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A Mild One-Pot Conversion of Alkenes into Amines through Tandem Ozonolysis and Reductive Amination

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Abstract

The selective reduction of hydroperoxyacetals to aldehydes by sodium triacetoxyborohydride provides the basis for a mild one-pot synthesis of amines from alkenes.


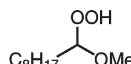
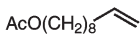
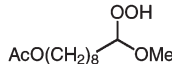
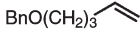
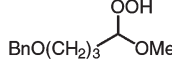
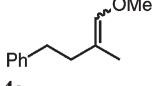
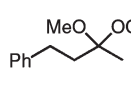
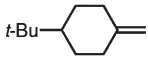
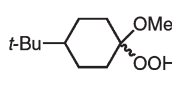
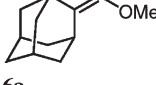
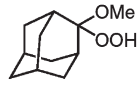
Keywords: ozonolysis, hydroperoxyacetal, reductive amination, amine, sodium triacetoxyborohydride

Reductive amination is a widely used transformation in organic synthesis.² The carbonyl precursors are often prepared from alkenes through ozonolysis followed by reduction of the ozonide or peroxide intermediates.^{3, 4} In the course of investigations into new transformations based upon fragmentation of ozonolysis intermediates,⁵ we became interested in the development of a mild method for a one-pot conversion of alkenes to amines. Reductive amination is frequently achieved through the reaction of carbonyls and amines in the presence of deactivated boron hydrides,^{2b, 6, 7} and NaCNBH₃-promoted reductive amination has been applied in tandem with ozonolysis.⁸ However, NaCNBH₃ reduces ozonides (1,2,4-trioxolanes) very slowly,⁹ and some of the reported transformations may actually involve amine-promoted E₁cb fragmentation of terminal ozonides.^{10, 11} We investigated the reaction of NaCNBH₃ with more reactive hydroperoxyacetals, but observed significant formation of alcohols. Our attention was therefore turned to the less reactive triacetoxyborohydride, which has been investigated little in ozonolysis/amination sequences.^{12, 13} We now report that NaBH(OAc)₃ allows rapid and efficient reduction of ozonolysis-derived hydroperoxyacetals to aldehydes, enabling a mild and convenient one-pot synthesis of amines from alkenes based upon ozonolysis and reductive amination.

The starting hydroperoxyacetals were readily available through ozonolysis of precursor alkenes or enol ethers in 3% MeOH-CH₂Cl₂ (Table 1).^{14, 15}

Reduction conditions were initially screened on hydroperoxyacetal **1b** using ¹H NMR of crude reaction mixtures to monitor the ratio of starting hydroperoxyacetal, aldehyde (nonanal), and the alcohol (nonanol) derived from overreduction. It was found that NaBH(OAc)₃ offered high selectivity for aldehyde formation in several different solvent systems (CH₂Cl₂, DCE, THF) and under several sets of conditions. Performing the reduction with one equivalent of NaBH(OAc)₃ proved optimal; and excess of reducing reagent (1.5–3 equiv) led to slow overreduction of the aldehyde. In contrast, the use of the more reactive NaCNBH₃ in stoichiometric amounts cleanly generated alcohol, while a mixture of alcohol and starting material was observed even with only 0.33 equivalent of reagent.

Table 1. Preparation of Hydroperoxyacetals

Alkene	Hydroperoxyacetal	Yield
		1b (70%)
		2b (67%)
		3b (69%)
		4b (38%) ^a
		5b (74%)
		6b (63%)

^a Partial decomposition upon chromatography.

Reduction with NaBH(OAc)₃ was next applied to a wider group of hydroperoxyacetals (Table 2). Hydroperoxyacetals reacted faster (2 h) than hydroperoxyketals (5–8 h). In both cases, a high yield of the carbonyl was obtained with little or no overreduction.

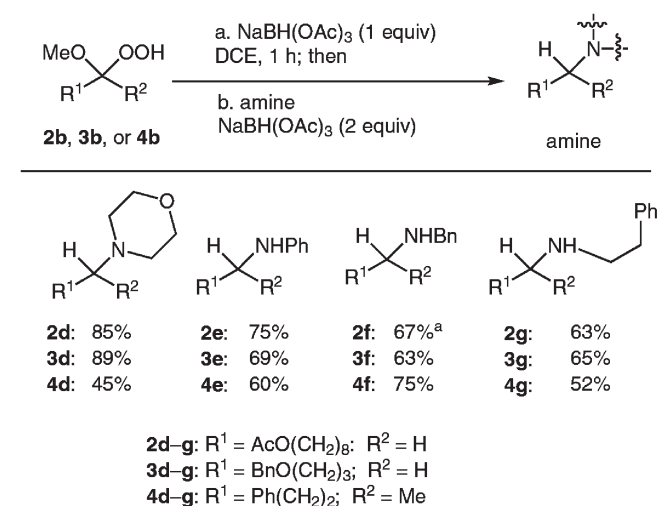
Reductive amination was easily conducted through reduction of the hydroperoxyacetal with NaBH(OAc)₃ (1 equiv), followed by addition of amine and additional hydride (Scheme 1). The reactions were found to give good yields of monoalkylated amines. For the amination of aldehyde **2b** with benzylamine, 8% of the tertiary amine derived from overalkylation was isolated, in addition to the expected secondary amine **2f**.

Finally, it was found that ozonolysis and reductive amination could be easily combined into a one-pot transformation

Table 2. Reduction of Hydroperoxyacetals with NaBH(OAc)₃

hydroperoxyacetal	$\xrightarrow[\text{DCE, r.t.}]{\text{NaBH(OAc)}_3 \text{ (1 equiv)}}$	carbonyl
Hydroperoxy acetal		Carbonyl
1b		1c (63%, 2 h)
2b		2c (85%, 2 h)
3b		3c (91%, 2 h)
4b		4c (70%, 5 h)
5b		5c (68%, 5 h)
6b		6c (68%, 8 h) ^a

^a Based upon 15% recovered starting material.



^a 8% of 3° amine was also isolated.

Scheme 1. Stepwise reduction-reductive amination

(Table 3). Ozonolysis of an alkene substrate was conducted in methanol-CH₂Cl₂ until the alkene could no longer be detected (TLC). The crude reaction mixture was then submitted to one of two protocols. In the first (method A), addition of acetoxyborohydride (1 equiv) was followed, after 45 minutes to 1 hour, by addition of amine and additional acetoxyborohydride. More conveniently (method B), the crude ozonolysis product was treated with acetoxyborohydride (3 equiv) and amine (1-2 equiv). Similar yields were obtained from the two sequences. Under these conditions, cyclic alkenes un-

Table 3. Stepwise versus One-Pot Reductive Amination

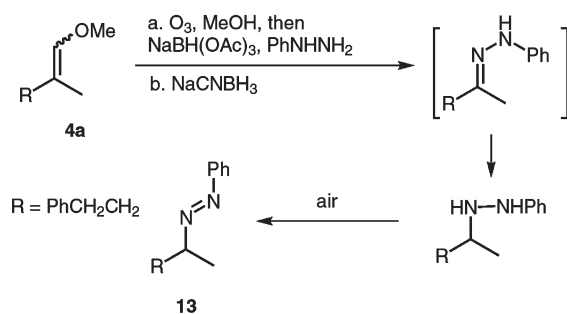
alkene	Reactant amine	Method (yield)	product
	PhCH ₂ NH ₂	A (57%)	
	Ph(CH ₂) ₂ NH ₂	A (63%)	
3a	morpholine	A (64%)	3d
	PhCH ₂ NH ₂	A (65%)	
	Ph(CH ₂) ₂ NH ₂	A (72%) B (62%)	
2a	morpholine	B (66%)	2d
	PhCH ₂ NH ₂	B (60%)	
	Ph(CH ₂) ₂ NH ₂	A (65%) B (65%)	

O₃, CH₂Cl₂-MeOH; then:
 A) NaBH(OAc)₃; then reactant amine, additional NaBH(OAc)₃
 B) NaBH(OAc)₃ (3 equiv) plus reactant amine

11a R = H 50%
11b R = Me 10%

dergo a tandem oxidation/reduction amination sequence to provide a variety of heterocyclic amines with an average yield of ~90% per reaction. In some cases, condensation of the product secondary amine with residual formaldehyde, a by-product of the ozonolysis of terminal olefins, gave rise to a tertiary amine (e.g., **11b**).

A very similar protocol can be applied to the synthesis of hydrazones, hydrazines, and diazenes. Ozonolysis of **4a**, followed by treatment of the crude hydroperoxyacetal with phenylhydrazine and NaBH(OAc)₃, cleanly furnished a phenylhydrazone (TLC). Addition of NaCNBH₃ resulted in the formation of the phenylhydrazide, which underwent rapid air oxidation during isolation to furnish the diazene as the isolated material in 57% yield (Scheme 2).



Scheme 2

While there are many procedures for conversion of alkenes to amines, most require separate oxidation and amination steps, each involving isolation and purification. The procedure reported here, which allows a one-pot conversion of alkenes to a variety of amines while avoiding any accumulation of peroxide intermediates, may be of particular use for preparation of an array of amines from a common precursor, or in procedures limited by concerns with isolation or handling of peroxide intermediates. Utilization of $\text{NaBH}(\text{OAc})_3$ in place of NaCNBH_3 provides a more economical, safe, and selective protocol for the one-pot tandem ozonolysis/reductive amination sequence for the conversion of alkenes to amines. The speed and selectivity of the reduction of the hydroperoxyacetals suggests the formation and decomposition of a peroxyborate, $\text{ROOBH}(\text{OAc})_2$.

All reagents were used as received from commercial vendors, with the exception of CH_2Cl_2 , which was distilled from CaH_2 , and THF, which was distilled from Na/benzophenone . TLC was performed on 0.25 mm hard-layer silica G plates; developed plates were visualized with a hand-held UV lamp or by staining: 1% $\text{Ce}(\text{SO}_4)_2$ and 10% $(\text{NH}_4)_2\text{MoO}_4$ in 10% H_2SO_4 (general stain, after charring); 1% N,N' -dimethyl-*p*-phenylenediamine solution in 1:20:100 $\text{AcOH-H}_2\text{O-MeOH}$ (specific for peroxides);¹⁶ 1% aq KMnO_4 (for unsaturated compounds); 3% vanillin in 3% H_2SO_4 in EtOH (general stain after charring). NMR spectra were recorded at 400 MHz (^1H) or 100 MHz (^{13}C) and in CDCl_3 unless otherwise indicated; peaks are reported as: chemical shift (multiplicity, *J* couplings in Hz, number of protons). IR spectra were recorded as neat films (ZnSe , ATR mode) with selected absorbances reported in wavenumbers (cm^{-1}). Melting points were collected using a melting point apparatus and all values are uncorrected unless otherwise noted.

Hydroperoxyacetals from Alkenes; General Procedure 1 (GP 1)

A solution of alkene (~1 mmol) and MeOH (0.3 mL) in CH_2Cl_2 (10 mL) in a round-bottomed flask was cooled to -78°C and a gaseous stream of 2% O_3/O_2 (approximately 1 mmol O_3/min) was bubbled through the solution. Once the pale blue color of ozone appeared, the ozonizer voltage was set to zero and the reaction was sparged with O_2 until the blue color dissipated. The reaction was quenched with sat. aq NaHCO_3 (15 mL) and the resulting mixture was allowed to warm to r.t. The separated aqueous layer was extracted with CH_2Cl_2 (2×25 mL) and the combined organic layers were dried (Na_2SO_4). The residue obtained upon concentration in vacuo was purified by flash chromatography using EtOAc-hexanes .

Carbonyl Compounds from Hydroperoxyacetals; General Procedure 2 (GP 2)

To a r.t. solution of hydroperoxyacetal (~0.5 mmol) in DCE (10 mL) was added $\text{NaBH}(\text{OAc})_3$ (0.5 mmol). After product formation was complete (TLC, eluent: EtOAc-hexanes), the reaction was diluted with CH_2Cl_2 (~40 mL) and washed sequentially with sat. aq NaHCO_3 (~30 mL) and brine (~30 mL). The organic layer was dried (Na_2SO_4) and the residue obtained upon concentration in vacuo was purified by flash chromatography using EtOAc-hexanes .

Amines from Hydroperoxyacetals; General Procedure 3 (GP 3)

To a solution of hydroperoxyacetal (~0.5 mmol) in DCE (10 mL) was added $\text{NaBH}(\text{OAc})_3$ (0.5 mmol). The reaction mixture was stirred for an hour at r.t., after which additional $\text{NaBH}(\text{OAc})_3$ (1 mmol) was added followed by amine (0.5 mmol). After the reaction appeared complete (TLC, eluent: EtOAc-hexanes), the solution was diluted with CH_2Cl_2 (40 mL), and washed sequentially with sat. aq NaHCO_3 (30 mL) and brine (30 mL). The organic layer was dried (Na_2SO_4) and the residue obtained upon concentration was purified by flash chromatography with $\text{CH}_2\text{Cl}_2\text{-MeOH}$ (1-2%) containing a few drops of Et_3N .

Direct Synthesis of Amines from Alkenes; General Procedure 4 (GP 4)

Stepwise (Method A): A solution of alkene (~1 mmol) and MeOH (0.3 mL) in CH_2Cl_2 (10 mL) in a round-bottomed flask was ozonized as in GP 1. Following removal of free ozone, the cooling bath was removed and $\text{NaBH}(\text{OAc})_3$ (1 mmol) was added. The reaction mixture was stirred at r.t. for 45 min, after which amine (~1 mmol) and $\text{NaBH}(\text{OAc})_3$ (2 mmol) were sequentially added. The mixture was stirred at r.t. for 1-2 h and then filtered through Celite and the pad washed with CH_2Cl_2 (20 mL). The filtrate was concentrated on a rotary evaporator, and the residue was purified as in GP 3.

One Step (Method B): A solution of alkene (~1 mmol) and MeOH (0.3 mL) in CH_2Cl_2 (10 mL) in a round-bottomed flask was ozonized as in GP 1. Following removal of free ozone, the cooling bath was removed and $\text{NaBH}(\text{OAc})_3$ (3 mmol) and the amine (1-2 mmol) were sequentially added. The reaction mixture was stirred at r.t. for 1-2 h and then filtered through Celite and the pad washed with CH_2Cl_2 (20 mL). The filtrate was concentrated on a rotary evaporator, and the residue was purified as in GP 3.

Hydroperoxyacetals from Alkenes-1-Methoxynonyl Hydroperoxide (1b) [20525-41-1]

Prepared from **1a** (1.40 g, 10 mmol) by GP 1; yield: 1.33 g (70%); $R_f = 0.22$ (10% EtOAc-hexanes).

^1H NMR: $\delta = 8.19$ (s, 1 H), 4.76 (t, $J = 5.6$ Hz, 1 H), 3.52 (s, 3 H), 1.60-1.80 (m, 2 H), 1.20-1.50 (m, 12 H), 0.90 (t, $J = 6.8$ Hz, 3 H).

^{13}C NMR: $\delta = 108.8, 55.8, 31.9, 31.3, 29.43, 29.38, 29.3, 24.6, 22.7, 14.1$.

9-Hydroperoxy-9-methoxynonanol Acetate Ester (2b) [1255126-65-8]

Prepared from **2a** (2.0 g, 10 mmol) by GP 1; yield: 1.66 g (67%); $R_f = 0.33$ (25% EtOAc-hexanes).

^1H NMR (300 MHz): $\delta = 9.46$ (s, 1 H), 4.66 (t, $J = 5.8$ Hz, 1 H), 3.97 (t, $J = 6.7$ Hz, 2 H), 3.43 (s, 3 H), 1.98 (s, 3 H), 1.66-1.48 (m, 4 H), 1.33-1.23 (m, 10 H).

^{13}C NMR (75 MHz): $\delta = 171.6, 108.5, 64.7, 55.7, 31.3, 29.2, 29.1, 28.9, 28.4, 25.7, 24.5, 20.9$.

4-Benzyloxy-1-methoxybutyl Hydroperoxide (3b) [1255126-67-0]

Prepared from **3a** (1.76 g, 10 mmol) by GP 1; yield: 1.56 g (69%); $R_f = 0.37$ (25% EtOAc-hexanes).

^1H NMR: δ = 8.93 (s, 1 H), 7.39-7.29 (m, 5 H), 4.77-4.74 (m, 1 H), 4.54 (s, 2 H), 3.58-3.50 (s, 5 H), 1.93-1.71 (m, 4 H).

^{13}C NMR: δ = 138.1, 128.4, 127.8, 127.7, 108.4, 72.9, 69.8, 55.9, 28.0, 24.6.

(3-Hydroperoxy-3-methoxybutyl)benzene (4b)

Prepared from 4a (1.0 g, 5.6 mmol) by GP 1; yield: 0.427 g (38%); R_f = 0.36 (25% EtOAc-hexanes).

IR (neat): 3345, 2943, 1102, 1069, 697 cm^{-1} .

^1H NMR: δ = 7.49 (s, 1 H), 7.31-7.22 (m, 5 H), 3.37 (s, 3 H), 2.76-2.69 (m, 2 H), 2.08-2.02 (m, 2 H), 1.45 (s, 3 H).

^{13}C NMR: δ = 141.7, 128.5, 128.3, 126.0, 106.9, 49.1, 36.9, 30.5, 19.1.

HRMS-ESI: m/z calcd for $\text{C}_{11}\text{H}_{16}\text{O}_3 + \text{Na}$ (M + Na) $^+$: 219.0997; found: 219.1007.

4-(*t*-Butyl)-1-hydroperoxy-1-methoxycyclohexane (5b) [169294-55-7]

Prepared as a mixture of diastereomers from 5a (1.52 g, 10 mmol) by GP 1; yield: 1.49 g (74%); R_f = 0.41 (20% EtOAc-hexanes).

^1H NMR: δ = 7.50 (s, 0.25 H), 7.48 (s, 0.68 H), 3.34 (s, 0.79 H), 3.31 (s, 2.18 H), 2.20-2.28 (m, 0.54 H), 2.09-2.19 (m, 1.52 H), 1.66-1.77 (m, 2 H), 1.33-1.46 (m, 2 H), 1.12-1.32 (m, 2 H), 1.00-1.11 (m, 1 H), 0.89 (s, 9 H).

^{13}C NMR: δ = 105.7, 105.4, 48.5, 48.3, 47.6, 47.5, 32.3, 31.5, 30.9, 27.6, 23.6, 23.4.

2-Hydroperoxy-2-methoxyadamantane (6b) [55975-21-8]

Prepared from 6a (1.0 g, 5.61 mmol) by GP 1; yield: 0.700 g (63%); R_f = 0.50 (25% EtOAc-hexanes).

^1H NMR: δ = 7.34 (s, 1 H), 3.32 (s, 3 H), 2.23 (m, 2 H), 2.01-1.86 (m, 6 H), 1.70-1.66 (m, 6 H).

^{13}C NMR: δ = 107.5, 47.4, 37.1, 33.71, 33.68, 31.9, 27.0, 26.9.

Carbonyls from HydroperoxyacetalsNonanal (1c) [124-19-6]

Prepared from 1b (0.095 g, 0.5 mmol) by GP 2; reaction time: 2 h; yield: 0.045 g (63%); R_f = 0.66 (10% EtOAc-hexanes).

^1H NMR: δ = 9.78 (t, J = 1.8 Hz, 1 H), 2.43 (dt, J = 1.8, 7.3 Hz, 2 H), 1.64 (m, 2 H), 1.31-1.28 (m, 10 H), 0.89 (t, J = 6.9 Hz, 3 H).

^{13}C NMR: δ = 203.0, 43.9, 31.8, 29.3, 29.2, 29.1, 22.6, 22.0, 14.0.

9-Hydroxy-nonanal Acetate Ester (2c) [29541-97-7]

Prepared from 2b (0.496 g, 2 mmol) by GP 2; reaction time: 2 h; yield: 0.341 g (85%); R_f = 0.44 (25% EtOAc-hexanes).

^1H NMR: δ = 9.78 (t, J = 1.8 Hz, 1 H), 4.07 (t, J = 6.8 Hz, 2 H), 2.45 (dt, J = 1.8, 7.3 Hz, 2 H), 2.07 (s, 3 H), 1.64 (m, 4 H), 1.34 (m, 8 H).

^{13}C NMR: δ = 202.7, 171.1, 64.5, 43.8, 29.1, 28.97, 28.95, 28.5, 25.8, 21.9, 20.9.

4-(Benzyloxy)butanal (3c) [5470-84-8]

Prepared from 3b (0.107 g, 0.47 mmol) by GP 2; reaction time: 2 h; yield: 0.076 g (91%); R_f = 0.33 (25% EtOAc-hexanes).

^1H NMR: δ = 9.79 (t, J = 1.5 Hz, 1 H), 7.39-7.27 (m, 5 H), 4.51 (s, 2 H), 3.53 (t, J = 6.0 Hz, 2 H), 2.58 (dt, J = 1.32, 7.1 Hz, 2 H), 1.98 (quint, J = 6.5 Hz, 2 H).

^{13}C NMR: δ = 202.3, 138.3, 128.4, 127.6, 72.9, 69.1, 40.9, 22.6.

4-Phenylbutan-2-one (4c) [2550-26-7]

Prepared from 4b (0.098 g, 0.5 mmol) by GP 2; reaction time: 5 h; yield: 0.052 g (70%); R_f = 0.37 (25% EtOAc-hexanes).

^1H NMR: δ = 7.33-7.29 (m, 2 H), 7.24-7.20 (m, 3 H), 2.92 (t, J = 7.6 Hz,

2 H), 2.79 (t, J = 7.6 Hz, 2 H), 2.16 (s, 3 H).

^{13}C NMR: δ = 207.9, 141.0, 128.5, 128.3, 126.1, 45.2, 30.1, 29.7.

4-(*tert*-Butyl)cyclohexanone (5c) [98-53-3]

Prepared from 5b (0.101 g, 0.5 mmol) by GP 2; reaction time: 5 h; yield: 0.053 g (68%); R_f = 0.50 (25% EtOAc-hexanes).

^1H NMR: δ = 2.40-2.26 (m, 4 H), 2.09-2.06 (m, 2 H), 1.52-1.38 (m, 3 H), 0.91 (s, 9 H).

^{13}C NMR: δ = 212.5, 46.7, 41.3, 32.4, 27.6.

Adamantan-2-one (6c) [700-58-3]

Prepared from 6b (0.099 g, 0.5 mmol) by GP 2; reaction time: 8 h; yield: 0.044 g [68% based on 0.015 g (15%) starting material recovery]; R_f = 0.47 (25% EtOAc-hexanes).

^1H NMR: δ = 2.54 (m, 2 H), 2.09-1.92 (m, 12 H).

^{13}C NMR: δ = 218.4, 46.9, 39.2, 36.3, 27.4.

Amines from Hydroperoxyacetals9-Morpholinononanol Acetate Ester (2d)

Prepared from 2b (0.124 g, 0.5 mmol) with morpholine (0.5 mmol) by GP 3; reaction time: 5 h; yield: 0.115 g (85%); R_f = 0.32 (10% MeOH- CH_2Cl_2).

IR (neat): 2927, 2853, 1738, 1118 cm^{-1} .

^1H NMR: δ = 4.05 (t, J = 6.8 Hz, 2 H), 3.72 (t, J = 4.4 Hz, 4 H), 2.44 (m, 4 H), 2.32 (t, J = 7.4 Hz, 2 H), 2.04 (s, 3 H), 1.65-1.57 (m, 2 H), 1.48 (m, 2 H), 1.35-1.29 (m, 10 H).

^{13}C NMR: δ = 171.2, 66.9, 64.6, 59.2, 53.8, 29.42, 29.38, 29.1, 28.5, 27.4, 26.5, 25.8, 20.9.

HRMS-FAB: m/z calcd for $\text{C}_{15}\text{H}_{30}\text{NO}_3$ (M + H) $^+$: 272.2226; found: 272.2213.

N-Phenyl-9-aminononanol Acetate Ester (2e)

Prepared from 2b (0.124 g, 0.5 mmol) with aniline (0.5 mmol) by GP 3; reaction time: 5 h; yield: 0.104 g (75%); R_f = 0.50 (10% EtOAc-hexanes).

IR (neat): 3401, 2926, 2854, 1734, 1236 cm^{-1} .

^1H NMR: δ = 7.22-7.18 (m, 2 H), 6.74-6.62 (m, 3 H), 4.09 (t, J = 6.76 Hz, 2 H), 3.66 (s, 1 H), 3.13 (t, J = 7.16 Hz, 2 H), 2.08 (s, 3 H), 1.68-1.63 (m, 4 H), 1.43-1.36 (m, 10 H).

^{13}C NMR: δ = 171.3, 148.6, 129.2, 117.0, 112.7, 64.6, 43.9, 29.6, 29.5, 29.4, 29.2, 28.6, 27.2, 25.9, 21.1.

HRMS-FAB: m/z calcd for $\text{C}_{17}\text{H}_{28}\text{NO}_2$ (M + H) $^+$: 278.2115; found: 278.2123.

N-Benzyl-9-aminononanol Acetate Ester (2f)

Prepared from 2b (0.124 g, 0.5 mmol) with benzylamine (2.5 mmol) by GP 3; reaction time: 5 h; yield: 0.098 g (67%); R_f = 0.42 (10% MeOH- CH_2Cl_2).

IR (neat): 2923, 2856, 1737, 1236 cm^{-1} .

^1H NMR: δ = 7.39-7.25 (m, 5 H), 4.21 (s, 1 H), 4.04 (t, J = 6.8 Hz, 2 H), 3.84 (s, 2 H), 2.65 (t, J = 7.5 Hz, 2 H), 2.04 (s, 3 H), 1.62-1.59 (m, 4 H), 1.28 (m, 10 H).

^{13}C NMR: δ = 171.2, 138.2, 128.6, 128.5, 127.4, 64.6, 53.3, 48.7, 29.4, 29.3, 29.1, 29.1, 28.6, 27.2, 25.9, 21.0.

HRMS-ESI: m/z calcd for $\text{C}_{15}\text{H}_{29}\text{NO}_2 + \text{Na}$ (M + Na) $^+$: 314.2091; found: 314.2110.

***N,N'*-(Bis-9-acetoxynonyl)benzylamine**

The tertiary amine, *N,N'*-(bis-9-acetoxynonyl)benzylamine, resulting from the reductive amination of **2f** by **2b** was also isolated in 8% yield; $R_f = 0.65$ (10% MeOH-CH₂Cl₂).

IR (neat): 2925, 2855, 1738, 1232, 1043 cm⁻¹.

¹H NMR: $\delta = 7.35$ -7.22 (m, 5 H), 4.06 (t, $J = 6.8$ Hz, 4 H), 3.56 (s, 2 H), 2.40 (t, $J = 7.2$ Hz, 4 H), 2.06 (s, 6 H), 1.66-1.59 (m, 4 H), 1.47-1.45 (m, 4 H), 1.34-1.27 (20 H).

¹³C NMR: $\delta = 171.2$, 140.5, 128.8, 128.0, 126.6, 64.6, 58.6, 53.8, 29.5, 29.4, 29.2, 28.6, 27.4, 27.0, 25.9, 21.0.

HRMS-FAB Calcd for C₂₉H₅₀NO₄ (M + H)⁺: 476.3740; found: 476.3725.

***N*-Phenethyl-9-aminononanol Acetate Ester (2g)**

Prepared from **2b** (0.124 g, 0.5 mmol) with phenethylamine (2 mmol) by GP 3; reaction time: 5 h; yield: 0.096 g (63%); $R_f = 0.60$ (10% MeOH-CH₂Cl₂).

IR (neat): 3676, 2988, 2901, 1736 cm⁻¹.

¹H NMR: $\delta = 7.32$ -7.19 (m, 5 H), 4.06 (t, $J = 6.7$ Hz, 2 H), 2.91-2.87 (m, 2 H), 2.84-2.80 (m, 2 H), 2.62 (t, $J = 7.3$ Hz, 2 H), 2.06 (s, 3 H), 1.62 (q, $J = 7.2$ Hz, 2 H), 1.47 (m, 2 H), 1.29 (m, 10 H).

¹³C NMR: $\delta = 171.3$, 140.2, 128.7, 128.4, 126.1, 64.6, 51.3, 49.9, 36.5, 30.1, 29.5, 29.4, 29.2, 28.6, 27.3, 25.9, 21.0.

HRMS-FAB: m/z calcd for C₁₉H₃₂NO₂ (M + H)⁺: 306.2428; found: 306.2439.

***4*-[4-(Benzyloxy)butyl]morpholine (3d)**

Prepared from **3b** (0.107 g, 0.47 mmol) with morpholine (0.5 mmol) by GP 3; reaction time: 5 h; yield: 0.104 g (89%); $R_f = 0.33$ (5% MeOH-CH₂Cl₂).

IR (neat): 2942, 2853, 2806, 1116 cm⁻¹.

¹H NMR: $\delta = 7.34$ -7.27 (m, 5 H), 4.50 (s, 2 H), 3.72 (t, $J = 4.7$ Hz, 4 H), 3.49 (t, $J = 6.1$ Hz, 2 H), 2.44 (m, 4 H), 2.35 (t, $J = 7.2$ Hz, 2 H), 1.66-1.56 (m, 4 H).

¹³C NMR: $\delta = 138.6$, 128.4, 127.6, 127.5, 72.9, 70.1, 66.9, 58.8, 53.7, 27.7, 23.2.

HRMS-FAB: m/z calcd for C₁₅H₂₄NO₂ (M + H)⁺: 250.1802; found: 250.1812.

***N*-[4-(Benzyloxy)butyl]aniline (3e)**

Prepared from **3b** (0.107 g, 0.47 mmol) with aniline (0.5 mmol) by GP 3; reaction time: 4 h; yield: 0.083 g (69%); $R_f = 0.51$ (20% EtOAc-hexanes).

IR (neat): 3403, 3026, 2935, 2858, 1601, 1505 cm⁻¹.

¹H NMR: $\delta = 7.38$ -7.30 (m, 5 H), 7.21-7.17 (m, 2 H), 6.71 (t, $J = 7.3$ Hz, 1 H), 6.61-6.59 (m, 2 H), 4.54 (s, 2 H), 3.73 (s, 1 H), 3.55 (t, $J = 5.9$ Hz, 2 H), 3.18-3.15 (m, 2 H), 1.79-1.57 (m, 4 H).

¹³C NMR: $\delta = 148.6$, 138.6, 129.3, 128.5, 127.8, 127.7, 117.2, 112.8, 73.1, 70.2, 43.8, 27.5, 26.5.

HRMS-FAB: m/z calcd for C₁₇H₂₂NO (M + H)⁺: 256.1696; found: 256.1692.

***N*-[4-(Benzyloxy)butyl]benzylamine (3f) [60058-23-3]**

Prepared from **3b** (0.107 g, 0.47 mmol) with benzylamine (2.5 mmol) by GP 3; reaction time: 5 h; yield: 0.079 g (63%); $R_f = 0.51$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.39$ -7.24 (m, 10 H), 4.52 (s, 2 H), 3.80 (s, 2 H), 3.50 (t, $J = 6.2$ Hz, 2 H), 2.67 (t, $J = 6.9$ Hz, 2 H), 1.73-1.59 (m, 4 H), 1.55 (s, 1 H).

¹³C NMR: $\delta = 140.5$, 138.6, 128.4, 128.3, 128.1, 127.6, 127.5, 126.9, 72.9, 70.3, 54.0, 49.2, 27.6, 26.8.

***N*-[4-(Benzyloxy)butyl]-2-phenethylamine (3g)**

Prepared from **3b** (0.107 g, 0.47 mmol) with phenethylamine (2.5 mmol) by GP 3; reaction time: 4 h; yield: 0.087 g (65%); $R_f = 0.22$ (5% MeOH-CH₂Cl₂).

IR (neat): 3026, 2932, 2854, 1097, 696 cm⁻¹.

¹H NMR: $\delta = 7.37$ -7.22 (m, 10 H), 4.52 (s, 2 H), 3.50 (t, $J = 6.2$ Hz, 2 H), 2.92-2.88 (m, 2 H), 2.84-2.80 (m, 2 H), 2.68-2.65 (t, $J = 7.1$ Hz, 2 H), 1.69-1.55 (m, 4 H), 1.37 (s, 1 H).

¹³C NMR: $\delta = 140.1$, 138.6, 128.7, 128.5, 128.4, 127.6, 127.5, 126.1, 72.9, 70.3, 51.2, 49.7, 36.5, 27.6, 26.9.

HRMS-FAB: m/z calcd for C₁₉H₂₆NO (M + H)⁺: 284.2009; found: 284.2018.

***4*-[4-(Phenylbutan-2-yl)morpholine (4d) [2832-95-3]**

Prepared from **4b** (0.098 g, 0.5 mmol) with morpholine (0.5 mmol) by GP 3; reaction time: 24 h; yield: 0.049 g (45%); $R_f = 0.37$ (5% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.32$ -7.18 (m, 5 H), 3.75-3.72 (m, 4 H), 2.75-2.62 (m, 2 H), 2.60-2.52-2.44 (m, 5 H), 1.90-1.83 (m, 1 H), 1.66 (m, 1 H), 1.64-1.54 (m, 1 H), 1.03 (d, $J = 6.7$ Hz, 2 H).

¹³C NMR: $\delta = 142.6$, 128.4, 128.3, 125.7, 67.5, 58.4, 48.7, 35.3, 32.8, 13.9.

***N*-[4-(Phenylbutan-2-yl)aniline (4e) [72641-00-0]**

Prepared from **4b** (0.098 g, 0.5 mmol) with aniline (0.5 mmol) by GP 3; reaction time: 36 h; yield: 0.068 g (60%); $R_f = 0.51$ (10% EtOAc-hexanes).

¹H NMR: $\delta = 7.33$ -7.28 (m, 2 H), 7.23-7.15 (m, 5 H), 6.69 (t, $J = 7.3$ Hz, 1 H), 6.56 (d, $J = 8.2$ Hz, 2 H), 3.52 (m, 1 H), 3.45 (s, 1 H), 2.75 (t, $J = 7.8$ Hz, 2 H), 1.95-1.86 (m, 1 H), 1.84-1.75 (m, 1 H), 1.24 (d, $J = 6.3$ Hz, 3 H).

¹³C NMR: $\delta = 147.6$, 142.0, 129.3, 128.5, 128.4, 125.9, 116.9, 113.2, 47.9, 38.8, 32.5, 20.9.

***N*-Benzyl-4-phenylbutan-2-amine (4f) [68164-04-5]**

Prepared from **4b** (0.098 g, 0.5 mmol) with benzylamine (0.5 mmol) by GP 3; reaction time: 36 h; yield: 0.089 g (75%); $R_f = 0.25$ (5% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.36$ -7.20 (m, 10 H), 3.87 (d, $J = 13.0$ Hz, 1 H), 3.77 (d, $J = 13.0$ Hz, 1 H), 2.82-2.64 (m, 3 H), 1.90-1.81 (m, 1 H), 1.76-1.66 (m, 1 H), 1.41 (s, 1 H), 1.19 (d, $J = 6.4$ Hz, 3 H).

¹³C NMR: $\delta = 142.5$, 140.7, 128.44, 128.39, 128.2, 127.9, 126.9, 125.7, 52.0, 51.3, 38.7, 32.3, 20.4.

***N*-Phenethyl-4-phenylbutan-2-amine (4g) [161012-69-7]**

Prepared from **4b** (0.138 g, 0.7 mmol) with phenethylamine by GP 3; reaction time: 30 h; yield: 0.092 g (52%); $R_f = 0.30$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.36$ -7.15 (m, 10 H), 2.99-2.91 (m, 1 H), 2.88-2.79 (m, 3 H), 2.72-2.66 (m, 1 H), 2.64-2.57 (m, 2 H), 1.84-1.75 (m, 1 H), 1.68-1.59 (m, 1 H), 1.30 (s, 1 H), 1.13 (d, $J = 6.3$ Hz, 3 H).

¹³C NMR: $\delta = 142.3$, 140.1, 128.7, 128.5, 128.4, 128.3, 126.2, 125.7, 55.4, 48.5, 38.7, 36.6, 32.3, 20.3.

Amines from Alkenes 1-Benzylazapine (7) [20422-13-3]

Prepared by reacting cyclohexene (0.082 g, 1 mmol) with benzylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.107 g (57%); $R_f = 0.57$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.40$ -7.27 (m, 5 H), 3.69 (s, 2 H), 2.67-2.66 (m, 4 H), 1.67 (m, 8 H).

¹³C NMR: $\delta = 140.1$, 128.8, 128.1, 126.7, 62.7, 55.6, 28.2, 27.0.

1-Phenethylazapine (8) [65530-43-0]

Prepared by reacting cyclohexene (0.082 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.127 g (63%); $R_f = 0.57$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.32$ -7.21 (m, 5 H), 2.83-2.80 (m, 8 H), 1.72 (m, 4 H), 1.64 (m, 4 H).

¹³C NMR: $\delta = 140.2, 128.8, 128.4, 126.0, 59.9, 55.1, 33.6, 27.2, 27.1$.

4-[4-(Benzyloxy)butyl]morpholine (3d)

Prepared from 3a (0.176 g, 1.0 mmol) with morpholine (1 mmol) by GP 4A; reaction time: 2 h; yield: 0.159 g (64%). Spectral data were identical with those of compound 3d described above.

1-Benzylpiperidine (9) [2905-56-8]

Prepared by reacting cyclopentene (0.068 g, 1 mmol) with benzylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.113 g (65%); $R_f = 0.50$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.35$ -7.24 (m, 5 H), 3.50 (s, 2 H), 2.40-2.39 (m, 4 H), 1.64-1.57 (m, 4 H), 1.49-1.46 (m, 2 H).

¹³C NMR: $\delta = 138.7, 129.2, 128.0, 126.8, 63.9, 54.5, 26.0, 24.4$.

1-Phenethylpiperidine (10) [332-14-9]

Prepared by reacting cyclopentene (0.068 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.136 g (72%); $R_f = 0.55$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.32$ -7.20 (m, 5 H), 2.86-2.82 (m, 2 H), 2.60-2.50 (m, 2 H), 2.40 (m, 4 H), 1.68-1.62 (m, 4 H), 1.51-1.48 (m, 2 H).

¹³C NMR: $\delta = 140.7, 128.7, 128.3, 125.9, 61.5, 54.6, 33.7, 26.1, 24.5$.

Compound 10 was also prepared by reacting cyclopentene (0.068 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4B; yield: 0.117 g (62%).

9-(N-Morpholino)nonyl Acetate Ester (2d)

Prepared from 2a (0.099 g, 0.5 mmol) with morpholine (1 mmol) by GP 4B; reaction time: 6 h. The product was identical to 2d obtained as above; yield: 0.089 g (66%).

N-Benzylcyclohexylmethanamine (11a) [4352-47-0] and N-benzyl-N-methylcyclohexylmethanamine (11b) [79952-95-7]

Prepared from the reaction of vinylcyclohexane (0.11 g, 1 mmol) with benzylamine (2 mmol) by GP 4B; reaction time: 6 h; yield: 0.102 g (50%); $R_f = 0.50$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.36$ -7.26 (m, 5 H), 3.80 (s, 2 H), 2.49 (d, $J = 6.7$ Hz, 2 H), 1.84-1.68 (m, 5 H), 1.57-1.46 (m, 1 H), 1.32-1.13 (m, 4 H), 0.99-0.90 (m, 2 H).

¹³C NMR: $\delta = 140.6, 128.4, 128.0, 126.8, 56.2, 54.1, 37.9, 31.5, 26.7, 26.1$.

The reaction also yielded 10% of 11b.

11b

$R_f = 0.63$ (10% MeOH-CH₂Cl₂).

¹H NMR (600 MHz): $\delta = 7.35$ -7.24 (m, 5 H), 3.47 (s, 2 H), 2.18-2.16 (m, 5 H), 1.84 (d, $J = 12.4$ Hz, 2 H), 1.74-1.67 (m, 3 H), 1.57-1.51 (m, 1 H), 1.28-1.13 (m, 3 H), 0.89-0.82 (m, 2 H).

¹³C NMR (150 MHz): $\delta = 139.8, 128.9, 128.0, 126.7, 64.7, 62.7, 42.9, 35.8, 31.8, 26.9, 26.2$.

3-Phenethyl-3-azabicyclo[3.2.1]octane (12)

Prepared by reacting norbornene (0.094 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.139 g (65%); $R_f = 0.63$ (10% MeOH-CH₂Cl₂).

IR (neat): 2931, 2757, 1496, 1406 cm⁻¹.

¹H NMR: $\delta = 7.37$ -7.24 (m, 5 H), 2.84-2.80 (m, 4 H), 2.64-2.60 (m, 2 H), 2.20-2.14 (m, 4 H), 1.76-1.63 (m, 4 H), 1.56-1.53 (m, 1 H), 1.44-1.42 (m, 1 H).

¹³C NMR: $\delta = 140.9, 128.8, 128.2, 125.8, 60.2, 60.1, 37.7, 35.2, 33.3, 28.6$.

HRMS-FAB: m/z calcd for C₁₅H₂₂N (M + H)⁺: 216.1747; found: 216.1748.

This product was also prepared by reacting norbornene (0.041 g, 0.43 mmol) with phenethylamine (0.43 mmol) according to GP 4B; yield: 0.062 g (65%).

1-Phenyl-2-(4-phenylbutan-2-yl)diazene (13) [343222-12-8]

A solution of alkene 4a (0.176 g, 1 mmol) and MeOH (0.3 mL) in CH₂Cl₂ (10 mL) in a round-bottomed flask was cooled to -40 °C and a stream of 2% O₃/O₂ (approximately 1 mmol O₃/min) was bubbled through the reaction solution. Once the pale blue color of ozone was observed, the ozonizer voltage was set to zero and the reaction was sparged for 1 min with N₂. The resulting solution was removed from dry ice/acetone bath, whereupon NaBH(OAc)₃ (2 mmol) and phenylhydrazine (1 mmol) were sequentially added. The reaction was stirred for 2 h at r.t. and then treated with NaCNBH₃ (1 mmol). After stirring for another 30 min, the reaction was filtered through neutral alumina with CH₂Cl₂, and the residue obtained upon concentration was purified by chromatography (alumina) using 10% EtOAc-hexanes to furnish a light yellow oil, which darkened very rapidly. Air was passed over the oil for 2 h to furnish diazene 13 as a yellow oil; yield: 0.135 g (57%); $R_f = 0.50$ (10% EtOAc-hexanes).

¹H NMR: $\delta = 7.73$ -7.70 (m, 2 H), 7.53-7.46 (m, 3 H), 7.33-7.28 (m, 2 H), 7.24-7.20 (m, 3 H), 3.90-3.81 (m, 1 H), 2.66 (t, $J = 8.1$ Hz, 2 H), 2.41-2.32 (m, 1 H), 2.13-2.04 (m, 1 H), 1.42 (d, $J = 6.6$ Hz, 3 H).

¹³C NMR: $\delta = 152.1, 141.9, 130.3, 129.0, 128.5, 128.4, 125.8, 122.2, 72.7, 36.9, 32.6, 19.0$.

Supporting Information for this article follows the References.

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Supporting Information: A Mild One-pot Conversion of Alkenes to Amines through Tandem Ozonolysis and Reductive Amination.

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Table of Contents

Preparation of hydroperoxyacetals

1b) 1-Methoxynonyl hydroperoxide	2
2b) 9-Hydroperoxy-9-methoxynonanol, acetate ester	3
3b) 4-Benzyloxy-1-methoxybutyl hydroperoxide.....	4
4b) (3-Hydroperoxy-3-methoxybutyl)benzene	5
5b) 4-(<i>tert</i> -Butyl)-1-Hydroperoxy-1-methoxycyclohexane	6
6b) 2-Hydroperoxy-2-methoxyadamantane	7

Synthesis of carbonyls from hydroperoxy acetals:

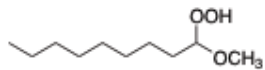
1c) Nonanal	8
2c) 9-Hydroxynonanal, acetate ester	9
3c) 4-Benzyloxybutanal	10
4c) 4-Phenylbutan-2-one	11
5c) 4- <i>tert</i> -Butylcyclohexanone	12
6c) Adamantan-2-one	13

Synthesis of amines from hydroperoxy acetals:

2d) 9-Morpholinononanol, acetate ester	14
2e) <i>N</i> -Phenyl-9-aminononanol, acetate ester.....	15
2f) <i>N</i> -Benzyl-9-aminononanol, acetate ester	16
<i>N,N'</i> -bis(9-acetoxynonyl)benzylamine	17
2g) <i>N</i> -Phenethyl-9-aminononanol, acetate ester	18
3d) 4-(4-(Benzyloxy)butyl)morpholine	19
3e) <i>N</i> -(4-(Benzyloxy)butyl)aniline	20
3f) <i>N</i> -(4-(Benzyloxybutyl)benzylamine	21
3g) <i>N</i> -(4-(Benzyloxybutyl)phenethylamine.....	22
4d) 4-(4-Phenylbutan-2-yl)morpholine	23
4e) <i>N</i> -(4-Phenylbutan-2-yl)aniline	24
4f) <i>N</i> -Benzyl-4-phenylbutan-2-amine	25
4g) <i>N</i> -Phenethyl-4-phenylbutan-2-amine	26

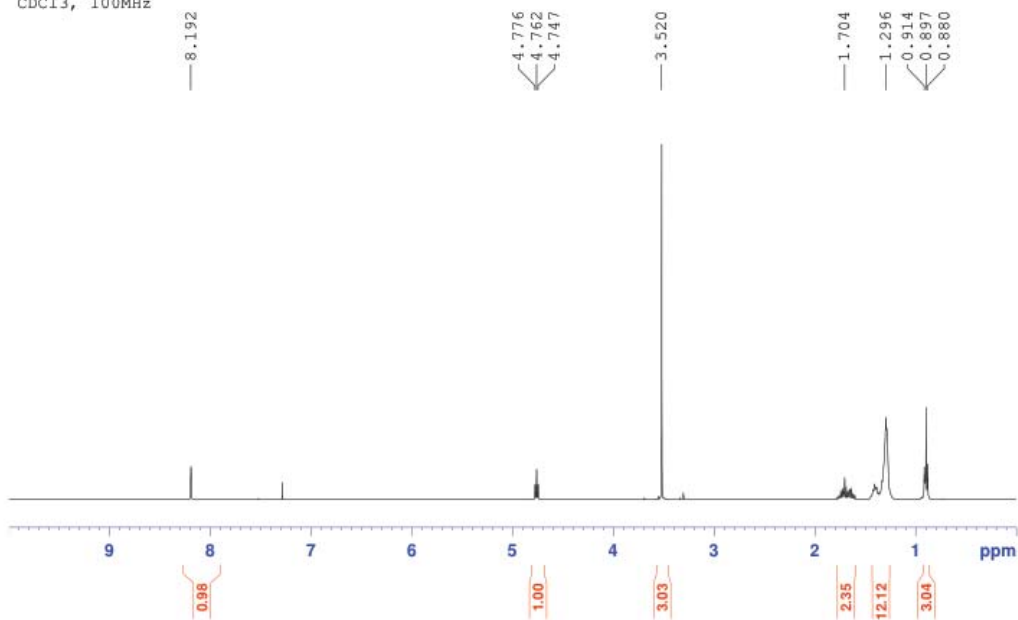
Synthesis of amines from alkenes:

7) 1-Benzylazapine	27
8) 1-Phenethylazapine	28
9) 1-Benzylpiperidine	29
10) 1-Phenethylpiperidine	30
11a) <i>N</i> -Benzyl-cyclohexylmethanamine.....	31
11b) <i>N</i> -Benzyl- <i>N</i> -methyl-cyclohexylmethanamine	32
12) 3-Phenethyl 3-azabicyclo (3.2.1) octane	33
13) 1-Phenyl-2-(4-phenylbutan-2-yl)diazene	34

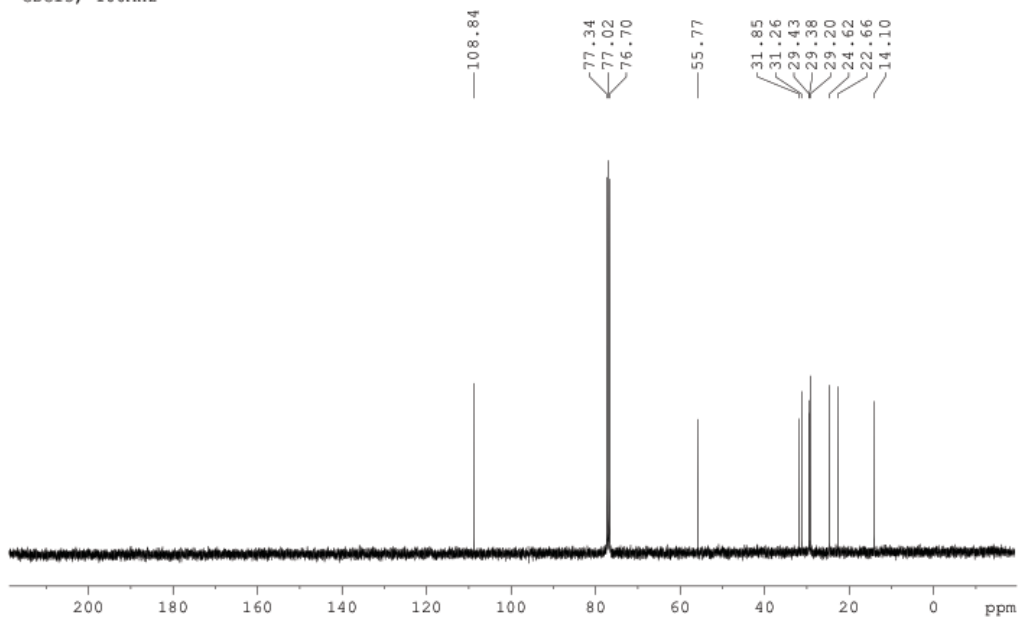


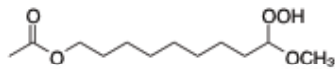
1b

1D Proton NMR
TFish0460
CDCl₃, 100MHz



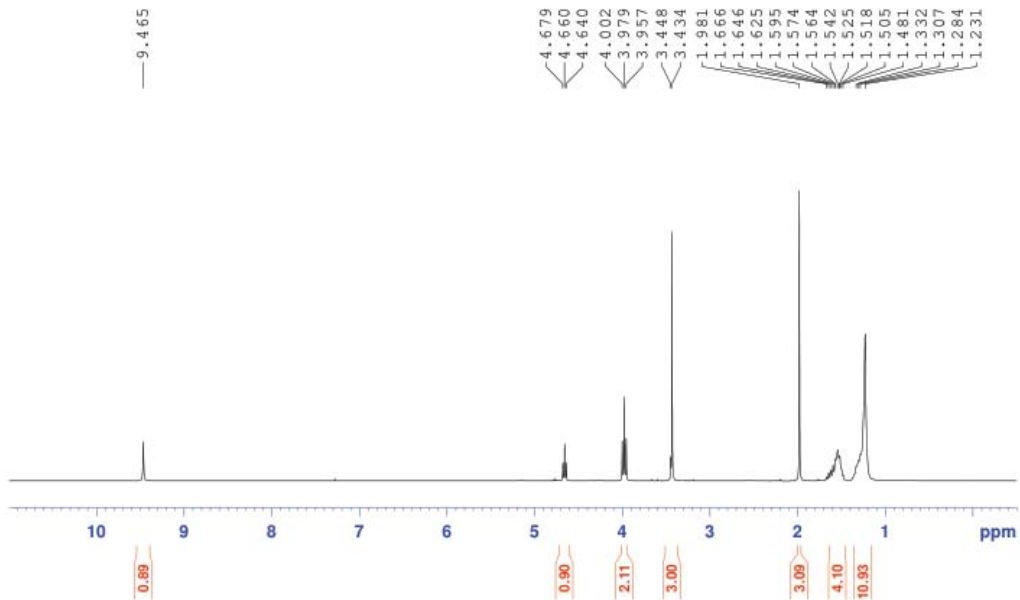
13C
TFish0460
CDCl₃, 100MHz



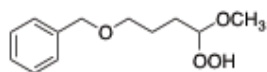
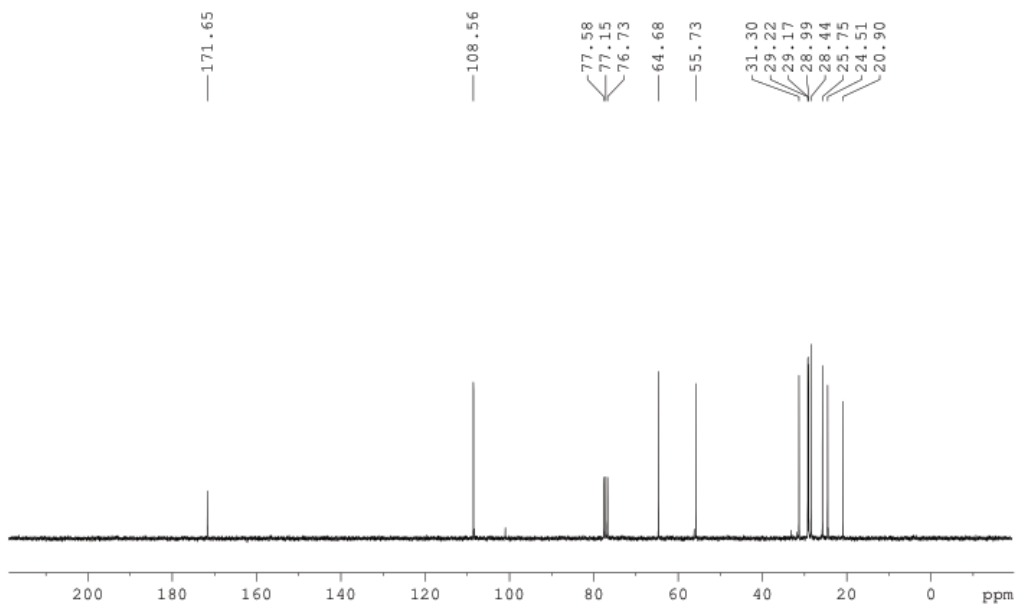


2b

1D Proton
ksk01-08

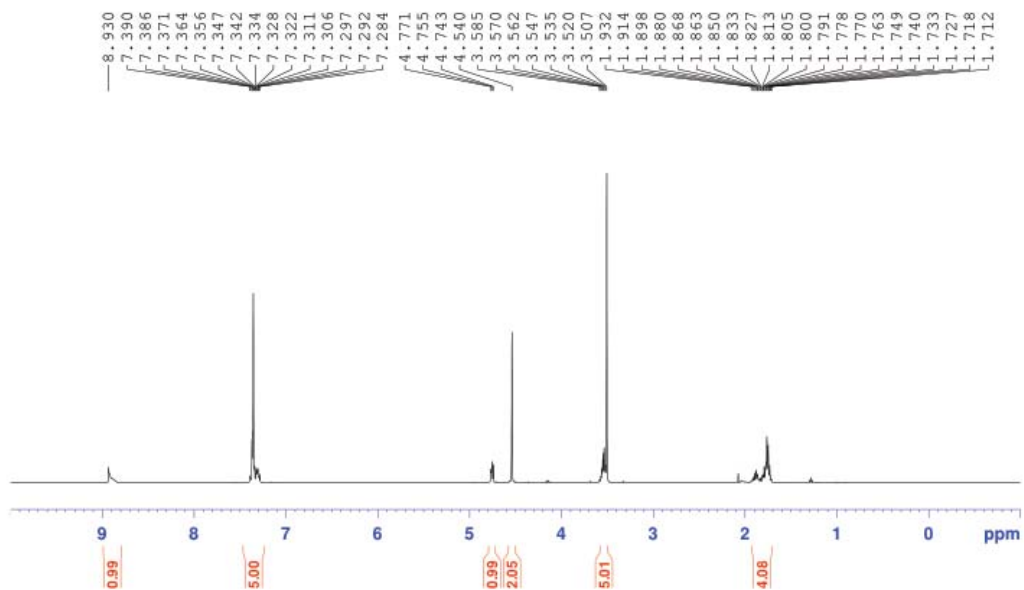


13C
ksk01-08

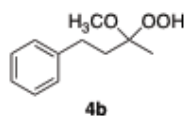
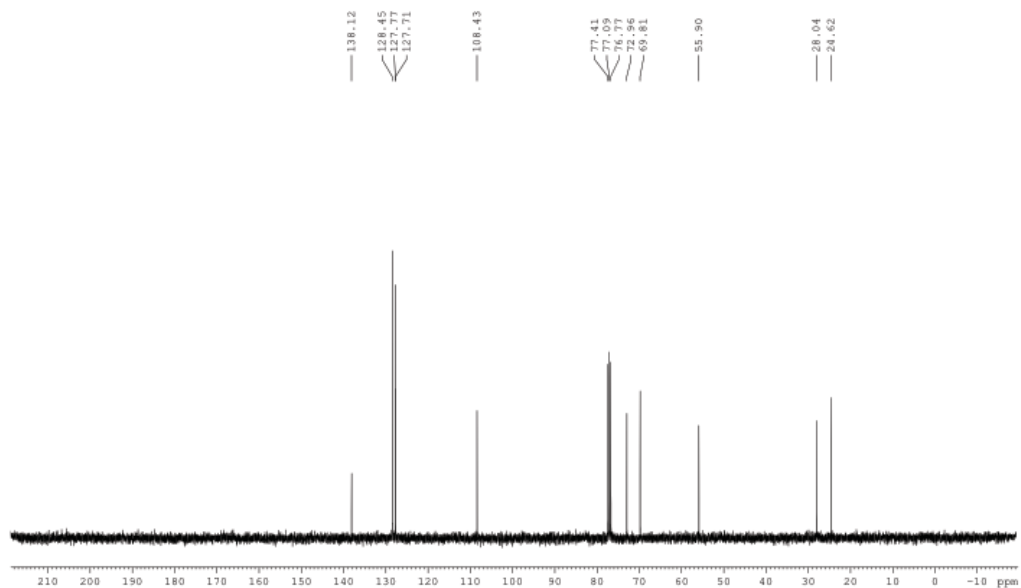


3b

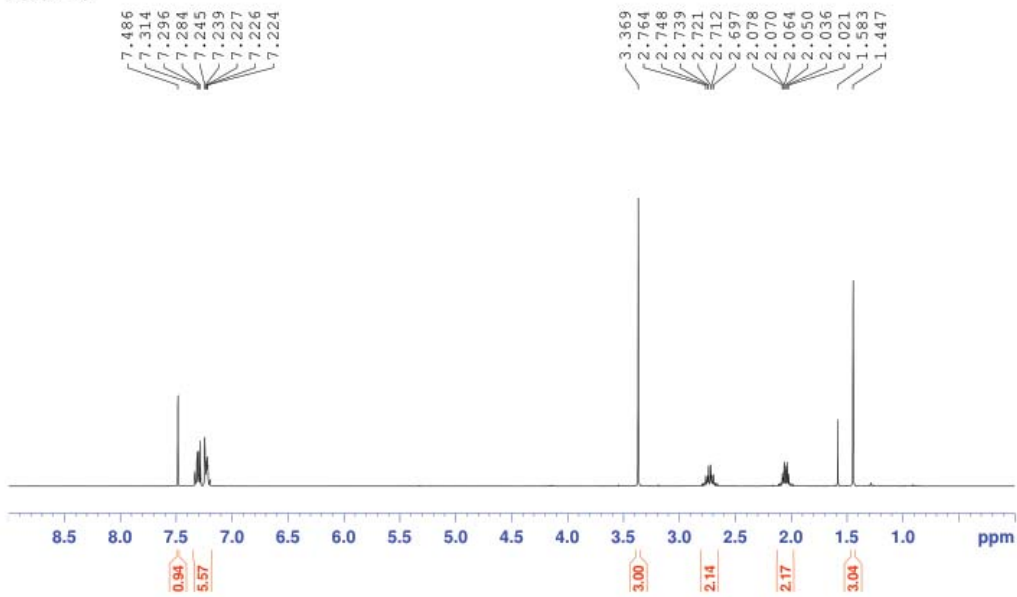
1D Proton NMR
ksk01-94
400 MHz, CDCl₃



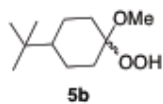
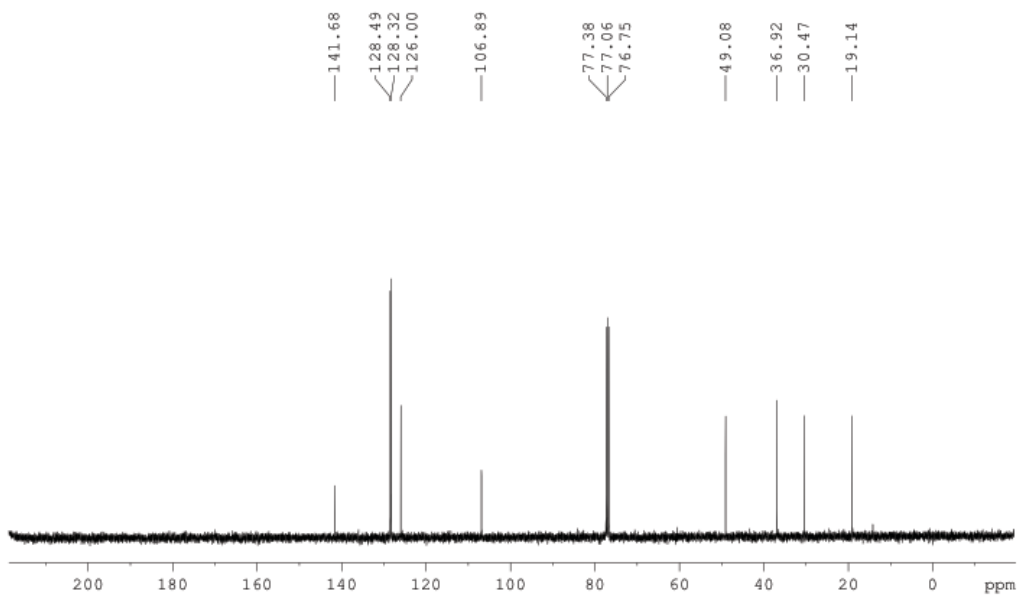
¹³C
ksk01-94
CDCl₃, 400MHz



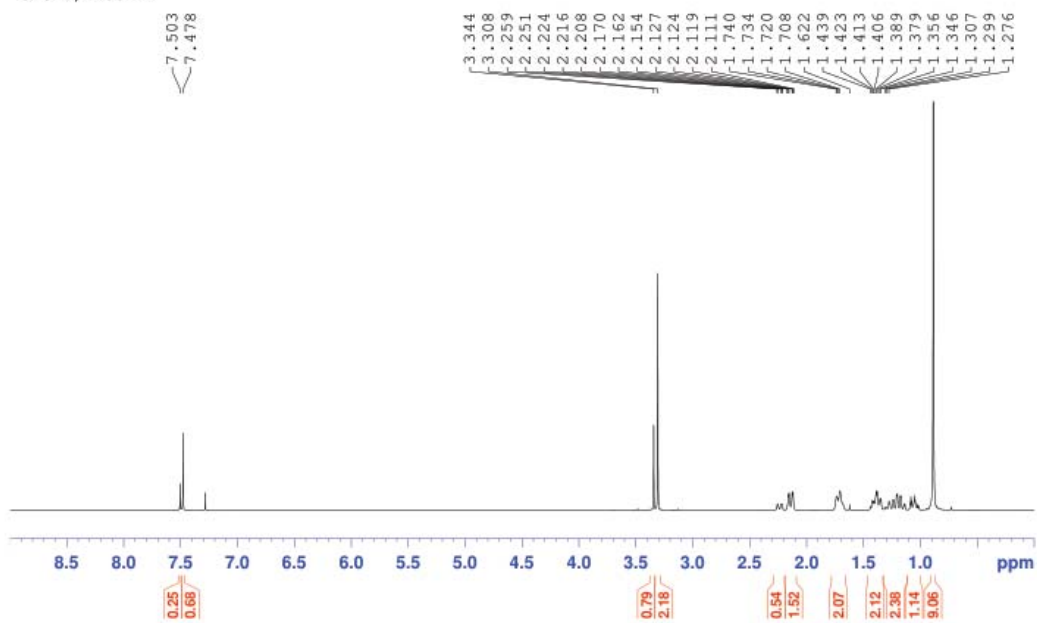
1D Proton NMR
ksk01-79



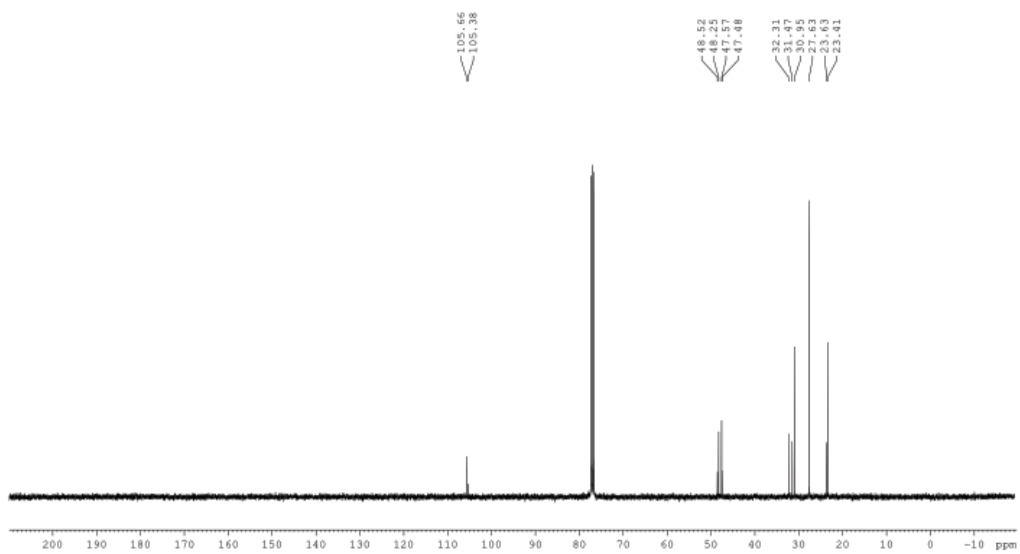
13C
ksk01-79

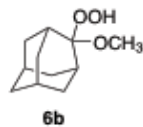


1D Proton NMR
TFish0580
CDCl3, 400MHz

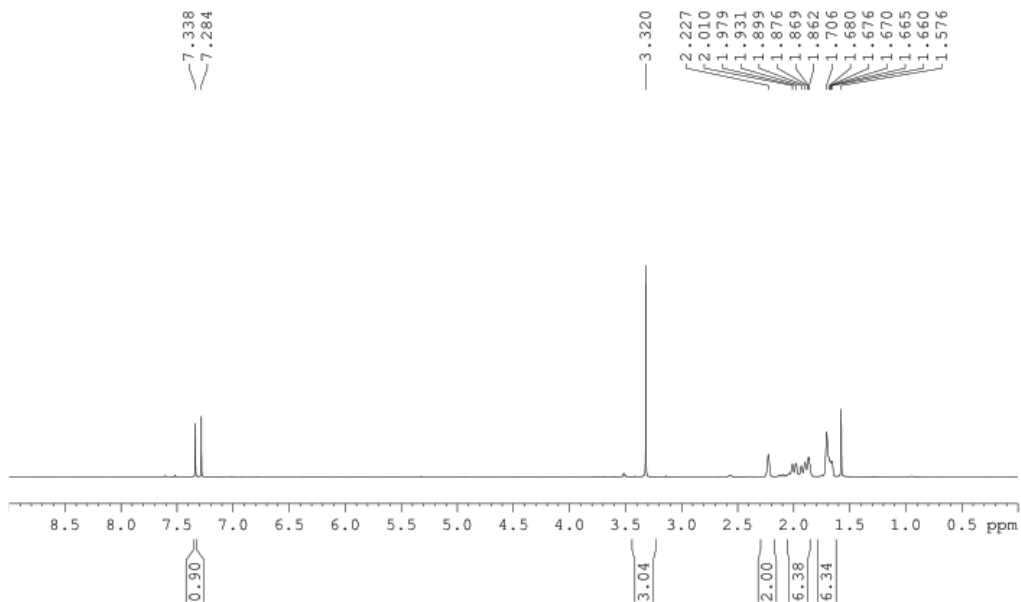


13C
TFish 0580
CDCl3, 400MHz

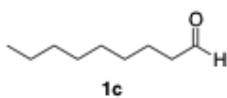
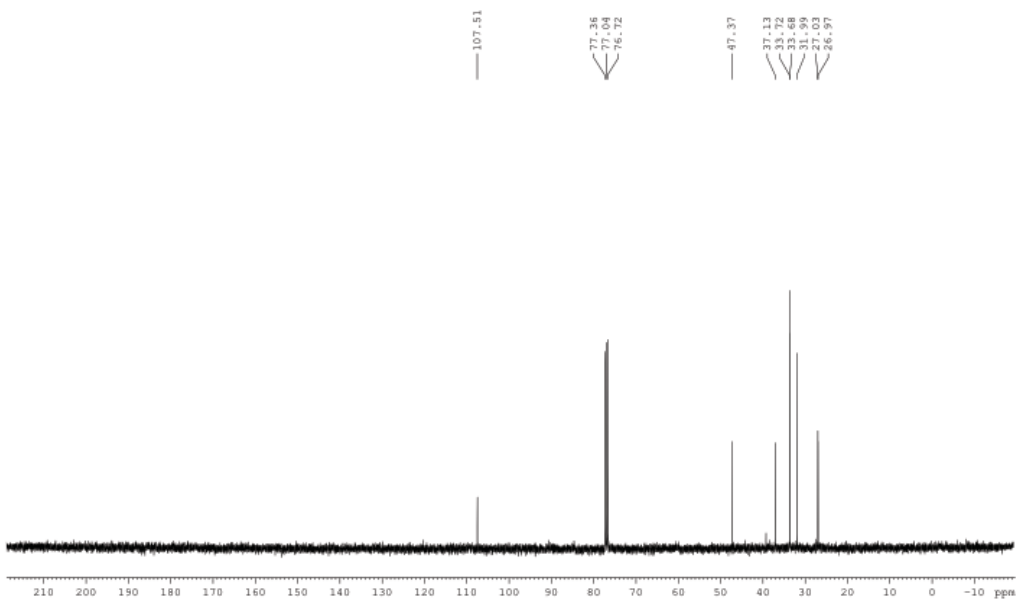




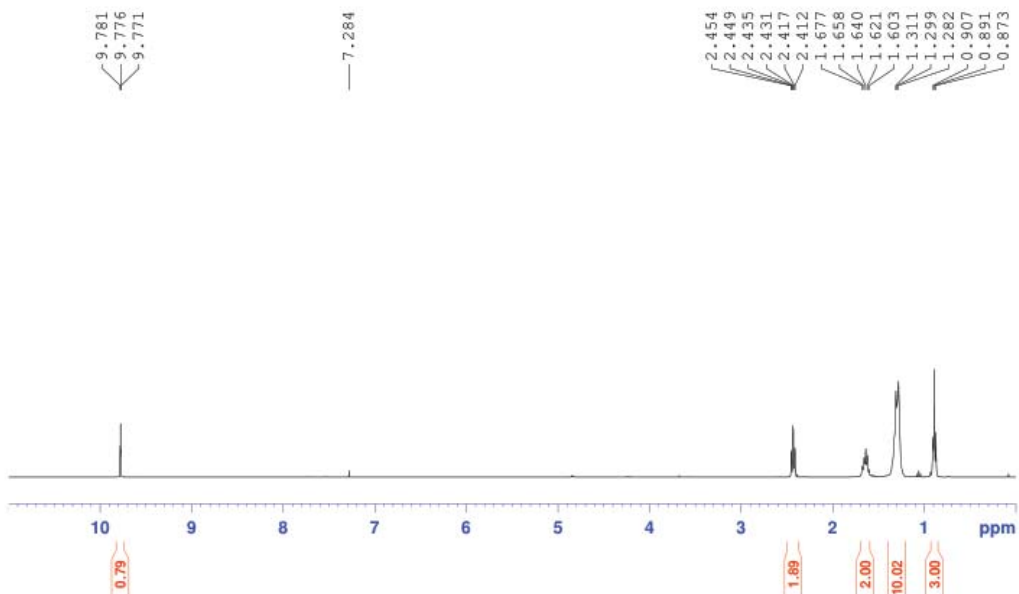
1D Proton NMR
KSK01-77



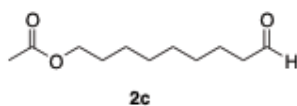
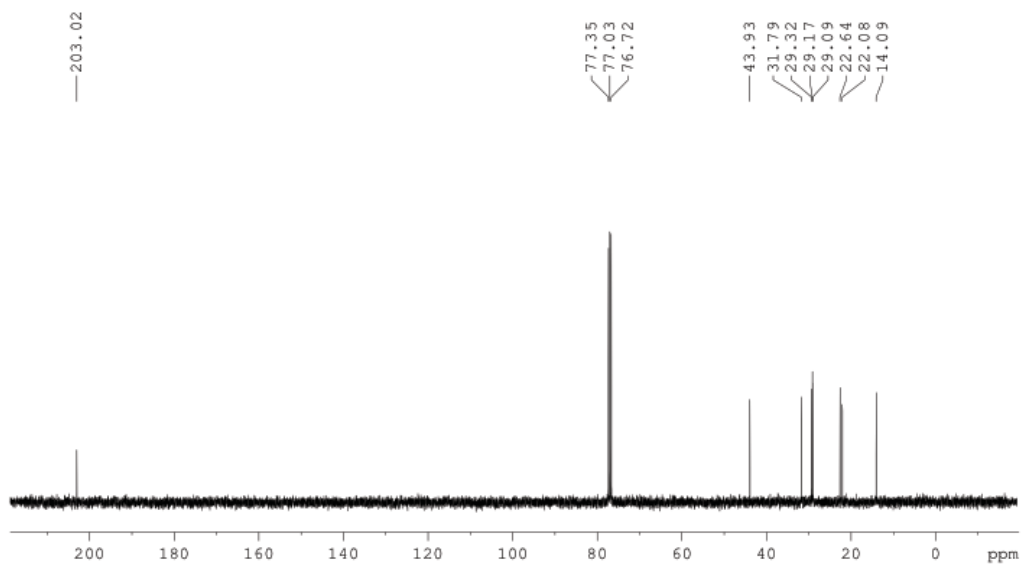
¹³C
KSK01-77



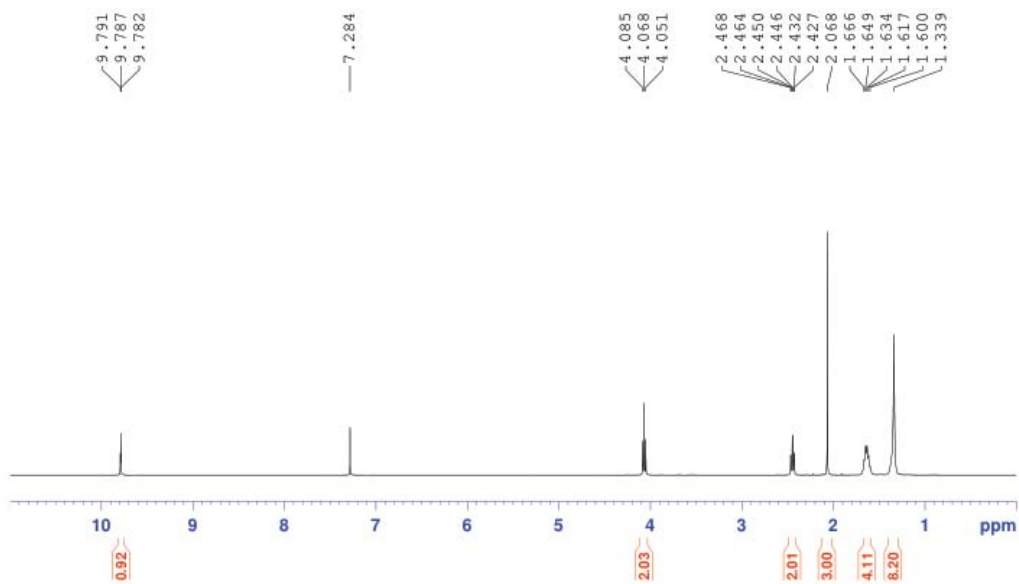
1D Proton NMR
ksk01-66
CDCl₃, 400 MHz



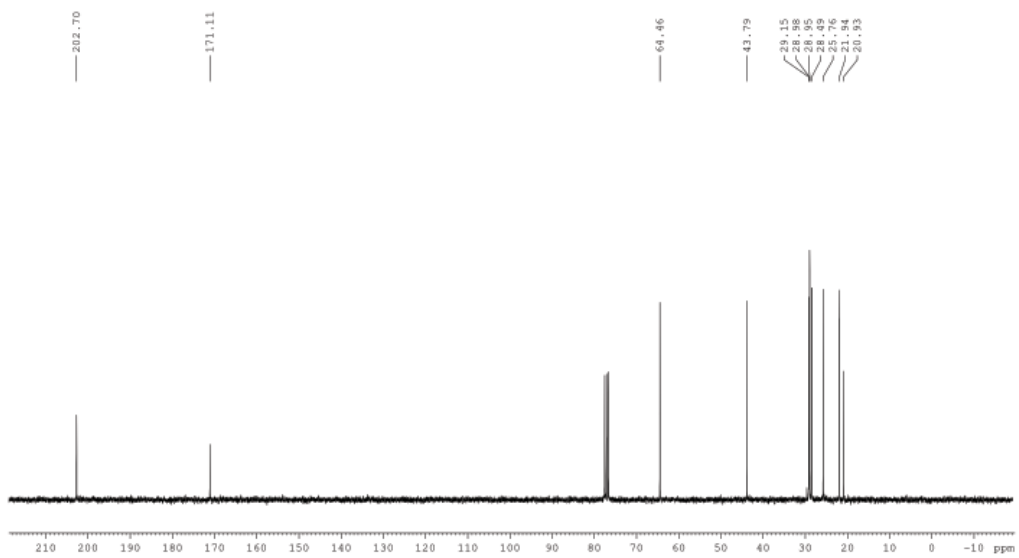
13C
ksk01-66
CDCl₃, 400 MHz

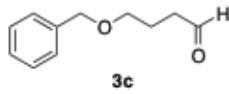


1D Proton NMR
ksk01-20
CDCl₃, 400 MHz

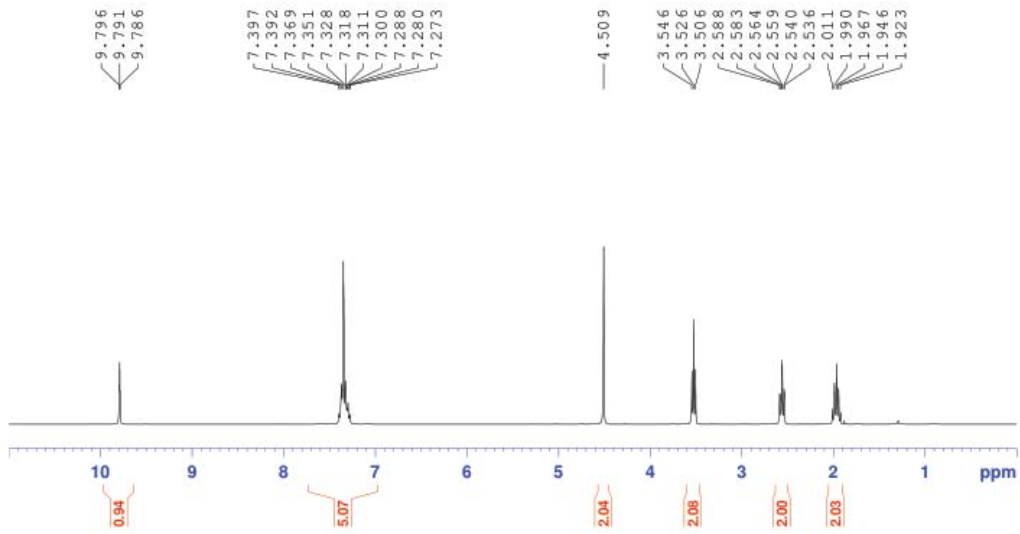


¹³C
ksk01-20

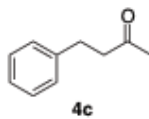
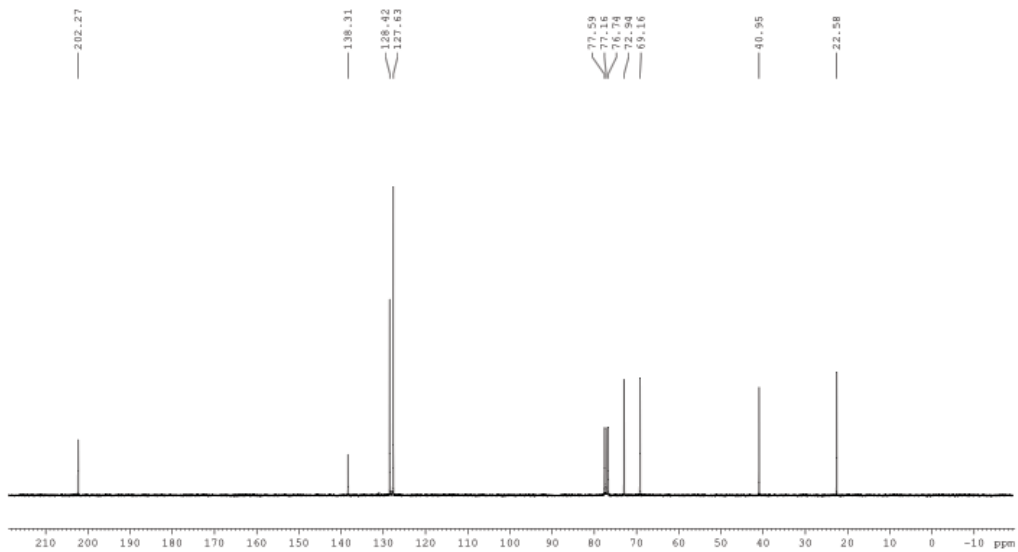




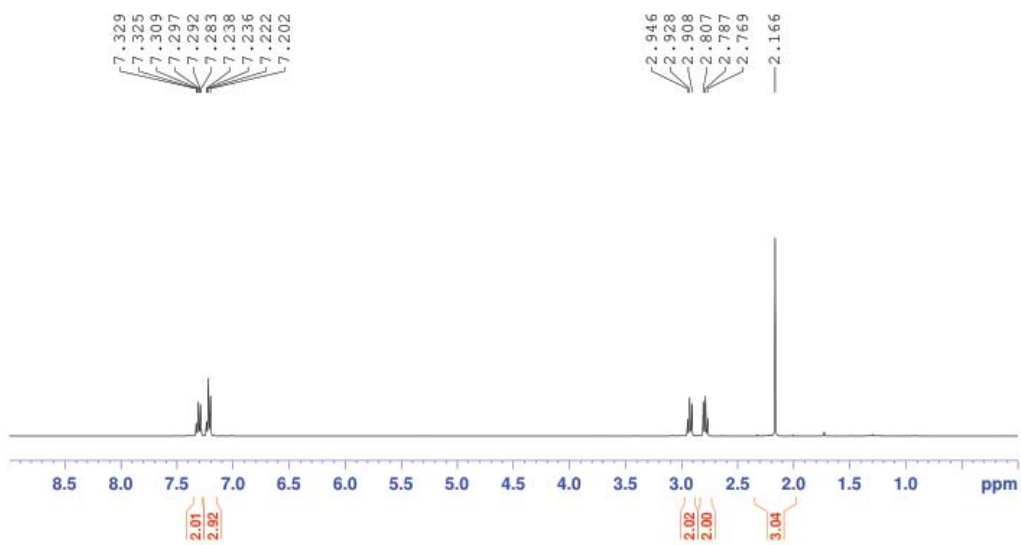
CDC13, 400 MHz
 ID Proton
 ksk01-68



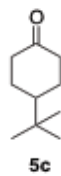
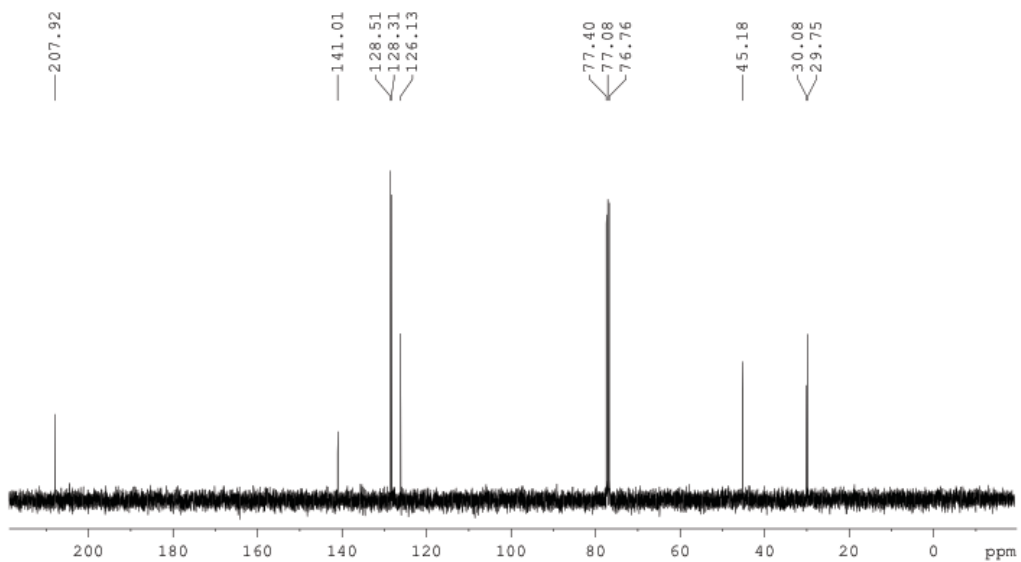
¹³C
 ksk01-68



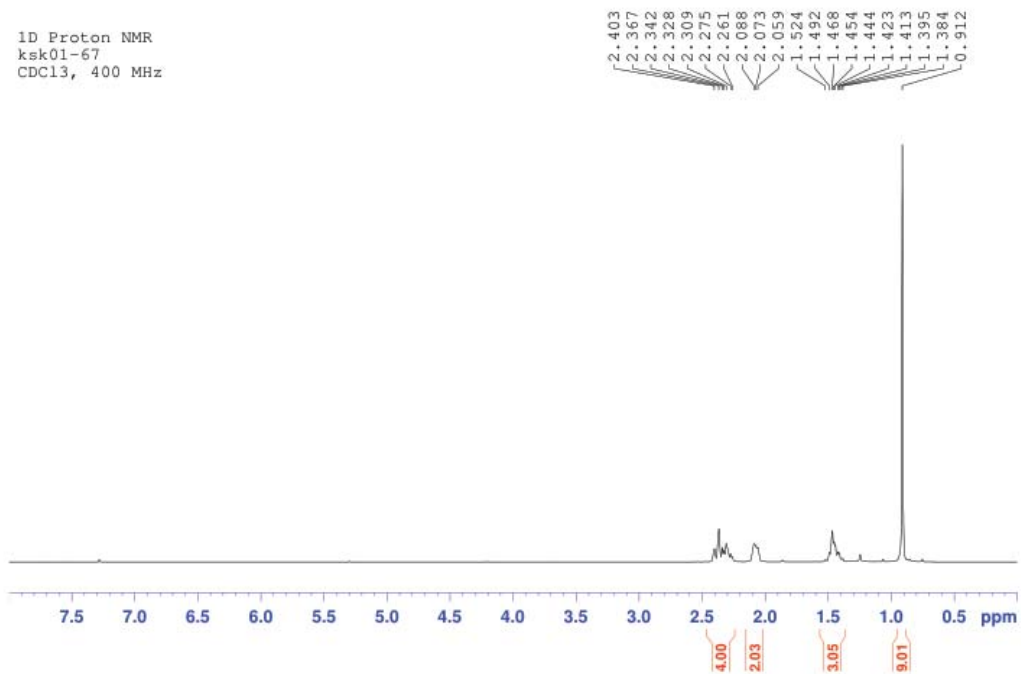
1D Proton NMR
ksk01-80
CDCl₃, 400 MHz



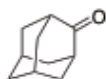
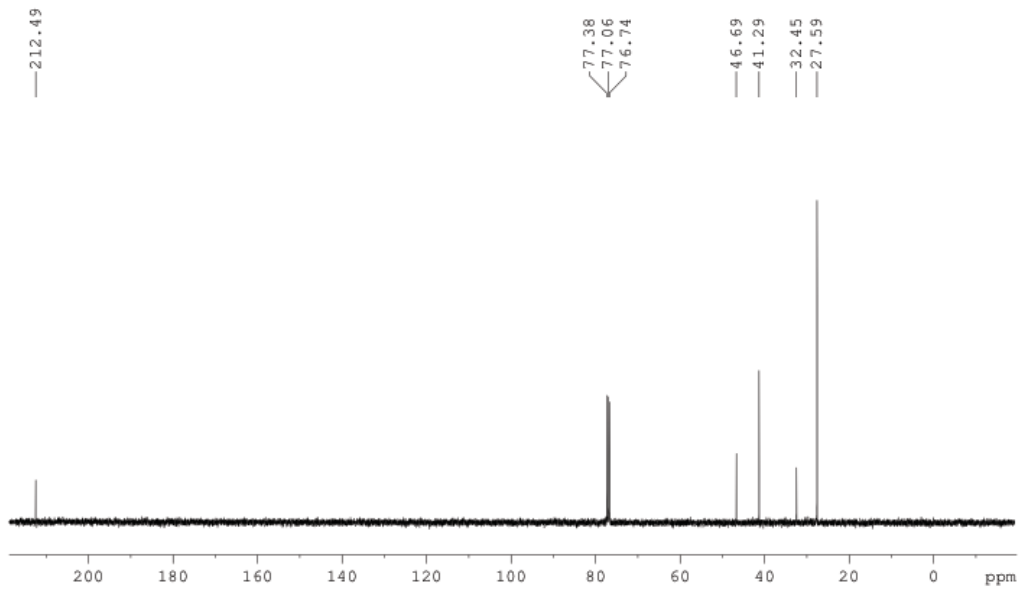
¹³C
ksk01-80



1D Proton NMR
ksk01-67
CDC13, 400 MHz

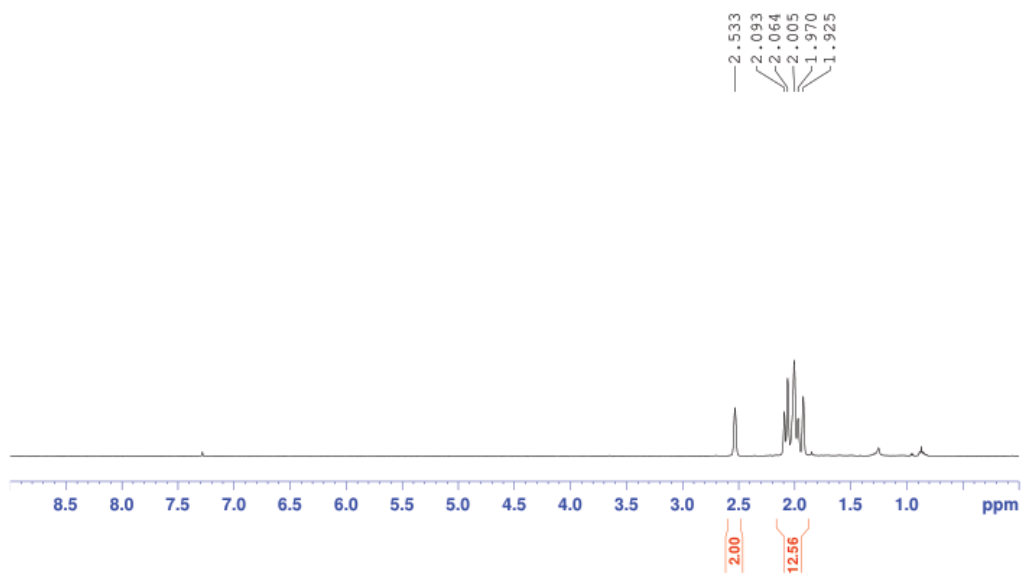


¹³C
ksk01-67

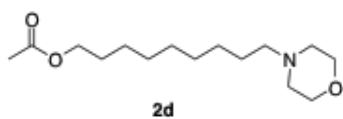
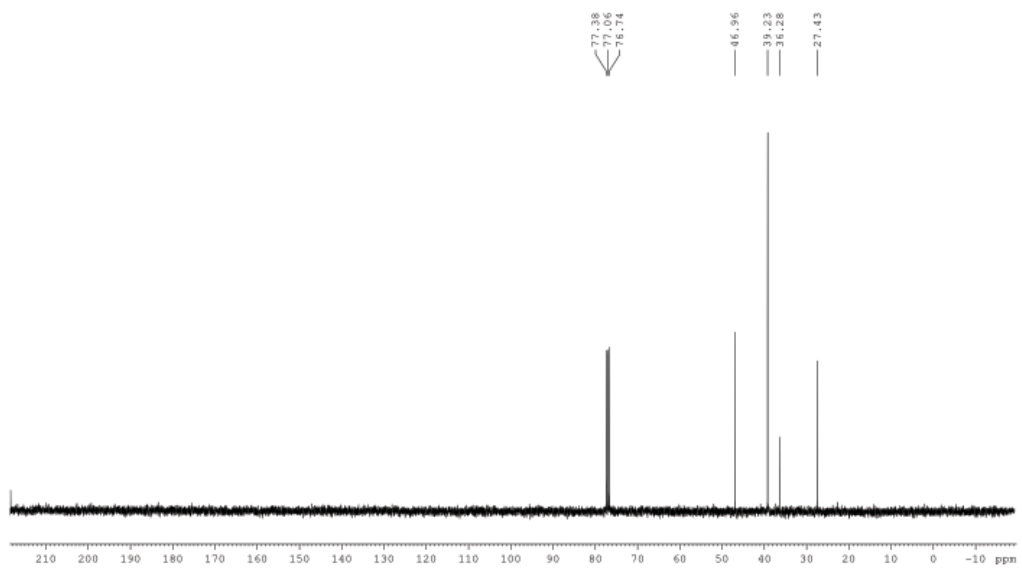


6c

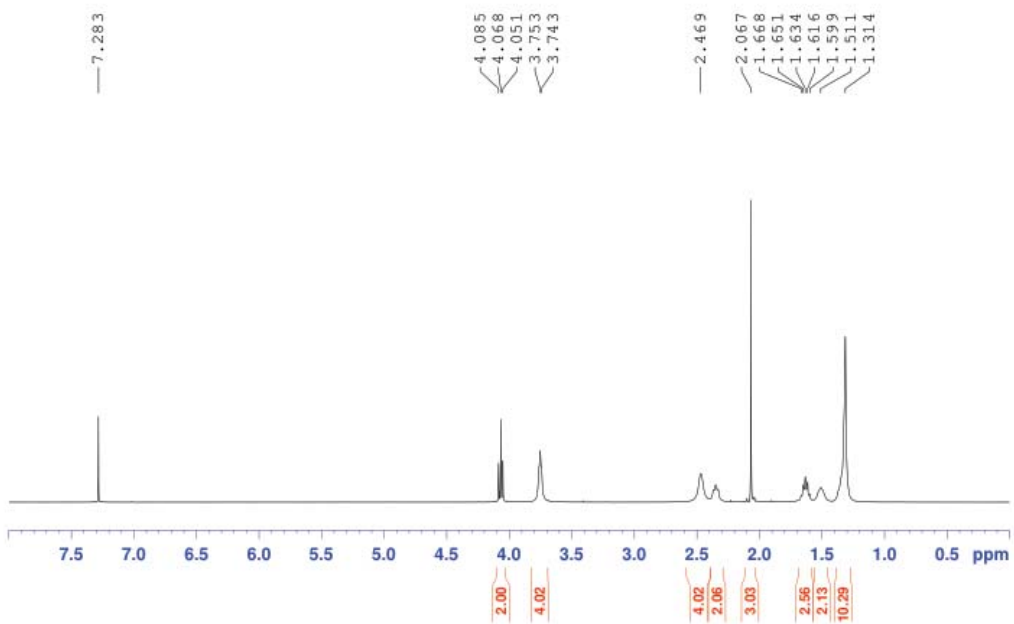
1D Proton NMR
ksk01-78



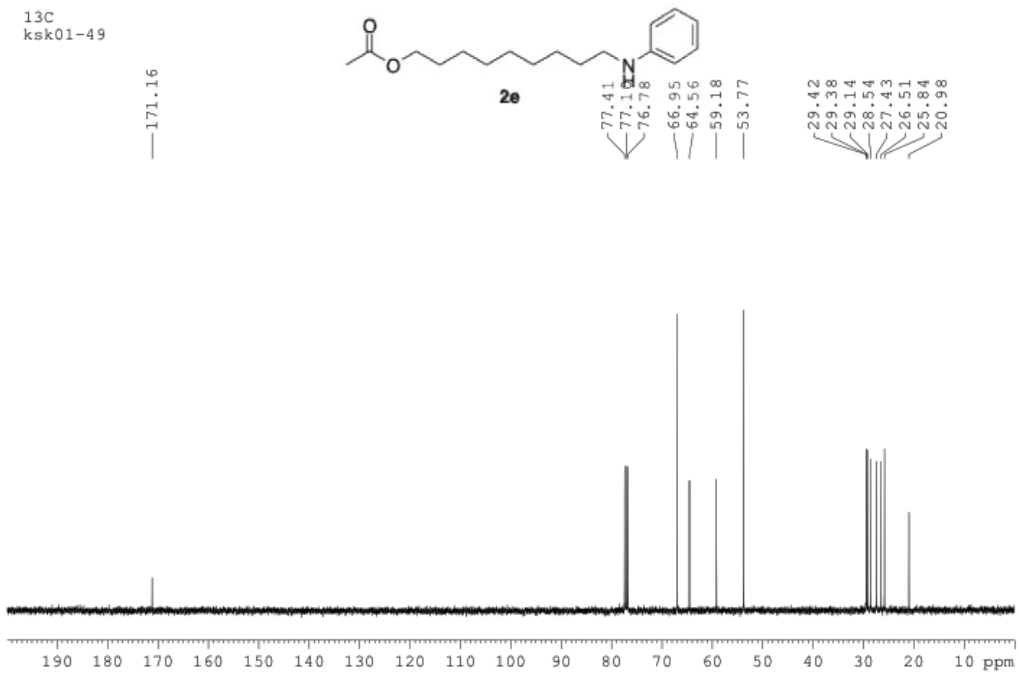
13C
ksk01-78



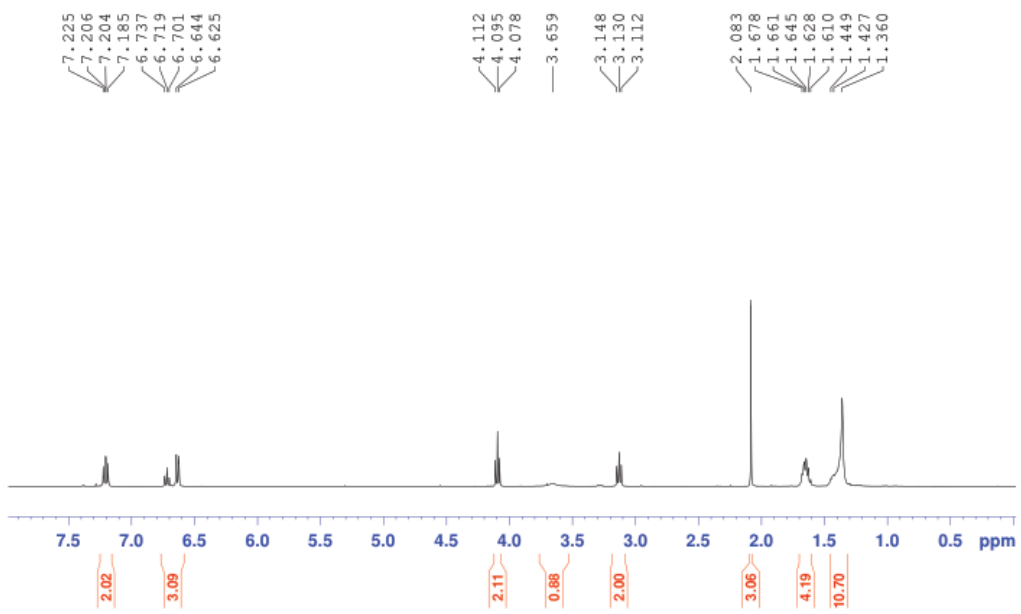
1D Proton NMR
ksk01-49



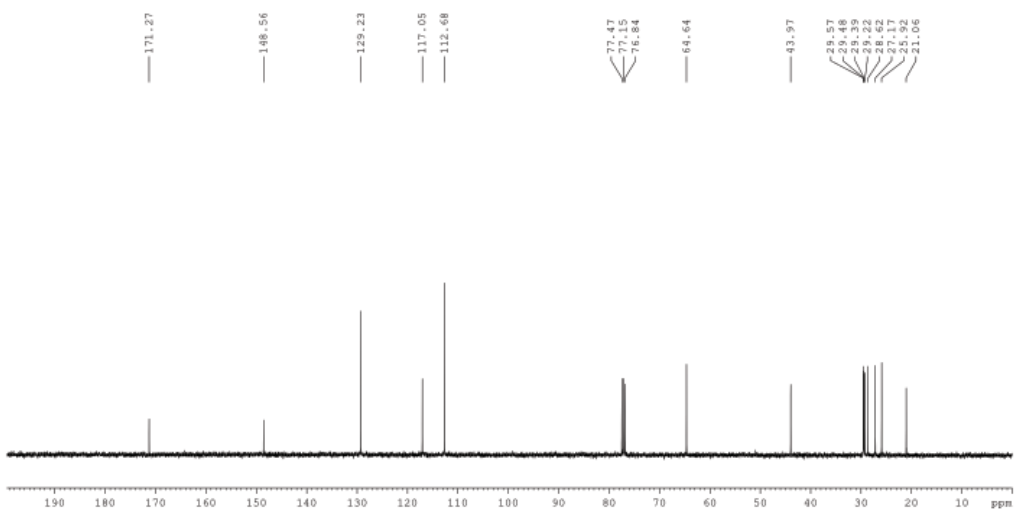
¹³C
ksk01-49

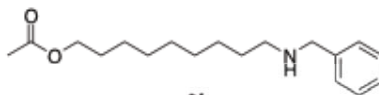


1D Proton NMR
ksk01-82

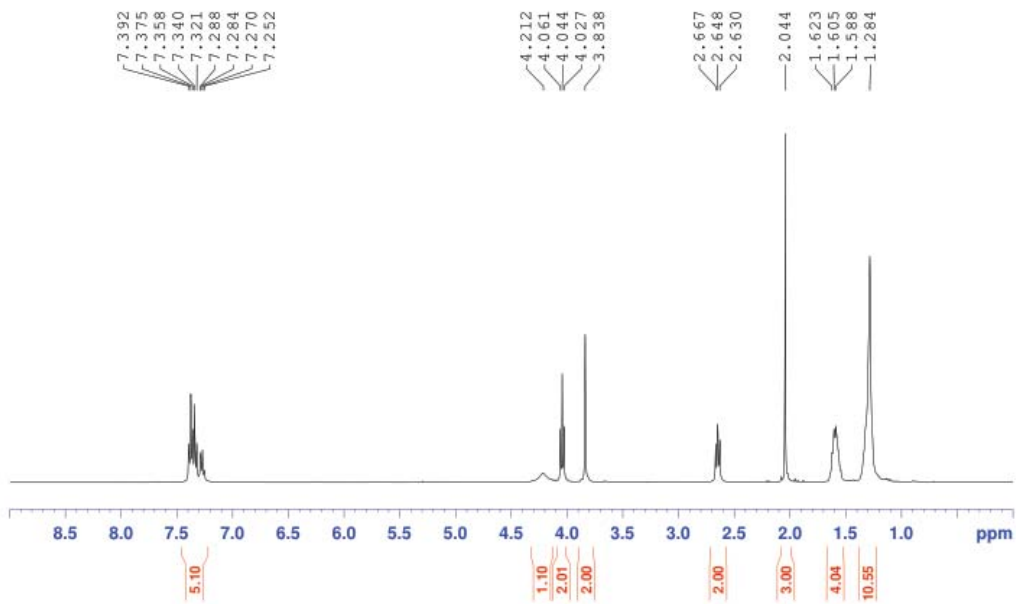


¹³C
ksk01-82

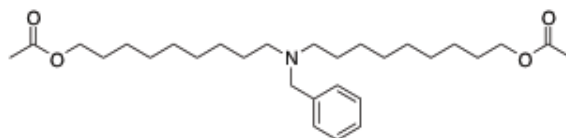
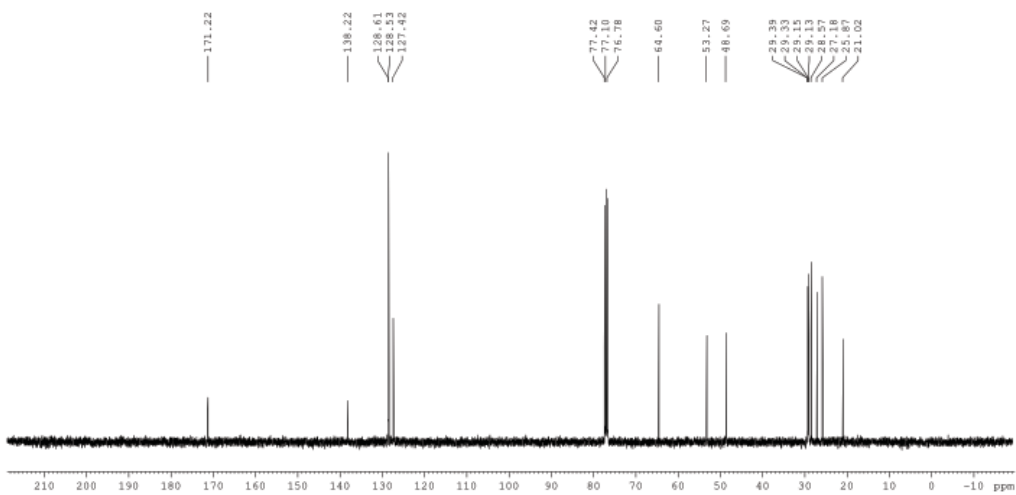




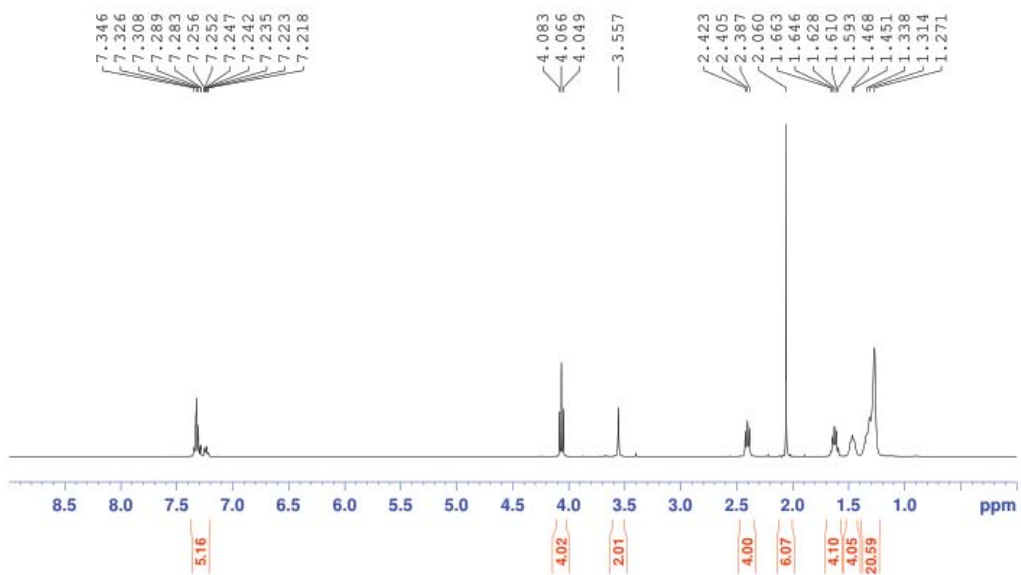
1D Proton NMR
ksk01-32 f2



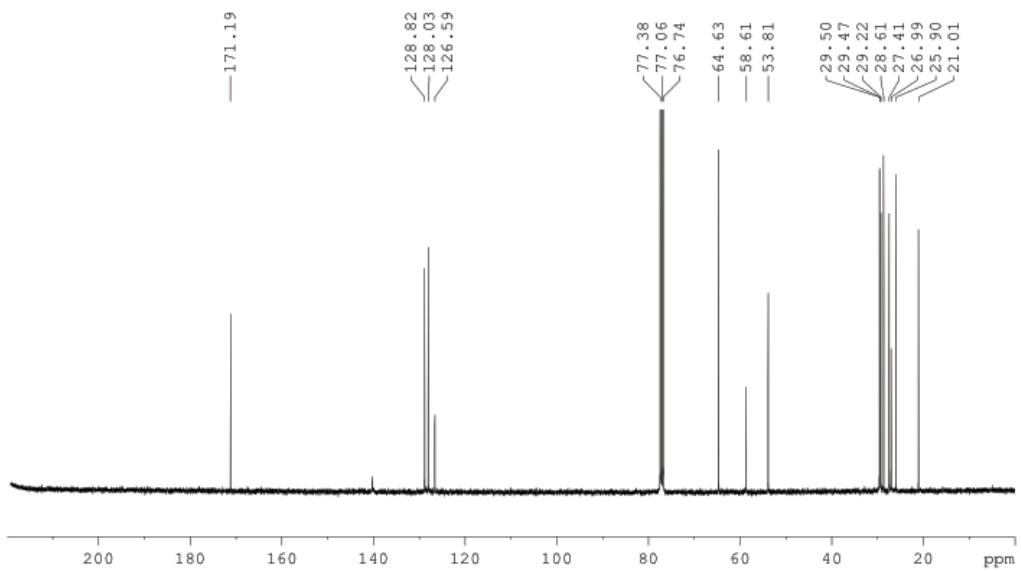
13C
ksk01-32 f2

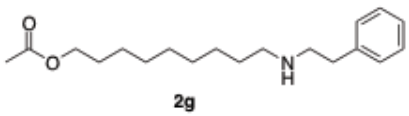


1D Proton NMR
ksk01-32 f1

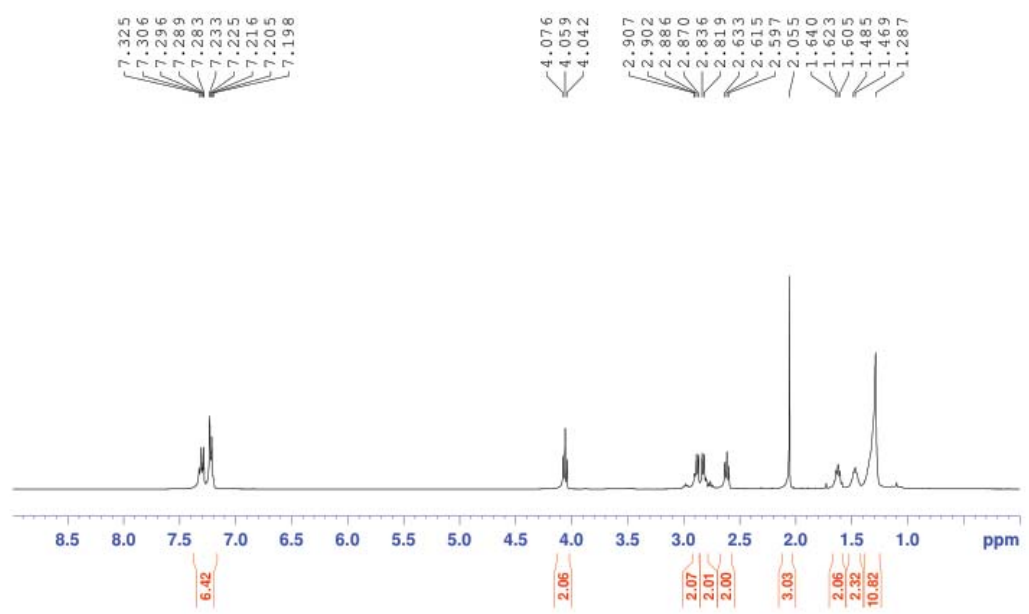


13C
ksk01-32 f1

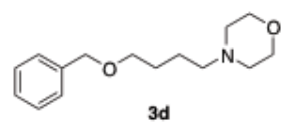
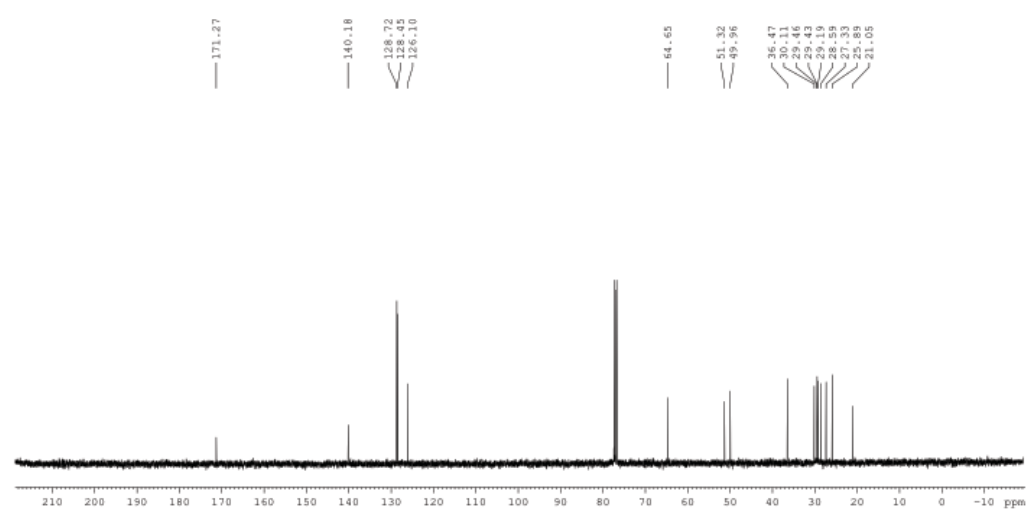




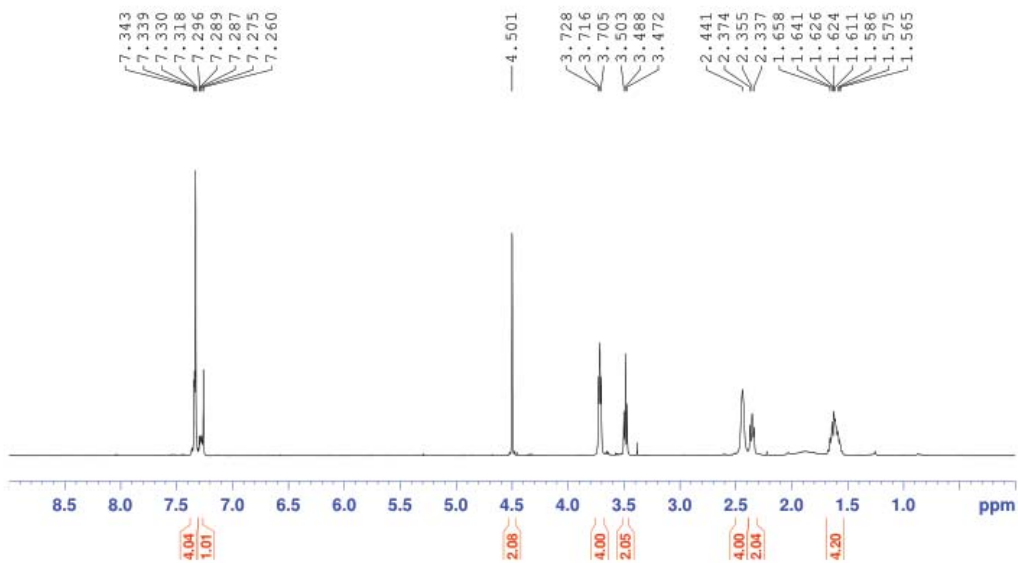
1D Proton NMR
ksk01-90



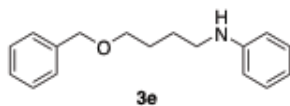
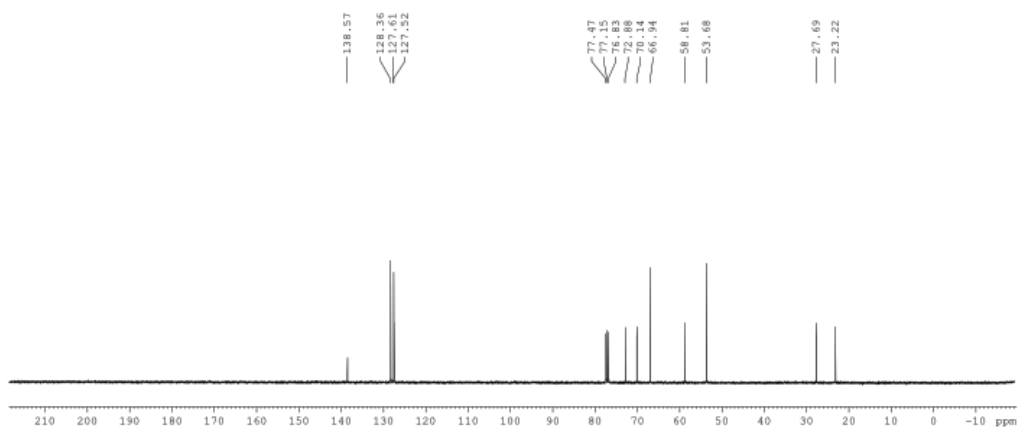
13C
ksk01-90



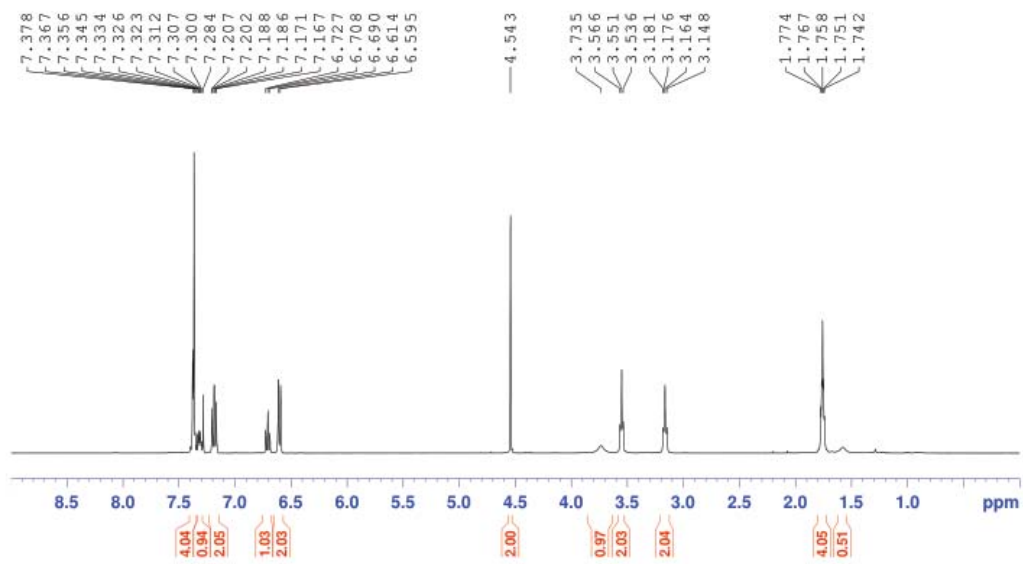
1D Proton NMR
ksk01-71
400MHz, CDCl3



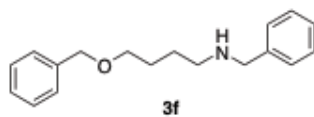
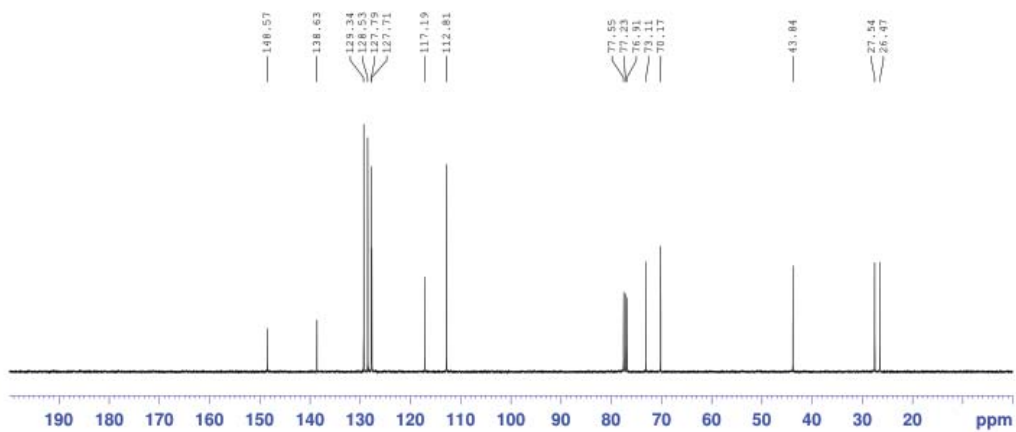
13C
ksk01-71



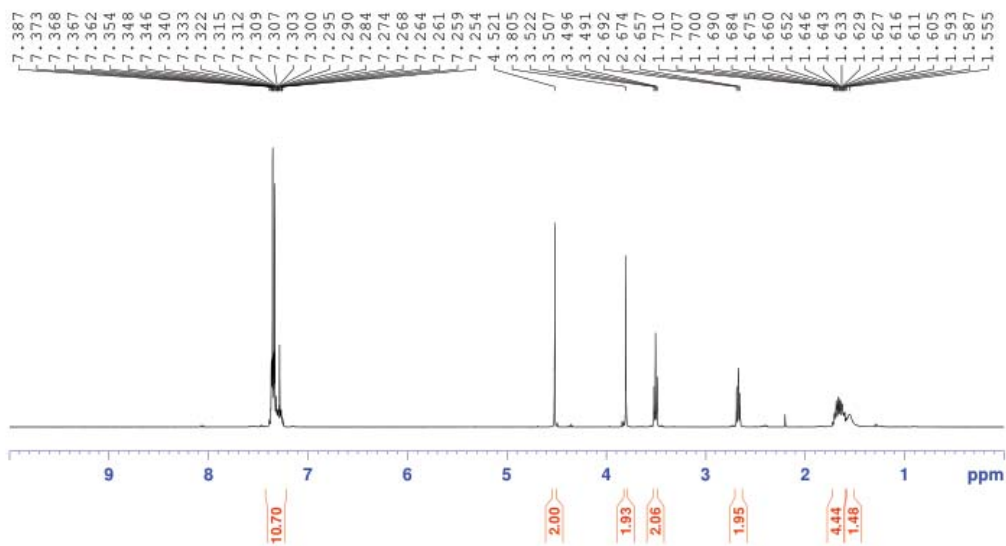
1D Proton NMR
ksk01-81
CDCl₃, 400 MHz



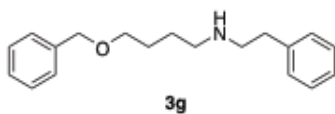
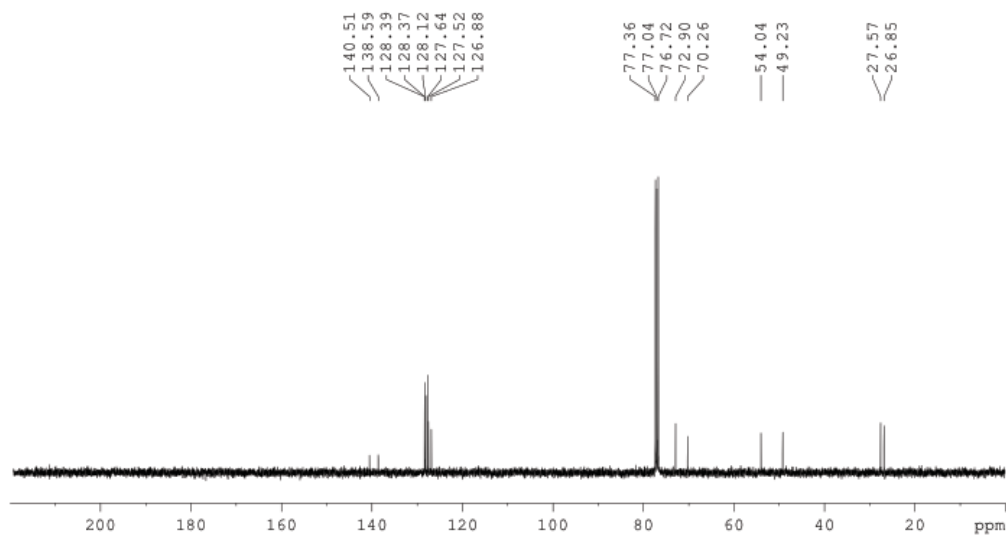
13C ksk01-81
CDCl₃



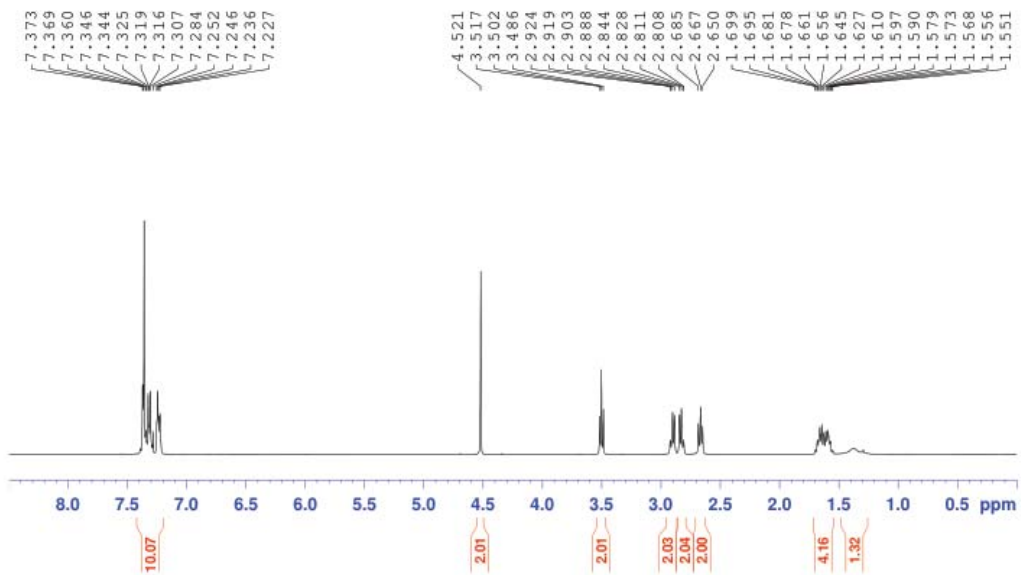
1D Proton NMR
ksk01-83



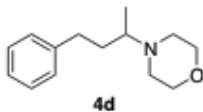
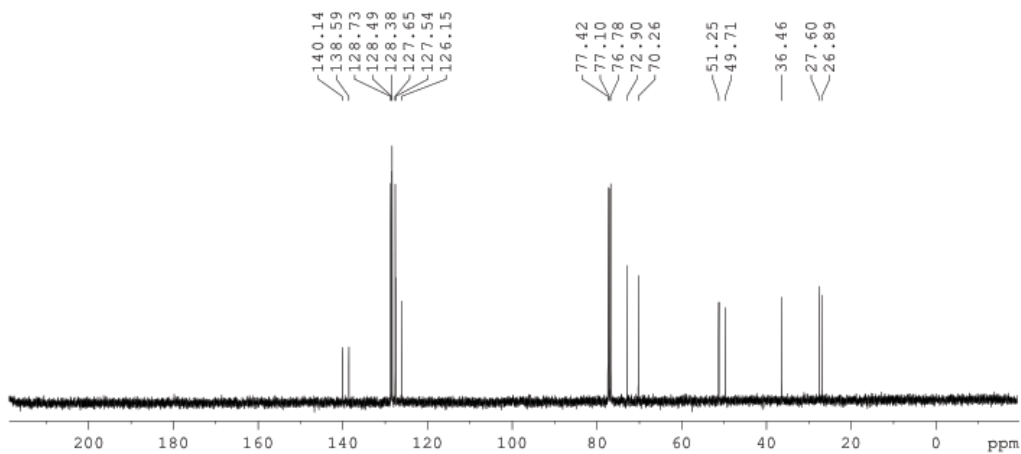
¹³C
ksk01-83



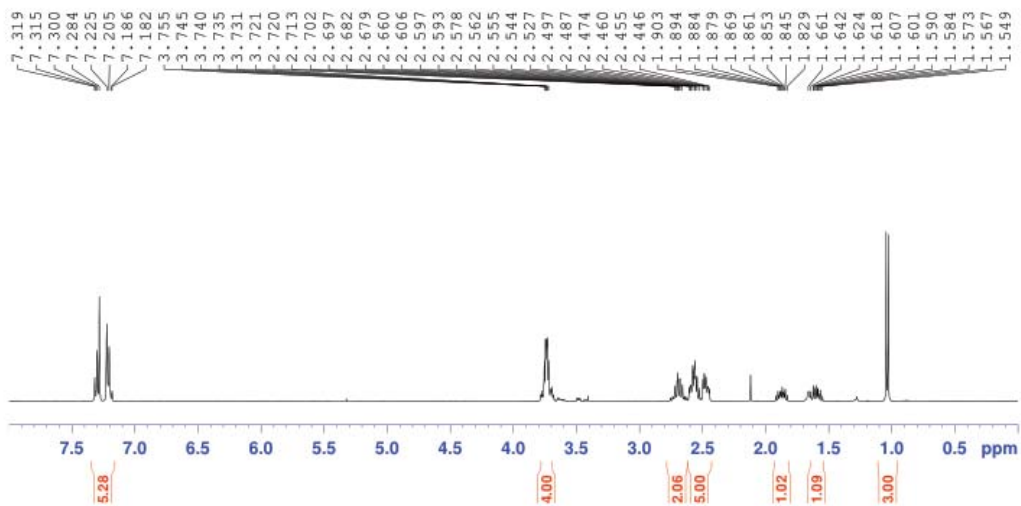
1D Proton NMR
ksk01-95



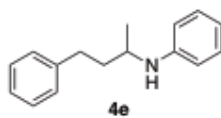
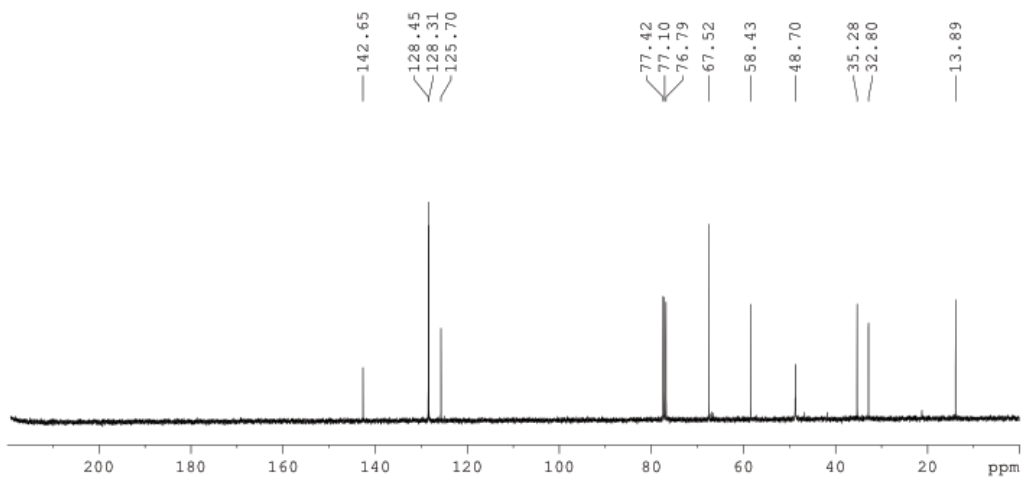
¹³C
ksk01-95



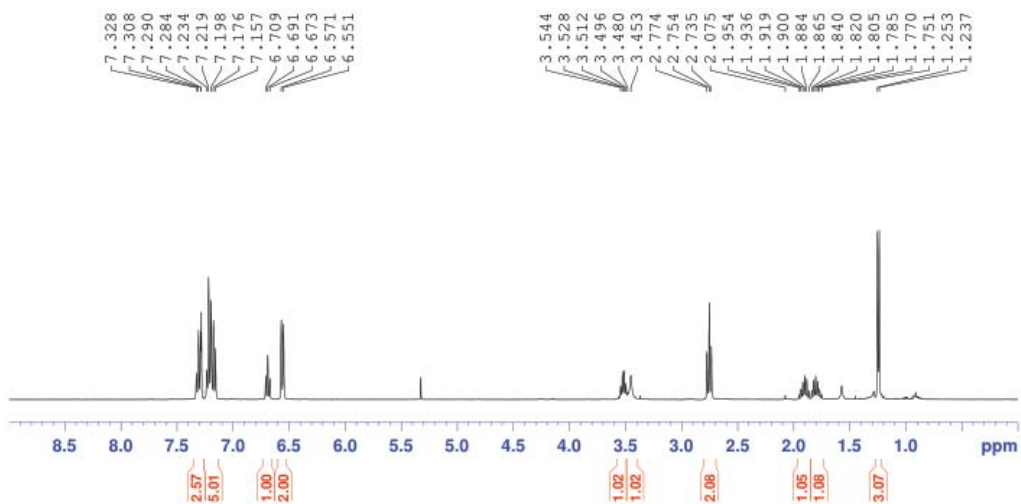
1D Proton NMR
ksk01-86



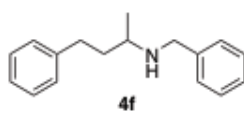
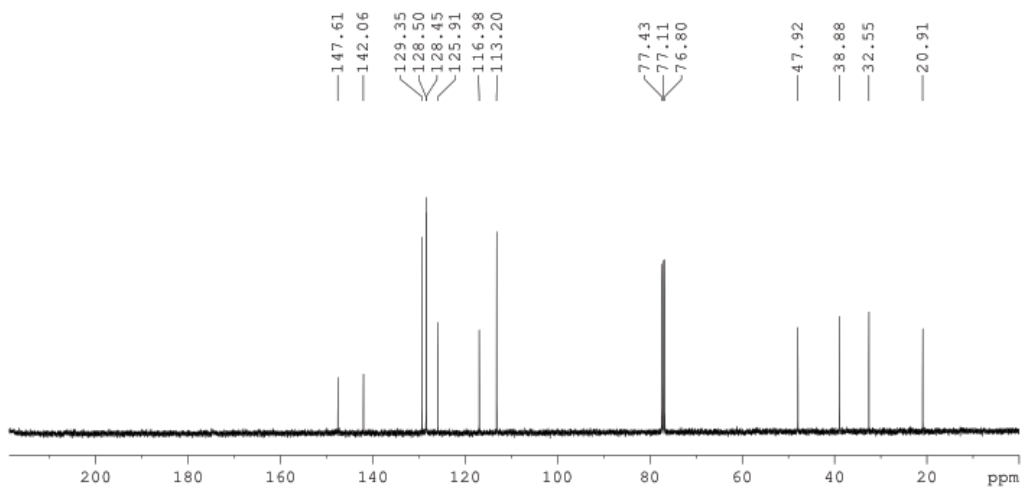
13C
ksk01-86



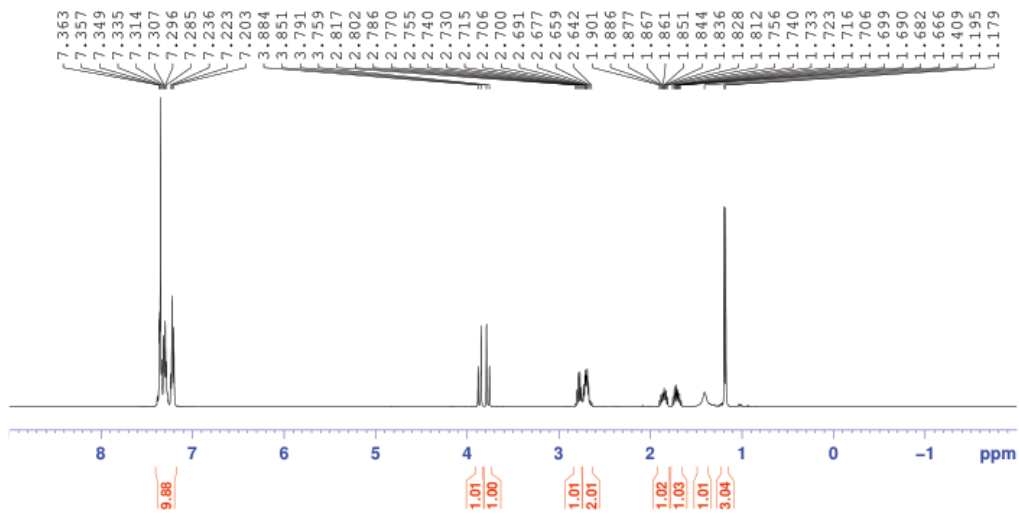
1D Proton NMR
ksk01-87



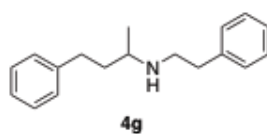
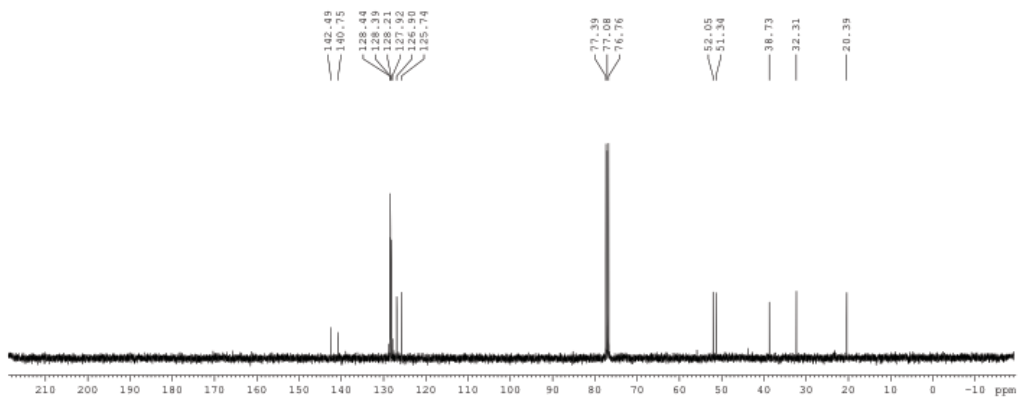
13C
ksk01-87



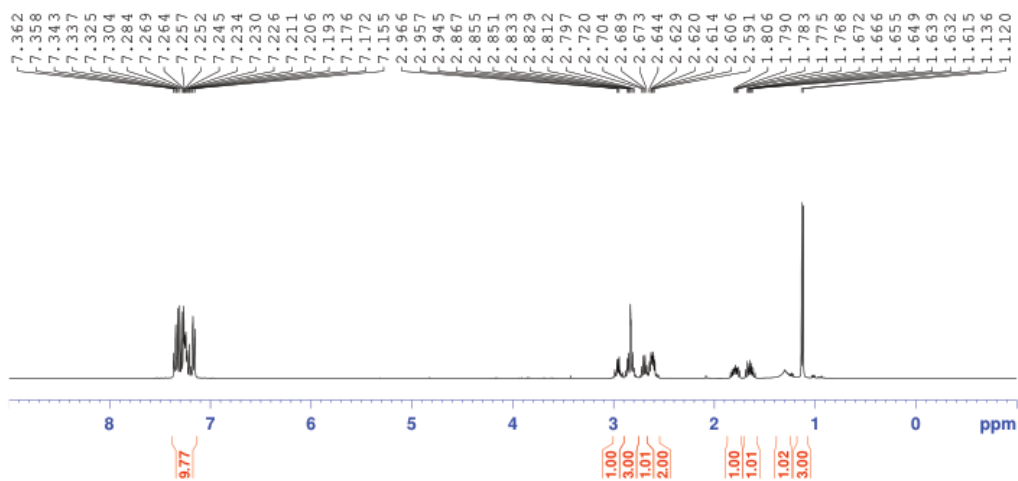
1D Proton NMR
kks01-91



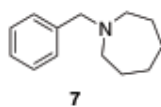
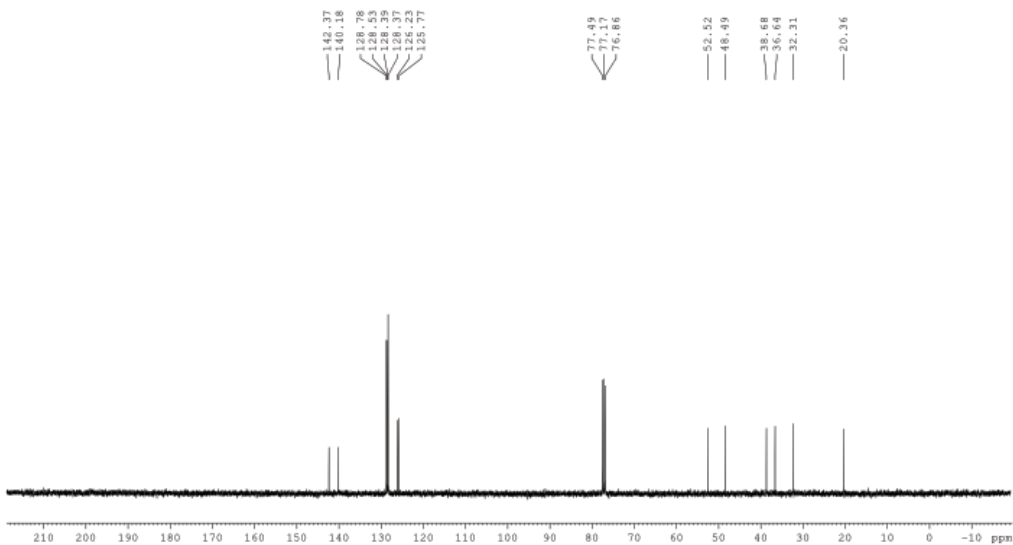
13C
kks01-91



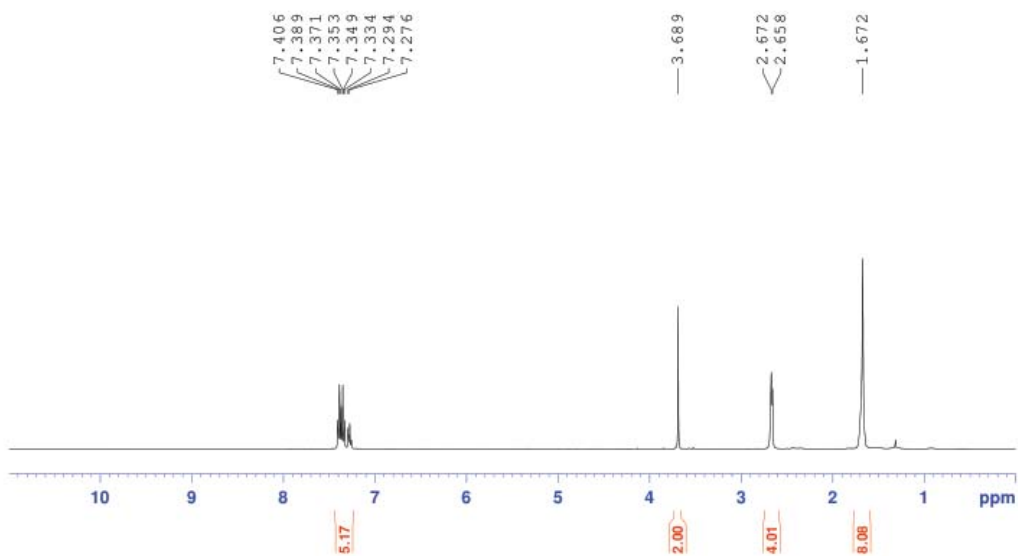
1D Proton NMR
ksk01-96



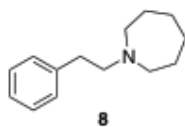
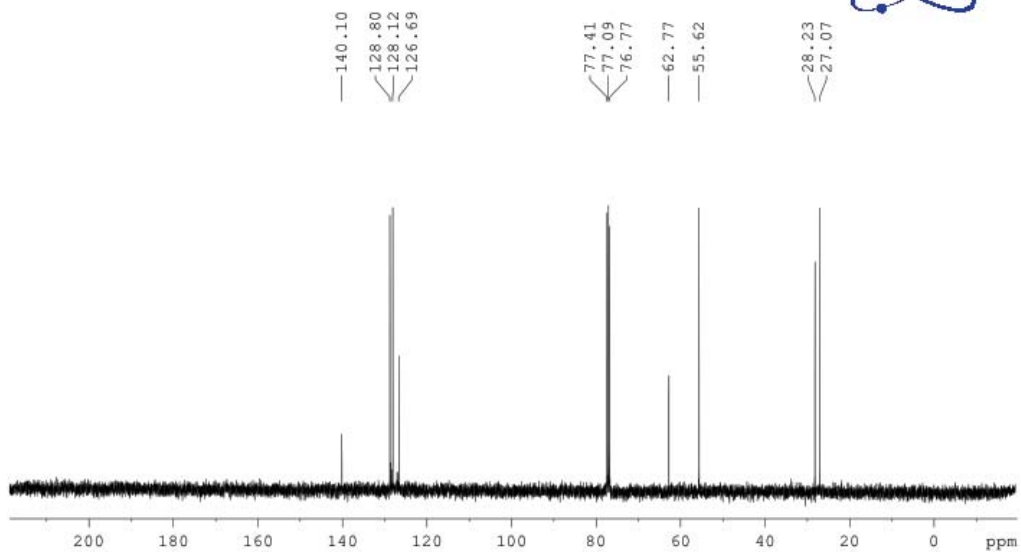
13C
ksk01-96



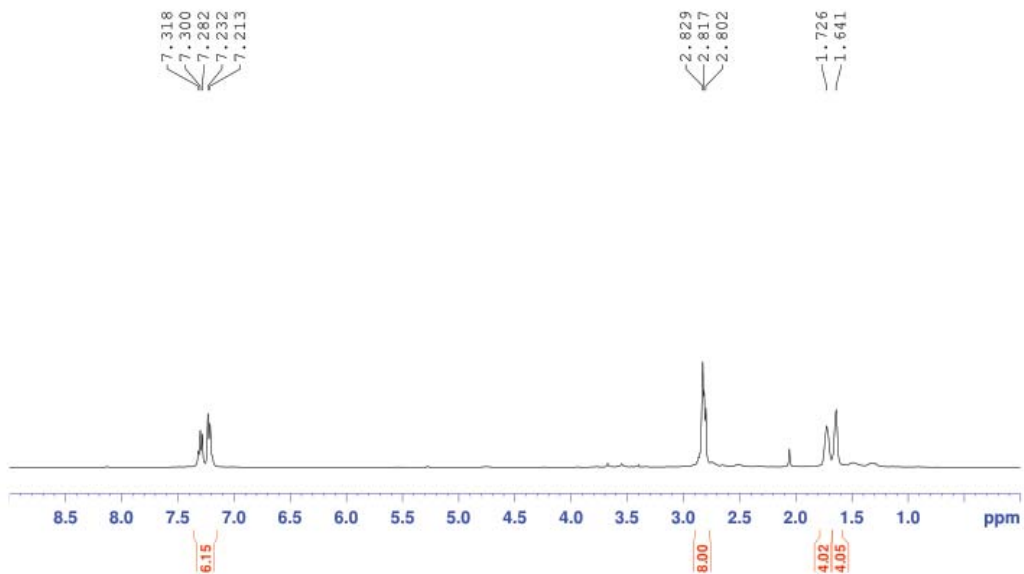
1D Proton NMR
ksk02-63
400 MHz, CDCl₃



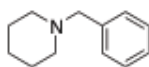
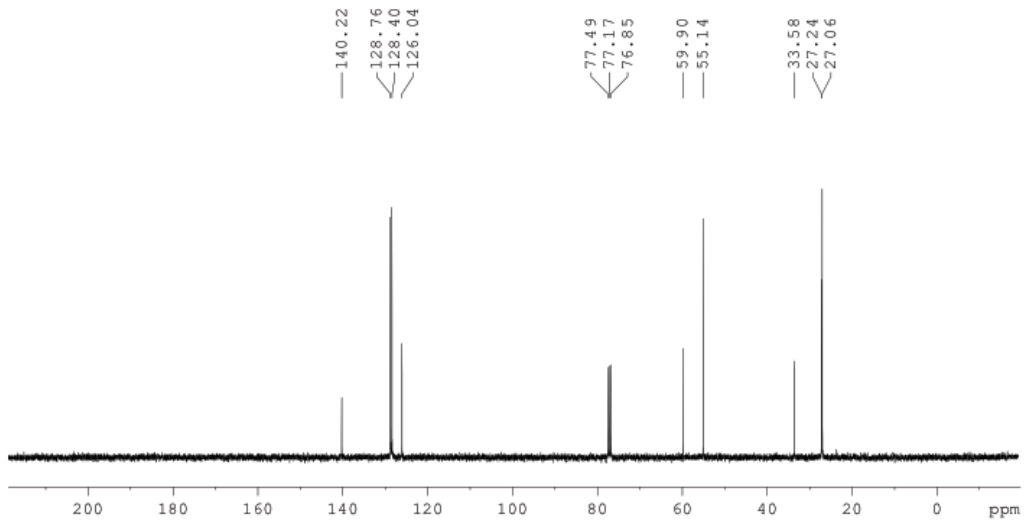
¹³C
ksk02-63
400 MHz, CDCl₃



1D Proton NMR
ksk02-64
400 MHz, CDCl₃

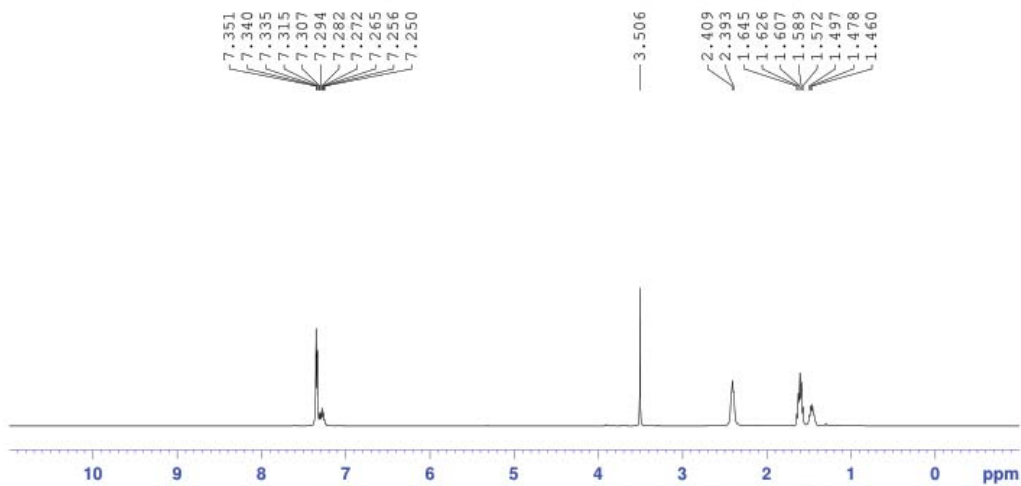


13C
ksk02-64
400MHz, CDCl₃

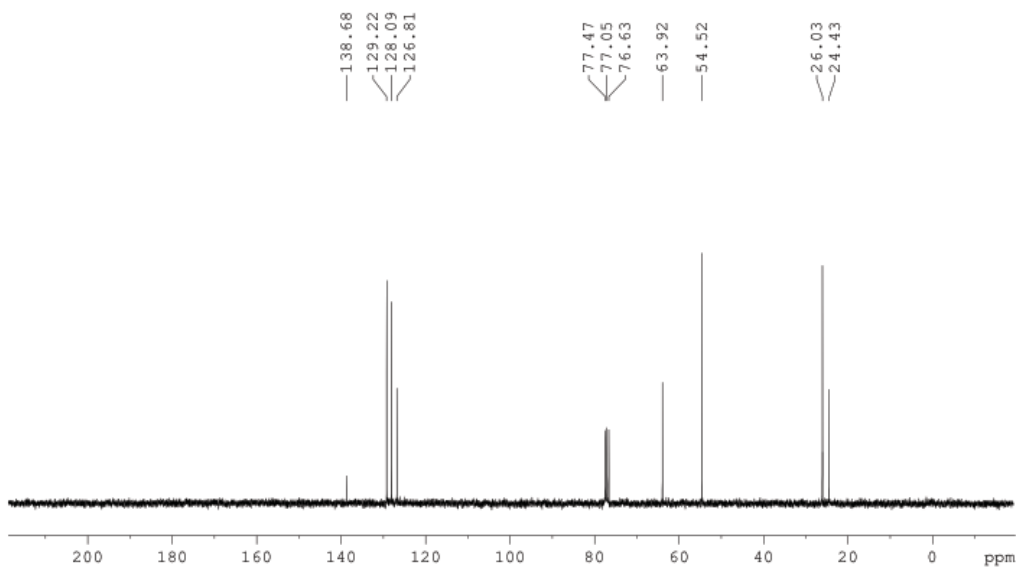


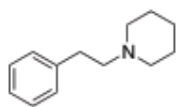
9

1D Proton
ksk02-60
CDCl₃, 400 MHz



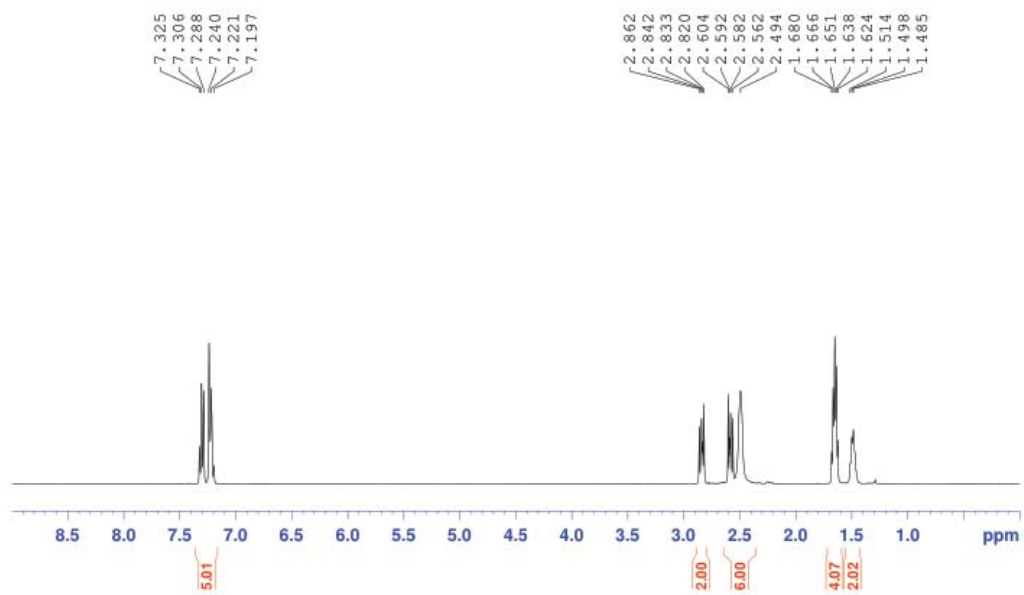
13C
ksk02-60
400 MHz, CDCl₃



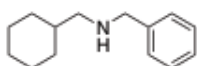
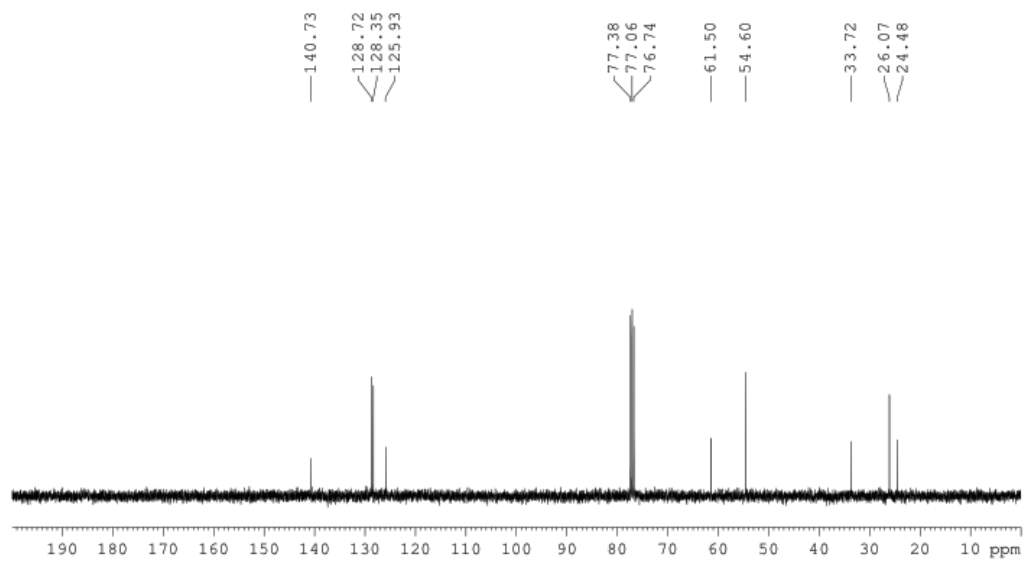


10

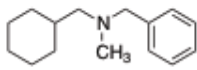
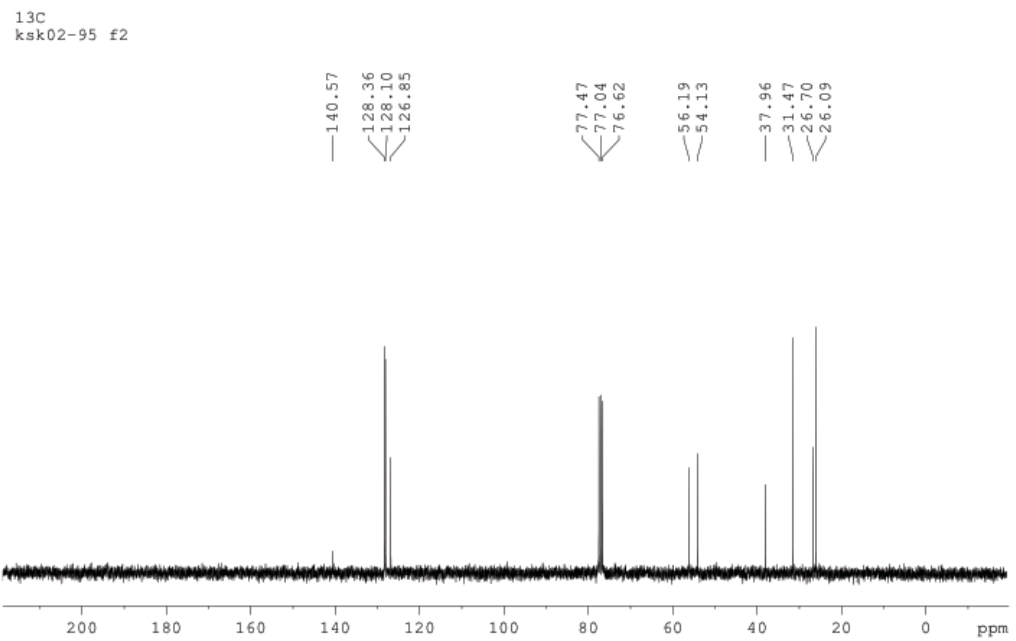
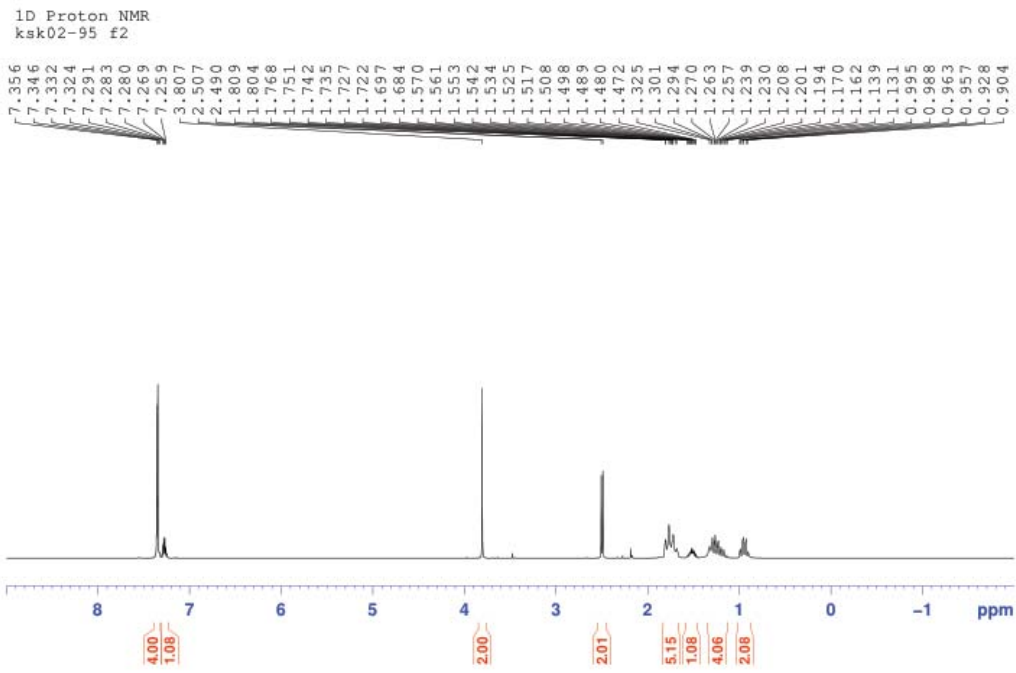
1D Proton NMR
ksk01-92



13C
ksk01-92

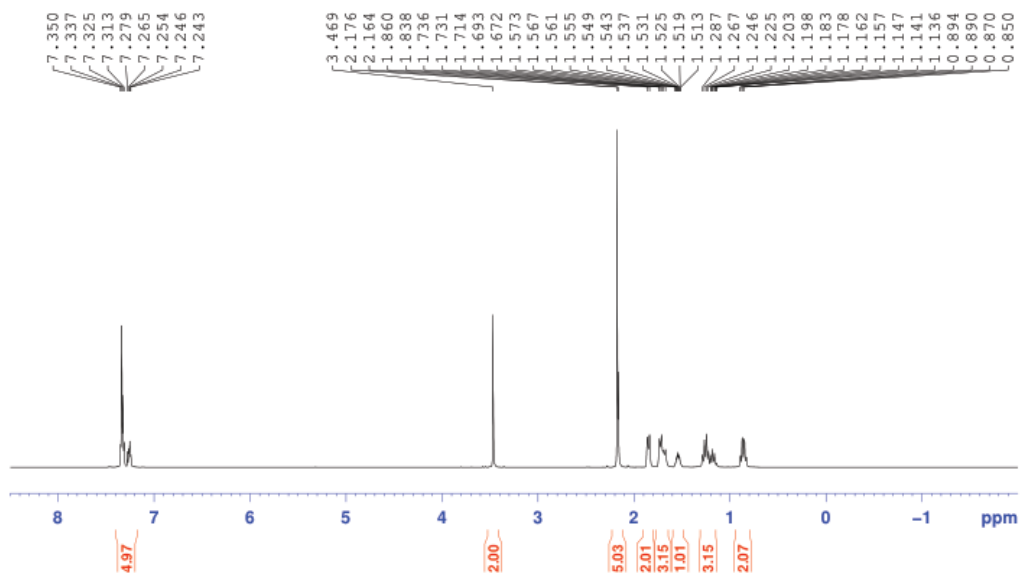


11a

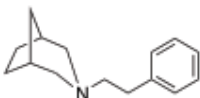
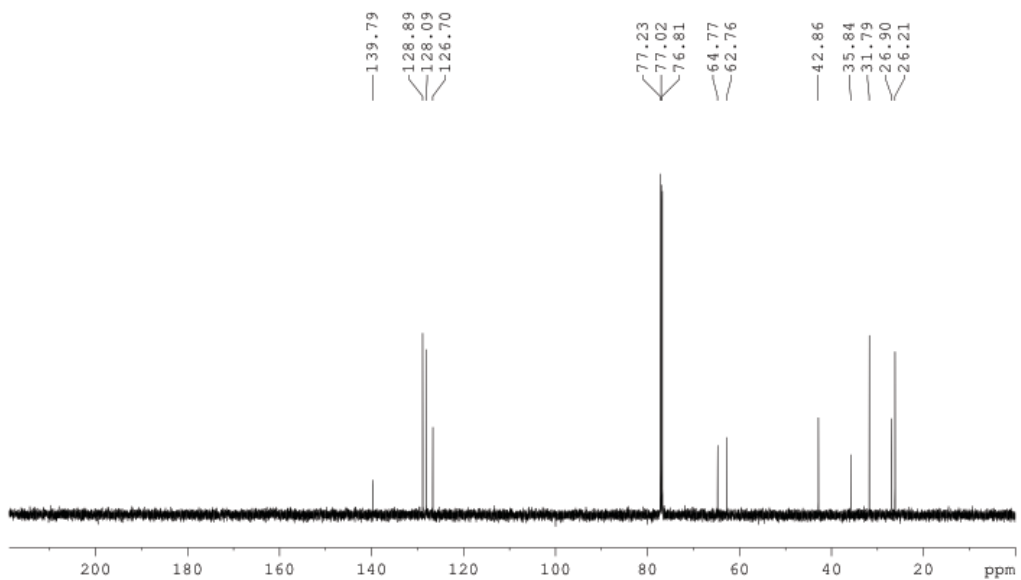


11b

1D Proton NMR
ksk02-95 f1

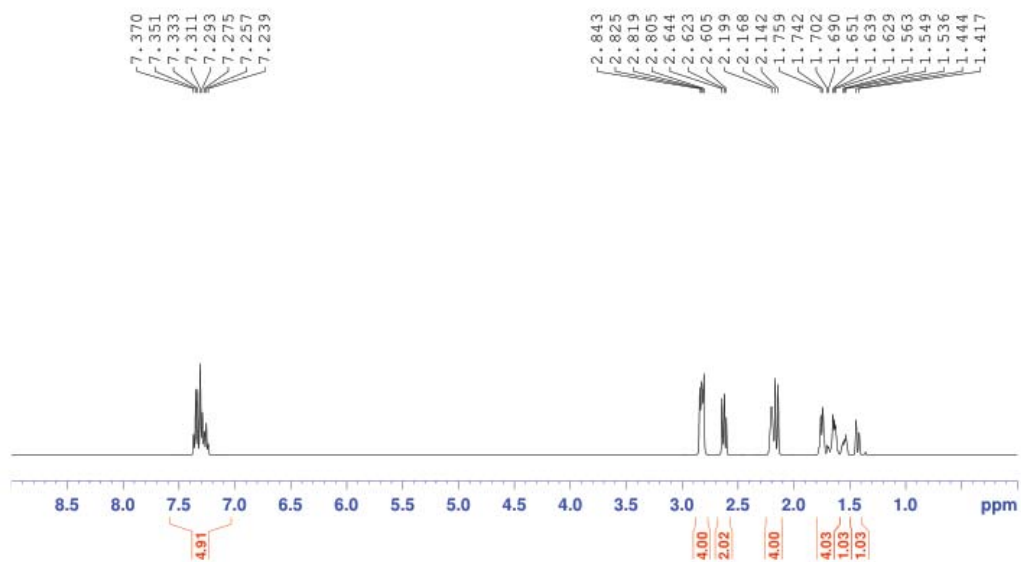


13C NMR
ksk02-95 f1

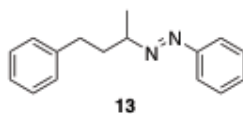
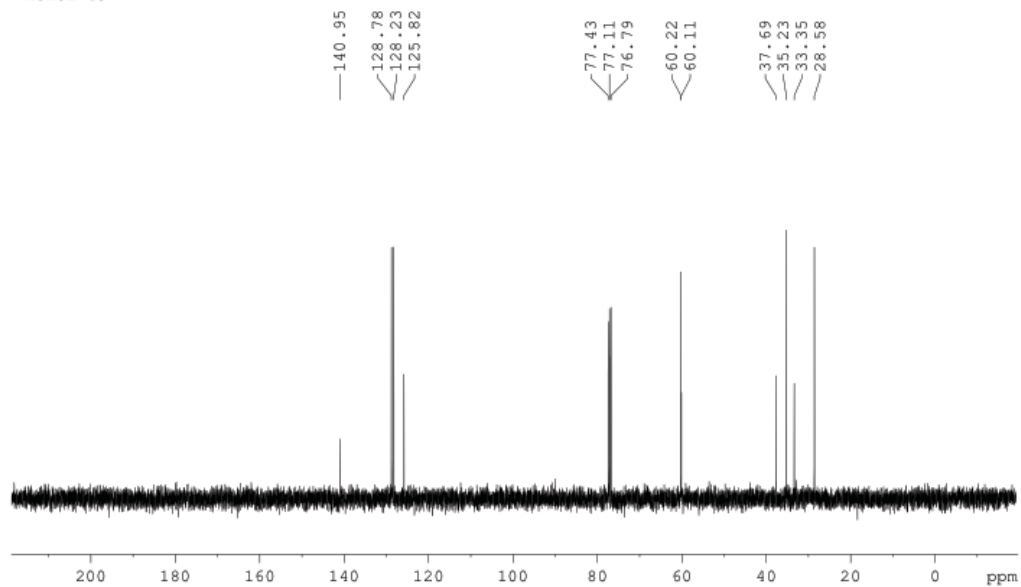


12

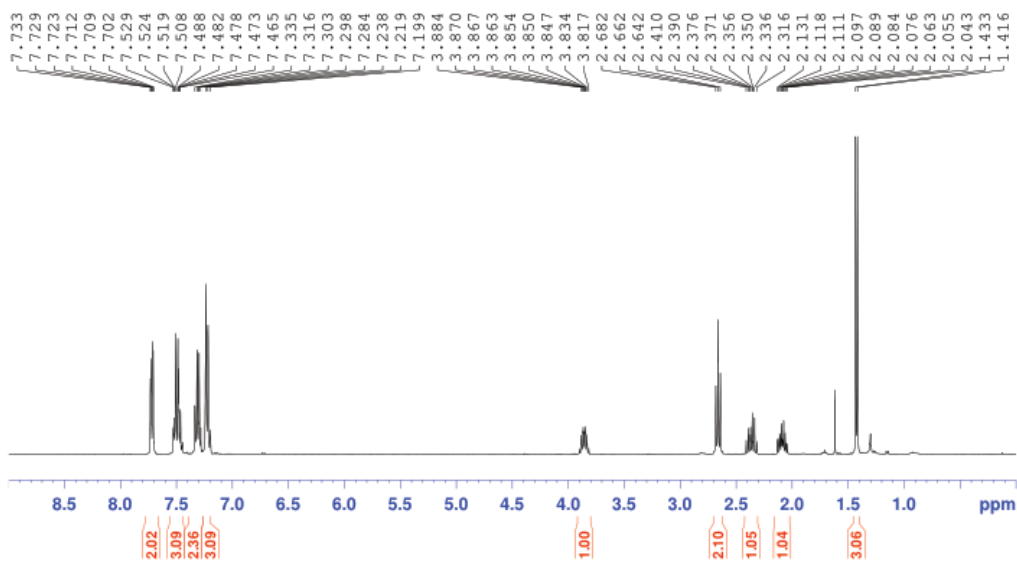
1D Proton NMR
ksk02-89



¹³C
ksk02-89



1D Proton NMR
ksk01-40
CDCl₃, 400 MHz



13C
ksk02-40
CDCl₃, 400 MHz

