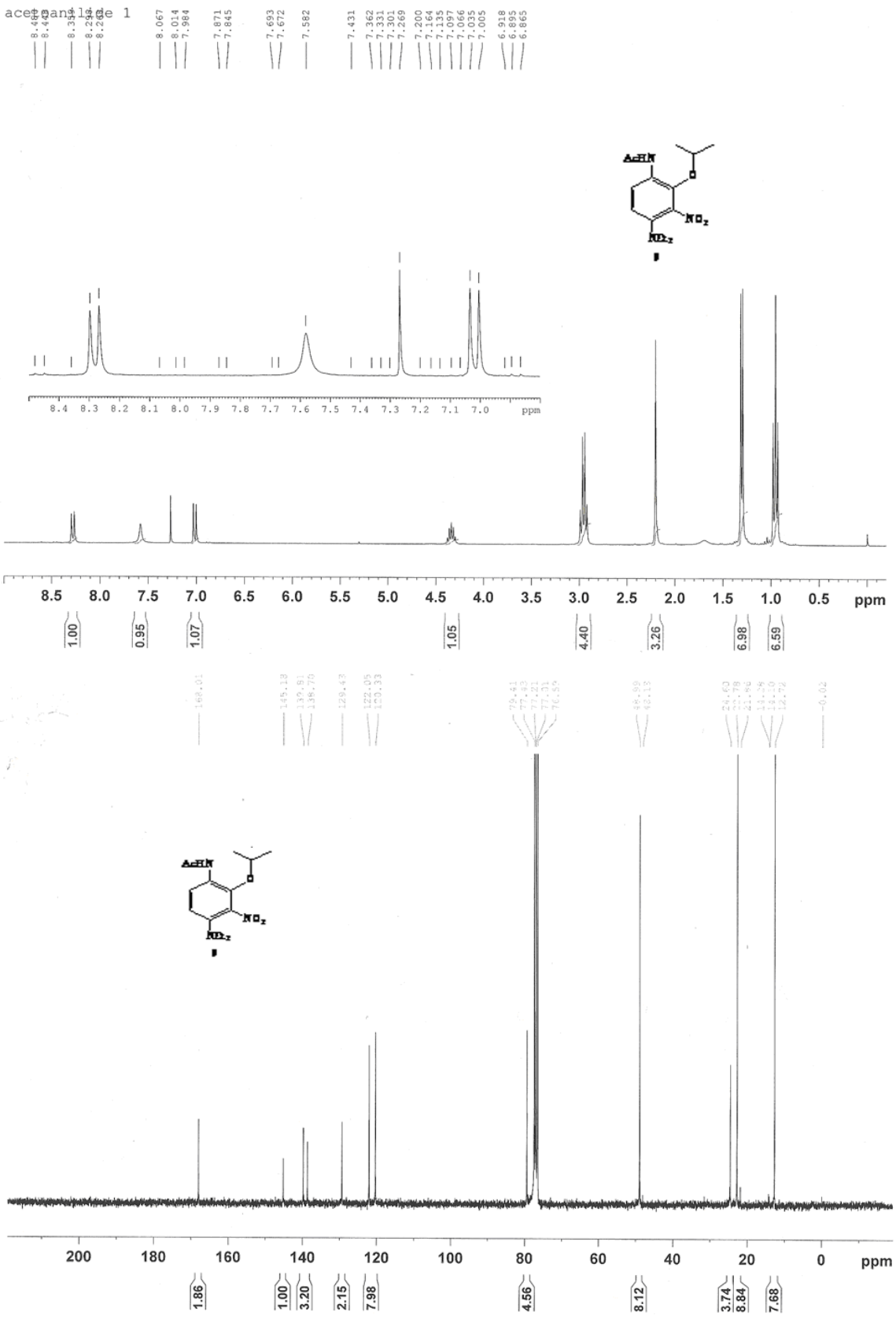
Spectrum I

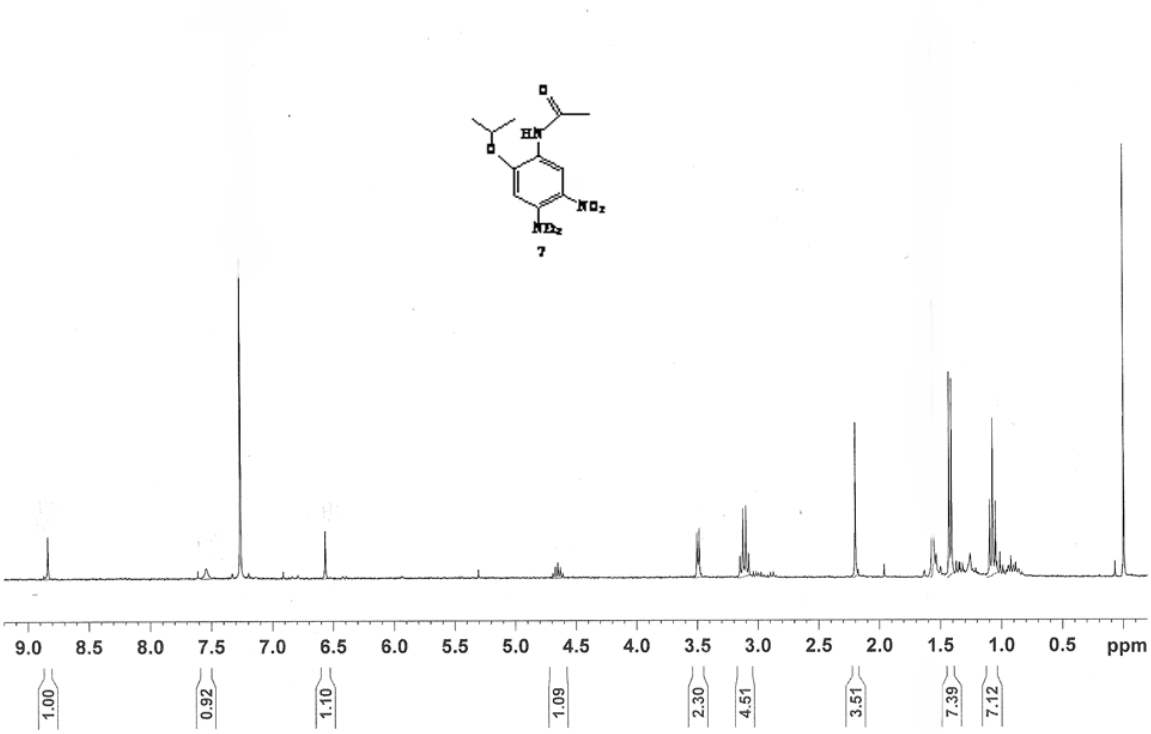

VV 1 in 1.7mm MATCH Tube
Selective NOE
Irradiate aromatic away from N-ethyl
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

# Spectrum II

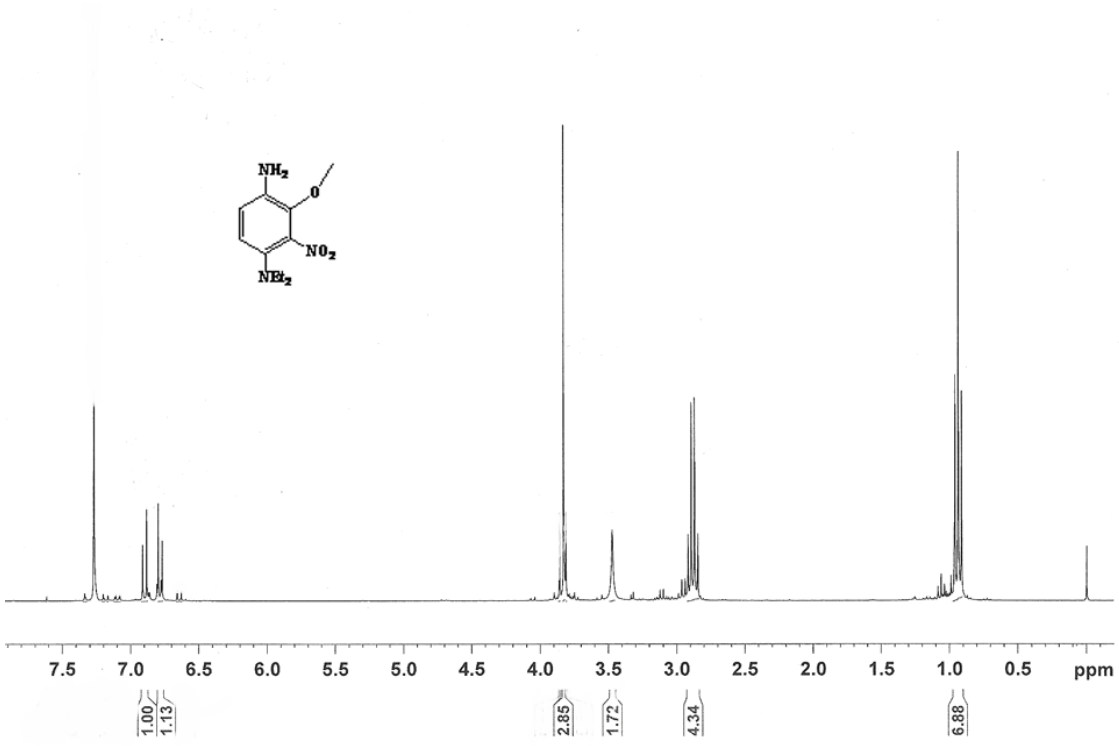
VV 1 in 1.7mm MATCH Tube
Selective NOE
Irradiate methyls of isopropyl

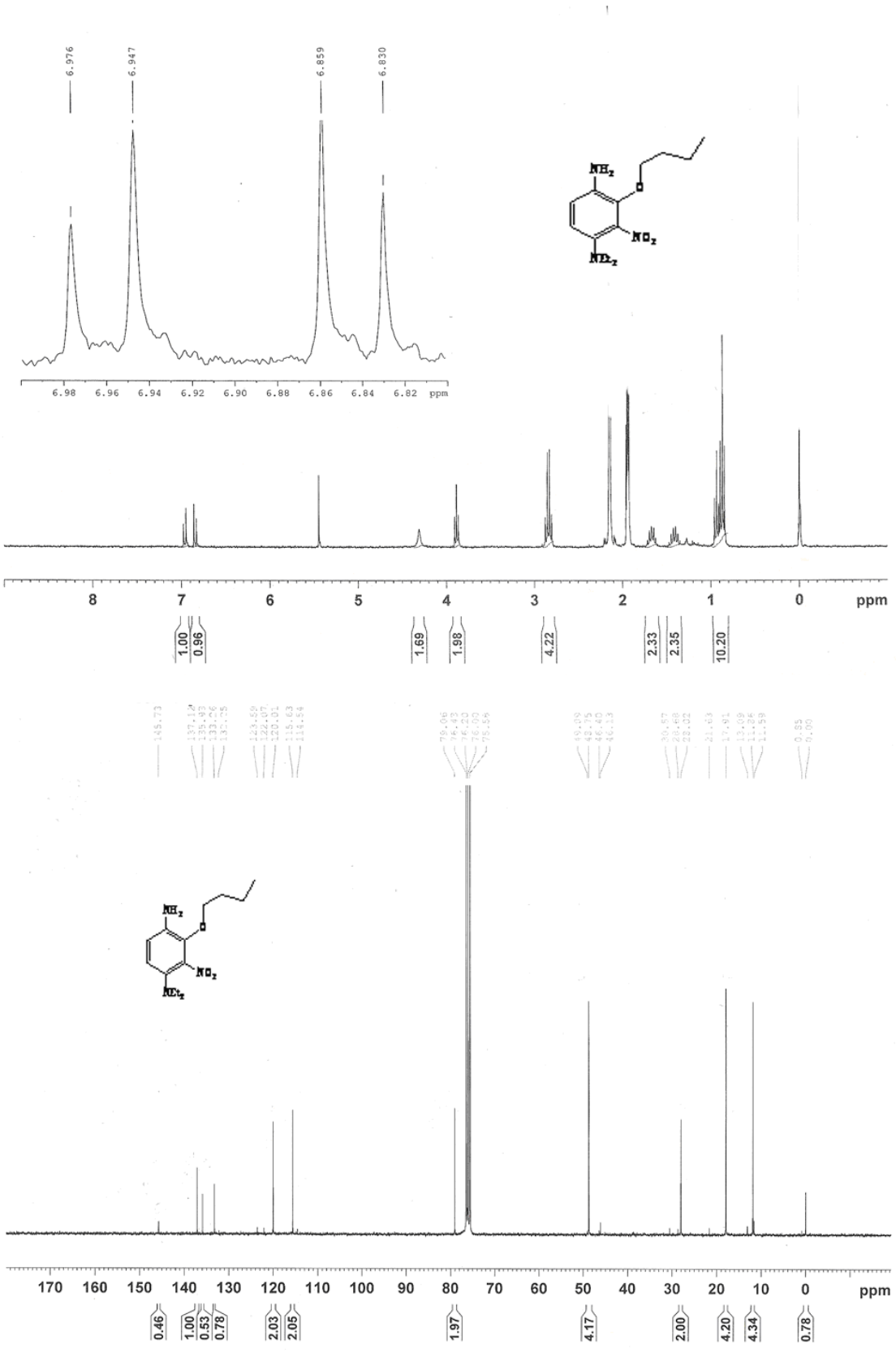


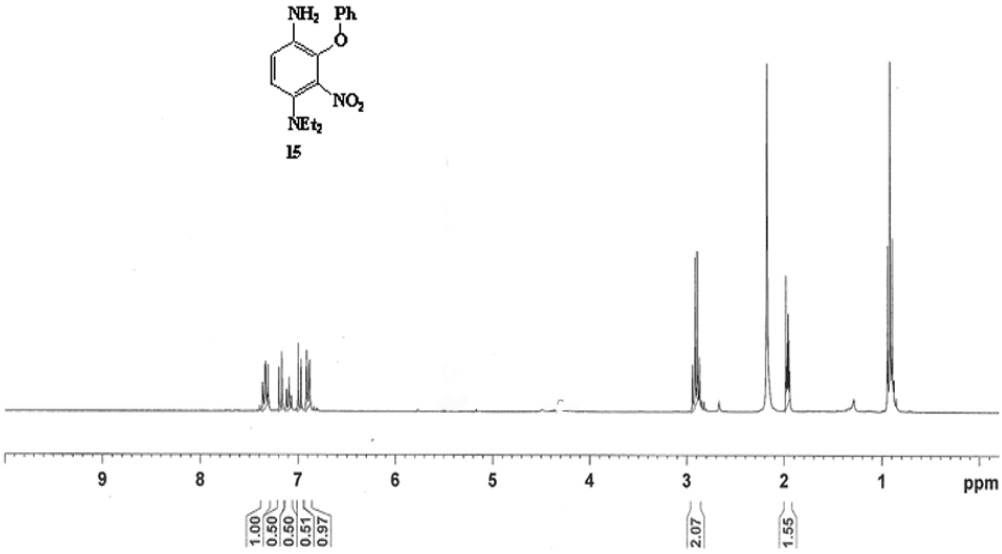

acetanilide 1
8.067
8.014
7.984
7.871
7.845
7.693
7.672
7.582
7.431
7.362
7.331
7.301
7.269
7.200
7.164
7.135
7.097
7.066
7.035
7.005
6.918
6.895
6.865
AcHN
NO2
NO2
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 ppm
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.95
1.07
1.05
4.40
3.26
6.98
6.59
168.01
145.18
137.51
136.75
129.43
122.05
120.33
79.41
77.00
77.21
77.01
76.54
68.99
68.13
24.63
21.78
21.96
19.36
19.10
12.72
-0.02
AcHN
NO2
NO2
200 180 160 140 120 100 80 60 40 20 0 ppm
1.86
1.00
3.20
2.15
7.98
4.56
8.12
3.74
8.84
7.68

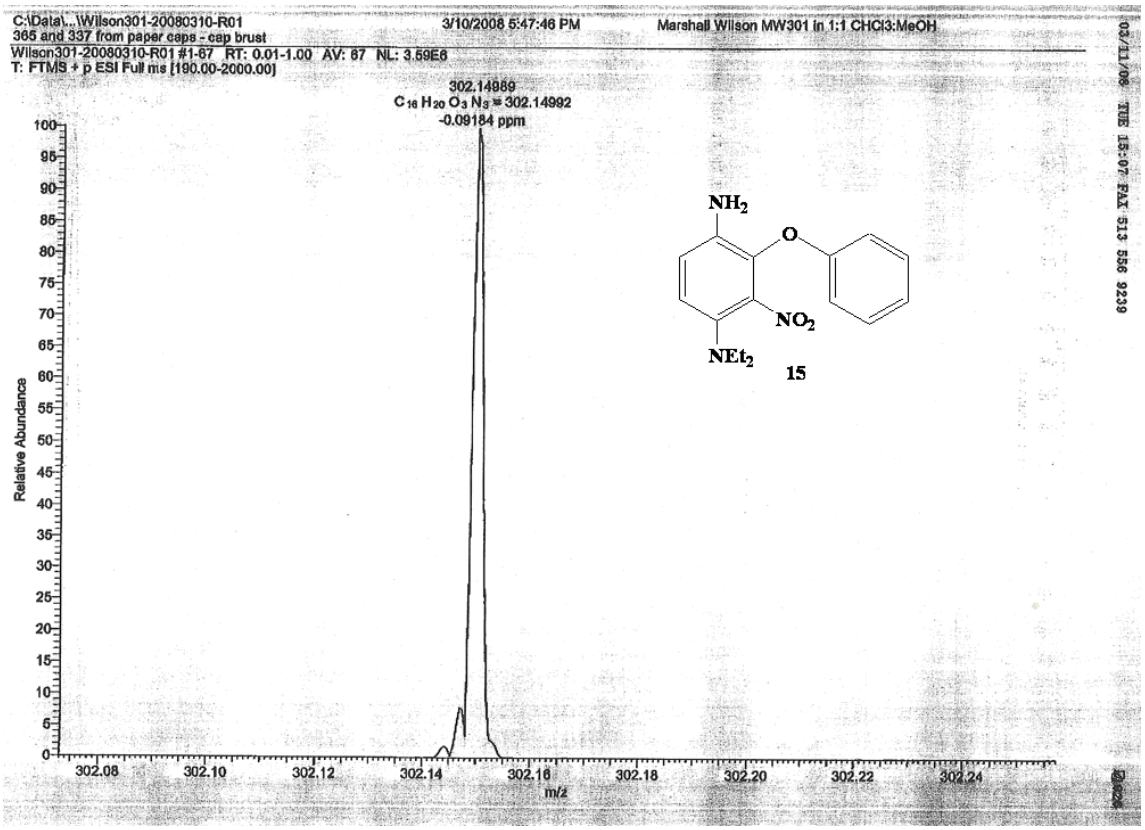

9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.92
1.10
1.09
2.30
4.51
3.51
7.39
7.12

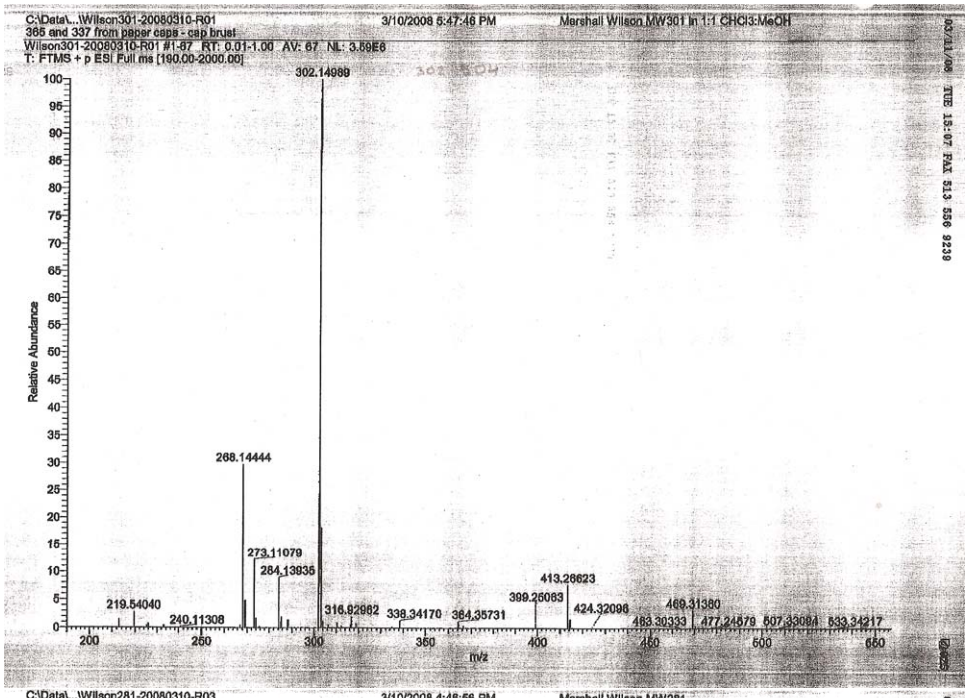
7

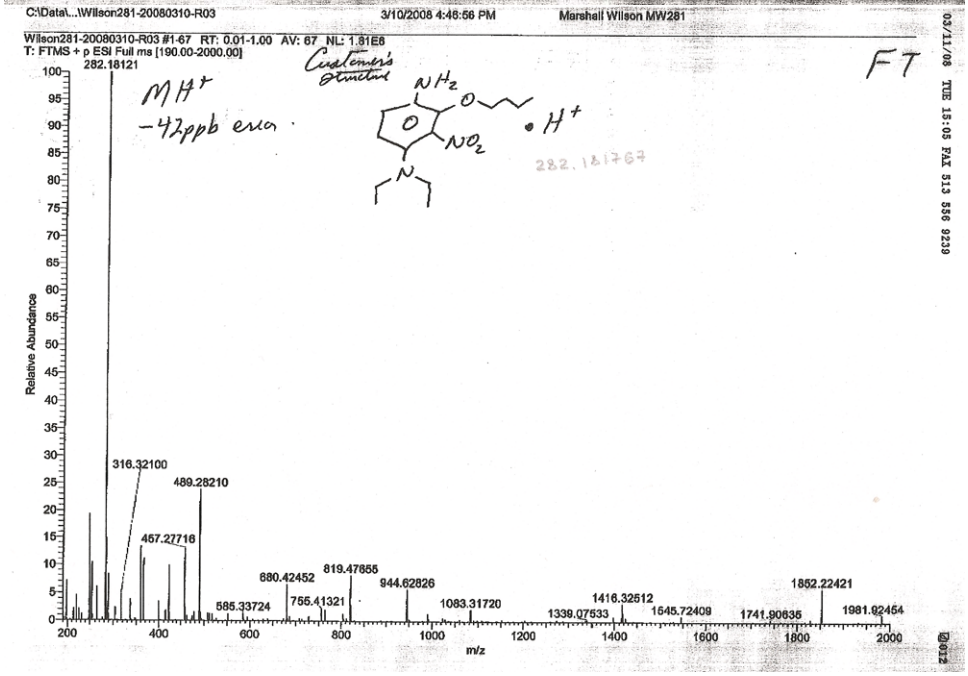

7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.13
2.85
1.72
4.34
6.88

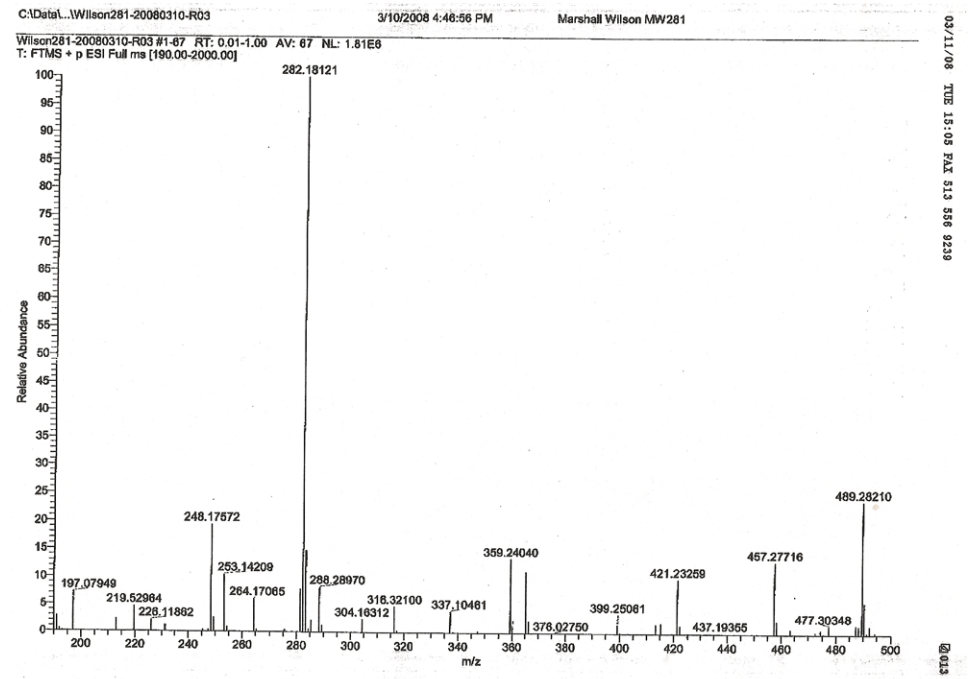

6.976
6.947
6.859
6.830
6.98 6.96 6.94 6.92 6.90 6.88 6.86 6.84 6.82 ppm
NH2
NO2
NH2
8 7 6 5 4 3 2 1 0 ppm
1.00 0.96 1.69 1.98 4.22 2.33 2.35 10.20
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
0.46 1.00 0.53 0.78 2.03 2.05 1.97 4.17 2.00 4.20 4.34 0.78

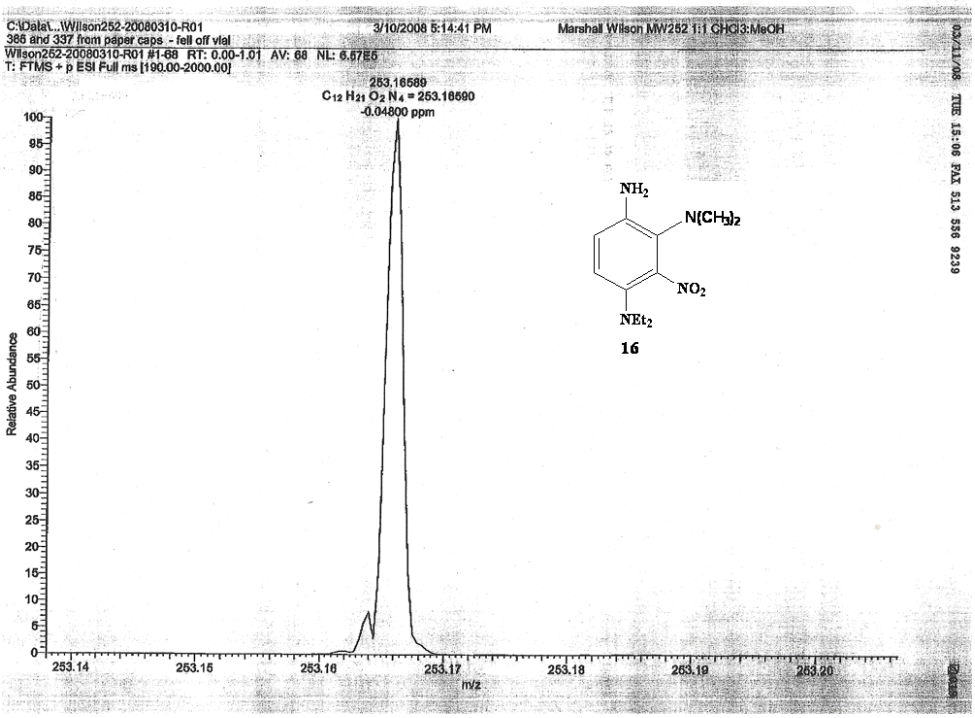

NH2
Ph
O
NO2
NEt2
15
9
8
7
6
5
4
3
2
1
ppm
1.00
0.50
0.50
0.51
0.97
2.07
1.55


C:\Data\...\Wilson301-20080310-R01
365 and 337 from paper caps - cap brust
3/10/2008 5:47:46 PM
Marshall Wilson MW301 in 1:1 CHCl3:MeOH
Wilson301-20080310-R01 #1-67 RT: 0.01-1.00 AV: 67 NL: 3.59E6
T: FTMS + p ESI Full ms [190.00-2000.00]
302.14989
C16 H20 O3 N3 = 302.14992
-0.09184 ppm
Relative Abundance
m/z
NH2
O
NO2
NEt2
15
03/11/08 TUE 15:07 FAX 513 556 9239


C:\Data\...\Wilson301-20080310-R01
3/10/2008 5:47:46 PM
Marshall Wilson MW301 in 1:1 CHCl3:MeOH
365 and 337 from paper caps - cap brust
Wilson301-20080310-R01 #1-67 RT: 0.01-1.00 AV: 67 NL: 3.59E6
T: FTMS + p ESI Full ms [190.00-2000.00]
302.14989
268.14444
273.11079
284.13835
219.54040
240.11308
316.82982
338.34170
364.35731
399.26083
413.28623
424.32098
469.31380
483.30333
477.24579
507.33084
533.34217
Relative Abundance
m/z
03/11/08 TUE 15:07 FAX 513 556 9239


C:\Data\...\Wilson281-20080310-R03
3/10/2008 4:46:56 PM
Marshall Wilson MW281
Wilson281-20080310-R03 #1-67 RT: 0.01-1.00 AV: 67 NL: 1.81E6
T: FTMS + p ESI Full ms [190.00-2000.00]
282.18121
MH+
-42ppb error
Customer's Structure
NH2
O
O
NO2
N
• H+
282.181767
FT
316.32100
489.28210
457.27716
680.42452
819.47855
944.62826
585.33724
755.41321
1083.31720
1339.07533
1416.32512
1645.72409
1741.90635
1852.22421
1981.92454
Relative Abundance
m/z
03/11/08 TUE 15:05 FAX 513 556 9239


C:\Data\...\Wilson281-20080310-R03
3/10/2008 4:46:56 PM
Marshall Wilson MW281
Wilson281-20080310-R03 #1-67 RT: 0.01-1.00 AV: 67 NL: 1.81E6
T: FTMS + p ESI Full ms [190.00-2000.00]
282.18121
248.17572
489.28210
359.24040
457.27716
421.23259
253.14209
288.28970
197.07949
264.17065
219.52964
316.32100
337.10461
228.11862
304.16312
399.25081
376.02750
437.19355
477.30348
Relative Abundance
m/z
03/11/08 TUE 15:05 FAX 513 556 9239


C:\Data\...\Wilson252-20080310-R01
3/10/2008 5:14:41 PM
Marshall Wilson MW252 1:1 CHCl3:MeOH
386 and 337 from paper caps. - fell off vial
Wilson252-20080310-R01 #1-68 RT: 0.00-1.01 AV: 68 NL: 6.67E5
T: FTMS + p ESI Full ms [190.00-2000.00]
253.16589
C12 H21 O2 N4 = 253.16590
-0.04800 ppm
NH2
N(CH3)2
NO2
NEt2
16
Relative Abundance
m/z
03/11/08 TUE 15:06 FAX 513 556 9239