A catalytic and *tert*-butoxide ion-mediated amidation of aldehydes with *para*-nitro azides

Giorgio Carbone, James Burnley and John E. Moses*

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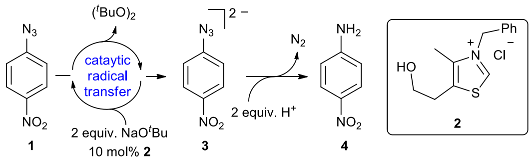
We report here a new catalytic reaction in which, *p*-nitro azides are acylated by aldehydes to produce amides and molecular nitrogen in a single step. The transformation is believed to proceed *via* an electron transfer process mediated by the *tert*-butoxide ion, and catalysed by a thiazolium salt derived species.

Amide bond forming reactions are among the most executed in organic chemistry, forming key linkages in peptides, proteins, synthetic polymers and drugs.[1] Traditional approaches to amide synthesis involving the coupling of activated carboxylic acid derivatives (anhydrides and acyl chlorides) with nucleophilic amines are expensive, wasteful and often produce toxic by-products necessitating lengthy purification.[2] The demand for sustainable and greener[3] approaches to amide synthesis has stimulated intense activity in the development of new and creative catalytic methods.[1d]
Straightforward and desirable methods involving the direct coupling of carboxylic acids and amines with certain boronic acid catalysts have been reported.[4] Other strategies generally involve either the catalytic or oxidative acylation of an amine, or occur by a suitable combination of complementary reaction partners following a unique pathway. For example, the catalytic generation of activated carboxylates from functionalised aldehydes by *N*-heterocyclic carbene (NHC) catalysts with a co-catalyst, followed by their conversion to amides has been shown to work with a variety of amines.[5] Oxidative processes utilising NHC's[6] and metal based catalysts have been used for amide formation from aldehydes with stoichiometric oxidants.[7] The ruthenium catalysed conversion of alcohols and primary amines with loss of H_2 developed by Milstein *et al.*[8] represents an example of an atom economic and green approach to amide synthesis, whereas the oxidative coupling of α-bromo nitroalkanes with amines offers a quite different pathway.[9]
The formation of amides by the intermolecular coupling of thioacids and electron poor anilines that require no activating or coupling reagents has been investigated.[10,11] The mechanism is believed to involve the nucleophilic attack of the thioacetate onto the azide *N*-3 followed by formation of a cyclic thiatriazoline intermediate which collapses to give the amide, nitrogen gas and elemental sulfur.[11] In seeking to further develop the theme of this chemistry, we were intrigued by the possibility of using aldehydes directly in a redox azido-amidation type-process, thus broadening the range of available substrates and eliminating the sulfur by-product.

In recent studies, we observed that in the presence of the thiazolium salt (**2**), the *tert*-butoxide ion selectively reduces the azide group of *para*-azidonitrobenzene (Scheme 1).[12] The reaction is believed to proceed by an electron transfer process *via* the dianion **3**, followed by concomitant loss of nitrogen gas to give aniline **4**.



Scheme 1 A proposed mechanism to explain the catalytic reduction of **1** to aniline **4** by the -butoxide ion.

We now report the *tert*-butoxide ion mediated amidation of aldehydes[13] with *p*-nitro azides, catalysed by a thiazolium salt derived catalyst. This clean transformation allows the synthesis of 4-nitroaromatic amides in one step with high atom economy, and driven by loss of environmentally benign nitrogen gas.[14]

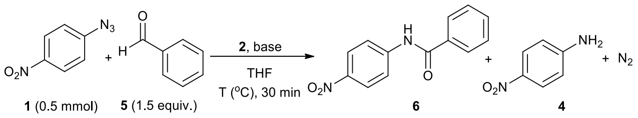
For the reaction development we opted to use the electron deficient 1-azido-4-nitrobenzene **1**, with benzaldehyde **5** as the acyl donor. Using previously optimised conditions for the azide-reduction as a starting point (2 equiv. *tert*-BuONa, THF, rt),[12] a number of preliminary reactions were performed (Table 1).

Thus, treatment of the azide **1** and aldehyde **5** in THF with *tert*-BuONa at room temperature immediately resulted in the evolution of a gas and, complete conversion of the starting material **1**. After chromatography, the corresponding target amide **6** was isolated in 56% yield along with *p*-nitroaniline **4** (39%) (Table 1, Entry 1). Similar results were achieved when the potassium salt of *tert*-butoxide ion was employed; giving amide **6** (51%) and aniline **4** (36%) (Table 1, Entry 2). However, when the same reaction was carried out in the presence of 1 equiv. of the thiazolium salt **2**, the yield of the amide **6** decreased (30%) whereas the aniline **4** was isolated in higher yield (62%) (Table 1, Entry 3).

When a solution of **1** and **5** was treated with *tert*-BuONa (2 equiv.) at -25 °C without catalyst, unreacted starting materials were recovered (Table 1, Entry 4). In contrast, the addition of **2** (1 equiv.) to the reaction mixture at -25 °C followed by *tert*-BuONa (3 equiv.) gave the target amide **6** in 80% yield with no observed aniline **4** (Table 1, Entry 5). A reduced loading of **2** (0.1 equiv.) resulted in a similar yield of amide **6** (81%) (Table 1, Entry 6).

Stirring a mixture (30 min) of the azide **1**, the aldehyde **5** and *tert*-BuONa (2 equiv.) at -25 °C; no reaction was observed (TLC) until the addition of **2** (0.1 equiv.), then after a further 30 min, the amide **6** was isolated in 83% yield (Table 1, Entry 7).

Table 1 Optimisation of the reaction conditions and control experiments.



Entry	**2** (equiv.)	base (equiv.)	T (°C)	**6** yield[a] (%)	**4** yield[a] (%)
1	-	NaO^{t}Bu (2.0)	rt	56	39
2	-	KO^{t}Bu (2.0)	rt	51	36
3	1.0	NaO^{t}Bu (3.0)	rt	30	62
4	-	NaO^{t}Bu (2.0)	-25	0	0
5	1.0	NaO^{t}Bu (3.0)	-25	80	0
6	0.1	NaO^{t}Bu (2.0)	-25	81	0
7	0.1[b]	NaO^{t}Bu (2.0)	-25	83	0

[a]isolated yield after chromatography; [b]**2** added after stirring **1** and **2** at -25 °C in the presence of *tert*-BuONa.

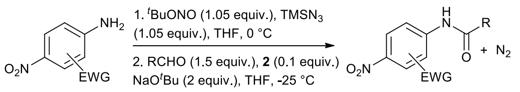
We next investigated the one pot diazotisation-azidation-amidation reaction, since this would negate the isolation of the azide substrate.[15] This proved viable; the diazotisation/azidation of *p*-nitroaniline **4** with ^{t}BuONO/TMSN$_3$ at 0 °C in THF,[16] followed by catalytic amidation at -25 °C provided the amide **6** in an excellent 94 % yield (Table 1, Entry 1). The scope of the tandem reaction was next investigated with a number of electron deficient anilines and several aldehydes (Table 2, Entries 1-15). Both *ortho*- and *meta*-substituents on the *para*-nitroaromatic azide were well tolerated with benzaldehyde (Table 2, Entries 2-4). The reaction worked best when electron deficient anilines were coupled with electron rich aromatic aldehydes, including *ortho*- and *para*-substitution on the aromatic aldehyde (Table 2, Entries 5-7).

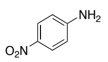
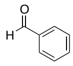
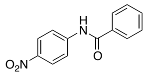
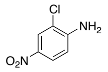
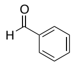
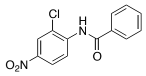
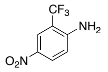
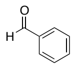
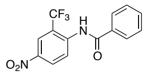
When an electron deficient aromatic aldehyde was used, the yield of amide dropped significantly (38%) (Table 2, Entry 8). The heteroaromatic thiophene-2-carbaldehyde was tolerated, giving the corresponding amide isolated in good yield (82%) (Table 2, Entry 9). When alkyl aldehydes were employed; high yields were consistently obtained (78-94% yield, entries 10-15).

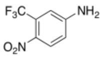
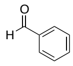
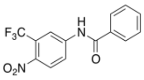
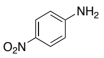
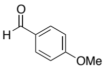
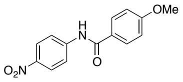
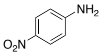
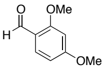
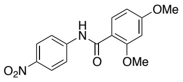
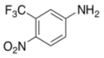
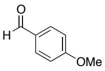
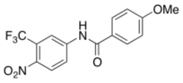
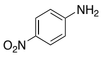
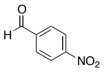
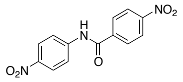
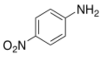
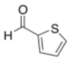
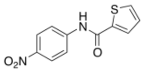
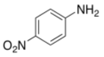
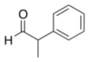
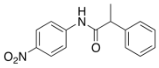
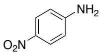
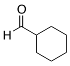
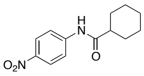
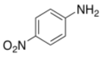
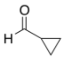
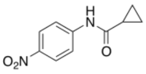
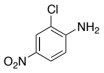
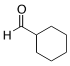
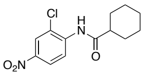
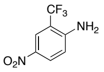
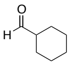
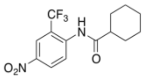
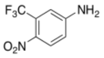
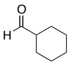
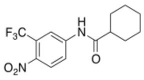
The reaction raises a number of interesting questions with regard to the mechanism. During the 1980's, Guthrie and co-workers published a number of detailed papers on the reaction mechanism between the *tert*-butoxide ion and nitrobenzene, involving SET processes.[17] Indeed, the function of *tert*-butoxide ion as an electron transfer agent is well documented.[18]

When the amidation reaction of the azide **1** and benzaldehyde (**2**) was attempted under an atmosphere of oxygen or in the presence of the radical trap TEMPO, no reaction was observed with complete recovery of the azide **1**. These results, which are consistent with our earlier findings[12] provide support for a radical based mechanism in the amidation process (See Scheme S1, supporting information (SI)).

Table 2 Reaction of *para*-nitro aromatic azides with various aldehydes



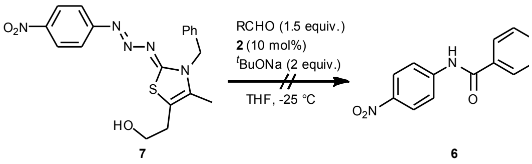
Entry	Aniline	Aldehyde	Amide	Yield%[a]
1				94
2				85
3				77

4				84
5				91
6				79
7				89
8				38
9				82
10				94
11				88
12				78
13				92
14				83
15				87

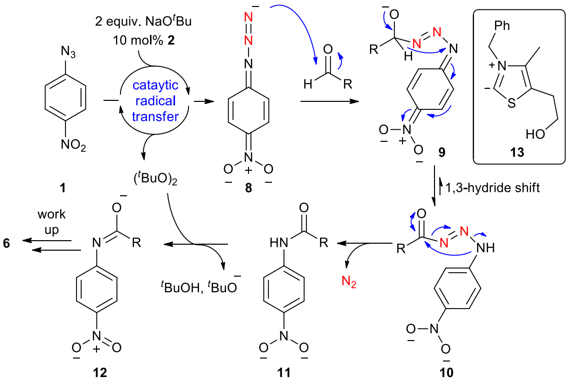
^{a}isolated yield after chromatography.

The relative nitrogen connectivity between the starting azide and the corresponding amide was determined by performing experiments with ^{15}N-labelled 1-azido-4-nitrobenzene. The reaction proceeded with retention of ^{15}N directly attached to the aromatic ring (See Scheme S2, SI).

The role of the thiazolium salt **2** is uncertain and warrants further investigation. However, preliminary mechanistic studies with the thiazolium derived triazene **7**[12,20] demonstrated that such speices are stable to the amidation conditions and hence ruled out as a possible intermediate (Scheme 2).



Scheme 2 Subjecting the triazene **7** to the preferred amidation conditions did not lead to the formation of **6**.



Scheme 3 A plausible amidation mechanism **1** (R=Ph).

Based upon the available experimental data, we present a working hypothesis to explain the mechanism. Thus, electron transfer from the *tert*-butoxide ion gives the dianion **8**, a process that we tentatively propose is catalysed by a thiazolium anion relay derived from **2** (**13**).[12] Whereas protonation and loss of nitrogen from **8** would deliver the corresponding aniline (Scheme 1), interception of the intermediate **8** by an electrophilic aldehyde would deliver intermediate **9**.[14] A subsequent 1,3-hydride shift leads to intermediate **10** that is poised to extrude nitrogen gas and deliver the dianion **11**. Next, in a manner consistent with the observations of Guthrie,[17] we speculate that the loss of two electrons from **11** occurs through two SET processes with concomitant formation of two equivalents of the *tert*-butoxide anion from *tert*-butylperoxide. Overall imidate **12**, *tert*-butanol and *tert*-butoxide are generated which, upon protic work-up delivers the corresponding amide **6** and *tert*-butanol (Scheme 3).

Conclusions

In summary, we have documented a new and straightforward synthetic method for the catalytic synthesis of substituted *p*-nitroaromatic amides, based upon an azido-amidation mechanism. This method offers an orthogonal approach to current methodology and, in particular, the method works particularly well with electron deficient anilines, thus complementing existing methodologies involving activated carboxylic acid derivatives. Studies are currently ongoing to unravel the mechanistic details of the reaction.

Notes and references

[a] *School of Chemistry, University of Nottingham, England, UK, NG7 2RD; Te;: +44 (0)115 951 3533; E-mail: john.moses@nottingham.ac.uk*

† Electronic Supplementary Information (ESI) available: Full experimental procedures and characterisation with ^{1}H and ^{13}C NMRs and schemes referenced in the text.. See DOI: 10.1039/b000000x/

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Supporting Information

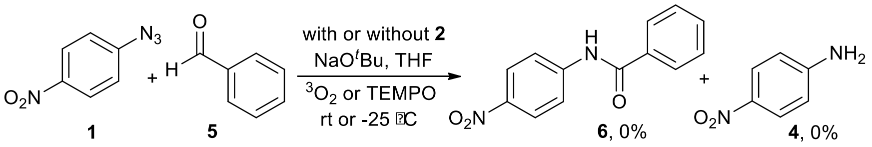
tert-Butoxide Promoted One-Pot Azidation-Amindation of *para*-nitroanilines with aldehydes

Giorgio Carbone, James Burnley and John E. Moses[*]

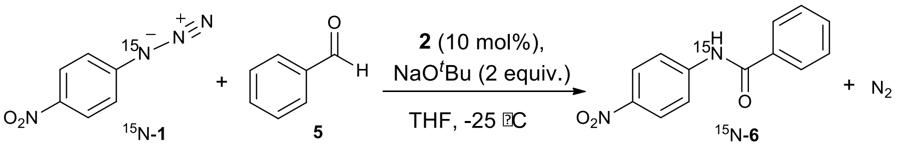
School of Chemistry, University of Nottingham, Nottingham, England,
UK, NG7 2RD

John.moses@nottingham.ac.uk To whom correspondence should be addressed.

Schemes and Figures



Scheme S1 Evidence for an electron transfer process in the azido-amidation reaction of azide **1** as both $^3O_2$ and TEMPO radical shut down the reaction.



Scheme S2 Experiment carried out using ^{15}N-labelled *p*-nitroaniline ^{15}N-**1** showed retention of the labeled ^{15}N atom in the corresponding amide ^{15}N-**6**.

Materials and Methods

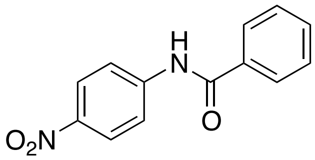
^{1}H and ^{13}C-NMR spectra were recorded on a Bruker AV (III) 400, Bruker AV 400,
Bruker DPX 400, AV 3500 (400MHz or 500 MHz (^{1}H), and 100 MHz or 125 MHz (^{13}C)) spectrometers. Chemical shifts are expressed in parts per million (ppm) and the spectra calibrated to residual solvent signals of DMSO (2.54 ppm (^{1}H) and 40.5 ppm (^{13}C)). Coupling constants are given in hertz (Hz) and the following notations indicate the multiplicity of the signals: s (singlet), d (doublet), brd (broad doublet), dd (double doublet), t (triplet), tt (triple triplet), q (quartet), m (multiplet). High Resolution Mass Spectra were recorded on a VG micron Autospec or Bruker microTOF. Fourier Transform Infrared Spectroscopy (FT-IR) spectra were obtained using a Perkin Elmer 1600 series or Bruker Tensor 27 spectrometer. Melting points were recorded using a STUART SMP3 apparatus and are uncorrected. Thin layer chromatography was carried out on Merck pre-coated silica gel plates (60F-254) and visualised using ultra violet light or $KMnO_4$ solution. THF was freshly distilled from sodium-benzophenone. Where necessary, reactions requiring anhydrous conditions were performed in dry solvents in flame dried or oven-dried apparatus under argon atmosphere.

Experimental Procedures

General procedure:

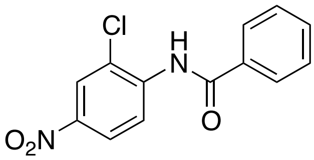
^{t}BuONO (0.53 mmol, 1.05 eq.) was added to a mixture of the aniline (0.50 mmol, 1.00 eq.) and $TMSN_3$ (0.53 mmol, 1.05 eq.) in THF (2 mL) at 0 ^{o}C. The mixture was stirred until deemed complete (TLC). Then, the solution was cooled to -25 ^{o}C at which temperature the aldehyde (0.75 mmol, 1.50 eq.), thiazolium salt **2** (0.05 mmol, 0.10 eq.) and NaO^{t}Bu (1.00 mmol, 2 eq.) were added sequentially. The resulting mixture was stirred until complete consumption of the azide (TLC). EtOAc (5 mL) and sat. $NaHSO_{3(aq)}$ (5 mL) were added and the layers separated. The organic layer was washed with $NaHSO_{3(aq)}$ (5 mL), 1 M $HCl_{(aq)}$ (5 mL), dried ($MgSO_4$), filtered and evaporated under reduced pressure to give the crude product. Purification of the crude residue by flash column chromatography on silica with [95:5:1-80:20:1 petrol:EtOAc:Et_3N] as eluent gave the corresponding amide.

***N*-(4-nitrophenyl)benzamide** (Table 2, Entry 1)



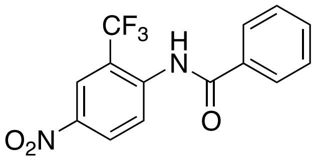
Yellow solid (0.139 g, 94 %); *R*f (70:30 petrol-EtOAc) 0.3; mp 198-199 ˚C (lit.,[1] 199-201 ˚C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3431 (NH), 1692 (C=O), 1507 (NO_2), 1405, 1345, 1243, 1113; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.8 (s, 1H), 8.29–8.26 (m, 2H) 8.08–8.06 (m, 2H), 7.99–7.97 (m, 2H), 7.66–7.64 (m, 1H), 7.59–7.55 (m, 2H); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ 166.5 (C), 145.7 (C), 142.7 (C), 134.2 (C), 132.4 (CH), 128.7 (CH), 128.1 (CH), 130.0 (CH), 120.9 (CH); ^{15}N NMR (100 MHz, DMSO-*d*$_6$) -248.5; HRMS ESI: calcd for $C_{13}H_{11}N_2O_3$ $[M+H]^+$, 243.0764; found, 243.0775; calcd for $C_{13}H_{10}NaN_2O_3$ $[M+Na]^+$, 265.0584; found 265.0588.

***N*-(3-chloro-4-nitrophenyl)benzamide** (Table 2, Entry 2)



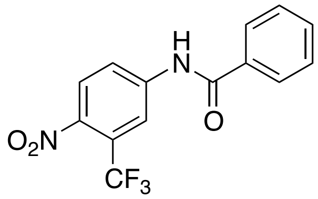
Yellow solid (0.117 g, 85%); *R*$_f$ (70:30 petrol-EtOAc) 0.3; mp 160 ˚C (lit.,[2] 162-164 ^{o}C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3417 (NH), 1697 (C=O), 1511 (NO_2), 1398, 1345, 1253, 1121; ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.3 (s, 1H), 8.42 (d, 1H, *J* = 2.6 Hz), 8.29 (dd, 1H, *J* = 8.9, 2.6 Hz), 8.26 (d, 1H, *J* = 8.9 Hz), 8.03–7.99 (m, 2H), 7.68–7.64 (m, 1H), 7.60–7.55 (m, 2H); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ 166.5 (C), 145.0 (C), 141.3 (C), 133.4 (C), 132.4 (CH), 128.6 (CH), 128.1 (C), 127.9 (CH), 166.6 (CH), 124.9 (CH), 122.9 (CH); HRMS ESI: calcd for $C_{13}H_{10}ClN_2O_3$ $[M+H]^+$, 277.0374; found, 277.0373; calcd for $C_{13}H_9ClN_2NaO_3$ $[M+Na]^+$, 299.0194; found 299.0189.

***N*-(4-nitro-2-(trifluoromethyl)phenyl)benzamide** (Table 2, Entry 3)



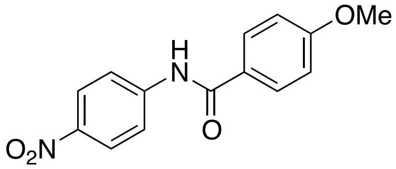
Yellow solid (0.119 g, 77%), *R*$_f$ (70:30 petrol-EtOAc) 0.3; mp 158–161 ˚C; IR (FTIR, $CHCl_3$) ν_{max} cm $^{-1}$: 3414 (NH), 1707 (C=O), 1534 (NO_2), 1508 (NO_2), 1398, 1345, 1161, 1050; ^{1}H NMR (500 MHz, DMSO-*d*$_6$) δ 10.42 (s, 1H), 8.57 (dd, *J* = 8.8, 2.5 Hz, 1H), 8.52 (d, *J* = 2.5 Hz, 1H), 7.98–7.95 (m, 3H), 7.67–7.63 (m, 1H), 7.51–7.55 (m, 2H); ^{13}C NMR (125 MHz, DMSO-*d*$_6$) δ 166.4 (C), 145.4 (C), 141.8 (C), 133.3 (C), 132.4 (CH), 131.8 (CH), 128.7 (CH), 128.0 (CH), 127.8 (CH), 126.3 (C, q, *J* = 33.1 Hz), 122.4 (CH); HRMS ESI: calcd for $C_{14}H_{10}F_3N_2O_3$ $[M+H]^+$, 311.0638; found, 311.0633; calcd for $C_{14}H_9F_3N_2NaO_3$ $[M+Na]^+$, 333.0457; found 333.0453.

***N*-(4-nitro-3-(trifluoromethyl)phenyl)benzamide** (Table 2, Entry 4)



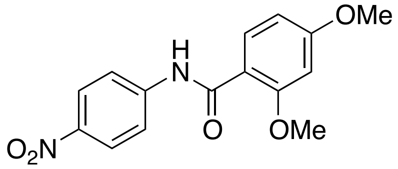
Yellow solid (0.130 g, 84%); R_f (70:30 petrol-EtOAc) 0.3; mp 129–131 ˚C (lit.,[3] 129 °C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3431 (NH), 1694 (C=O), 1520 (NO_2), 1415, 1322, 1250 (CF), 1163 (CF); ^1H NMR (500 MHz, DMSO-d_6) δ 11.0 (s, 1H), 8.49 (d, *J* = 2.2 Hz, 1H), 8.35 (dd, *J* = 9.0, 2.2 Hz, 1H), 8.26 (d, *J* = 9.0 Hz, 1H), 8.02–7.99 (m, 2H), 7.66–7.64 (m, 1H), 7.60–7.57 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) 170.0 (C), 144.4 (C), 142.1 (C), 134.2 (C), 133.0 (C), 129.7 (CH), 129.1 (CH), 128.4 (CH), 128.1 (CH), 123.7 (CH), 123.2 (C, q, *J* = 33.2 Hz), 118.8 (CH); HRMS ESI: calcd for $C_{14}H_9F_3N_2NaO_3$ $[M+Na]^+$, 333.0457; found 333.0450.

Methoxy-*N*-(4-nitrophenyl)benzamide (Table 2, Entry 5)



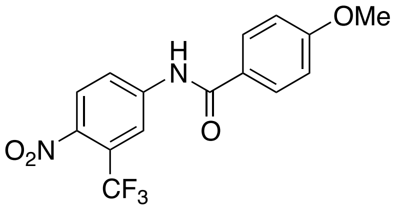
Yellow solid (0.124 g, 91 %); R_f (70:30 petrol-EtOAc) 0.3; mp 183-185 ˚C (lit.,[4] 184–185 °C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3435 (NH), 1685 (C=O), 1504 (NO_2), 1345, 1240, 1113, 1030; ^1H NMR (400 MHz, DMSO-d_6) δ 10.6 (s, 1H), 8.26–8.24 (m, 2H) 8.07–8.04 (m, 2H), 8.01–7.98 (m, 2H), 7.11–7.08 (m, 2H), 3.31 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 165.9 (C), 162.8 (C), 146.2 (C), 142.7 (C), 130.4 (CH), 126.5 (C), 125.2 (CH), 120.1 (CH), 114.2 (CH), 55.9 (CH_3); HRMS ESI: calcd for $C_{14}H_{13}N_2O_4$ $[M+H]^+$, 273.0870; found, 273.0869; calcd for $C_{14}H_{12}NaN_2O_4$ $[M+Na]^+$, 295.0689; found 295.0689.

3,5-Dimethoxy-*N*-(4-nitrophenyl)benzamide (Table 2, Entry 6)



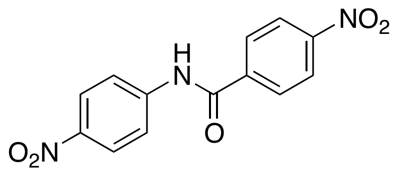
Yellow solid (0.119 g, 79%), R_f (70:30 petrol-EtOAc) 0.2; mp 198-199°C; IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3356 (NH), 1674 (C=O), 1604, 1548 (NO_2), 1342, 1249, 1027; ^1H NMR (400 MHz, DMSO-d_6) δ 10.45 (s, 1H), 8.26–8.22 (m, 2H), 8.01-7.98 (m, 2H), 7.72 (d, J = 8.6 Hz, 1H), 6.72 (d, J = 2.3 Hz, 1H), 6.68 (dd, J = 8.6, 2.3 1H), 3.94 (s, 3H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 164.5 (C), 163.4 (C), 158.5 (C), 145.2 (C), 142.2 (C), 132.0 (CH), 124.9 (CH), 119.4 (CH), 115.8 (C), 105.9 (CH), 95.6 (CH), 56.7 (CH_3), 56.1 (CH_3); HRMS ESI: calcd for $C_{15}H_{15}N_2O_5$ $[M+H]^+$, 303.0975; found, 303.0973; calcd for $C_{15}H_{14}N_2NaO_5$ $[M+Na]^+$, 325.0795; found 325.0792.

4-Methoxy-N-(4-nitro-3-(trifluoromethyl)phenyl)benzamide (Table 2, Entry 7)



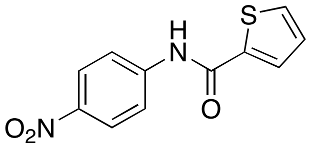
Yellow solid (0.138 g, 89%); R_f (70:30 petrol-EtOAc) 0.3; mp 110–113 °C (lit.,[3] 112–114 °C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3436 (NH), 1686 (C=O), 1508 (NO_2), 1250 (CF), 1175 (CF), 1097, 1030; ^1H NMR (500 MHz, DMSO-d_6) δ 10.80 (s, 1H), 8.48 (d, J = 2.3 Hz, 1H), 8.35 (dd, J = 9.0, 2.3 Hz, 1H), 8.23 (d, J = 9.0 Hz, 1H), 8.02–7.99 (m, 2H), 7.12–7.10 (m, 2H), 3.32 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 165.7 (C), 162.7 (C), 144.3 (C), 141.4 (C), 130.1 (CH), 127.7 (CH), 125.6 (C), 123.0 (CH), 122.5 (C, q, J = 33.2 Hz), 121.1 (C), 118.2 (CH), 133.9 (CH), 55.6 (CH_3); HRMS ESI: calcd for $C_{15}H_{12}F_3N_2O_4$ $[M+H]^+$, 341.0744; found, 341.0743; calcd for $C_{15}H_{11}F_3N_2NaO_4$ $[M+Na]^+$, 363.0563; found 363.0568.

4-Nitro-*N*-(4-nitrophenyl) benzamide (Table 2, Entry 8)



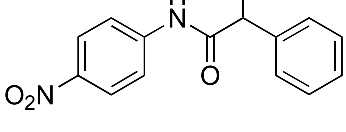
Yellow solid (0.052 g, 38%), R_f (70:30 petrol-EtOAc) 0.2; mp 266-270 ˚C (lit.,[5] 267-269 ^{o}C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3693 (NH), 1600 (C=O), 1530 (NO_2), 1511 (NO_2), 1346, 1241, 1113; ^{1}H NMR (500 MHz, DMSO-d_6) δ 11.11 (s, 1H), 8.42–8.41 (m, 2H), 8.39–8.38 (m, 2H), 8.23–8.22 (m, 2H), 8.09–8.05 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) 165.2 (C), 149.0 (C), 145.5 (C), 143.3 (C), 140,4 (C), 130.0 (CH), 125.4 (CH), 124.1 (CH), 120.6 (CH); HRMS ESI: calcd for $C_{13}H_{10}N_3O_5$ [M+H]$^+$, 288.2351; found, 288.9213; calcd for $C_{13}H_9N_3NaO_5$ [M+Na]$^+$, 310.2169; found 310.0425.

***N*-(4-nitrophenyl)thiophene-2-carboxamide** (Table 2, Entry 9)



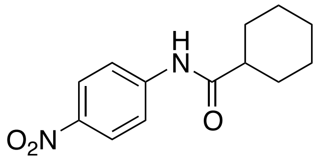
Yellow solid (0.102 g, 82%), R_f (70:30 petrol:EtOAc) 0.3; mp 221-223 ˚C (lit.,[6] 222-224 ^{o}C); IR (FTIR, $CHCl_3$) ν_{max} cm $^{-1}$: 3430 (NH), 1673 (amide), 1600, 1506 (NO_2), 1344, 1245, 1113; ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.75 (s, 1H), 8.29–8.25 (m, 2H), 8.10 (dd, *J* = 3.8, 1.1 Hz, 1H), 8.04–8.00 (m, 2H), 7.95 (dd, *J* = 5.0, 1.1 Hz, 1H), 7.27 (dd, *J* = 5.0, 3.8 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 160.5 (C), 145.2 (C), 142.5 (C), 139.1 (C), 133.1 (CH), 130.3 (CH), 128.3 (CH), 124.9 (CH), 119.8 (CH); HRMS ESI: calcd for $C_{11}H_9N_2O_3S$ [M+H]$^+$, 249.0328; found, 249.0315; calcd for $C_{11}H_8N_2NaO_3S$ [M+Na]$^+$, 271.0148; found 271.0140.

***N*-(4-nitrophenyl)-2-phenylpropanamide** (Table 2, Entry 10)



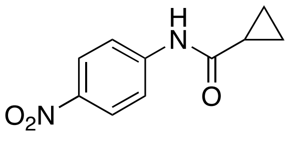
Yellow solid (0.127 g, 94%), R_f (70:30 petrol:EtOAc) 0.3; mp 168–169 ˚C; IR (FTIR, $CHCl_3$) ν_{max} cm $^{-1}$: 3401 (NH), 1702 (C=O), 1599, 1533 (NO_2), 1506 (NO_2), 1344, 1249, 1177, 1114; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.65 (s, 1H), 8.22–8.18 (m, 2H), 7.86–7.82 (m, 2H), 7.40–7.32 (m, 4H), 7.26–7.22 (m, 1H), 3.89 (q, *J* =7.0 Hz, 1H), 1.43 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 173.2 (C), 145.3 (C), 142.1 (C), 141.2 (C), 128.5 (CH), 127.3 (CH), 126.9 (CH), 124.9 (CH), 118.9 (CH), 46.2 (CH), 18.6 (CH_3); HRMS ESI: calcd for $C_{15}H_{15}N_2O_3$ $[M+H]^+$, 271.1077; found, 271.1070; calcd for $C_{15}H_{14}N_2NaO_3$ $[M+Na]^+$, 293.0897; found 293.0901.

***N*-(4-nitrophenyl)cyclohexanecarboxamide** (Table 2, Entry 11)



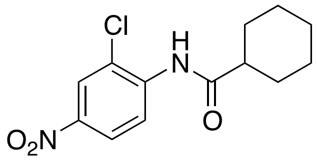
Colourless solid (0.109 g, 88%); R_f (70:30 petrol-EtOAc) 0.4; mp 160–163 ˚C (lit.,[7] 162-163 °C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3429 (NH), 1702 (C=O)), 1505 (NO_2), 1345, 1246, 1163, 1114; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.4 (s, 1H), 8.21–8.17 (m, 2H), 7.86–7.83 (m, 2H), 2.83 (tt, *J* = 11.5, 3.5 Hz, 1H), 1.84–1.81 (m, 2H), 1.78– 1.74 (m, 2H), 1.66–1.64 (m, 1H), 1.45–1.16 (m, 5H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.2 (C), 145.7 (C), 141.9 (C), 124.9 (CH), 118.6 (CH), 44.9 (CH), 28.9 (CH_2), 25.3 (CH_2), 25.1 (CH_2); HRMS ESI: calcd for $C_{13}H_{16}NaN_2O_3$ $[M+Na]^+$, 271.1053; found 271.1055.

***N*-(4-nitrophenyl)cyclopropanecarboxamide** (Table 2, Entry 12)[9]



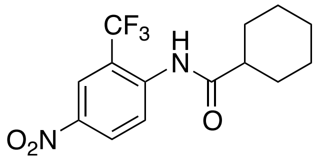
Yellow solid (0.102 g, 78%), R_f (70:30 petrol:EtOAc) 0.4; mp 145-147 ˚C (lit.,[8] 183-185 ^{o}C); IR (FTIR, $CHCl_3$) ν_{max} cm $^{-1}$: 3430 (NH), 1701 (C=O), 1507 (NO_2), 1343 (NO_2), 1159, 1035, 953, 853; ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.81 (s, 1H), 8.22–8.18 (m, 2H), 7.85–7.81 (m, 2H), 2.50 (app quintet, *J* = 1.8 Hz, 1H), 0.88–0.86 (m, 4H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 172.7 (C), 144.4 (C), 141.9 (C), 125.0 (CH), 118.6 (CH), 14.8 (CH), 7.9 (CH_2); HRMS ESI: calcd for $C_{10}H_{10}N_2NaO_3$ $[M+Na]^+$, 229.0584; found, 229.0581.

***N*-(2-chloro-4-nitrophenyl)cyclohexanecarboxamide** (Table 2, Entry 13)



Colourless solid (0.130 g, 92%); R_f (70:30 petrol-EtOAc) 0.4; mp 156-158 ˚C; IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3692 (NH), 1619 (C=O), 1524 (NO_2), 1443, 1241, 1025, 930; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.69 (s, 1H), 8.34–8.34 (m, 1H), 8.19–8.19 (m, 2H), 2.64 (tt, *J* = 11.4, 3.4 Hz, 1H), 1.86–1.83 (m, 2H), 1.76–1.74 (m, 2H), 1.67 – 1.64 (m, 1H), 1.45–1.16 (m, 5H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 175.2 (C), 143.1 (C), 141.3 (C), 124.9 (CH), 124.8 (C), 124.1 (CH), 123.0 (CH), 44.1 (CH), 29.1 (CH_2), 25.3 (CH_2), 25.1 (CH_2); HRMS ESI: calcd for $C_{13}H_{16}ClN_2O_3$ $[M+H]^+$, 283.0844; found, 283.0834; calcd for $C_{13}H_{15}ClN_2NaO_3$ $[M+Na]^+$, 305.0663; found 305.0653.

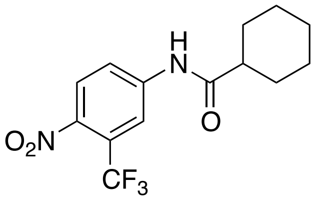
***N*-(4-nitro-2-(trifluoromethyl)phenyl)cyclohexanecarboxamide** (Table 2, Entry 14)



Colourless solid (0.131 g, 83%): R_f (70:30 petrol-EtOAc) 0.4; mp 142-145 ˚C; IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3428 (NH), 1706 (C=O), 1517 (NO_2), 1320, 1275 (CF), 1163 (CF), 1077; ^{1}H NMR (500 MHz, DMSO-d_6) δ 9.72 (s, 1H), 8.49 (dd, *J* = 8.9, 2.6 Hz, 1H), 8.44 (d, *J* = 2.6 Hz, 1H), 7.89 (d, *J* = 8.9 Hz, 1H), 2.55 (tt, *J* = 11.5, 3.4 Hz, 1H), 1.85–1.82 (m, 2H), 1.78–1.74 (m, 2H), 1.66–1.62 (m, 1H), 1.44–1.16 (m, 5H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 175.2 (C), 144.2 (C), 141.6 (C), 130.1 (CH), 127.8 (CH), 123.5 (C, q, *J* = 31.3 Hz), 122.2 (CH), 121.4 (C), 43.8 (CH), 28.9 (CH_2), 25.4 (CH_2), 25.1 (CH_2); HRMS ESI: calcd for

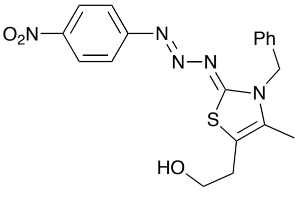
$C_{14}H_{16}F_3N_2O_3$ [M+H]$^+$, 317.1108; found, 317.1094; calcd for $C_{14}H_{15}F_3N_2NaO_3$ [M+Na]$^+$, 339.0927; found 339.0914.

***N*-(4-nitro-3-(trifluoromethyl)phenyl)cyclohexanecarboxamide** (Table 2, Entry 15)



Colourless solid (0.138 g, 87%); R_f (70:30 petrol-EtOAc) 0.4; mp 128-130 °C (lit.,[3] 129-131 °C); IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3415 (NH), 1706 (C=O), 1517 (NO_2), 1380, 1225 (CF), 1142 (CF), 1068; ^{1}H NMR (500 MHz, DMSO-*d*$_6$) δ 10.62 (s, 1H), 8.29 (d, *J* = 2.2 Hz, 1H), 8.17 (d, *J* = 8.9 Hz, 1H), 8.04 (dd, *J* = 8.9, 2.2 Hz, 1H), 2.36 (tt, *J* = 11.4, 3.4 Hz, 2H), 1.85–1.82 (m, 2H), 1.77-173 (m, 2H), 1.66–1.63 (m, 1H), 1.45–1.15 (m, 5H); ^{13}C NMR (125 MHz, DMSO-*d*$_6$) δ 175.6 (C), 144.2 (C), 141.1 (C), 127.8 (CH), 122.9 (C, q, *J* = 31.3 Hz), 122.0 (CH), 121.0 (C), 117.1 (CH), 45.1 (CH), 28.9 (CH_2), 25.3 (CH_2), 25.1 (CH_2); HRMS ESI: calcd for $C_{14}H_{16}F_3N_2O_3$ [M+H]$^+$, 317.1108; found, 317.1095; calcd for $C_{14}H_{15}F_3N_2NaO_3$ [M+Na]$^+$, 339.0927; found 339.0919.

2-((*Z*)-3-benzyl-4-methyl-2-((*E*)-(4-nitrophenyl)triaz-2-en-1-ylidene)-2,3-dihydrothiazol-5-yl) ethanol (7)



1-azido-4-nitrobenzene **1** (50.0 mg, 0.305 mmol) and the thiazolium salt **2** (82.0 mg, 0.305 mmol) were dissolved in THF (1 mL) and the corresponding mixture was cooled to -78 °C. NaH (30.5 mg, 60% w/w mineral oil, 0.761 mmol) was added to the mixture in one portion. The reaction mixture was allowed to warm to room temperature, at which point a bright red colour appeared. The reaction was stirred at room temperature until complete (TLC, 5 h). The reaction mixture was then poured onto saturated ammonium chloride solution (5 mL) and the products extracted with ethyl acetate (3 x 5 mL). The combined organic extracts were dried over anhydrous magnesium sulfate, filtered and the solvents removed *in vacuo*. The resulting residue was finally subjected to flash column chromatography (eluting with ethyl acetate) to deliver the product as a bright red solid (98.0 mg, 81%), R_f (EtOAc) 0.2; IR (FTIR, $CHCl_3$) ν_{max} cm^{-1}: 3108 (OH), 1520 (NO_2), 1428 (N=N), 1327, 1134, 1106; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.28 (d, *J* = 8.9 Hz, 2H), 7.64 (d, *J* = 8.9 Hz, 2H), 7.41-7.37 (m, 2H), 7.33-7.30 (m, 1H), 7.26-7.24 (m, 2H), 5.42 (s, 2H), 4.93 (t, *J* = 5.7 Hz, 1H), 3.59 (app q, *J* = 5.7 Hz, 2H), 2.76 (t, *J* = 5.7 Hz, 2H), 2.16 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 176.8 (C), 155.6 (C), 145.4 (C), 135.8 (C), 133.1 (C), 128.9 (CH), 127.7 (CH), 126.5 (CH), 125.0 (CH), 121.7 (CH), 116.5 (C), 60.5 (CH_2), 48.6 (CH_2), 29.7 (CH_2), 11.2 (CH_3); HRMS ESI: calcd for $C_{19}H_{20}N_5O_3S$ $[M+H]^+$, 398.1281; found, 398.1291; calcd for $C_{19}H_{19}N_5NaO_3S$ $[M+Na]^+$, 420.1101; found, 420.1122.

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NMR spectra

2-((*Z*)-3-benzyl-4-methyl-2-((*E*)-(4-nitrophenyl)triaz-2-en-1-ylidene)-2,3-dihydrothiazol-5-yl) ethanol (SI1)

12/10/2012 14:32:40
UserID j_bur SampleID jb1254 SupervisorID moses Lab Phone No. 13540 Slot Number 35

Acquisition Time (sec)	3.9846	**Comment**	UserID j_bur SampleID jb1254 SupervisorID moses Lab Phone No. 13540 Slot Number 35				
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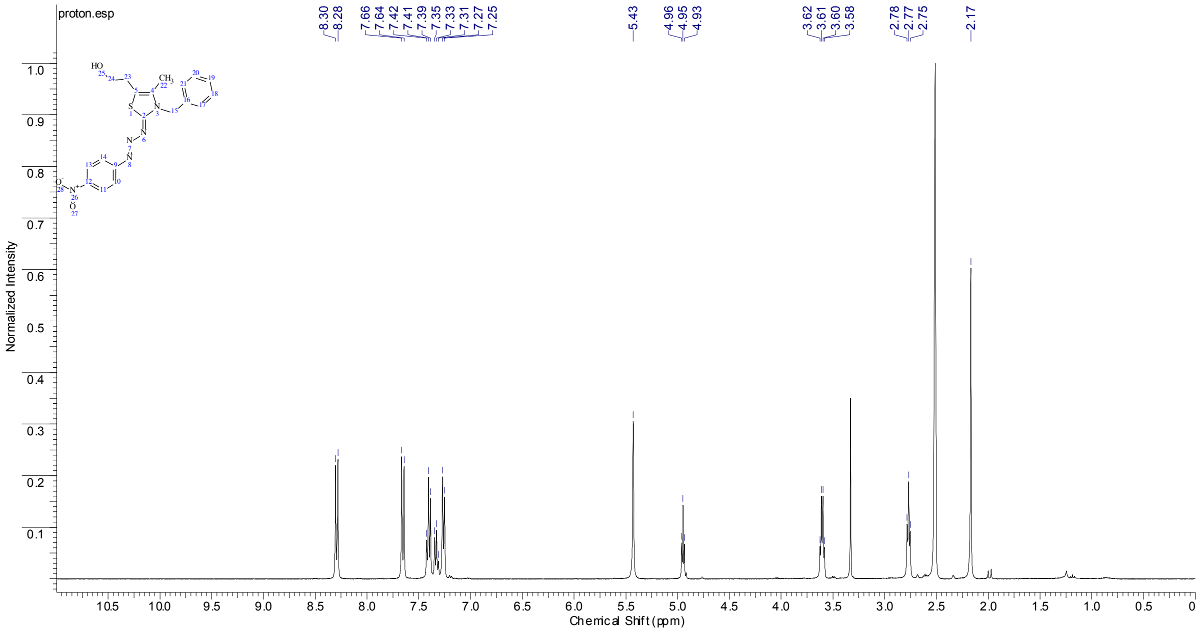
Frequency (MHz)	400.07	**Nucleus**	1H	**Number of Transients**	16	**Origin**	av3400
Original Points Count	32768	**Owner**	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30
Receiver Gain	406.00	**SW(cyclical) (Hz)**	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2470.5977
Spectrum Type	STANDARD	**Sweep Width (Hz)**	8223.56	**Temperature (degree C)**	25.000		



R:\Moses Group Work\James Burnley\azide reduction paper\NMR SI\triazene\proton.esp

UserID j_bur SampleID jb1254 SupervisorID moses Lab Phone No. 13540 Slot Number 12

Formula	$C_{19}H_{19}N_5O_3S$	**FW**	397.4509

Acquisition Time (sec)	0.6832	**Comment**	UserID j_bur SampleID jb1254 SupervisorID moses Lab Phone No. 13540 Slot Number 12				
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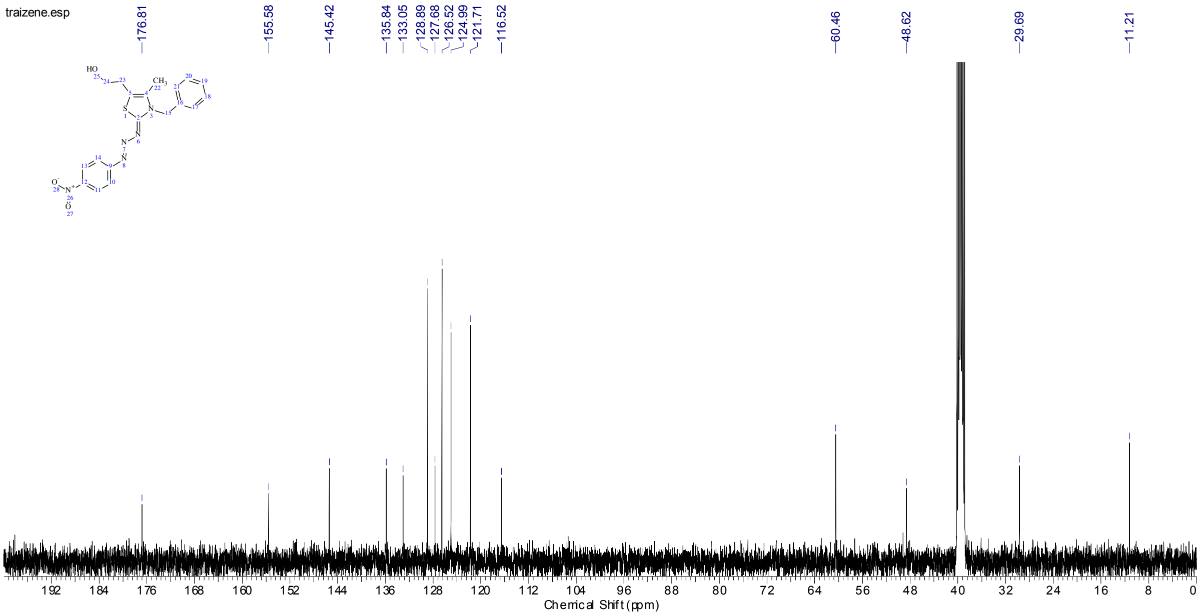
Original Points Count	16384	**Owner**	nmruser	**Points Count**	32768	**Pulse Sequence**	zgpg30
Receiver Gain	18390.40	**SW(cyclical) (Hz)**	23980.81	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	11017.3057
Spectrum Type	STANDARD	**Sweep Width (Hz)**	23980.08	**Temperature (degree C)**	25.160		

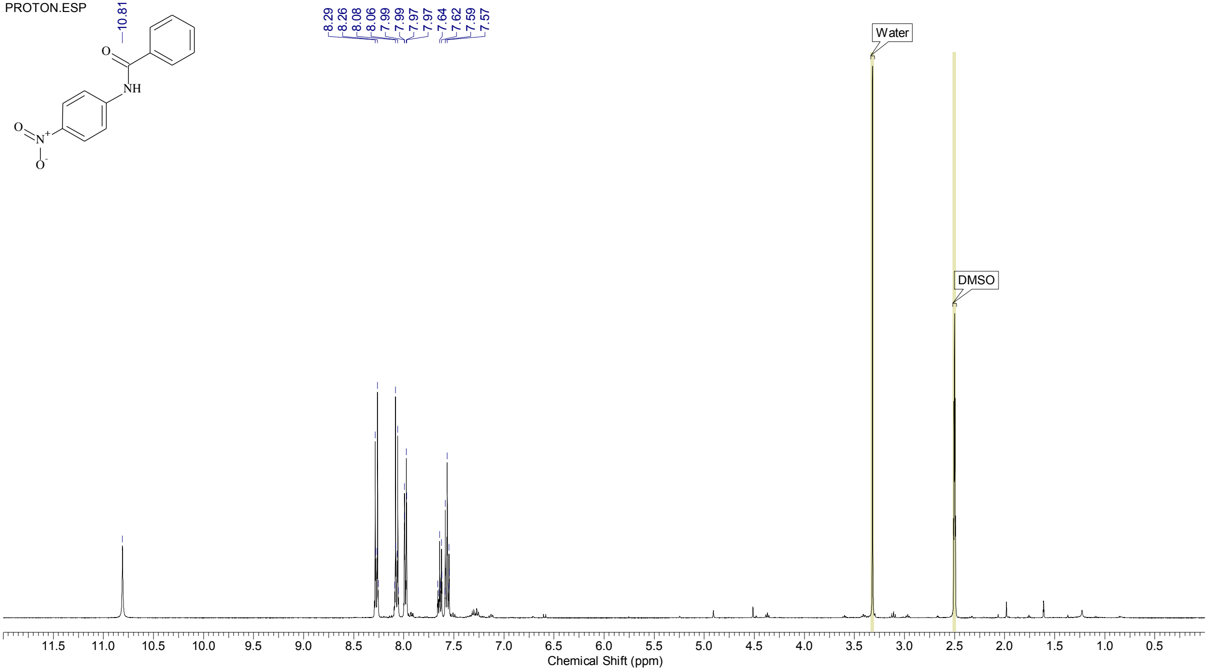


\\128.243.125.107\Public\Moses Group Work\James Burnley\azide reduction paper\NMR SI\triazene\traizene.esp

***N*-(4-nitrophenyl)benzamide** (Table 2, Entry 1)

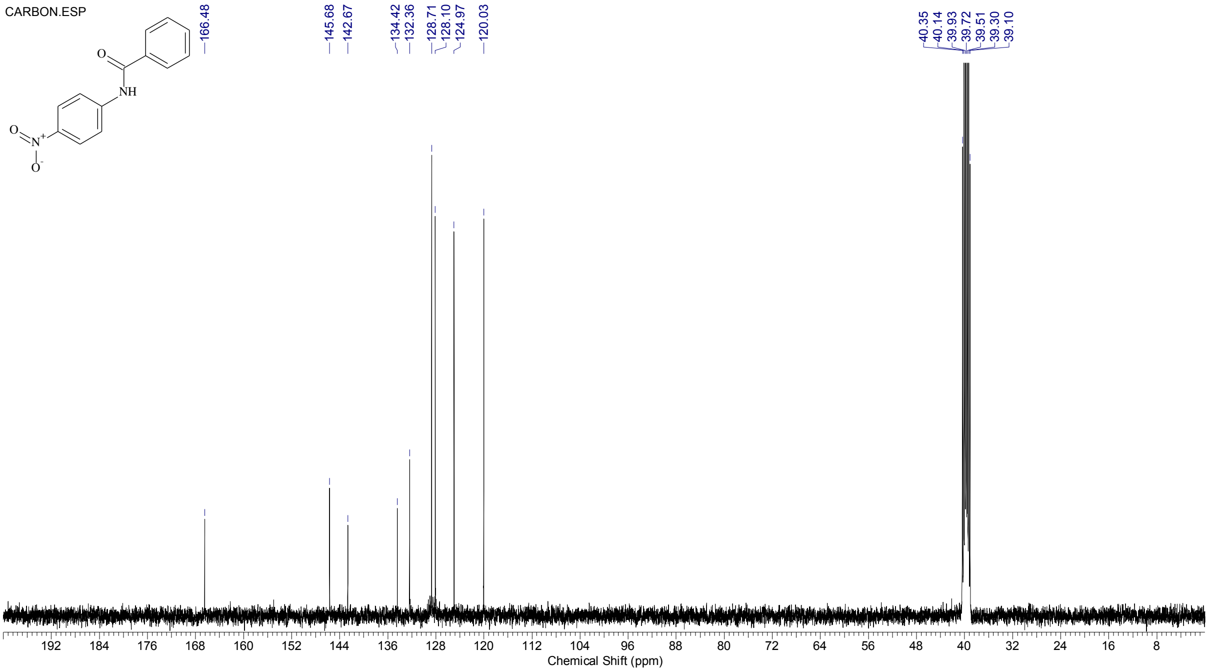
Formula $C_{13}H_{10}N_2O_3$	***FW*** 242.2301

Acquisition Time (sec)	3.9846	***Comment***	Slot No. 48 Sample ID gc3-48-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	29 Mar 2012 08:32:16			***Date Stamp***	29 Mar 2012 08:32:16		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC3-48-1\1\PDATA\1\1R					***Frequency (MHz)***	400.20
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	645.10
SW(cyclical) (Hz)	8223.68	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	2465.0803	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	25.000				



Formula $C_{13}H_{10}N_2O_3$	***FW*** 242.2301

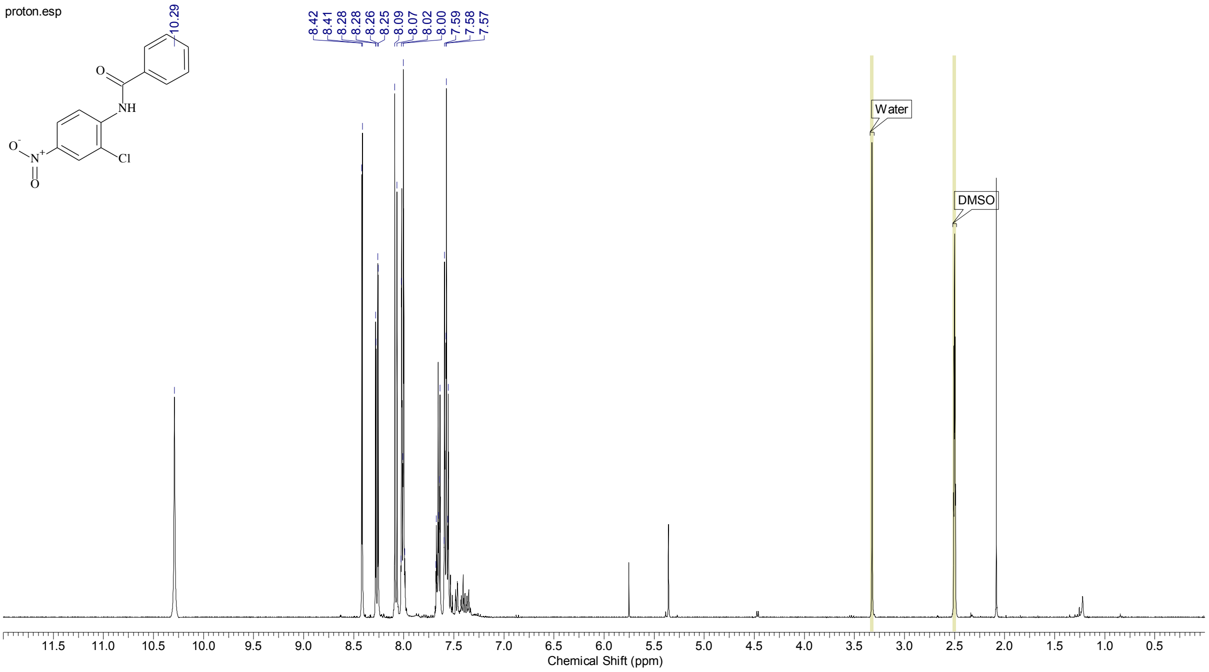
Acquisition Time (sec)	0.6521	***Comment***	Slot No. 48 Sample ID gc3-48-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	31 Mar 2012 19:16:32			***Date Stamp***	31 Mar 2012 19:16:32		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC3-48-1\2\PDATA\1\1R					***Frequency (MHz)***	100.63
Nucleus	13C	***Number of Transients***	2048	***Origin***	dpx400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	9195.20
SW(cyclical) (Hz)	25125.63	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	11039.0381	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.000				



***N*-(3-chloro-4-nitrophenyl)benzamide** (Table 2, Entry 2)

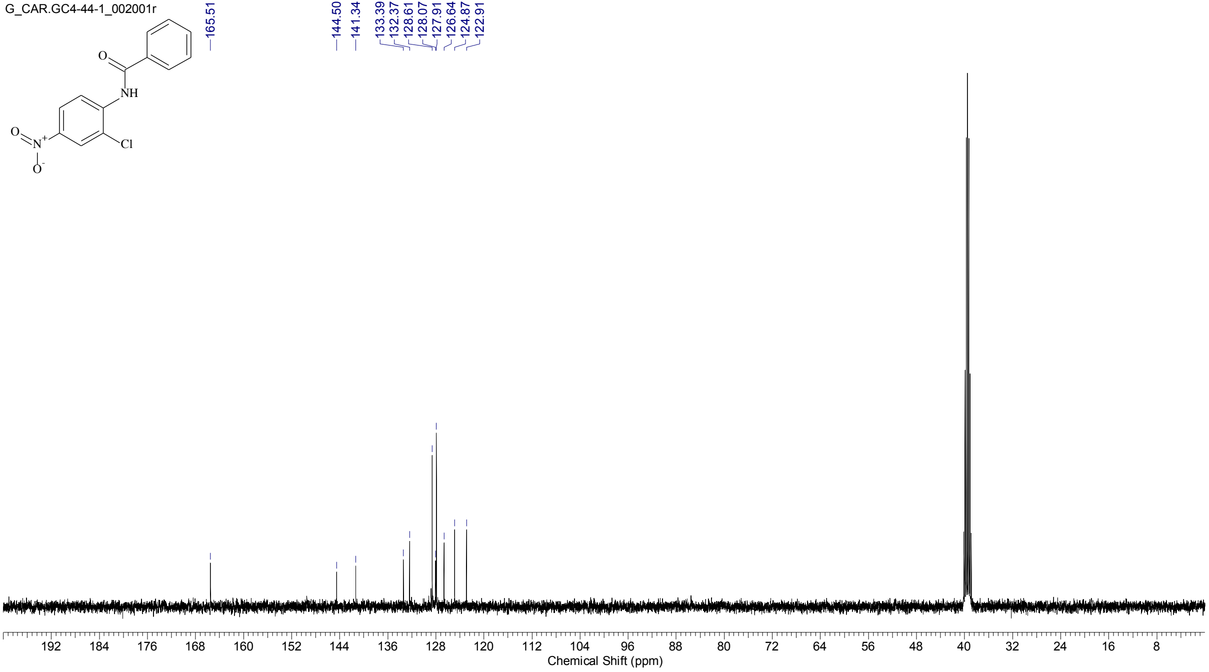
Formula $C_{13}H_9ClN_2O_3$	FW	276.6752

Acquisition Time (sec)	3.9846	***Comment***	Slot No. 24 Sample ID gc4-44-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	30 Mar 2012 09:34:08			***Date Stamp***	30 Mar 2012 09:34:08		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-44-1\1\PDATA\1\1R					***Frequency (MHz)***	400.20
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	456.10
SW(cyclical) (Hz)	8223.68	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	2465.0803	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	25.000				



Formula $C_{13}H_9ClN_2O_3$	FW	276.6752

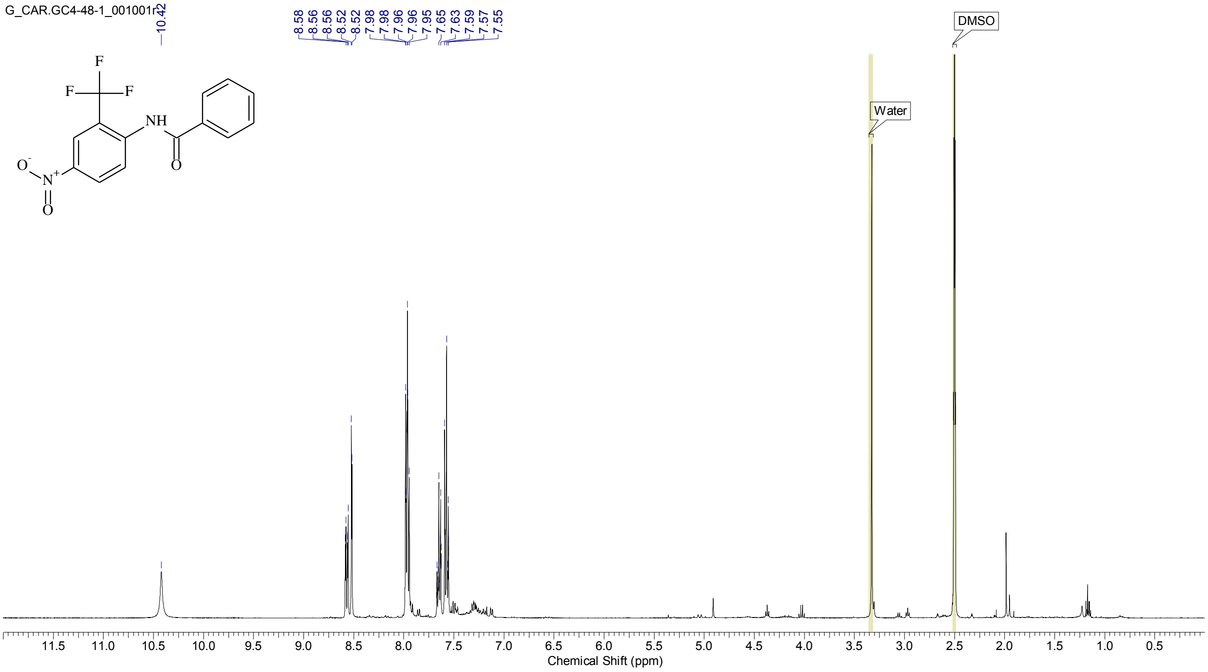
Acquisition Time (sec)	0.6521	***Comment***	Slot No. 24 Sample ID gc4-44-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	30 Mar 2012 09:40:32			***Date Stamp***	30 Mar 2012 09:40:32		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-44-1\2\PDATA\1\1R					***Frequency (MHz)***	100.63
Nucleus	13C	***Number of Transients***	128	***Origin***	dpx400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	4597.60
SW(cyclical) (Hz)	25125.63	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	11017.5684	***Spectrum Type***	STANDARD
Sweep Width (Hz)	25124.86	***Temperature (degree C)***	25.100				



***N*-(4-nitro-2-(trifluoromethyl)phenyl)benzamide** (Table 2, Entry 3)

Formula $C_{14}H_9F_3N_2O_3$	**FW** 310.2281

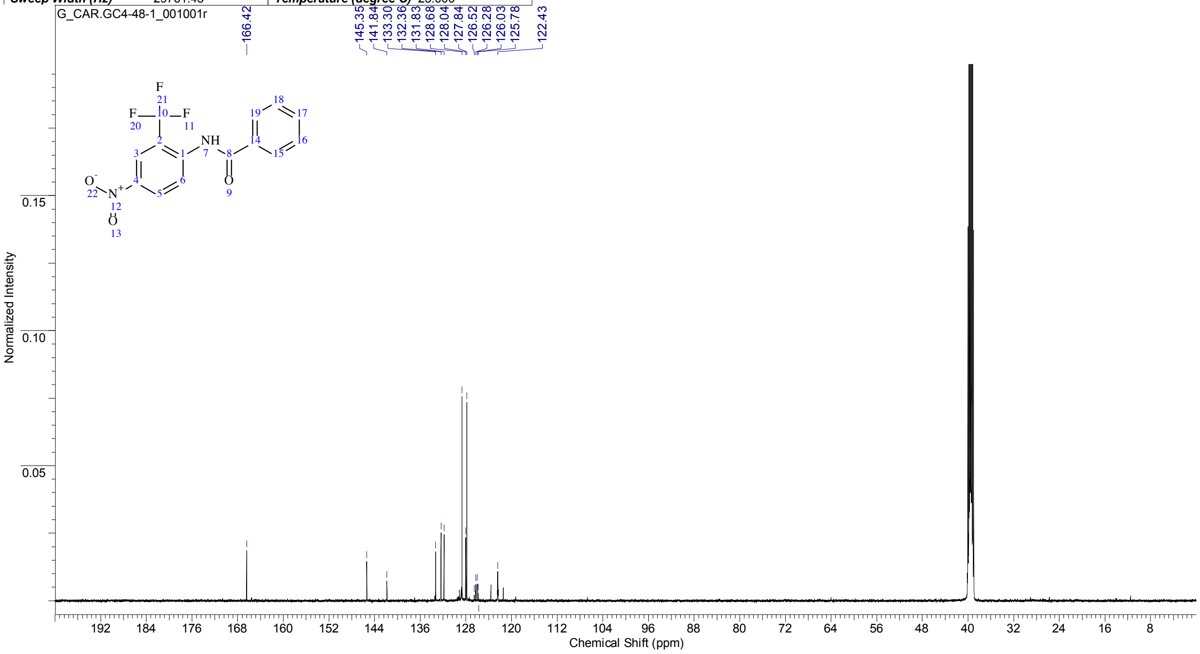
Acquisition Time (sec)	3.9846	**Comment**	Slot No. 39 Sample ID gc4-48-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	05 Apr 2012 05:50:08			**Date Stamp**	05 Apr 2012 05:50:08		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-48-1\1\PDATA\1\1R					**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400	**Original Points Count**	32768
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30	**Receiver Gain**	287.40
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.2061	**Spectrum Type**	STANDARD
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.000				



01/06/2012 16:41:09

Formula $C_{14}H_9F_3N_2O_3$	**FW** 310.2281

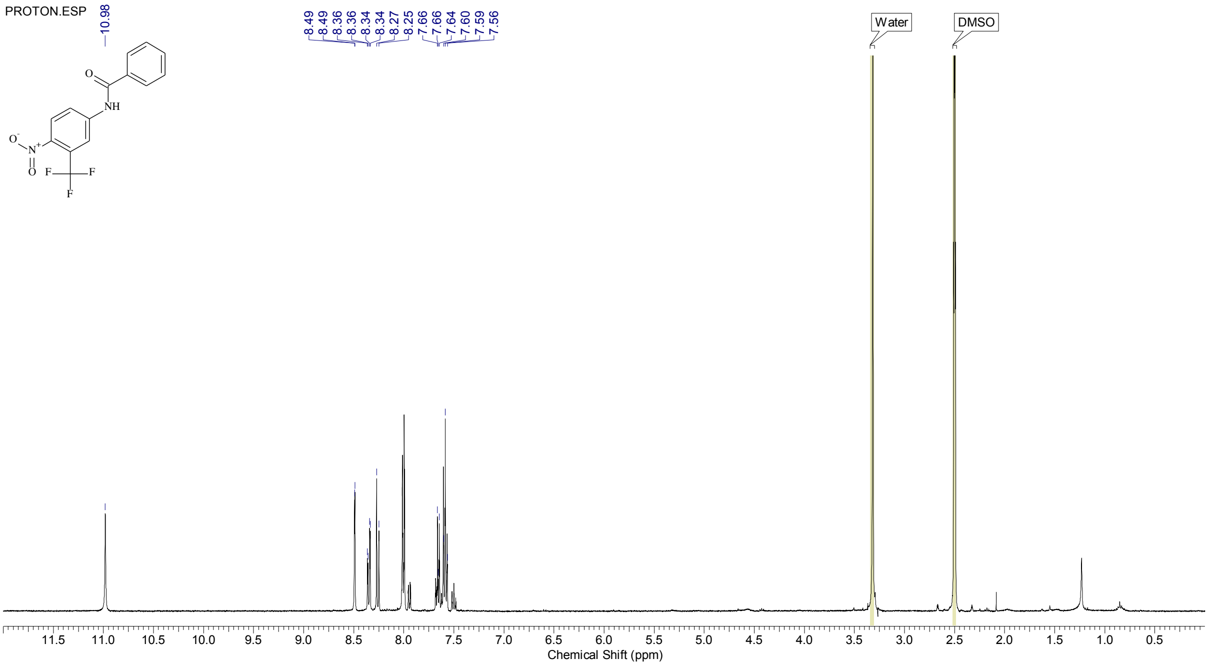
Acquisition Time (sec)	1.1010	**Comment**	UserID g_car SampleID gc4-48-1 SupervisorID moses Lab Phone No. 13540 Slot 1				
Date	22 May 2012 10:46:40			**Date Stamp**	22 May 2012 10:46:40		
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC4-48-1\1\PDATA\1\1R					**Frequency (MHz)**	125.76
Nucleus	13C	**Number of Transients**	320	**Origin**	av3500	**Original Points Count**	32768
Owner	service	**Points Count**	65536	**Pulse Sequence**	zgpg30	**Receiver Gain**	2050.00
SW(cyclical) (Hz)	29761.90	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	13777.2344	**Spectrum Type**	STANDARD
Sweep Width (Hz)	29761.45	**Temperature (degree C)**	25.000				



***N*-(4-nitro-3-(trifluoromethyl)phenyl)benzamide** (Table 2, Entry 4)

Formula $C_{14}H_{9}F_{3}N_{2}O_{3}$	**FW** 310.2281

Acquisition Time (sec)	3.9846	***Comment***	Slot No. 52 Sample ID gc4_28_2 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	22 Mar 2012 10:10:08			***Date Stamp***	22 Mar 2012 10:10:08		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4_28_2\6\PDATA\1\1R					***Frequency (MHz)***	400.20
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx400	***Original Points Count***	32768
Owner	nmruser	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	912.30
SW(cyclical) (Hz)	8223.68	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	2465.0803	***Spectrum Type***	STANDARD
Sweep Width (Hz)	8223.56	***Temperature (degree C)***	25.000				



01/06/2012 15:16:40

Formula $C_{14}H_{9}F_{3}N_{2}O_{3}$	**FW** 310.2281

Acquisition Time (sec)	1.1010	***Comment***	UserID g_car SampleID gc4-41-1 SupervisorID moses Lab Phone No. 13540 Slot 1				
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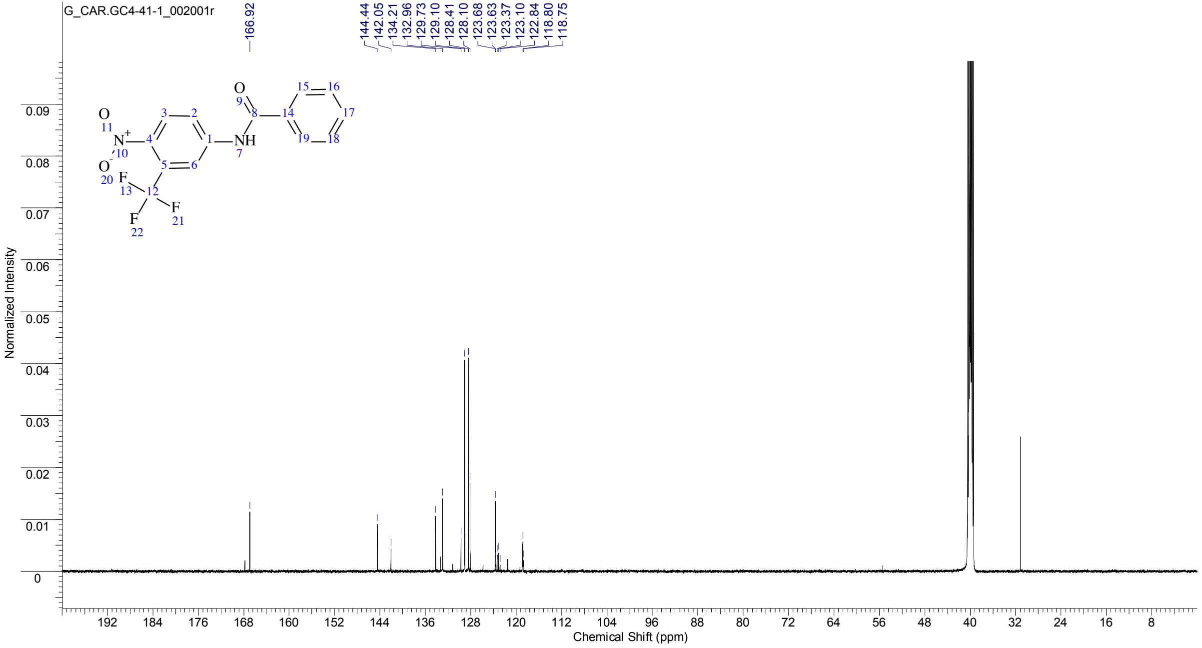
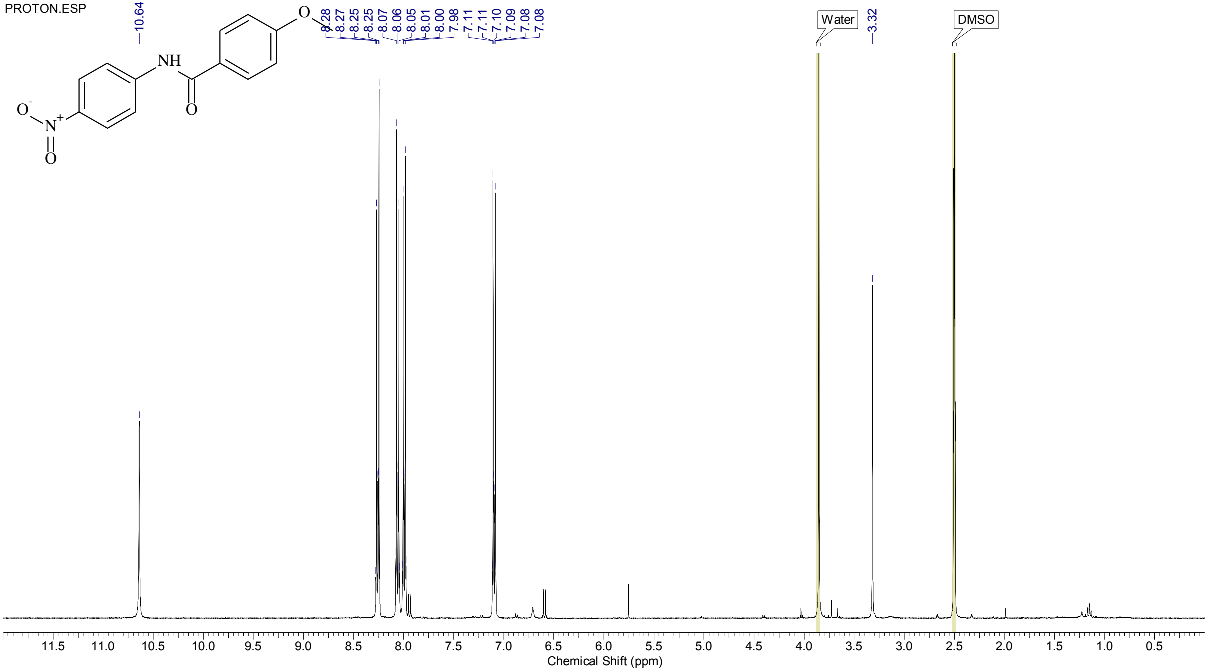
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC4-41-1\2\PDATA\1\1R					***Frequency (MHz)***	125.76
Nucleus	13C	***Number of Transients***	1024	***Origin***	av3500	***Original Points Count***	32768
Owner	service	***Points Count***	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	2050.00
SW(cyclical) (Hz)	29761.90	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	13833.3623	***Spectrum Type***	STANDARD
Sweep Width (Hz)	29761.45	***Temperature (degree C)***	25.000				



Methoxy-*N*-(4-nitrophenyl)benzamide (Table 2, Entry 5)

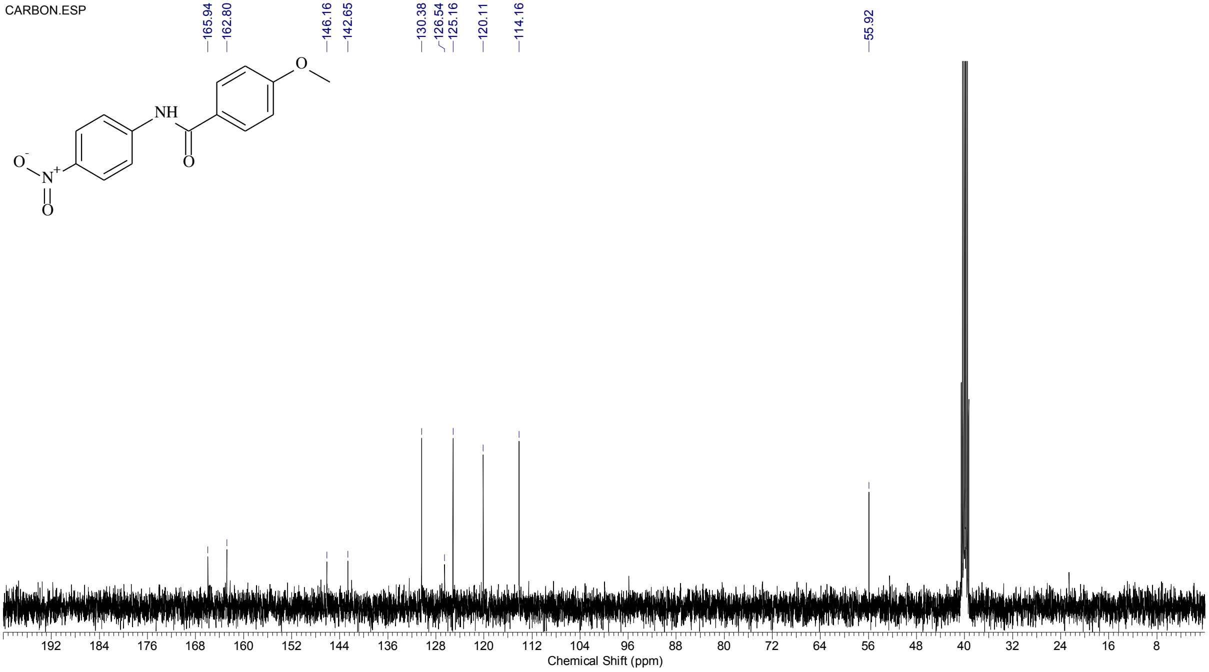
Formula	$C_{14}H_{12}N_2O_4$	FW	272.2561

Acquisition Time (sec)	3.9846	**Comment**	Slot No. 39 Sample ID gc4-39-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	28 Mar 2012 15:11:12			**Date Stamp**	28 Mar 2012 15:11:12		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-39-1\2\PDATA\1\1R					**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400	**Original Points Count**	32768
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30	**Receiver Gain**	645.10
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.0803	**Spectrum Type**	STANDARD
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.000				



Formula	$C_{14}H_{12}N_2O_4$	FW	272.2561

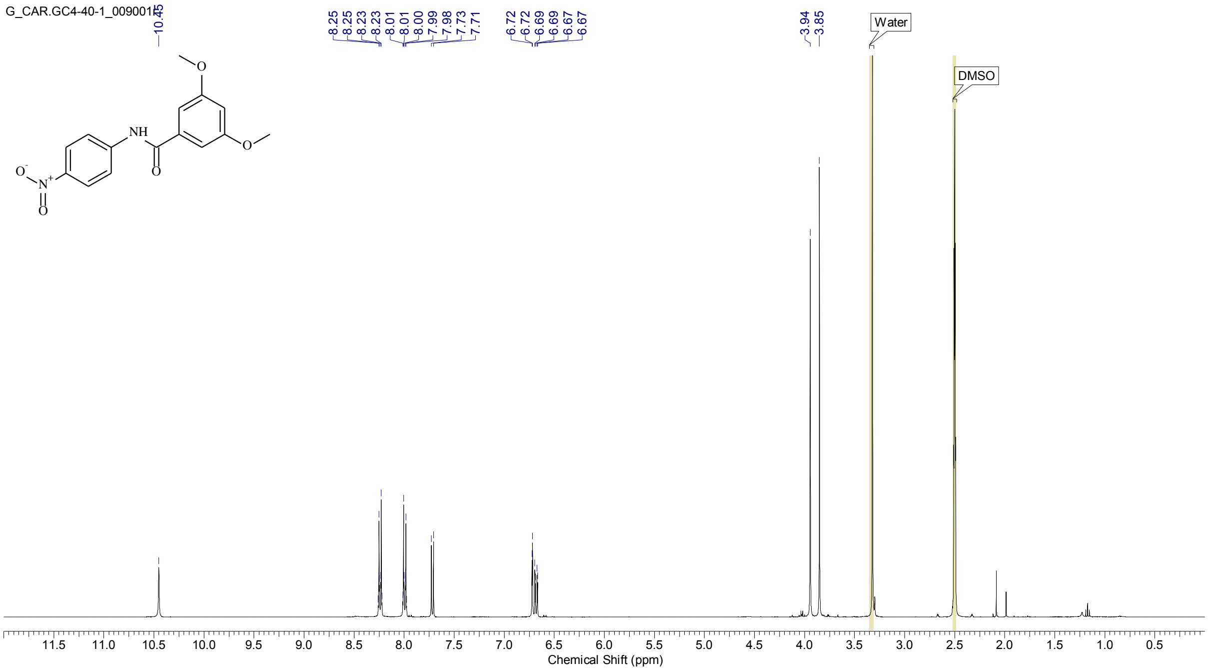
Acquisition Time (sec)	0.6521	**Comment**	Slot No. 39 Sample ID gc4-39-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	28 Mar 2012 15:41:04			**Date Stamp**	28 Mar 2012 15:41:04		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-39-1\3\PDATA\1\1R					**Frequency (MHz)**	100.63
Nucleus	13C	**Number of Transients**	128	**Origin**	dpx400	**Original Points Count**	16384
Owner	nmruser	**Points Count**	32768	**Pulse Sequence**	zgpg30	**Receiver Gain**	18390.40
SW(cyclical) (Hz)	25125.63	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	11060.5078	**Spectrum Type**	STANDARD
Sweep Width (Hz)	25124.86	**Temperature (degree C)**	25.100				



3,5-Dimethoxy-*N*-(4-nitrophenyl)benzamide (Table 2, Entry 6)

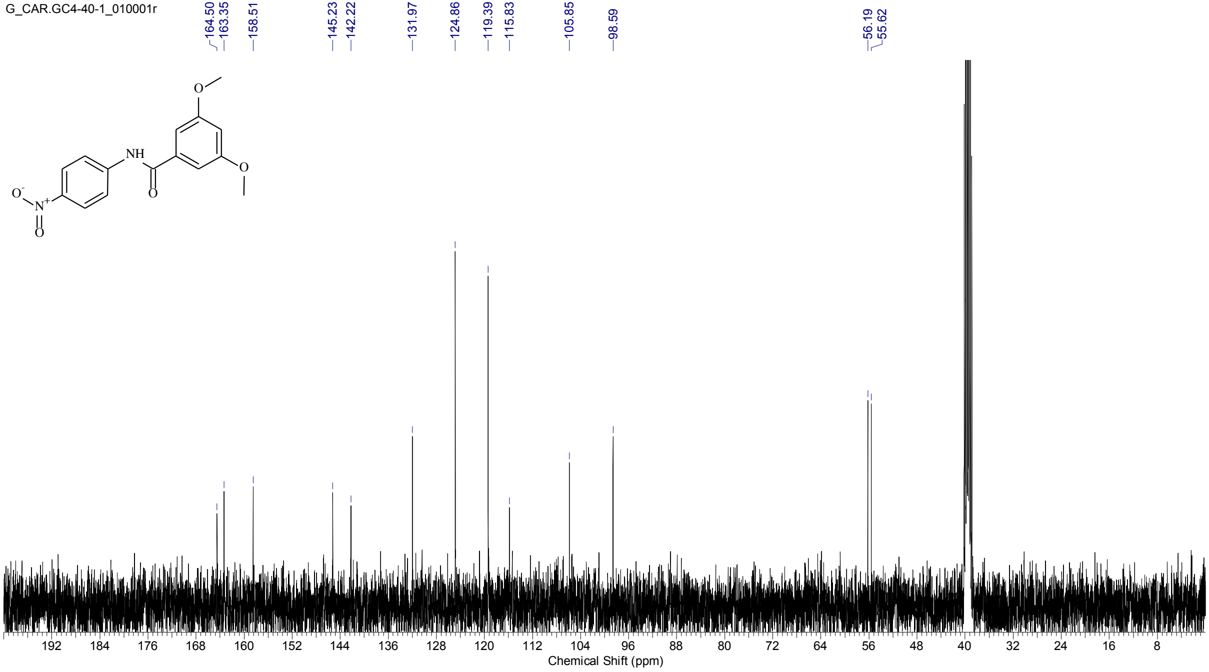
Formula	$C_{15}H_{14}N_2O_5$	**FW**	302.2821

Acquisition Time (sec)	3.9846	**Comment**	Slot No. 40 Sample ID gc4-40-1 SupervisorID moses Lab Phone No. 13540 UserID g_car		
Date	05 Apr 2012 11:29:20			**Date Stamp**	05 Apr 2012 11:29:20
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-40-1\9\PDATA\1\1R			**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400
Original Points Count	32768				
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30
Receiver Gain	322.50				
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.2061
Spectrum Type	STANDARD				
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.000		



Formula	$C_{15}H_{14}N_2O_5$	**FW**	302.2821

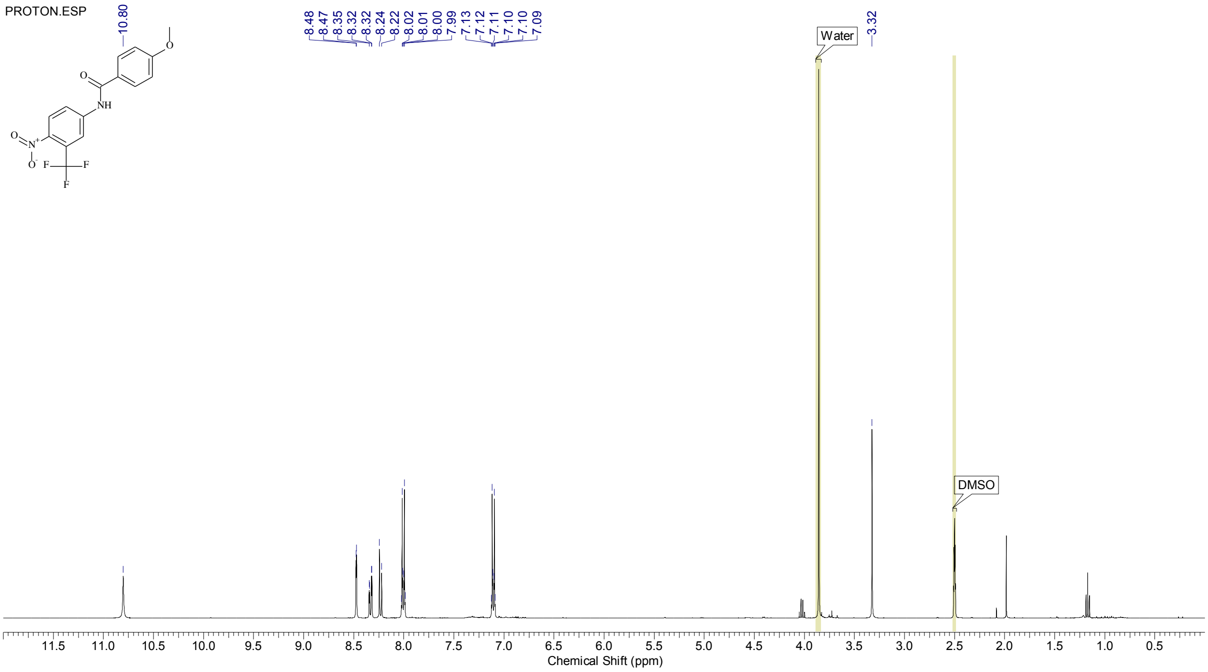
Acquisition Time (sec)	0.6521	**Comment**	Slot No. 40 Sample ID gc4-40-1 SupervisorID moses Lab Phone No. 13540 UserID g_car		
Date	05 Apr 2012 11:33:36			**Date Stamp**	05 Apr 2012 11:33:36
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-40-1\10\PDATA\1\1R			**Frequency (MHz)**	100.63
Nucleus	13C	**Number of Transients**	128	**Origin**	dpx400
Original Points Count	16384				
Owner	nmruser	**Points Count**	32768	**Pulse Sequence**	zgpg30
Receiver Gain	5792.60				
SW(cyclical) (Hz)	25125.63	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	11018.3350
Spectrum Type	STANDARD				
Sweep Width (Hz)	25124.86	**Temperature (degree C)**	25.000		



4-Methoxy-N-(4-nitro-3-(trifluoromethyl)phenyl)benzamide (Table 2, Entry 7)

Formula	$C_{15}H_{11}F_3N_2O_4$	FW	340.2540

Acquisition Time (sec)	3.9846	Comment	Slot No. 22 Sample ID gc4-42-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	30 Mar 2012 09:17:04			Date Stamp	30 Mar 2012 09:17:04		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-42-1\5\PDATA\1\1R					Frequency (MHz)	400.20
Nucleus	1H	Number of Transients	16	Origin	dpx400	Original Points Count	32768
Owner	nmruser	Points Count	65536	Pulse Sequence	zg30	Receiver Gain	456.10
SW(cyclical) (Hz)	8223.68	Solvent	DMSO-d6	Spectrum Offset (Hz)	2465.0803	Spectrum Type	STANDARD
Sweep Width (Hz)	8223.56	Temperature (degree C)	25.000				



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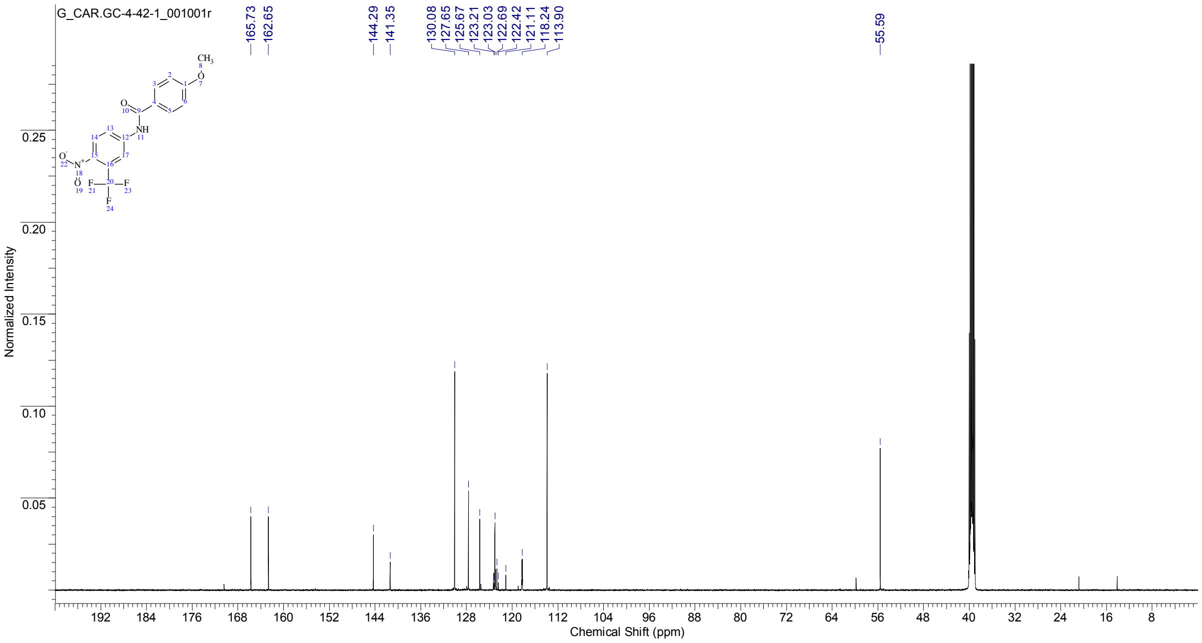
Formula	$C_{15}H_{11}F_3N_2O_4$	FW	340.2540

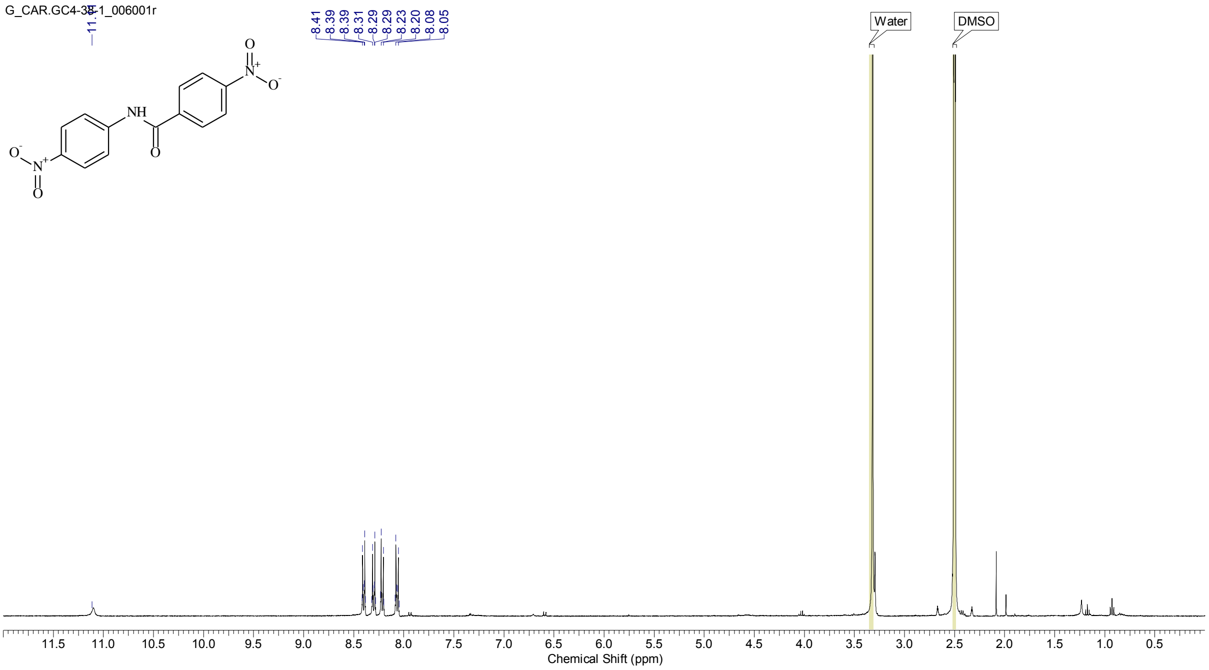
Acquisition Time (sec)	1.1010	Comment	UserID g_car SampleID gc-4-42-1 SupervisorID moses Lab Phone No. 13540 Slot 3				
Date	21 May 2012 16:38:40			Date Stamp	21 May 2012 16:38:40		
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC-4-42-1\1\PDATA\1\1R					Frequency (MHz)	125.76
Nucleus	13C	Number of Transients	512	Origin	av3500	Original Points Count	32768
Owner	service	Points Count	65536	Pulse Sequence	zgpg30	Receiver Gain	2050.00
SW(cyclical) (Hz)	29761.90	Solvent	DMSO-d6	Spectrum Offset (Hz)	13780.8672	Spectrum Type	STANDARD
Sweep Width (Hz)	29761.45	Temperature (degree C)	25.000				



4-Nitro-*N*-(4-nitrophenyl) benzamide (Table 2, Entry 8)

Formula	$C_{13}H_9N_3O_5$	**FW**	287.2277

Acquisition Time (sec)	3.9846	**Comment**	Slot No. 38 Sample ID gc4-38-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	05 Apr 2012 11:12:16			**Date Stamp**	05 Apr 2012 11:12:16		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-38-1\6\PDATA\1\1R					**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400	**Original Points Count**	32768
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30	**Receiver Gain**	645.10
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.2061	**Spectrum Type**	STANDARD
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.000				



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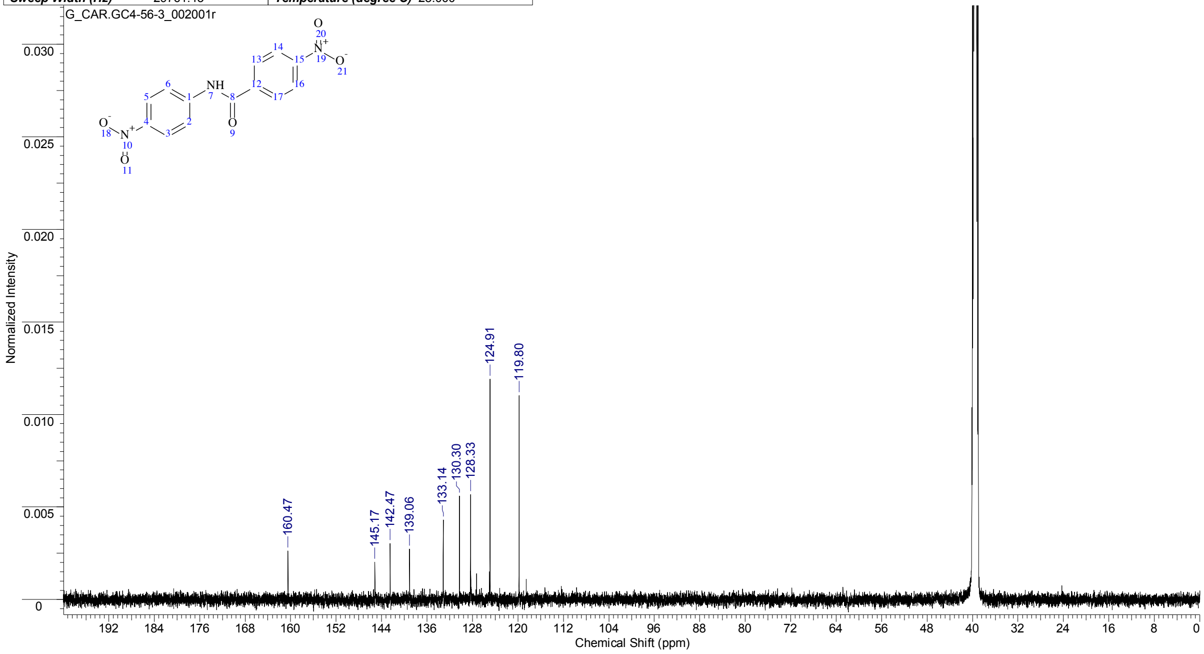
Formula	$C_{13}H_9N_3O_5$	**FW**	287.2277

Acquisition Time (sec)	1.1010	**Comment**	UserID g_car SampleID gc4-56-3 SupervisorID moses Lab Phone No. 13540 Slot 1				
Date	16 May 2012 10:53:04			**Date Stamp**	16 May 2012 10:53:04		
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC4-56-3\2\PDATA\1\1R					**Frequency (MHz)**	125.76
Nucleus	13C	**Number of Transients**	400	**Origin**	av3500	**Original Points Count**	32768
Owner	service	**Points Count**	65536	**Pulse Sequence**	zgpg30	**Receiver Gain**	2050.00
SW(cyclical) (Hz)	29761.90	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	13777.2344	**Spectrum Type**	STANDARD
Sweep Width (Hz)	29761.45	**Temperature (degree C)**	25.000				

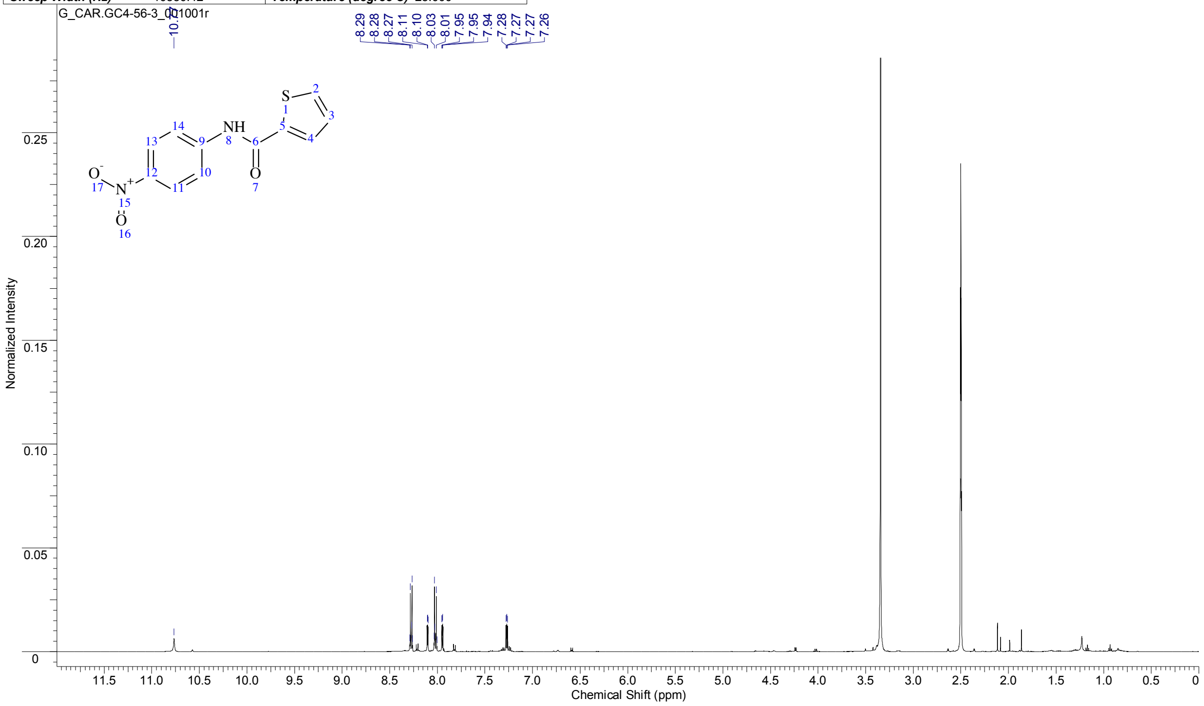


***N*-(4-nitrophenyl)thiophene-2-carboxamide** (Table 2, Entry 9)

25/05/2012 15:53:16

Formula $C_{11}H_8N_2O_3S$	***FW*** 248.2578

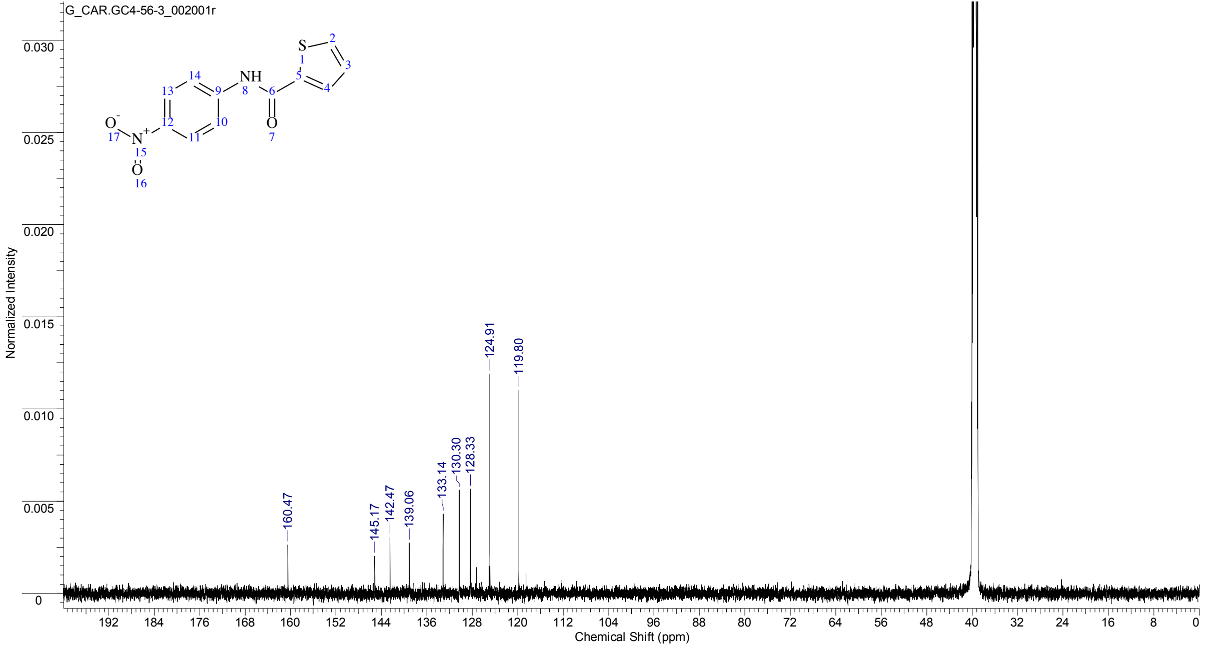
Acquisition Time (sec)	3.1719	***Comment***	UserID g_car SampleID gc4-56-3 SupervisorID moses Lab Phone No. 13540 Slot 1				
Date	16 May 2012 09:57:36			***Date Stamp***	16 May 2012 09:57:36		
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC4-56-3\1\PDATA\1\1R					***Frequency (MHz)***	500.13
Nucleus	1H	***Number of Transients***	16	***Origin***	av3500	***Original Points Count***	32768
Owner	service	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	575.00
SW(cyclical) (Hz)	10330.58	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	3083.0349	***Spectrum Type***	STANDARD
Sweep Width (Hz)	10330.42	***Temperature (degree C)***	25.000				



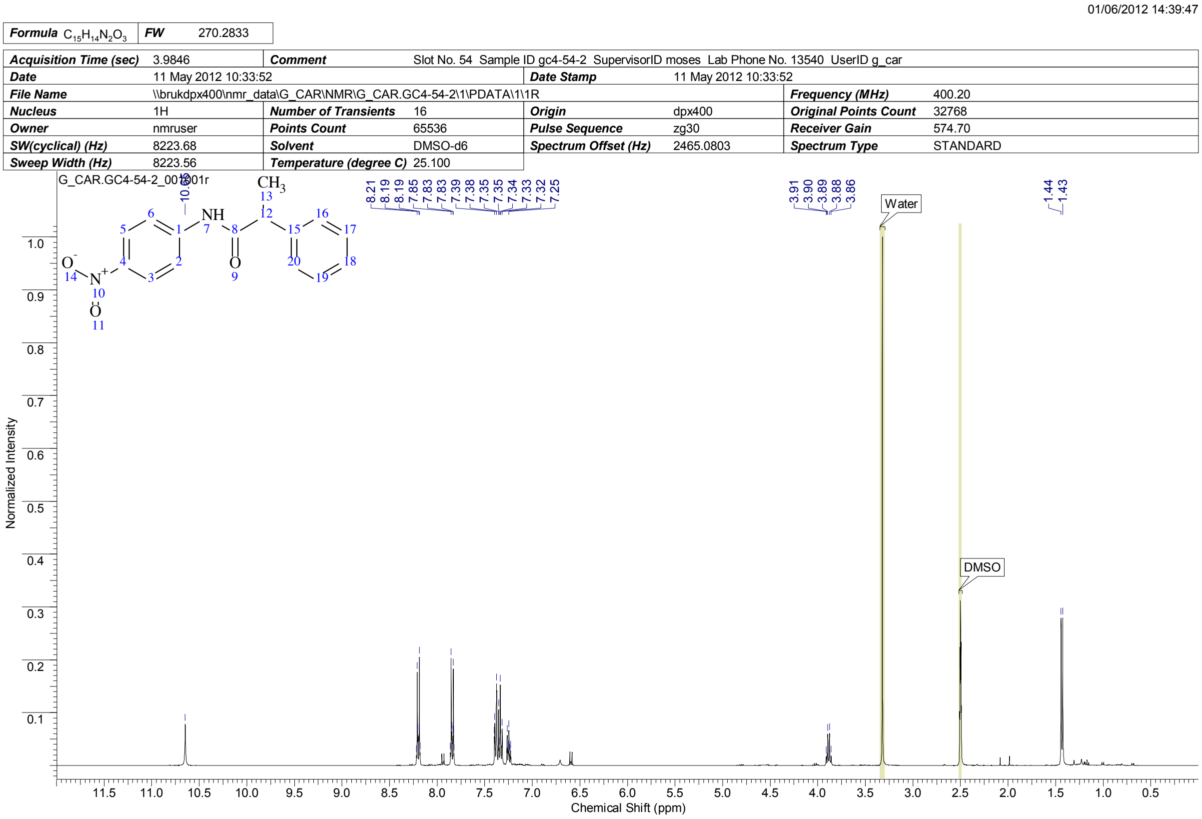
25/05/2012 15:34:00

Formula $C_{11}H_8N_2O_3S$	***FW*** 248.2578

Acquisition Time (sec)	1.1010	***Comment***	UserID g_car SampleID gc4-56-3 SupervisorID moses Lab Phone No. 13540 Slot 1				
Date	16 May 2012 10:53:04			***Date Stamp***	16 May 2012 10:53:04		
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC4-56-3\2\PDATA\1\1R					***Frequency (MHz)***	125.76
Nucleus	13C	***Number of Transients***	400	***Origin***	av3500	***Original Points Count***	32768
Owner	service	***Points Count***	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	2050.00
SW(cyclical) (Hz)	29761.90	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	13777.2344	***Spectrum Type***	STANDARD
Sweep Width (Hz)	29761.45	***Temperature (degree C)***	25.000				

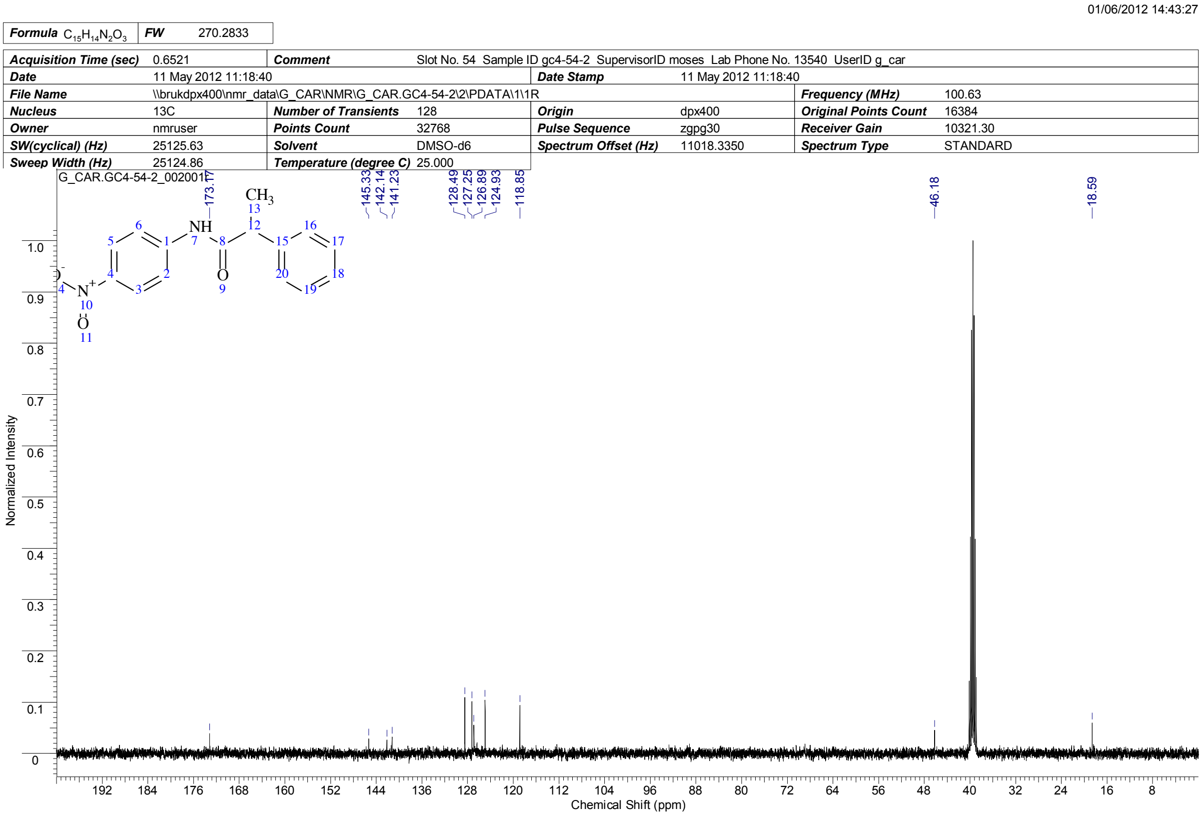


***N*-(4-nitrophenyl)-2-phenylpropanamide** (Table 2, Entry 10)


01/06/2012 14:39:47

Formula	$C_{15}H_{14}N_2O_3$	**FW**	270.2833

Acquisition Time (sec)	3.9846	**Comment**	Slot No. 54 Sample ID gc4-54-2 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	11 May 2012 10:33:52			**Date Stamp**	11 May 2012 10:33:52		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-54-2\1\PDATA\1\1R					**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400	**Original Points Count**	32768
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30	**Receiver Gain**	574.70
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.0803	**Spectrum Type**	STANDARD
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.100				


01/06/2012 14:43:27

Formula	$C_{15}H_{14}N_2O_3$	**FW**	270.2833

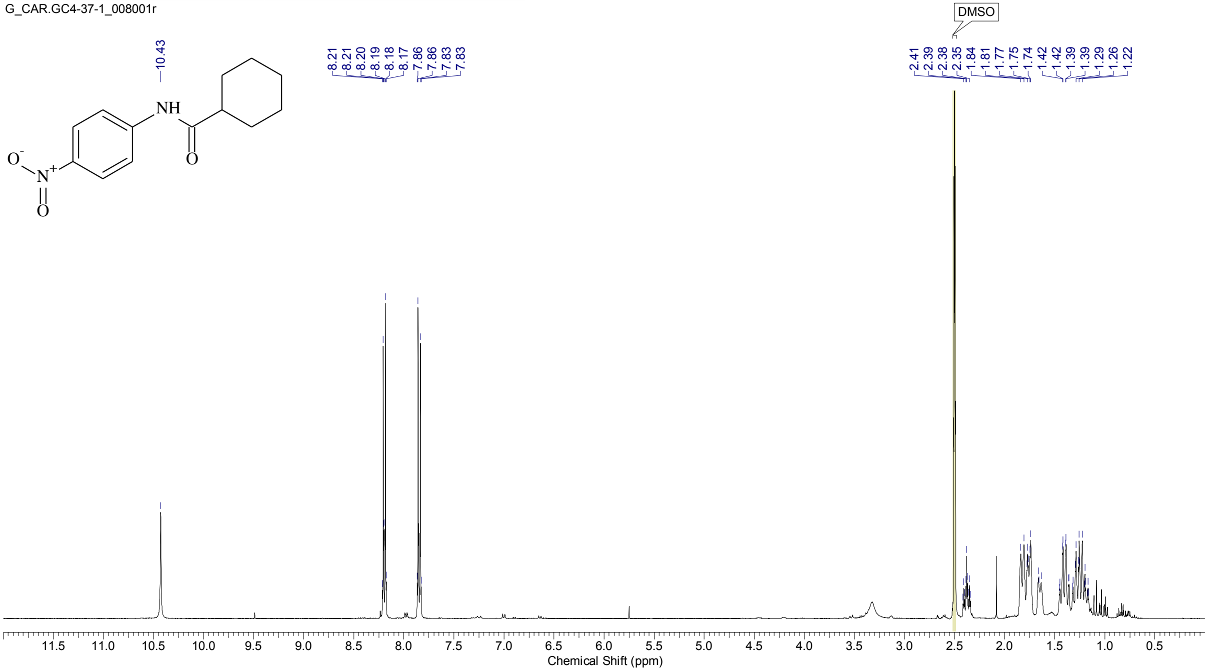
Acquisition Time (sec)	0.6521	**Comment**	Slot No. 54 Sample ID gc4-54-2 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	11 May 2012 11:18:40			**Date Stamp**	11 May 2012 11:18:40		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-54-2\2\PDATA\1\1R					**Frequency (MHz)**	100.63
Nucleus	13C	**Number of Transients**	128	**Origin**	dpx400	**Original Points Count**	16384
Owner	nmruser	**Points Count**	32768	**Pulse Sequence**	zgpg30	**Receiver Gain**	10321.30
SW(cyclical) (Hz)	25125.63	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	11018.3350	**Spectrum Type**	STANDARD
Sweep Width (Hz)	25124.86	**Temperature (degree C)**	25.000				

***N*-(4-nitrophenyl)cyclohexanecarboxamide** (Table 2, Entry 11)

Formula $C_{13}H_{16}N_2O_3$	**FW** 248.2777

Acquisition Time (sec)	3.9846	**Comment**	Slot No. 37 Sample ID gc4-37-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	05 Apr 2012 05:16:00			**Date Stamp**	05 Apr 2012 05:16:00		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-37-1\8\PDATA\1\1R					**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400	**Original Points Count**	32768
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30	**Receiver Gain**	256.00
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.2061	**Spectrum Type**	STANDARD
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.000				

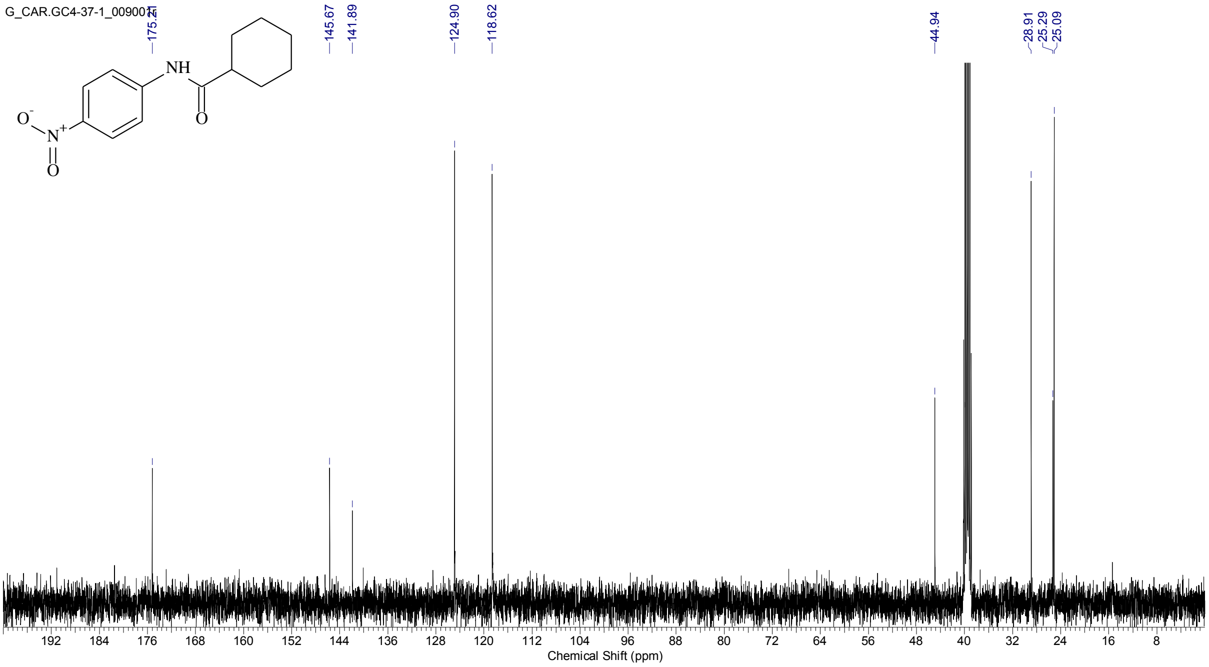
G_CAR.GC4-37-1_008001r



Formula $C_{13}H_{16}N_2O_3$	**FW** 248.2777

Acquisition Time (sec)	0.6521	**Comment**	Slot No. 37 Sample ID gc4-37-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	05 Apr 2012 05:22:24			**Date Stamp**	05 Apr 2012 05:22:24		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-37-1\9\PDATA\1\1R					**Frequency (MHz)**	100.63
Nucleus	13C	**Number of Transients**	128	**Origin**	dpx400	**Original Points Count**	16384
Owner	nmruser	**Points Count**	32768	**Pulse Sequence**	zgpg30	**Receiver Gain**	20642.50
SW(cyclical) (Hz)	25125.63	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	11018.3350	**Spectrum Type**	STANDARD
Sweep Width (Hz)	25124.86	**Temperature (degree C)**	25.000				

G_CAR.GC4-37-1_009001r

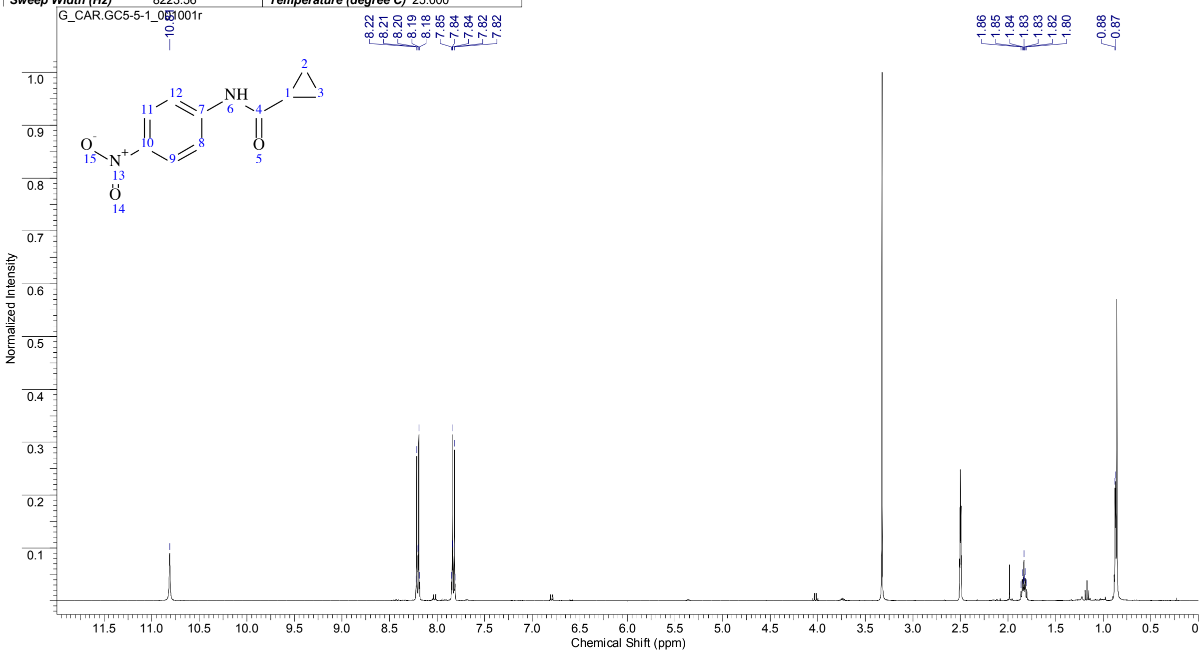


***N*-(4-nitrophenyl)cyclopropanecarboxamide** (Table 2, Entry 12)

25/05/2012 16:02:26

Formula $C_{10}H_{10}N_2O_3$	**FW** 206.1980

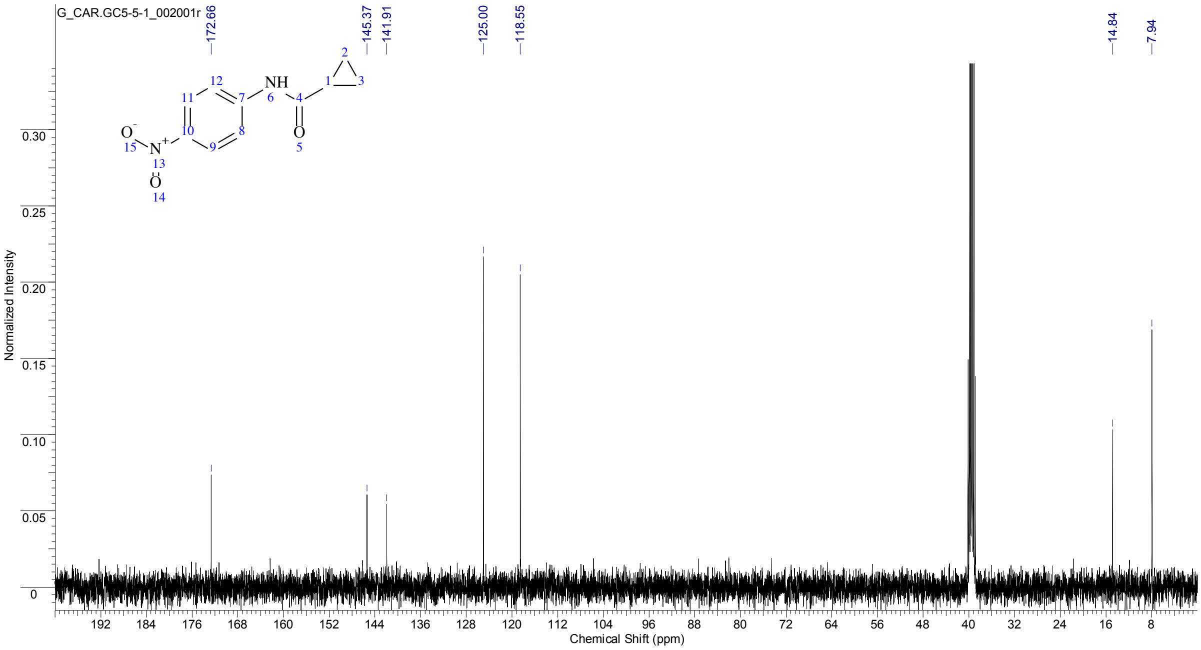
Acquisition Time (sec)	3.9846	**Comment**	Slot No. 21 Sample ID gc5-5-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	01 May 2012 18:04:00			**Date Stamp**	01 May 2012 18:04:00		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC5-5-1\1\PDATA\1\1R					**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400	**Original Points Count**	32768
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30	**Receiver Gain**	456.10
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.2061	**Spectrum Type**	STANDARD
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.000				



25/05/2012 16:09:08

Formula $C_{10}H_{10}N_2O_3$	**FW** 206.1980

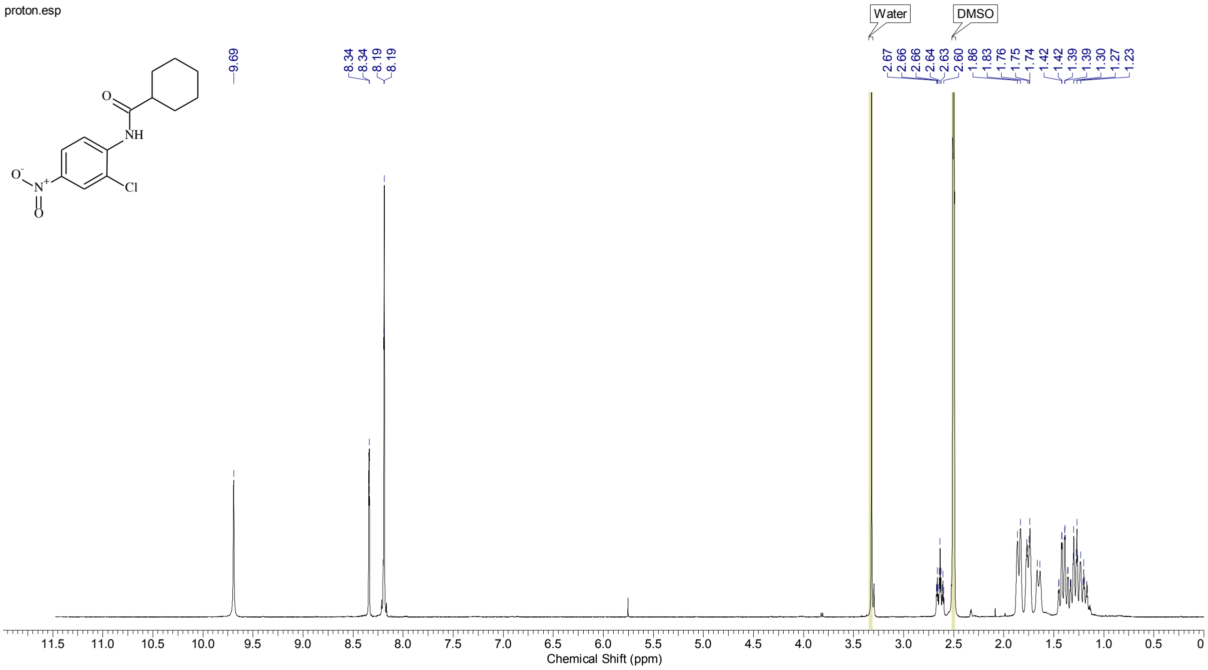
Acquisition Time (sec)	0.6521	**Comment**	Slot No. 21 Sample ID gc5-5-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	01 May 2012 18:33:52			**Date Stamp**	01 May 2012 18:33:52		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC5-5-1\2\PDATA\1\1R					**Frequency (MHz)**	100.63
Nucleus	13C	**Number of Transients**	128	**Origin**	dpx400	**Original Points Count**	16384
Owner	nmruser	**Points Count**	32768	**Pulse Sequence**	zgpg30	**Receiver Gain**	5160.60
SW(cyclical) (Hz)	25125.63	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	11019.1025	**Spectrum Type**	STANDARD
Sweep Width (Hz)	25124.86	**Temperature (degree C)**	25.000				



***N*-(2-chloro-4-nitrophenyl)cyclohexanecarboxamide** (Table 2, Entry 13)

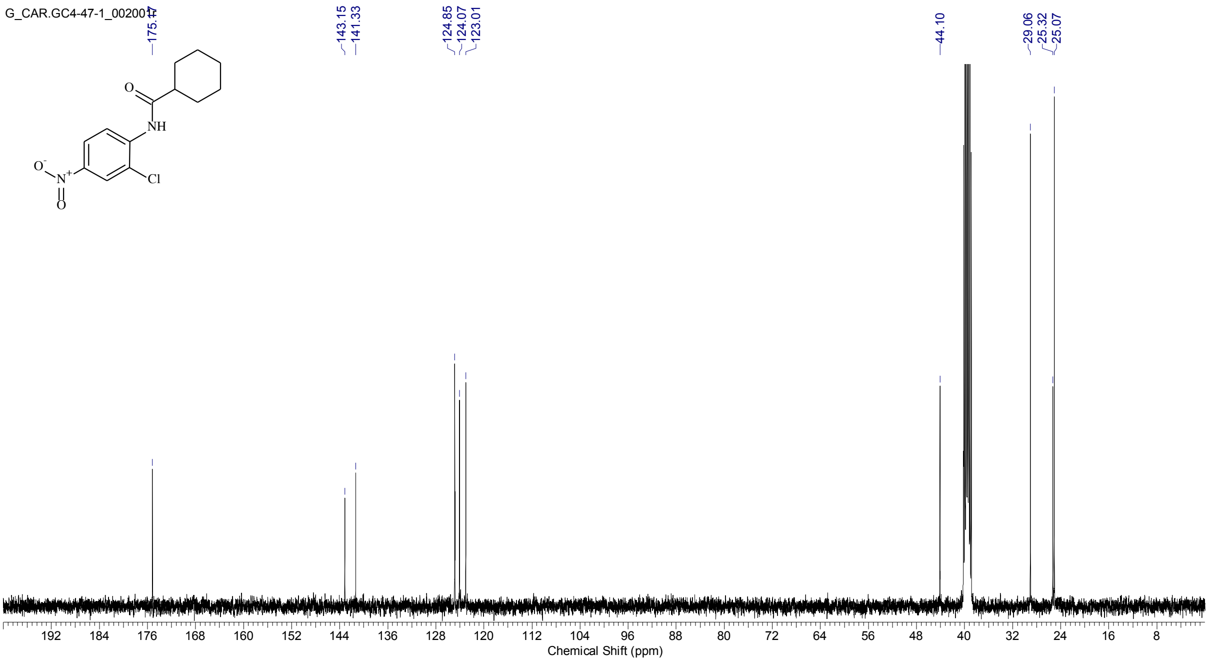
Formula	$C_{13}H_{15}ClN_2O_3$	**FW**	282.7228

Acquisition Time (sec)	3.4210	**Comment**	UserID g_car SampleID gc4-47-1 SupervisorID moses Lab Phone No. 13540 Slot Number 47				
Date	04 Apr 2012 04:54:40			**Date Stamp**	04 Apr 2012 04:54:40		
File Name	\\brukav400\nmr_data\G_CAR\NMR\G_CAR.GC4-47-1\1\PDATA\1\1R					**Frequency (MHz)**	400.13
Nucleus	1H	**Number of Transients**	16	**Origin**	av400	**Original Points Count**	16384
Owner	nmruser	**Points Count**	32768	**Pulse Sequence**	zg30	**Receiver Gain**	181.00
SW(cyclical) (Hz)	4789.27	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2197.4238	**Spectrum Type**	STANDARD
Sweep Width (Hz)	4789.13	**Temperature (degree C)**	25.160				



Formula	$C_{13}H_{15}ClN_2O_3$	**FW**	282.7228

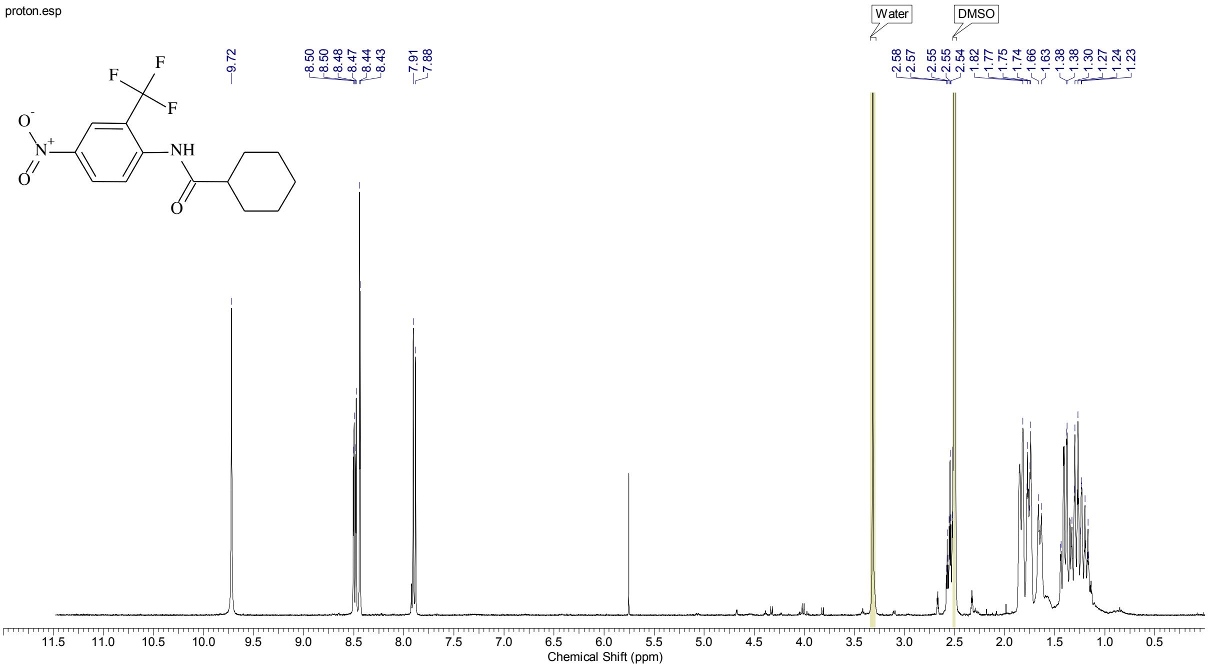
Acquisition Time (sec)	0.6832	**Comment**	UserID g_car SampleID gc4-47-1 SupervisorID moses Lab Phone No. 13540 Slot Number 47				
Date	04 Apr 2012 05:54:24			**Date Stamp**	04 Apr 2012 05:54:24		
File Name	\\brukav400\nmr_data\G_CAR\NMR\G_CAR.GC4-47-1\2\PDATA\1\1R					**Frequency (MHz)**	100.61
Nucleus	13C	**Number of Transients**	2048	**Origin**	av400	**Original Points Count**	16384
Owner	nmruser	**Points Count**	32768	**Pulse Sequence**	zgpg30	**Receiver Gain**	11585.20
SW(cyclical) (Hz)	23980.81	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	11017.3057	**Spectrum Type**	STANDARD
Sweep Width (Hz)	23980.08	**Temperature (degree C)**	25.160				



***N*-(4-nitro-2-(trifluoromethyl)phenyl)cyclohexanecarboxamide** (Table 2, Entry 14)

Formula $C_{14}H_{15}F_3N_2O_3$		***FW***	316.2757

Acquisition Time (sec)	3.4210	***Comment***	UserID g_car SampleID gc4-50-1 SupervisorID moses Lab Phone No. 13540 Slot Number 50				
Date	04 Apr 2012 07:09:04			***Date Stamp***	04 Apr 2012 07:09:04		
File Name	\\brukav400\nmr_data\G_CAR\NMR\G_CAR.GC4-50-1\1\PDATA\1\1R					***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	16	***Origin***	av400	***Original Points Count***	16384
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zg30	***Receiver Gain***	181.00
SW(cyclical) (Hz)	4789.27	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	2197.2776	***Spectrum Type***	STANDARD
Sweep Width (Hz)	4789.13	***Temperature (degree C)***	25.160				



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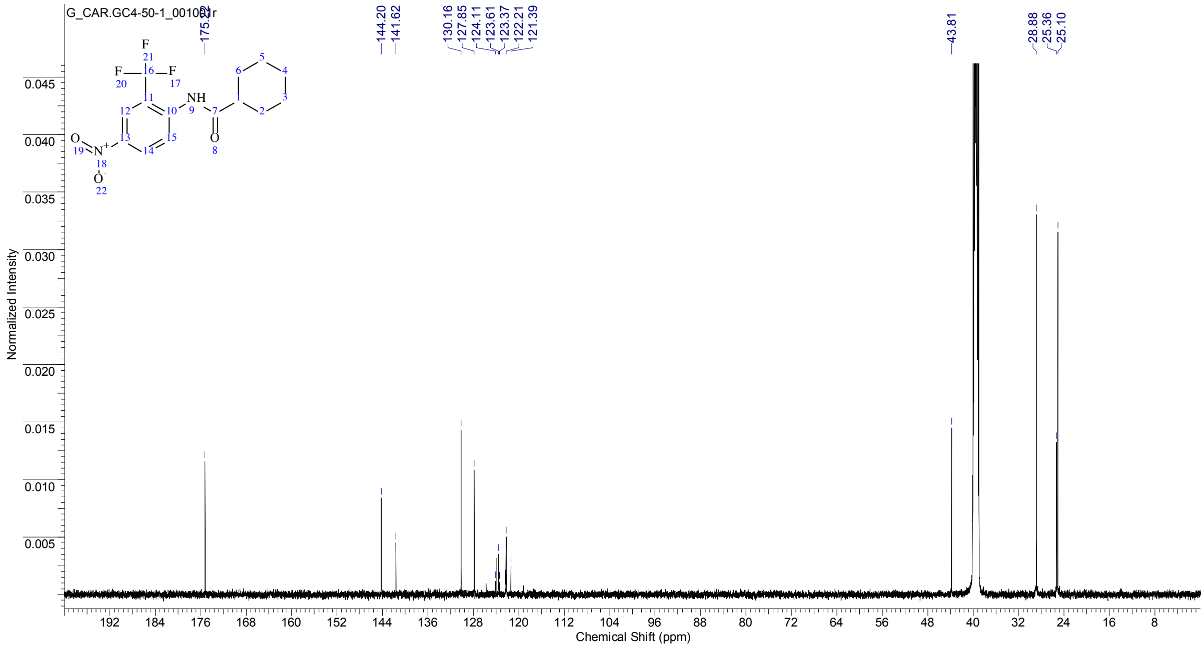
Formula $C_{14}H_{15}F_3N_2O_3$		***FW***	316.2757

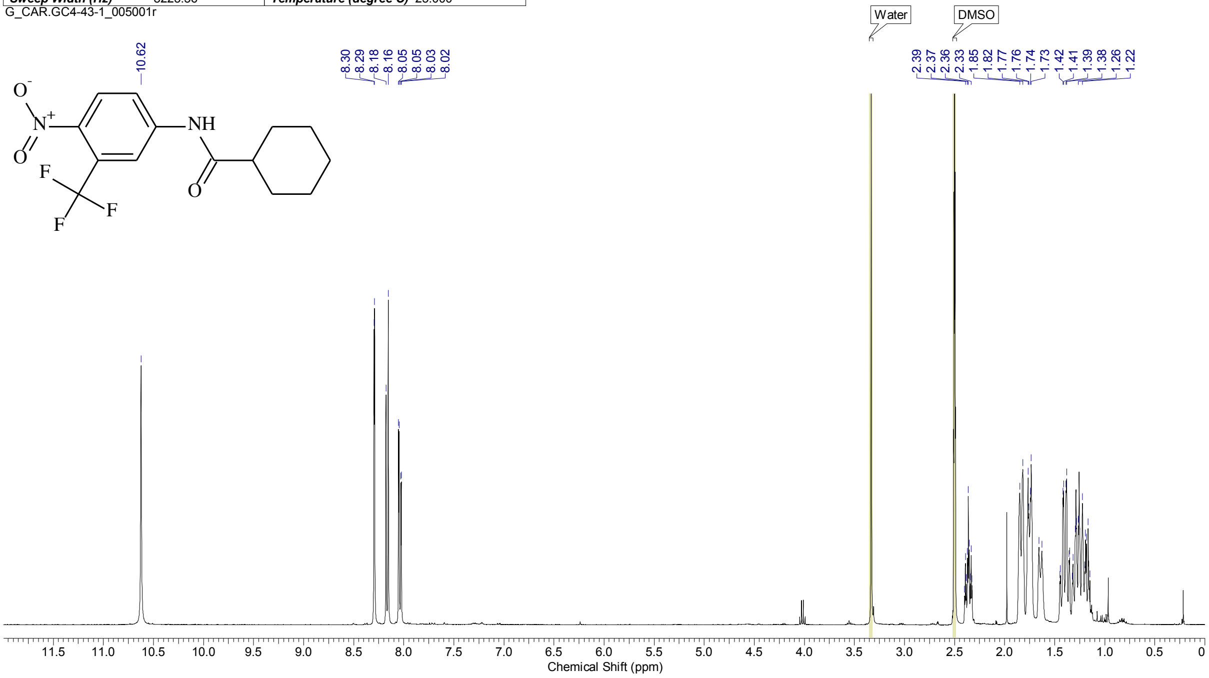
Acquisition Time (sec)	1.1010	***Comment***	UserID g_car SampleID gc4-50-1 SupervisorID moses Lab Phone No. 13540 Slot 2				
Date	22 May 2012 12:18:24			***Date Stamp***	22 May 2012 12:18:24		
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC4-50-1\1\PDATA\1\1R					***Frequency (MHz)***	125.76
Nucleus	13C	***Number of Transients***	512	***Origin***	av3500	***Original Points Count***	32768
Owner	service	***Points Count***	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	2050.00
SW(cyclical) (Hz)	29761.90	***Solvent***	DMSO-d6	***Spectrum Offset (Hz)***	13777.2344	***Spectrum Type***	STANDARD
Sweep Width (Hz)	29761.45	***Temperature (degree C)***	25.000				



***N*-(4-nitro-3-(trifluoromethyl)phenyl)cyclohexanecarboxamide** (Table 2, Entry 15)

Formula	$C_{14}H_{15}F_3N_2O_3$	**FW**	316.2757

Acquisition Time (sec)	3.9846	**Comment**	Slot No. 43 Sample ID gc4-43-1 SupervisorID moses Lab Phone No. 13540 UserID g_car				
Date	05 Apr 2012 14:17:52			**Date Stamp**	05 Apr 2012 14:17:52		
File Name	\\brukdpx400\nmr_data\G_CAR\NMR\G_CAR.GC4-43-1\5\PDATA\1\1R					**Frequency (MHz)**	400.20
Nucleus	1H	**Number of Transients**	16	**Origin**	dpx400	**Original Points Count**	32768
Owner	nmruser	**Points Count**	65536	**Pulse Sequence**	zg30	**Receiver Gain**	181.00
SW(cyclical) (Hz)	8223.68	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2465.0808	**Spectrum Type**	STANDARD
Sweep Width (Hz)	8223.56	**Temperature (degree C)**	25.000				



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Formula	$C_{14}H_{15}F_3N_2O_3$	**FW**	316.2757

Acquisition Time (sec)	1.1010	**Comment**	UserID g_car SampleID gc-4-43-1 SupervisorID moses Lab Phone No. 13540 Slot 2				
Date	21 May 2012 14:54:08			**Date Stamp**	21 May 2012 14:54:08		
File Name	\\brukav3500\nmr_data\G_CAR\NMR\G_CAR.GC-4-43-1\1\PDATA\1\1R					**Frequency (MHz)**	125.76
Nucleus	13C	**Number of Transients**	512	**Origin**	av3500	**Original Points Count**	32768
Owner	service	**Points Count**	65536	**Pulse Sequence**	zgpg30	**Receiver Gain**	1030.00
SW(cyclical) (Hz)	29761.90	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	13780.8672	**Spectrum Type**	STANDARD
Sweep Width (Hz)	29761.45	**Temperature (degree C)**	25.000