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SUPPORTING INFORMATION

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Title: Indium-Triflate-Catalyzed Ritter Reaction in Liquid Sulfur Dioxide
Author(s): Daniels Posevins, Krista Suta, Māris Turks*

Contents of Supporting Information:

1. General information

Solvents for the reactions were dried over standard drying agents and freshly distilled prior to use. Commercially available reagents were used as received. Reactions were monitored by GC/MS. Column chromatography was performed on silica gel (60 Å, 40-63 μm, *Merck*). Melting points were recorded with a *Fisher Digital Melting Point Analyzer Model 355* apparatus and are uncorrected. Optical rotation was measured at 25 °C on a *Anton Paar MCP 500* polarimeter (1 dm cell) using a sodium lamp as the light source (589 nm). ^{1}H and ^{13}C NMR spectra were recorded on a *Bruker* 300 MHz, in $CDCl_3$. Chemical shifts (δ) values are reported in ppm. The residual solvent peaks are used as internal reference ($CDCl_3$ 7.26 ppm for ^{1}H-NMR, $CDCl_3$ 77.0 ppm for ^{13}C-NMR), s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet); *J* in hertz.

2. Experimental procedures and characterization of products

Synthesis of *N*-benzhydrylacetamide 2 in liquid sulfur dioxide in the absence of catalyst

Acetonitrile (780 μL, 15.0 mmol, 5.0 equiv.) was injected under N_2 atmosphere into an oven-dried pressure reactor (nominal volume: 150 mL) equipped with a glass tube and a magnetic stirring bar and containing benzhydryl alohol (553 mg, 3.0 mmol, 1.0 equiv.). The reactor was sealed, carefully degassed by applying vacuum and cooled to –50 ^{o}C. Sulfur dioxide (~25 g) was added from a storage cylinder by distillation. The content of the reactor was allowed to reach room temperature and then the resulting mixture was heated and stirred in oil bath at +150 ^{o}C for 24 h. Then the reaction mixture was slowly cooled to room temperature and SO_2 was distilled into a storage cylinder. The reactor was opened and the resulting mixture was partitioned between DCM (30 mL) and water (15 mL). The aqueous phase was extracted with DCM (3 × 20 mL). The combined organic layers were dried (Na_2SO_4) and filtered. The residue was purified by silica flash chromatography (EtOAc/hexanes) to give product **2** (680 mg, 97%) as a colorless solid.

General procedure 1 for catalyst screening for the Ritter reaction in liquid sulfur dioxide:

The selected catalyst (1.85 mmol, 0.2 equiv.) and acetonitrile (2.42 mL, 46.2 mmol, 5.0 equiv.) were placed under N_2 atmosphere into an oven-dried pressure reactor (nominal volume: 150 mL) equipped with a glass tube and a magnetic stirring bar and

containing benzyl alcohol (1.00 g, 9.25 mmol, 1.0 equiv.). The reactor was sealed, carefully degassed by applying vacuum and cooled to –50 °C. Sulfur dioxide (~40 g) was added from a storage cylinder by distillation. The content of the reactor was allowed to reach room temperature and then the resulting mixture was heated and stirred in oil bath at +60 °C for time specified in Table 2 (see the main text of the paper) Then the reaction mixture was slowly cooled to room temperature and SO_2 was distilled into a storage cylinder. The reactor was opened and the resulting mixture was partitioned between EtOAc (30 mL) and water (15 mL). The aqueous phase was extracted with EtOAc (3 × 20 mL). The combined organic layers were dried (Na_2SO_4), filtered and evaporated to dryness. Around 50 mg of the crude sample were precisely weighted and diluted with EtOAc (50 mL) in a volumetric flask. The resulting solution was analyzed by GC/MS (see method below) and external calibration curves obtained for each of the components of interest were used to calculate the GC-MS conversion.

Reactions were monitored by GC/MS as follows:

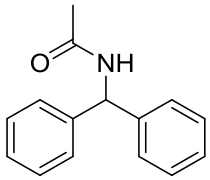
Instrument	Gas chromatograph equipped with MS detector and autosampler.
Column	Agilent 190917-433 HP5 5% Phenyl Methyl Siloxane, 30 m × 0.25 mm ID, 0.25 μm.
Column temp.	40 °C (hold for 2 minutes) to 310 °C @ 50 °C/ minute (hold at 310 °C for 3 minutes)
Injector/detector	250 °C/230 °C
Carrier gas	Helium @ 1.0 mL/min. flow rate
Injection mode	Splitless (Solvent delay: 3 min.)
Injection volume	1 μL

General procedure 2 for $In(OTf)_3$ catalyzed Ritter reaction in liquid sulfur dioxide:

$In(OTf)_3$ catalyst (0.15 mmol, 0.05 equiv. or 0.3 mmol, 0.1 equiv. or 0.6 mmol, 0.2 equiv.; according to Table 4) and a nitrile (15.0 mmol, 5.0 equiv. or 9.0 mmol, 3.0 equiv.; according to Table 4) were placed under N_2 atmosphere into an oven-dried pressure reactor (nominal volume: 150 mL) equipped with a glass tube and a magnetic stirring bar and containing alcohol (3.0 mmol, 1.0 equiv.). The reactor was sealed, carefully degassed by applying vacuum and cooled to –50 °C. Sulfur dioxide (~25 g) was added from a storage cylinder by distillation. The content of the reactor was allowed to reach room temperature and then the resulting mixture was heated and

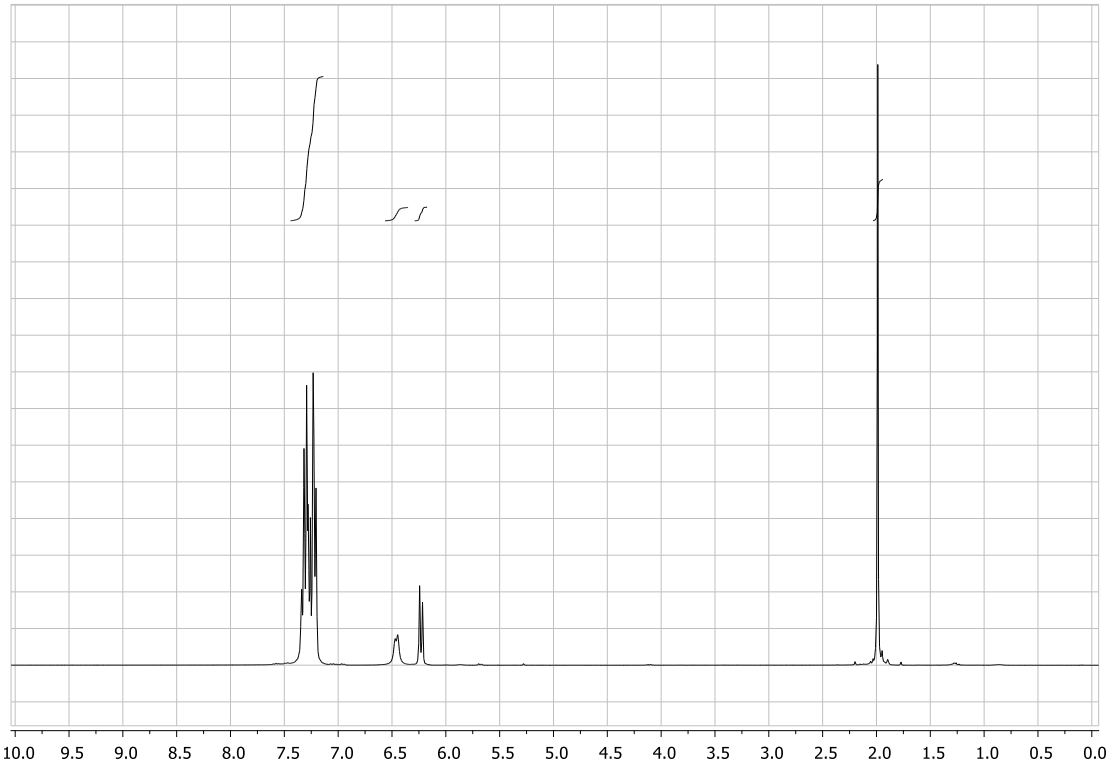
stirred in oil bath at +100 ^{o}C for 24h. Then the reaction mixture was slowly cooled to room temperature and SO_2 was distilled into a storage cylinder. The reactor was opened and the resulting mixture was partitioned between DCM (30 mL) and water (15 mL). The aqueous phase was extracted with DCM (3 × 20 mL). The combined organic layers were dried (Na_2SO_4) and filtered. The residue was purified by silica flash chromatography using hexanes and ethyl acetate in appropriate combination based on R_f of desired product.

***N*-Benzhydrylacetamide 2**

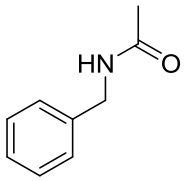


Prepared according to general procedure 2. White crystalline solid (570 mg, 85%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 – 7.35 (m, 10H), 6.46 (d, *J* = 7.3 Hz, 1H), 6.23 (d, *J* = 8.1 Hz, 1H), 1.99 (s, 3H) ppm; which is in agreement with that reported.[2]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-benzhydrylacetamide 2:

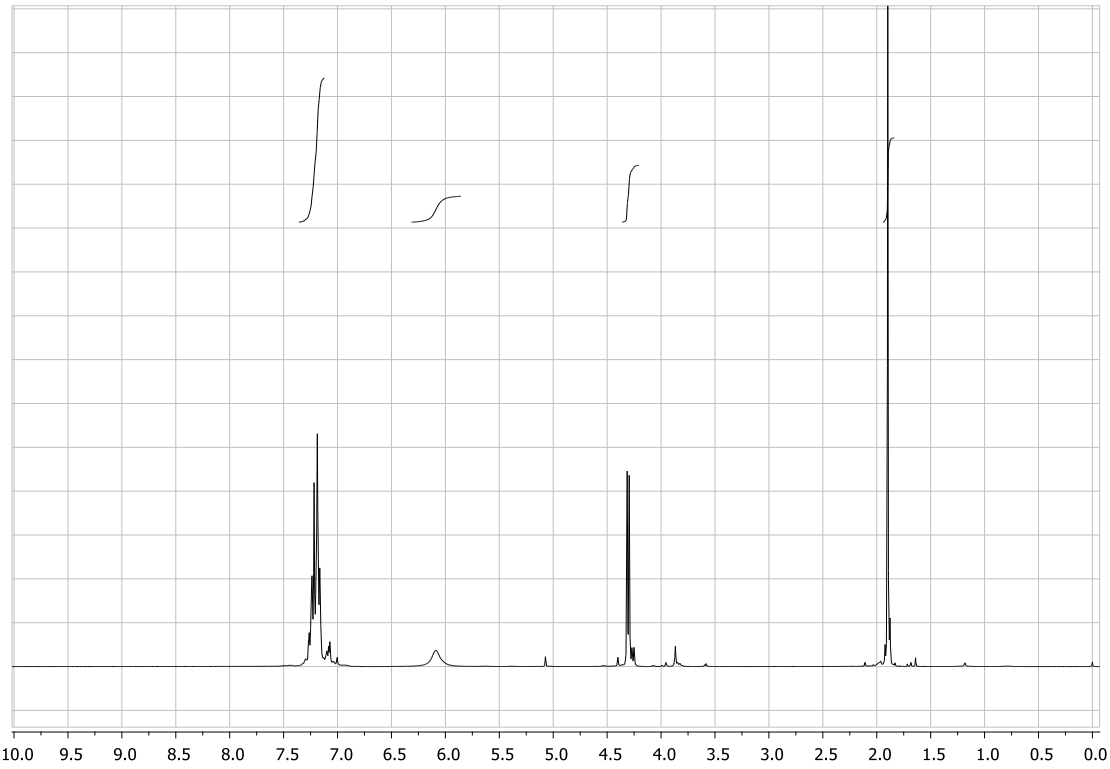


***N*-Benzylacetamide 6**

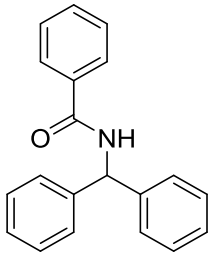


Prepared according to general procedure 2. White crystalline solid (785 mg, 57%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.21 (m, 5H), 6.17 (s, 1H), 4.38 (d, *J* = 5.7 Hz, 2H), 1.98 (s, 3H) ppm; which is in agreement with that reported.[1]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-benzylacetamide 6:

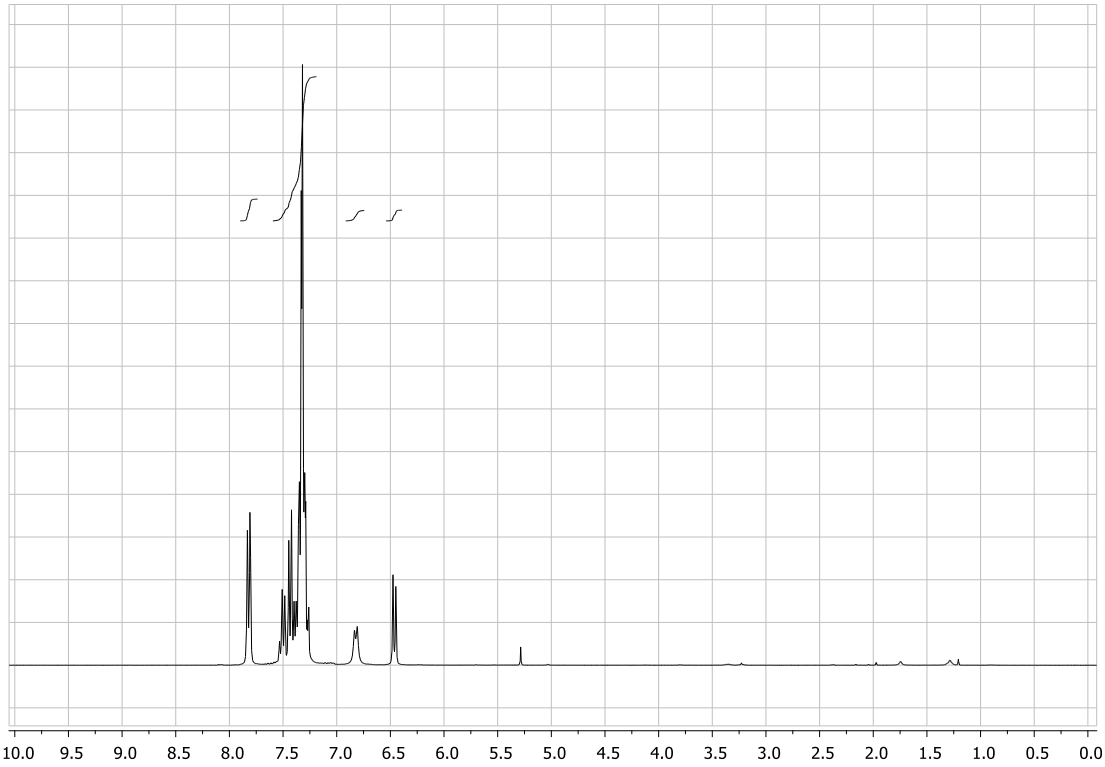


***N*-Benzhydrylbenzamide 11a**

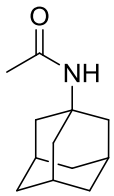


Prepared according to general procedure 2. Pale yellow crystalline solid (701 mg, 82%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 – 7.77 (m, 2H), 7.56–7.25 (m, 13H), 6.82 (d, *J* = 7.5 Hz, 1H), 6.46 (d, *J* = 7.8 Hz, 2H) ppm; which is in agreement with that reported.[3]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-benzhydrylbenzamide 11a:

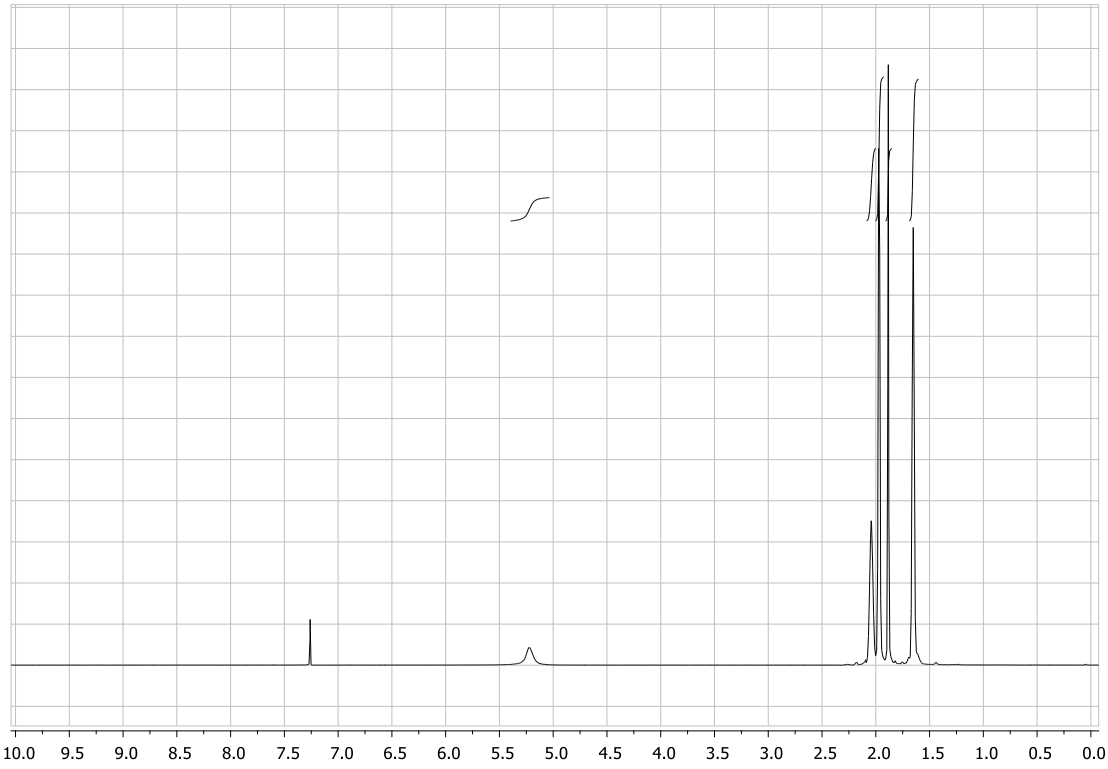


***N*-Acetyl-1-aminoadamantane 11b**

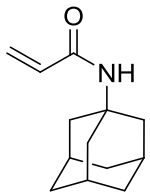


Prepared according to general procedure 2. White crystalline solid (510 mg, 86%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.22 (br s, 1H), 2.08 – 2.01 (m, 3H), 1.99 – 1.94 (m, 6H), 1.90 – 1.86 (m, 3H), 1.68 – 1.62 (m, 6H) ppm; which is in agreement with that reported.[1]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-acetyl-1-aminoadamantane 11b:

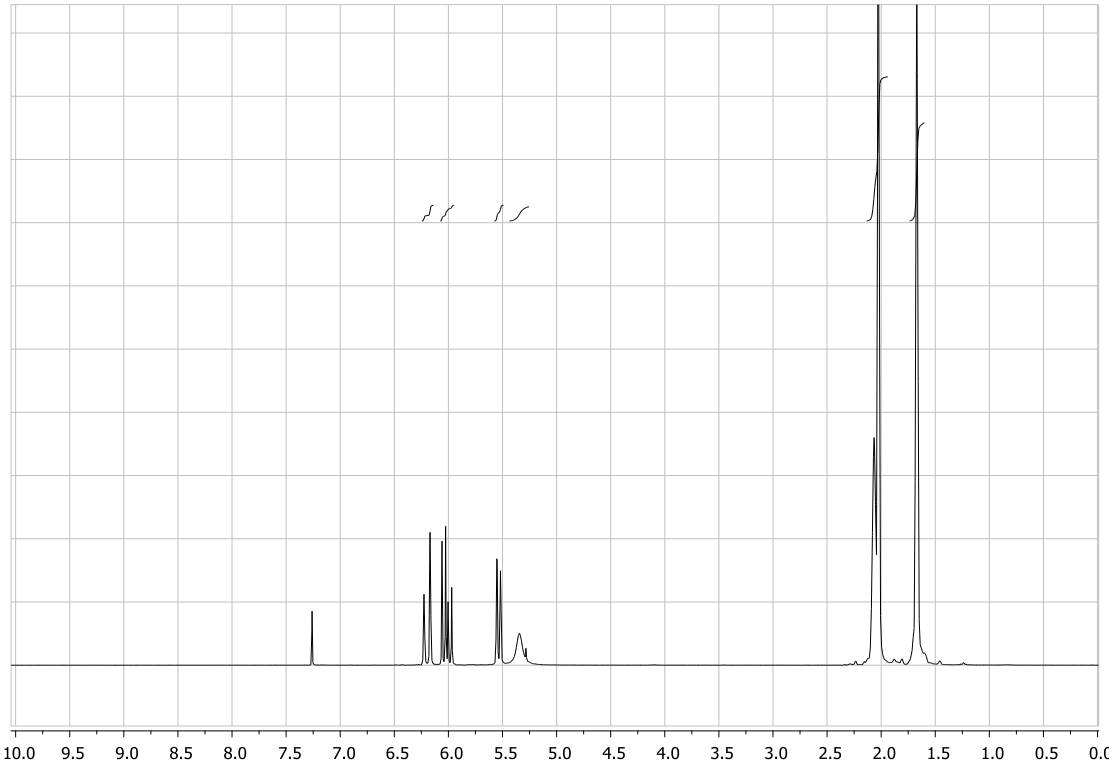


***N*-Acryloyl-1-aminoadamantane 11c**

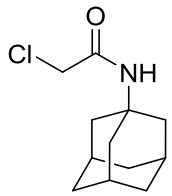


Prepared according to general procedure 2. White crystalline solid (510 mg, 83%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.24 – 6.15 (m, 1H), 6.01 (dd, *J* = 16.9, 10.1 Hz, 1H), 5.34 (br s, 1H), 2.10 – 2.00 (m, 9H), 1.70 – 1.64 (m, 6H) ppm; which is in agreement with that reported.[6]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-acryloyl-1-aminoadamantane 11c:

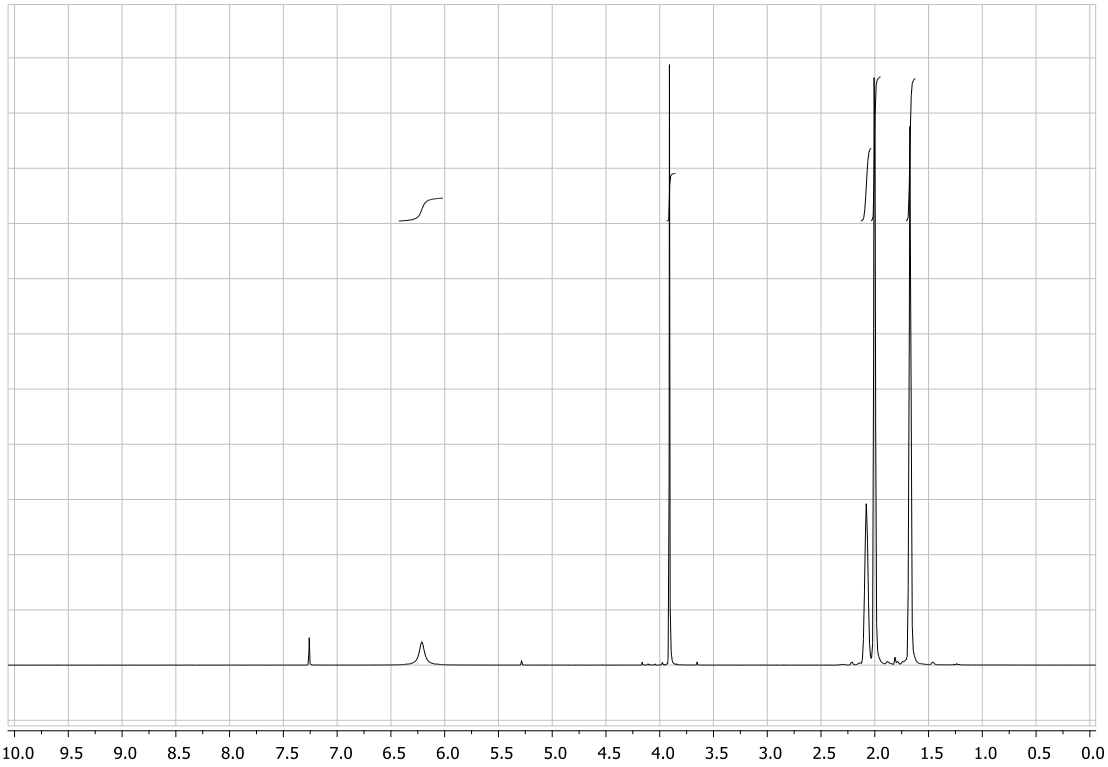


***N*-Chloroacetyl-1-aminoadamantane 11d**

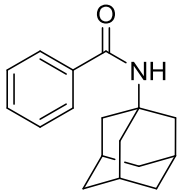


Prepared according to general procedure 2. White crystalline solid (542 mg, 79%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.21 (br s, 1H), 3.91 (s, 1H), 2.12 – 2.04 (m, 3H), 2.03 – 1.97 (m, 6H), 1.67 (t, J = 2.7 Hz, 1H) ppm; which is in agreement with that reported.[4]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-chloroacetyl-1-aminoadamantane 11d:

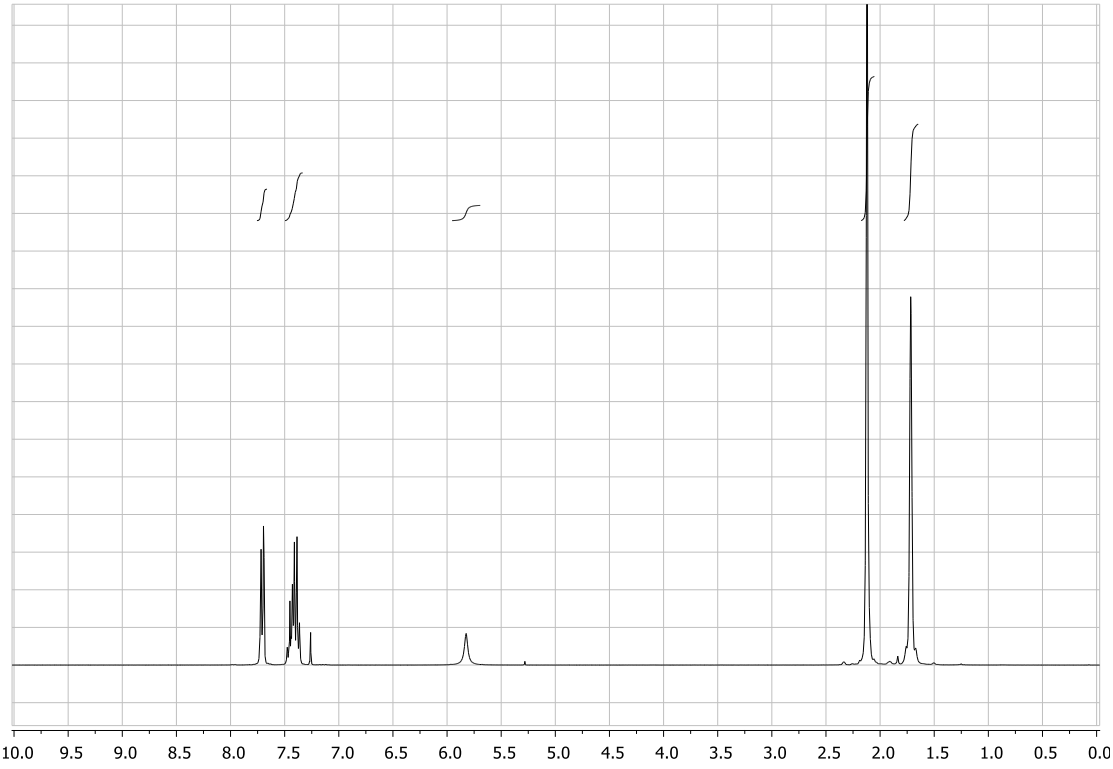


***N*-Benzoyl-1-aminoadamantane 11e**

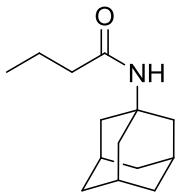


Prepared according to general procedure 2. Pale yellow crystalline solid (510 mg, 83%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 – 7.68 (m, 2H), 7.50 – 7.35 (m, 3H), 5.82 (br s, 1H), 2.15 – 2.09 (m, 9H), 1.78 – 1.65 (m, 6H) ppm; which is in agreement with that reported.[7]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-benzoyl-1-aminoadamantane 11e:

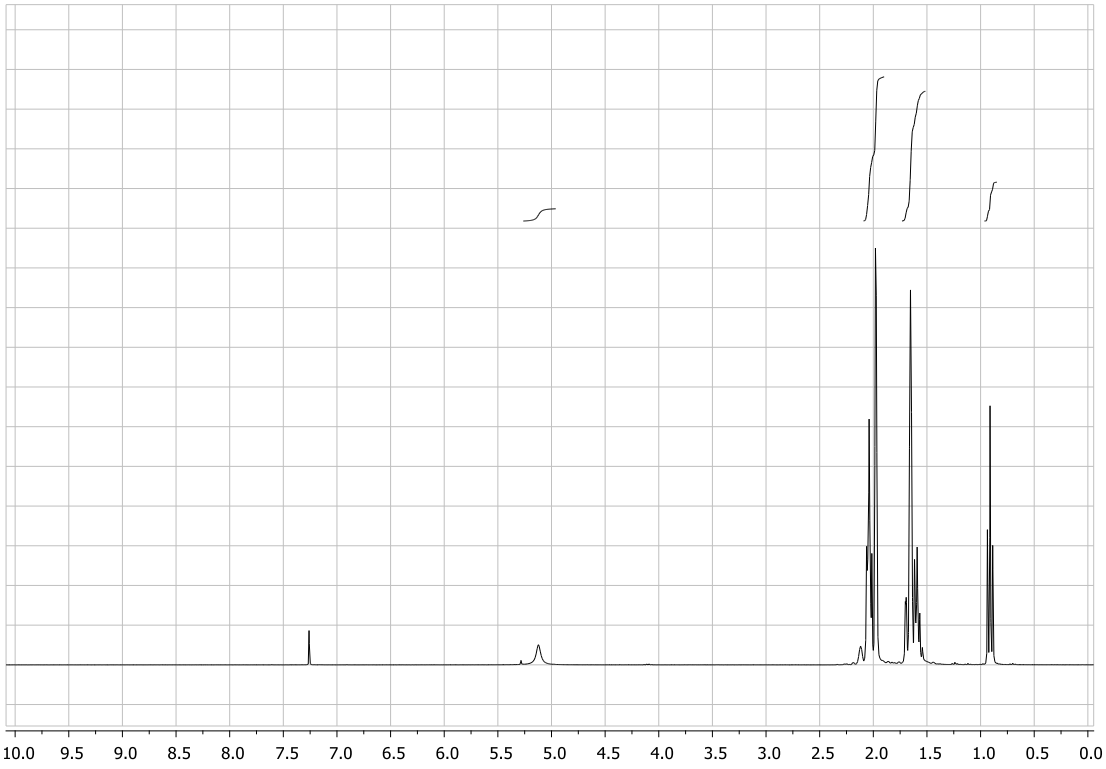


_N_-Butyryl-1-aminoadamantane 11f

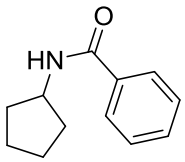


Prepared according to general procedure 2. Pale yellow amorphous solid (525 mg, 79%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.12 (br s, 1H), 2.09 – 1.93 (m, 11H), 1.72 – 1.51 (m, 8H), 0.91 (t, J = 7.4 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): 172.26, 41.82, 39.83, 36.49, 29.55, 19.32, 13.76, which is in agreement with that reported.[5]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of _N_-butyryl-1-aminoadamantane 11f:

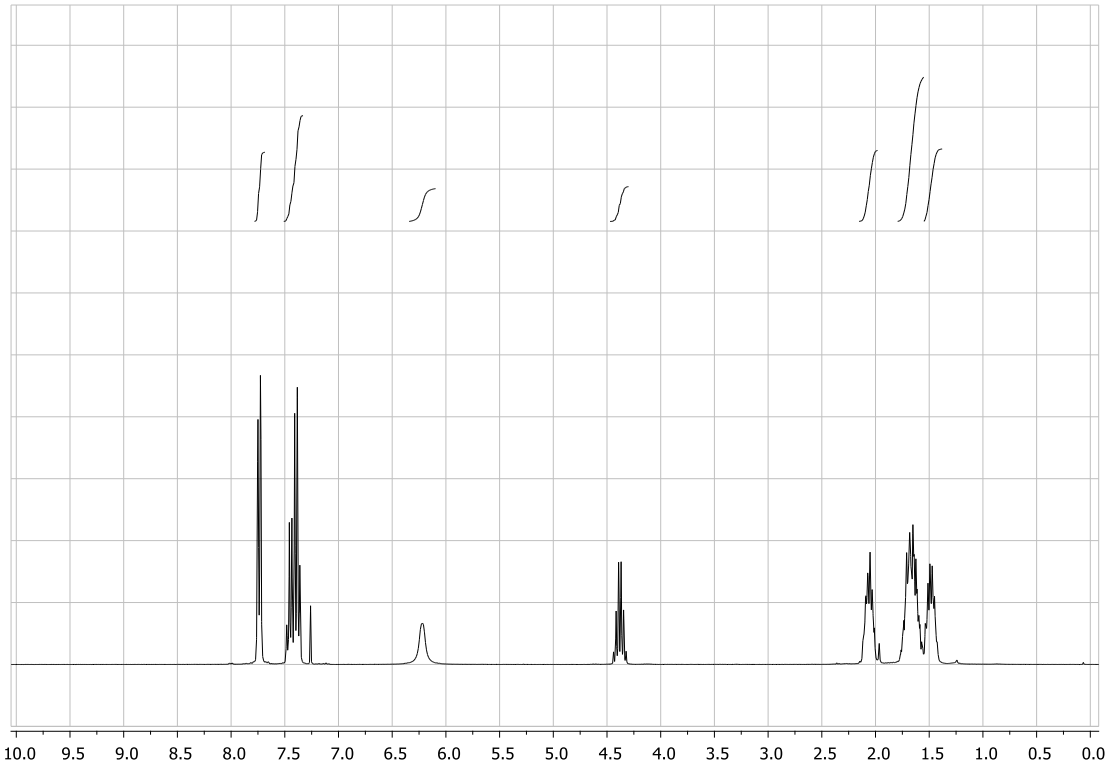


***N*-Cyclopentylbenzamide 11g**

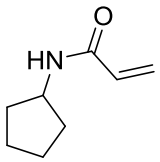


Prepared according to general procedure 2. White crystalline solid (342 mg, 60%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 – 7.71 (m, 2H), 7.50 – 7.34 (m, 3H), 6.22 (br s, 1H), 4.45-4.31 (m, 1H), 2.14 – 1.96 (m, 2H), 1.78 – 1.56 (m, 4H), 1.55 – 1.41 (m, 2H) ppm; which is in agreement with that reported.[12]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-cyclopentylbenzamide 11g:

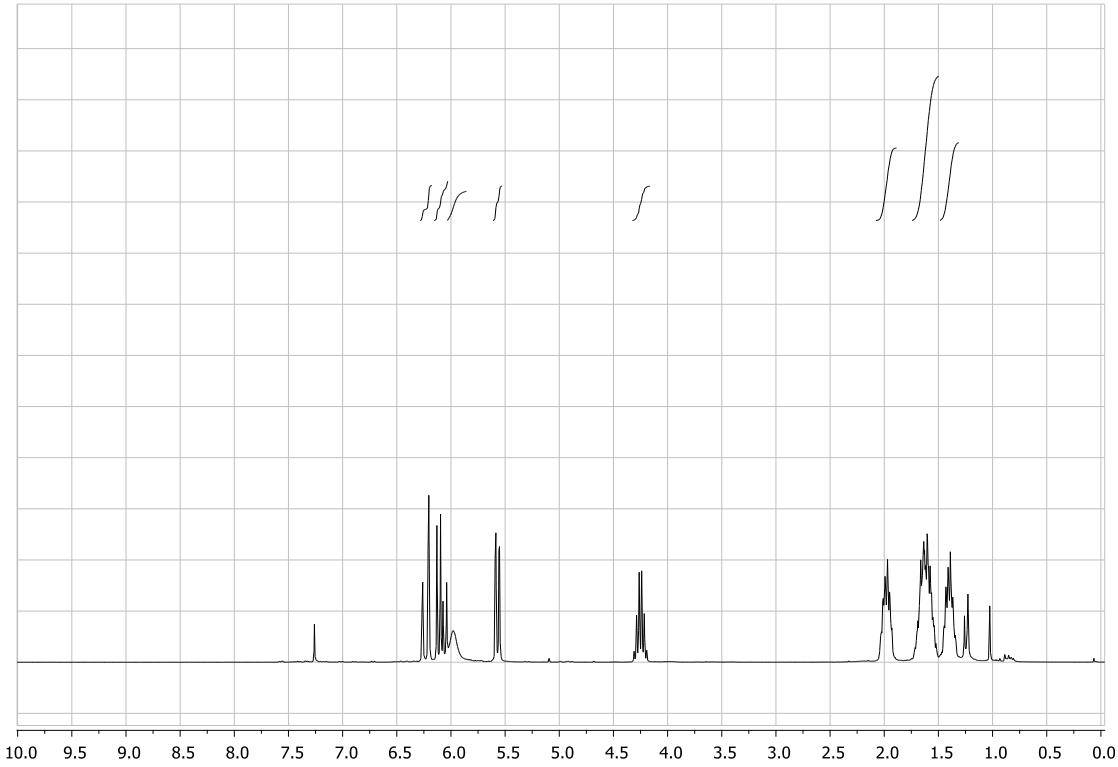


***N*-Cyclopentylacrylamide 11h**

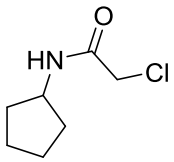


Prepared according to general procedure 2. Pale yellow crystalline solid (200 mg, 48%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.24 (dd, *J* = 17.0, 1.7 Hz, 1H), 6.08 (dd, *J* = 17.0, 10.0 Hz, 1H), 5.98 (br s, 1H), 5.57 (dd, *J* = 10.0, 1.7 Hz, 1H), 4.34 – 4.15 (m, 1H), 2.07 – 1.90 (m, 2H), 1.74 – 1.50 (m, 4H), 1.49 – 1.31 (m, 2H) ppm; which is in agreement with that reported.[10]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-cyclopentylacrylamide 11h:

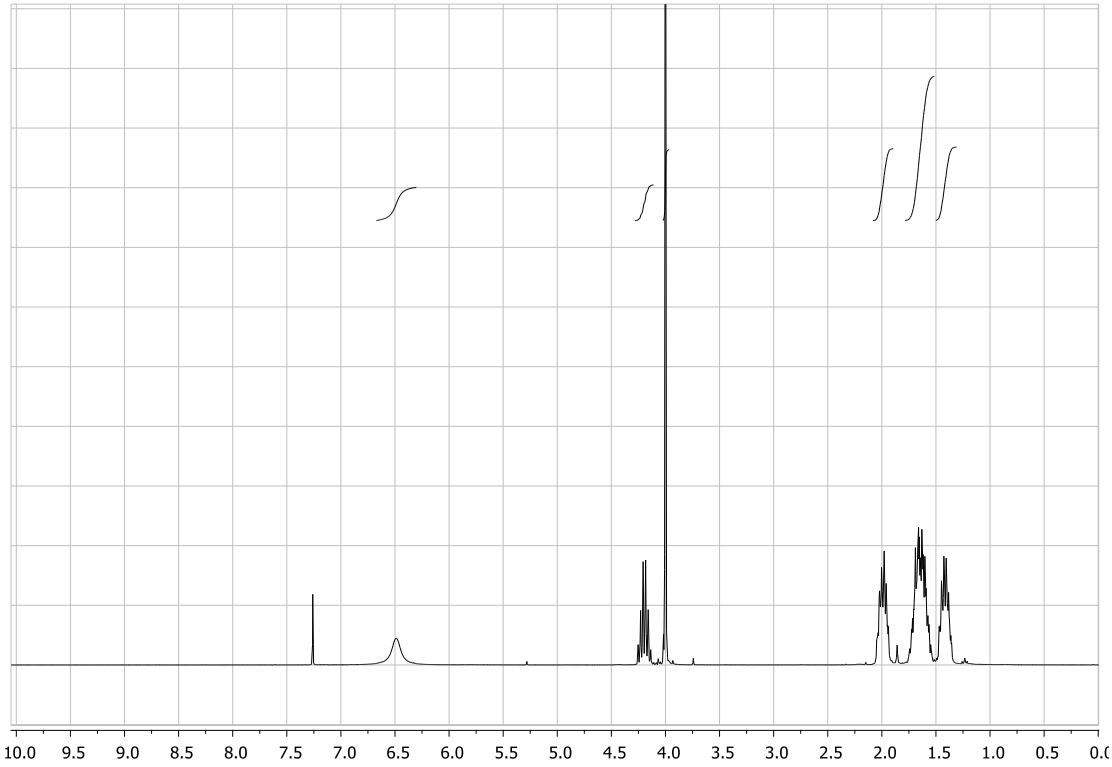


2-Chloro-*N*-cyclopentylacetamide 11i

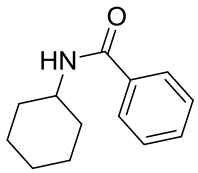


Prepared according to general procedure 2. Pale yellow amorphous solid (340 mg, 71%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.49 (br s, 1H), 4.27 – 4.12 (m, 1H), 4.00 (s, 2H), 2.06 – 1.92 (m, 2H), 1.76 – 1.53 (m, 4H), 1.48 – 1.35 (m, 2H) ppm; which is in agreement with that reported.[10]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of 2-chloro-*N*-cyclopentylacetamide 11i:

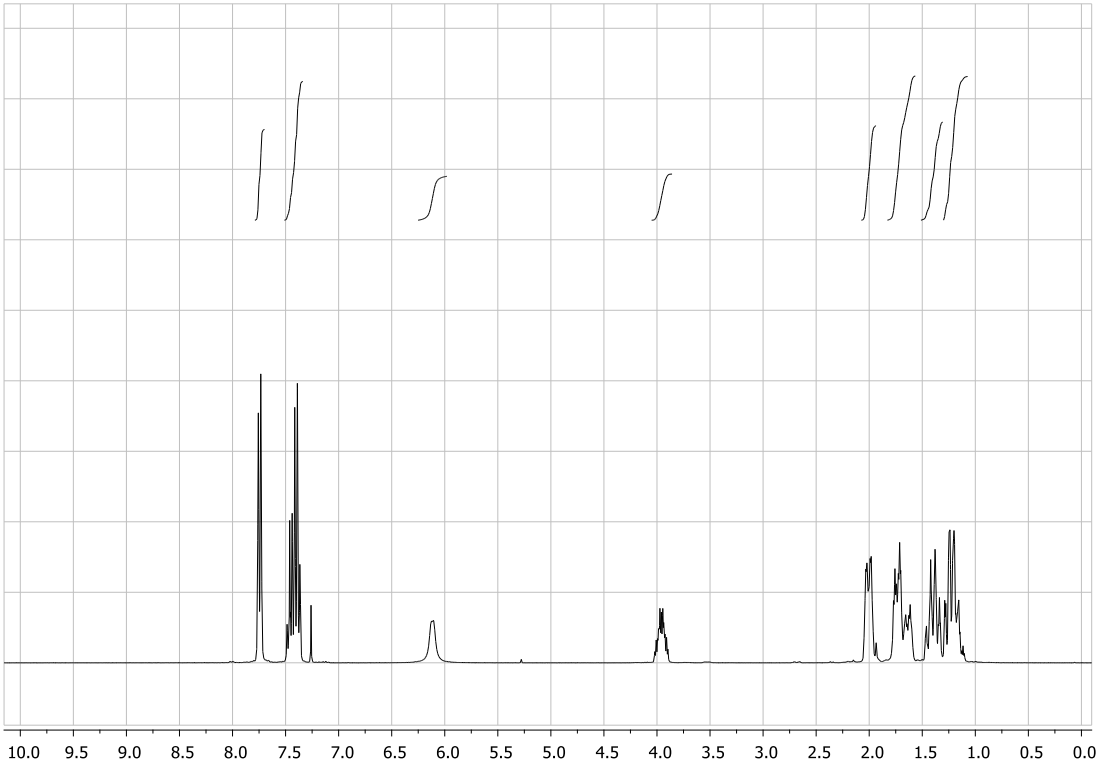


***N*-Cyclohexylbenzamide 11j**

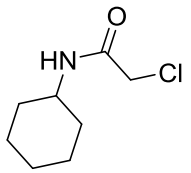


Prepared according to general procedure 2. White crystalline solid (372 mg, 61%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 – 7.71 (m, 2H), 7.50 – 7.35 (m, 3H), 6.12 (br s, 1H), 4.03 – 3.88 (m, 1H), 2.06 – 1.92 (m, 2H), 1.82 – 1.58 (m, 3H), 1.48 – 1.31 (m, 2H), 1.30 – 1.10 (m, 3H) ppm; which is in agreement with that reported.[12]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-cyclohexylbenzamide 11j:

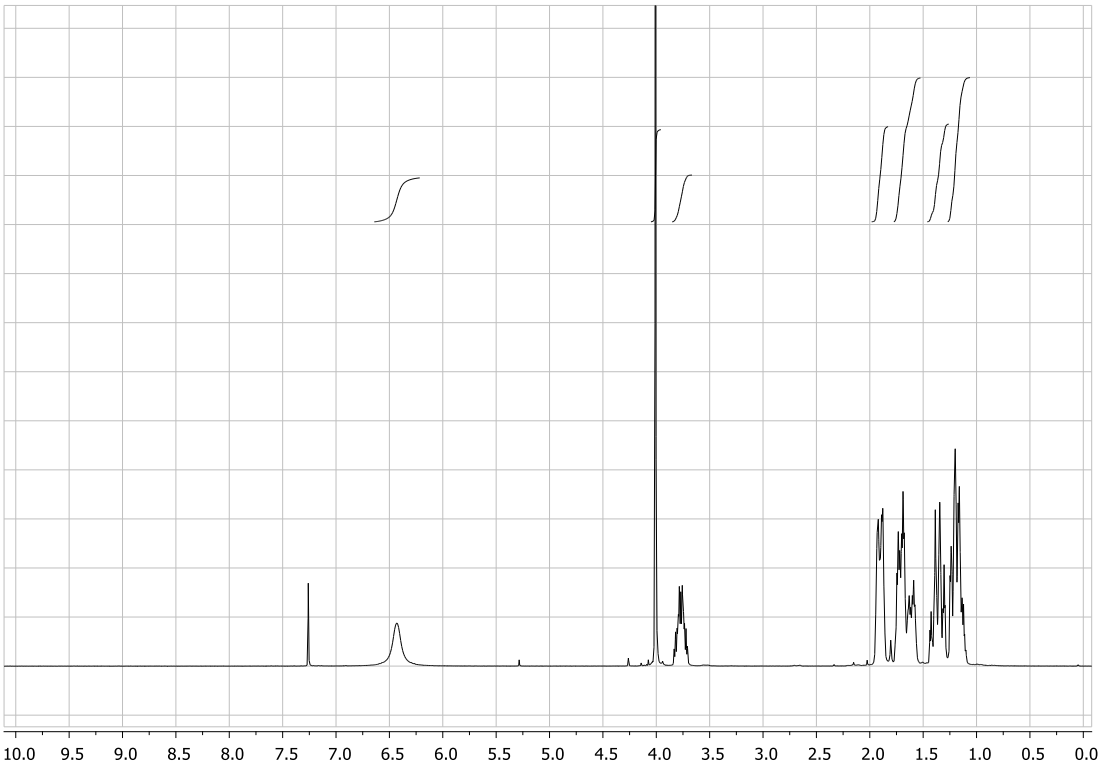


2-Chloro-*N*-cyclohexylacetamide 11k

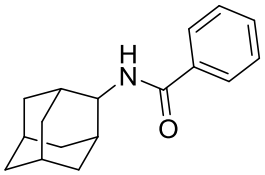


Prepared according to general procedure 2. Pale yellow amorphous solid (350 mg, 66%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.43 (br s, 1H), 4.01 (s, 2H), 3.85 – 3.69 (m, 1H), 1.96 – 1.85 (m, 2H), 1.78 – 1.54 (m, 3H), 1.44 – 1.28 (m, 2H), 1.26 – 1.09 (m, 3H) ppm; which is in agreement with that reported.[13]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of 2-chloro-*N*-cyclohexylacetamide 11k:

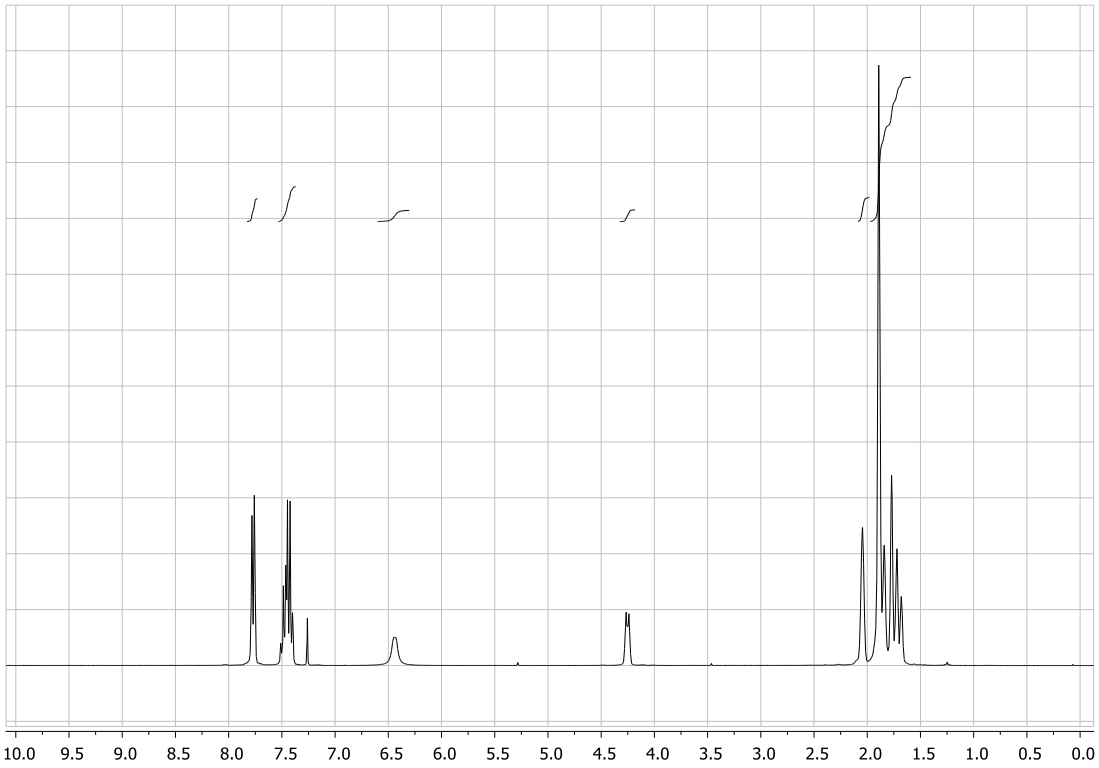


***N*-Benzoyl-2-aminoadamantane 11l**

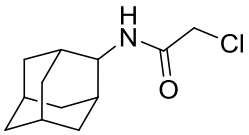


Prepared according to general procedure 2. White crystalline solid (740 mg, 96%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 – 7.73 (m, 2H), 7.53 – 7.38 (m, 3H), 6.43 (br s, 1H), 4.30 – 4.21 (m, 1H), 2.10 – 2.00 (m, 2H), 1.98 – 1.64 (m, 12H) ppm; which is in agreement with that reported.[15]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-benzoyl-2-aminoadamantane 11l:

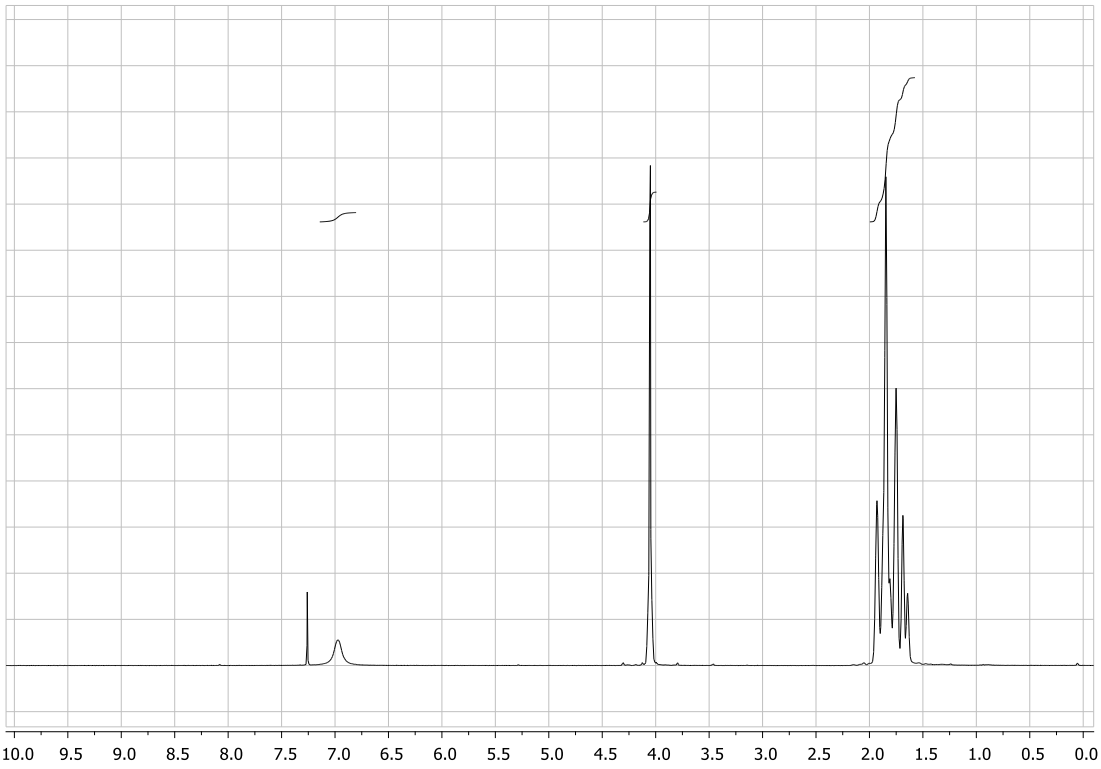


***N*-Chloroacetyl-2-aminoadamantane 11m**

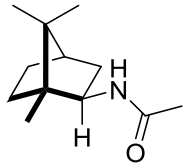


Prepared according to general procedure 2. White crystalline solid (660 mg, 97%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.97 (br s, 1H), 4.08 – 4.02 (m, 3H), 1.97 – 1.62 (m, 14H) ppm; which is in agreement with that reported.[14]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-chloroacetyl-2-aminoadamantane 11m:

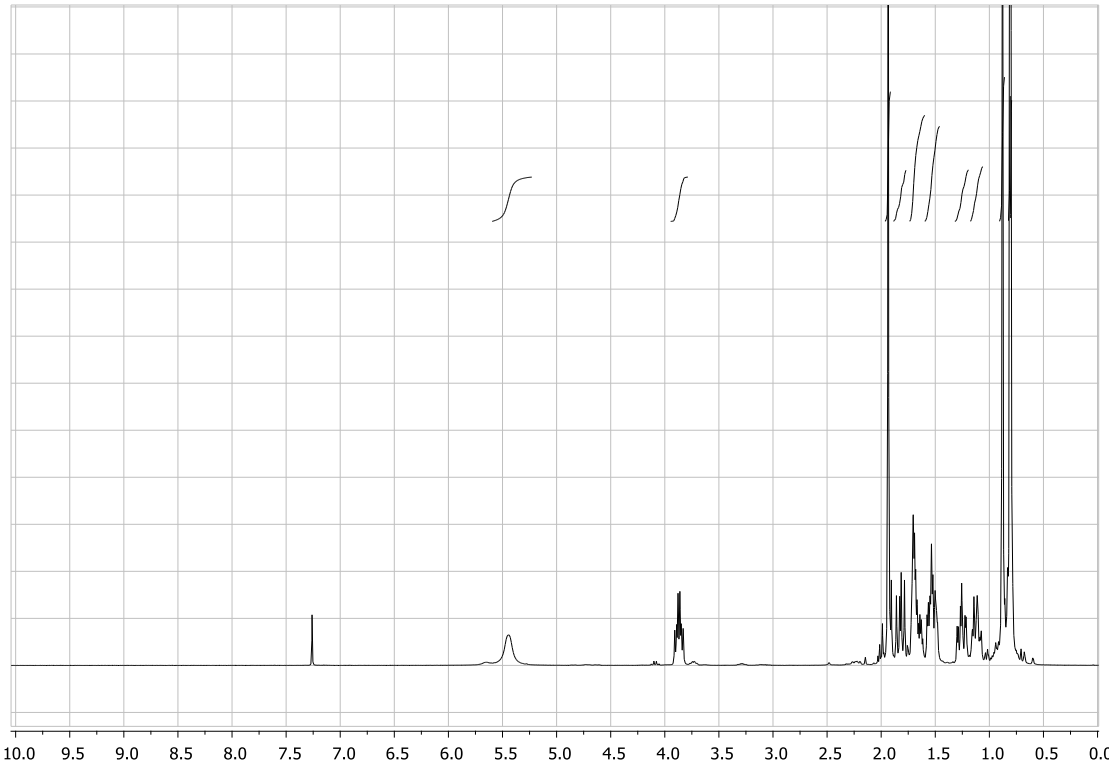


***N*-((1*SR*,2*RS*,4*RS*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl)acetamide 13a**

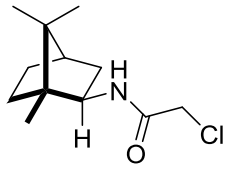


Prepared according to general procedure 2. White crystalline solid (510 mg, 87%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.44 (br s, 1H), 3.87 (td, *J* = 9.0, 5.2 Hz, 1H), 1.97 – 1.77 (m, 4H), 1.73 – 1.61 (m, 2H), 1.60 – 1.46 (m, 2H), 1.32 – 1.20 (m, 1H), 1.18 – 1.07 (m, 1H), 0.88 (s, 3H), 0.81 (s, 3H), 0.80 (s, 3H) ppm; which is in agreement with that reported.[9]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-((1*SR*,2*RS*,4*RS*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)acetamide 13a:

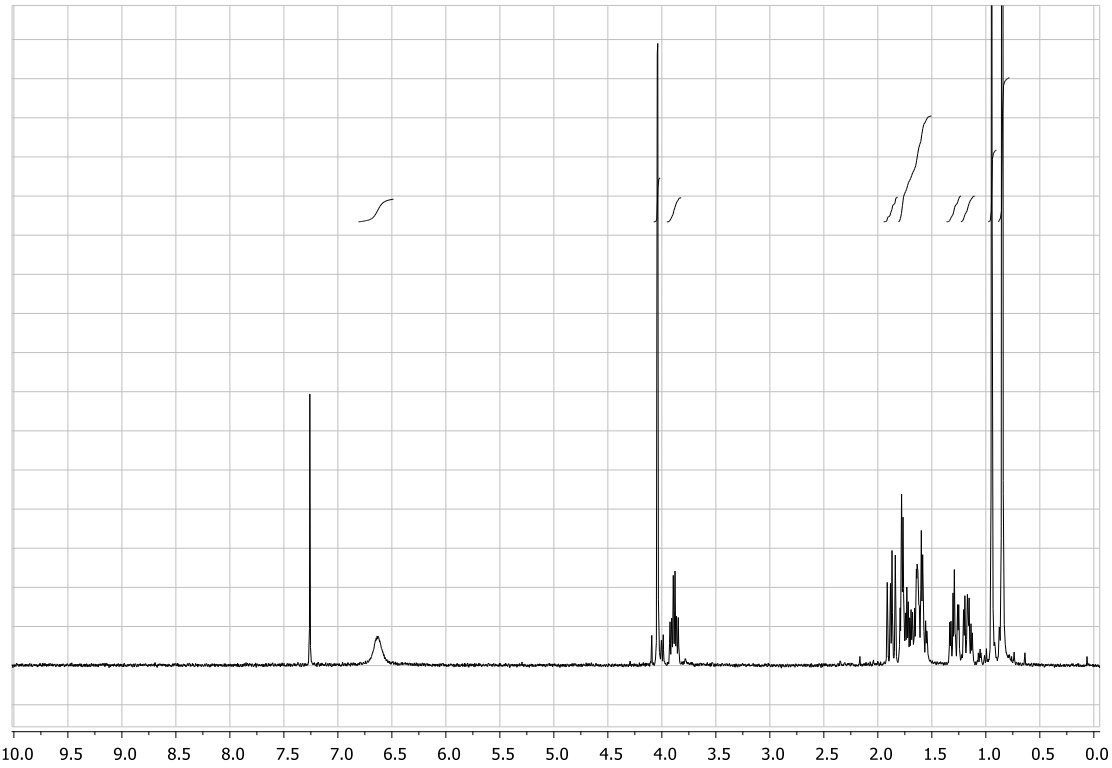


2-Chloro-*N*-((1*SR*,2*RS*,4*RS*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)acetamide 13b

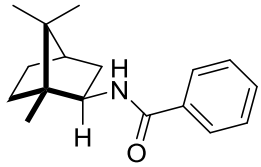


Prepared according to general procedure 2. White crystalline solid (213 mg, 71%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.63 (br s, 1H), 4.03 (d, *J* = 1.5 Hz, 2H), 3.88 (td, *J* = 9.1, 4.9 Hz, 1H), 1.87 (dd, *J* = 13.3, 9.1 Hz, 1H), 1.80 – 1.52 (m, 4H), 1.35 – 1.23 (m, 1H), 1.22 – 1.10 (m, 1H), 0.94 (s, 3H), 0.86-0.83 (m, 6H) ppm; which is in agreement with that reported.[9]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of 2-chloro-*N*-((1*SR*,2*RS*,4*RS*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)acetamide 13b:

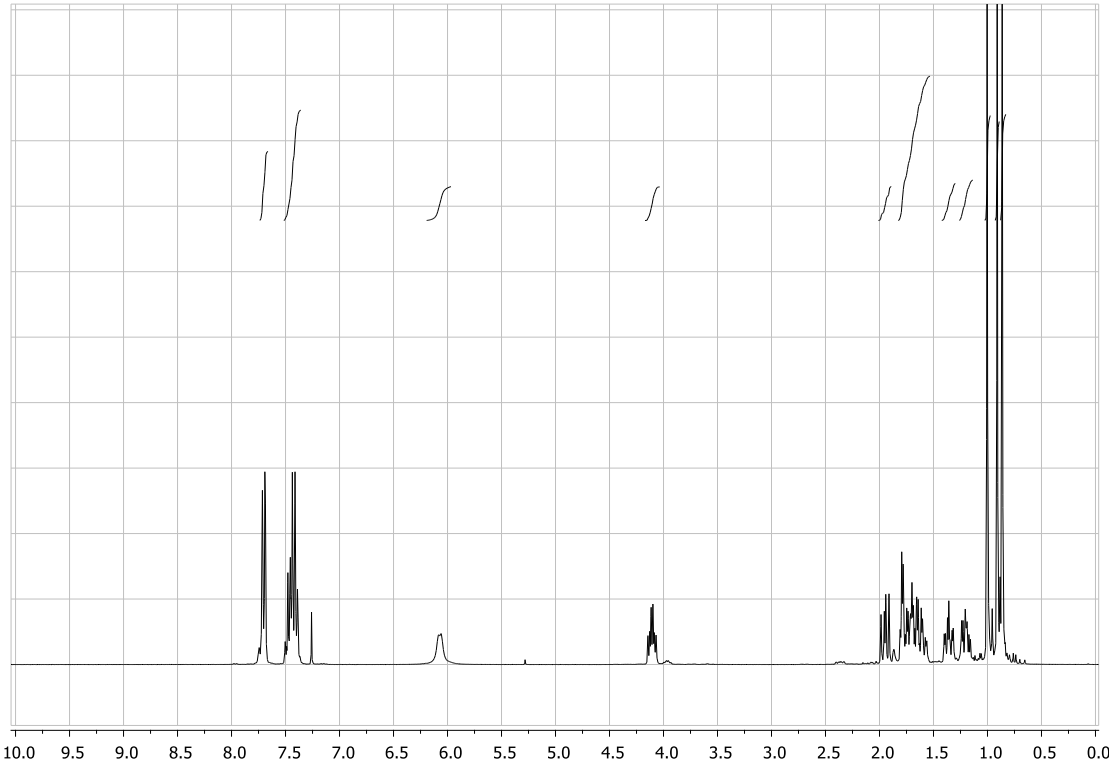


***N*-((1*SR*,2*RS*,4*RS*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl)benzamide 13c**

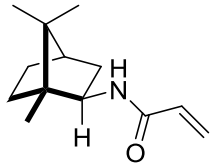


Prepared according to general procedure 2. White crystalline solid (505 mg, 66%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 – 7.66 (m, 2H), 7.52 – 7.36 (m, 3H), 6.07 (d, *J* = 6.6 Hz, 1H), 4.11 (td, *J* = 8.8, 4.9 Hz, 1H), 1.95 (dd, *J* = 13.1, 9.1 Hz, 1H), 1.82 – 1.54 (m, 4H), 1.42 – 1.29 (m, 1H), 1.27 – 1.14 (m, 1H), 1.00 (s, 3H), 0.91 (s, 3H), 0.86 (s, 3H) ppm; which is in agreement with that reported.[8]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-((1*SR*,2*RS*,4*RS*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)benzamide 13c:

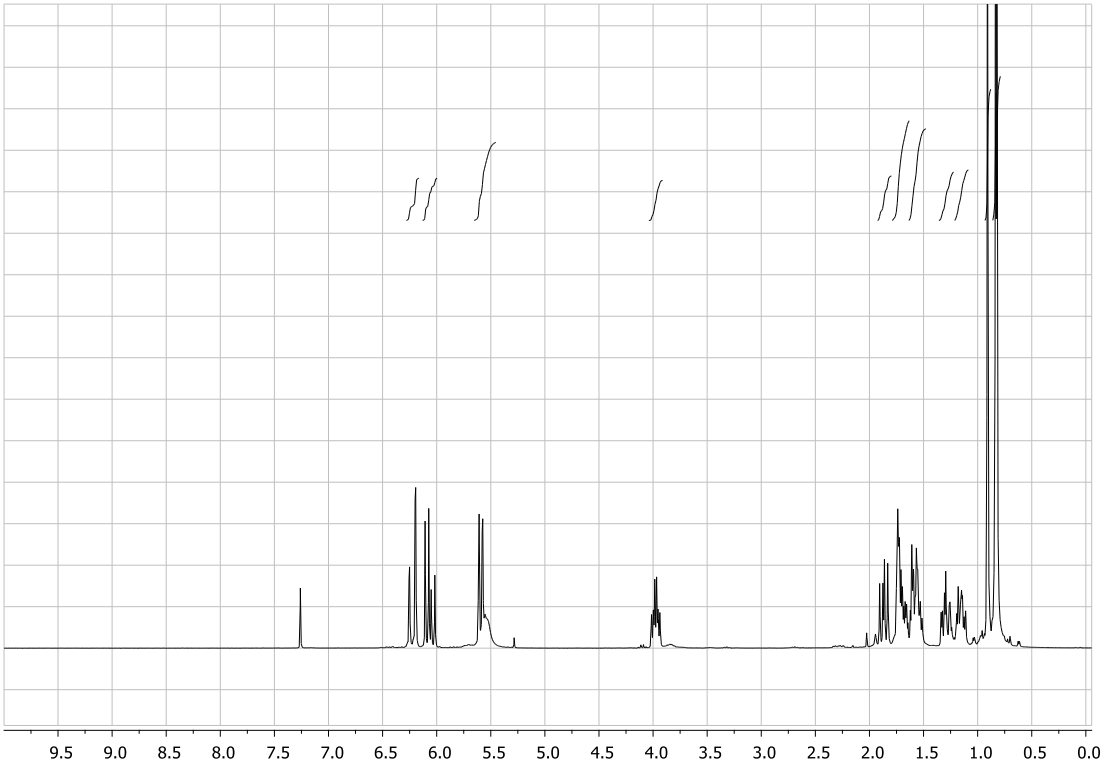


***N*-((1*SR*,2*RS*,4*RS*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl)acrylamide 13d**

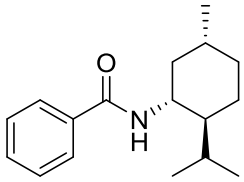


Prepared according to general procedure 2. Pale yellow amorphous solid (422 mg, 83%). 1H NMR (300 MHz, $CDCl_3$) δ 6.22 (dd, *J* = 16.9, 1.6 Hz, 1H), 6.06 (dd, *J* = 16.9, 10.1 Hz, 1H), 5.64 – 5.44 (m, 2H), 3.98 (td, *J* = 9.0, 5.1 Hz, 1H), 1.87 (dd, *J* = 13.3, 9.1 Hz, 1H), 1.77 – 1.64 (m, 2H), 1.63 – 1.50 (m, 2H), 1.35 – 1.23 (m, 1H), 1.21 – 1.09 (m, 1H), 0.91 (s, 3H), 0.83 (s, 3H), 0.82 (s, 3H) ppm; which is in agreement with that reported.[8]

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of *N*-((1*SR*,2*RS*,4*RS*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)acrylamide 13d:



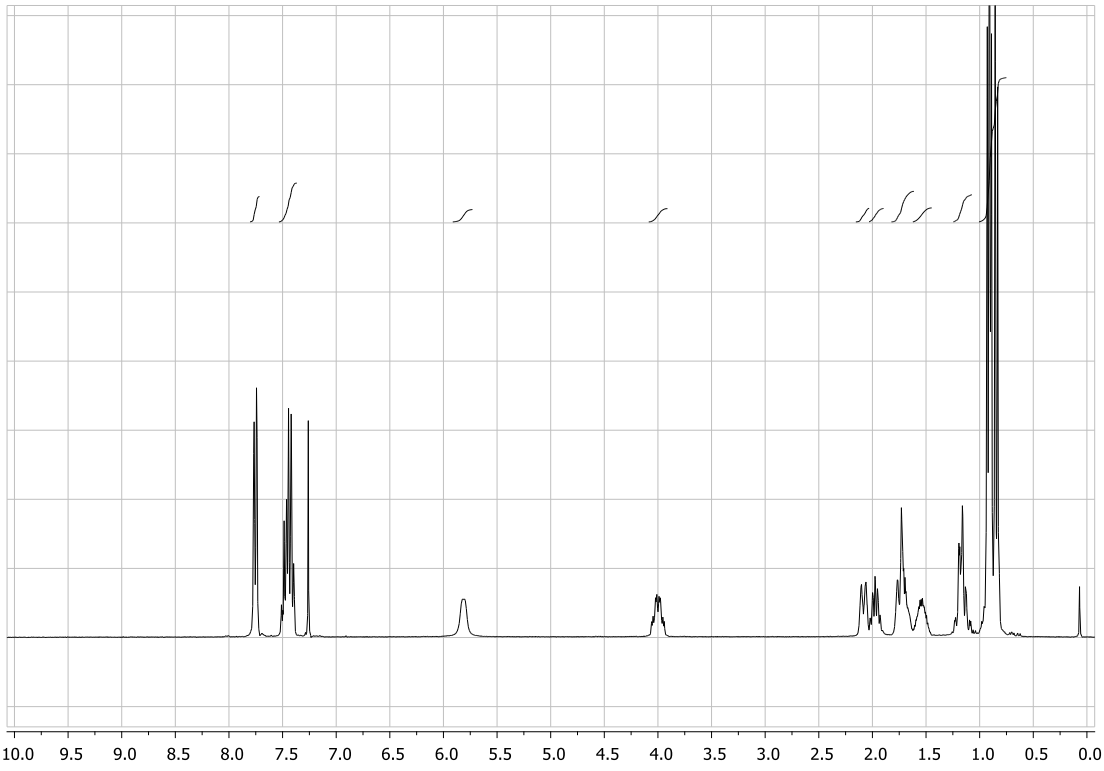
(-)-(1*R*,3*R*,4*S*)-*N*-Menthylbenzamide (-)-15a



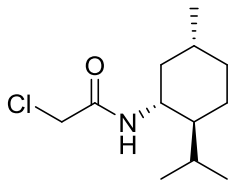
Prepared similar to general procedure 2 (+ 80 ^{o}C, 17h). White crystalline solid (231 mg, 30%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 – 7.71 (m, 2H), 7.53 – 7.37 (m, 3H), 5.82 (br s, 1H), 4.00 (ddd, *J* = 11.2, 11.1, 4.0 Hz, 1H), 2.14 – 2.04 (m, 1H), 2.03 – 1.89 (m, 1H), 1.81 – 1.64 (m, 2H), 1.62 – 1.45 (m, 1H), 1.26 – 1.07 (m, 2H), 1.00 – 0.77 (m, 11H) ppm; which is in agreement with that reported.[16]

$[\alpha]_D^{20}$ = -62.8 (c = 1, $CHCl_3$). [Lit][16]: $[\alpha]_D^{20}$ = -63.6 (c = 1, $CHCl_3$).

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of (-)-(1*R*,3*R*,4*S*)-*N*-menthylbenzamide (-)-15a:

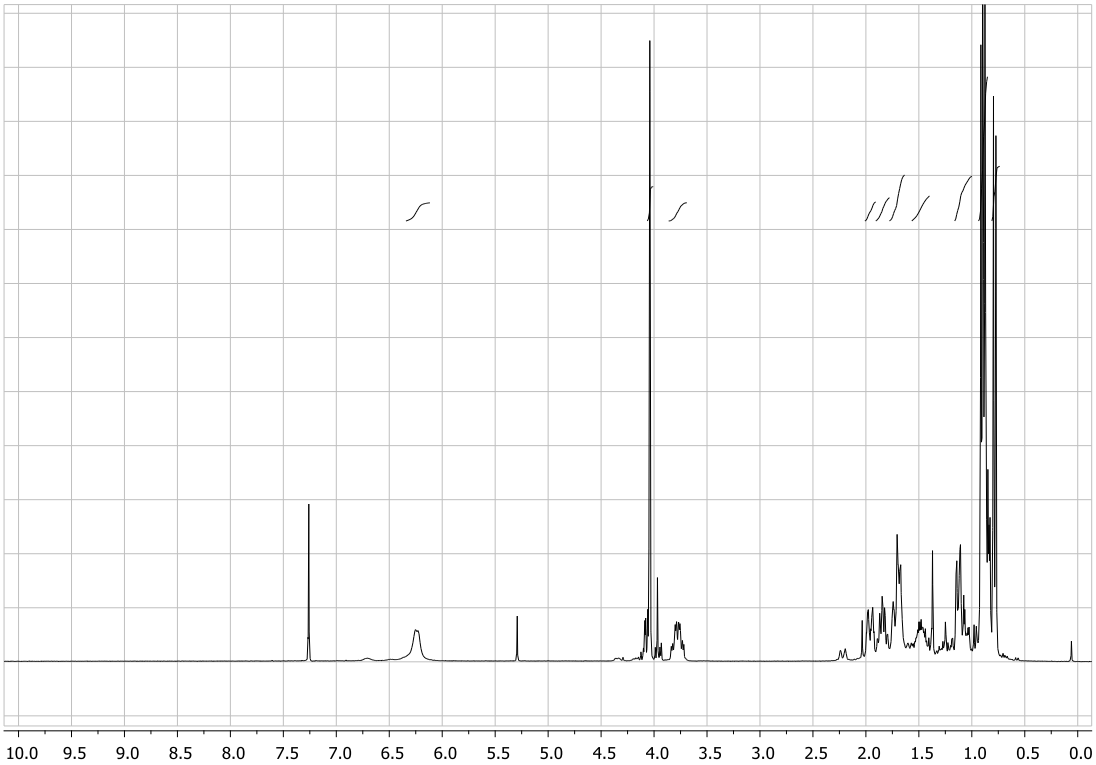


2-Chloro-*N*-[(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl]acetamide (-)-15b

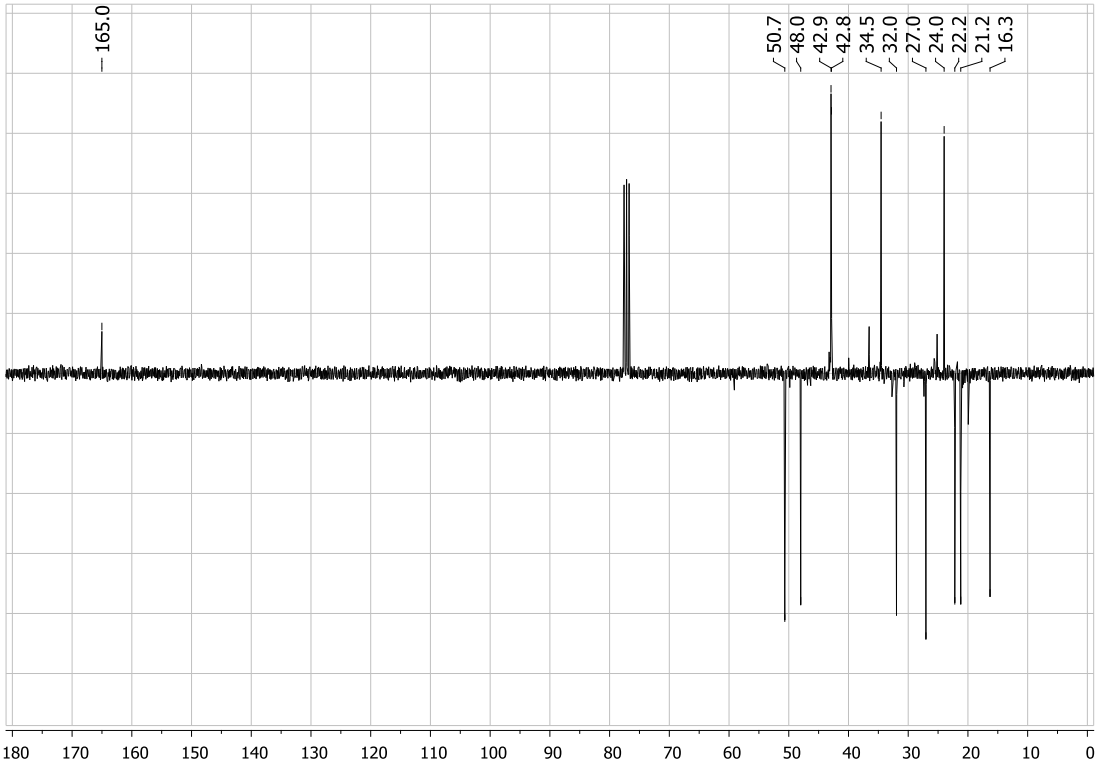


Prepared similar to general procedure 2 (+ 80 ^{o}C, 17h). White crystalline solid (210 mg, 30%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.24 (br s, 1H), 4.04 (s, 2H), 3.78 (ddd, *J* = 20.6, 11.1, 4.1 Hz, 1H), 2.01 – 1.91 (m, 1H), 1.90 – 1.78 (m, 1H), 1.77 – 1.63 (m, 2H), 1.56 – 1.40 (m, 1H), 1.16 – 1.02 (m, 2H), 0.93 – 0.85 (m, 8H), 0.81 – 0.76 (m, 3H) ppm; ^{13}C NMR (75.5 MHz, $CDCl_3$) δ 165.0, 50.7, 48.0, 42.9, 42.8, 34.5, 32.0, 27.0, 24.0, 22.2, 21.2, 16.3 ppm, which is in agreement with that reported.[17]
$[\alpha]_D^{25}$ = -56.9 (c = 0.65, $CHCl_3$). [Lit][17]: $[\alpha]_D^{25}$ = -52.9 (c = 0.65, $CHCl_3$).

^{1}H-NMR ($CDCl_3$, 300 MHz) spectrum of 2-Chloro-*N*-[(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl]acetamide (-)-15b:



^{13}C-NMR ($CDCl_3$, 75 MHz) spectrum of 2-Chloro-*N*-[(1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl]acetamide (-)-15b:



3. References

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