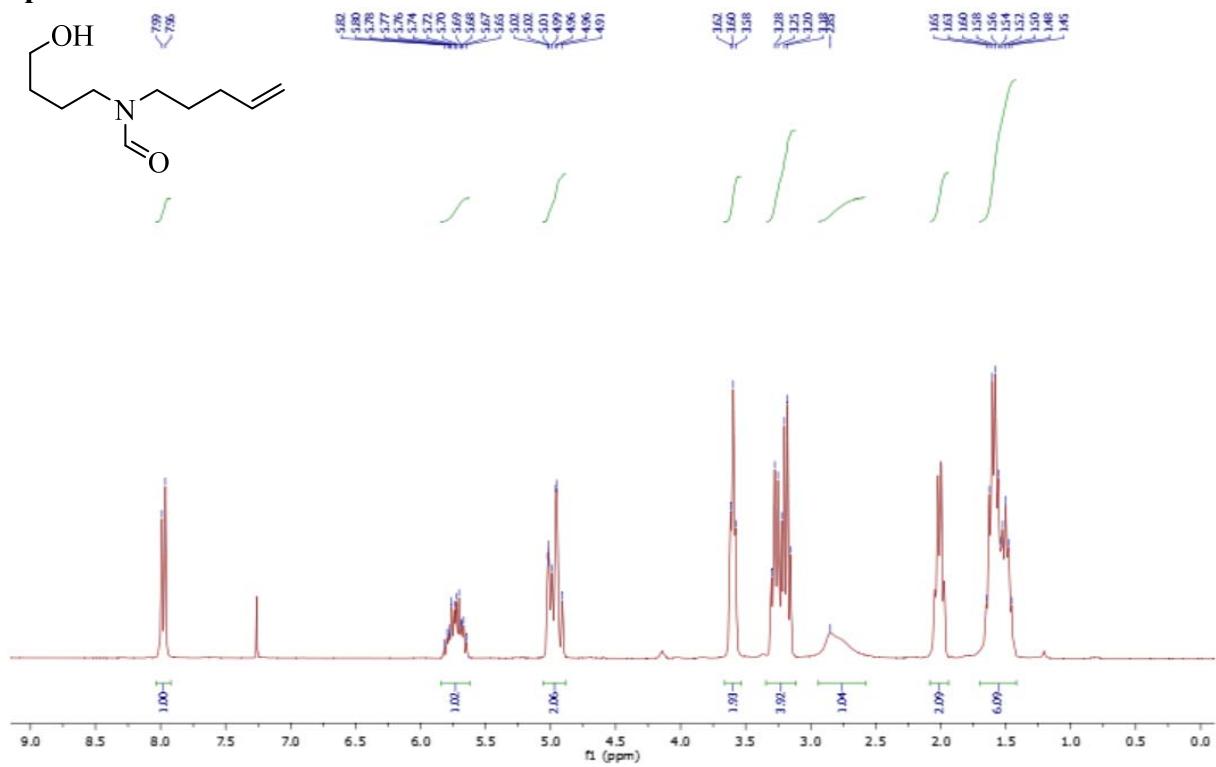
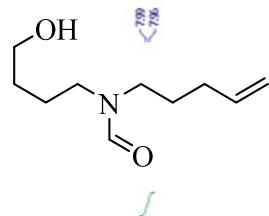


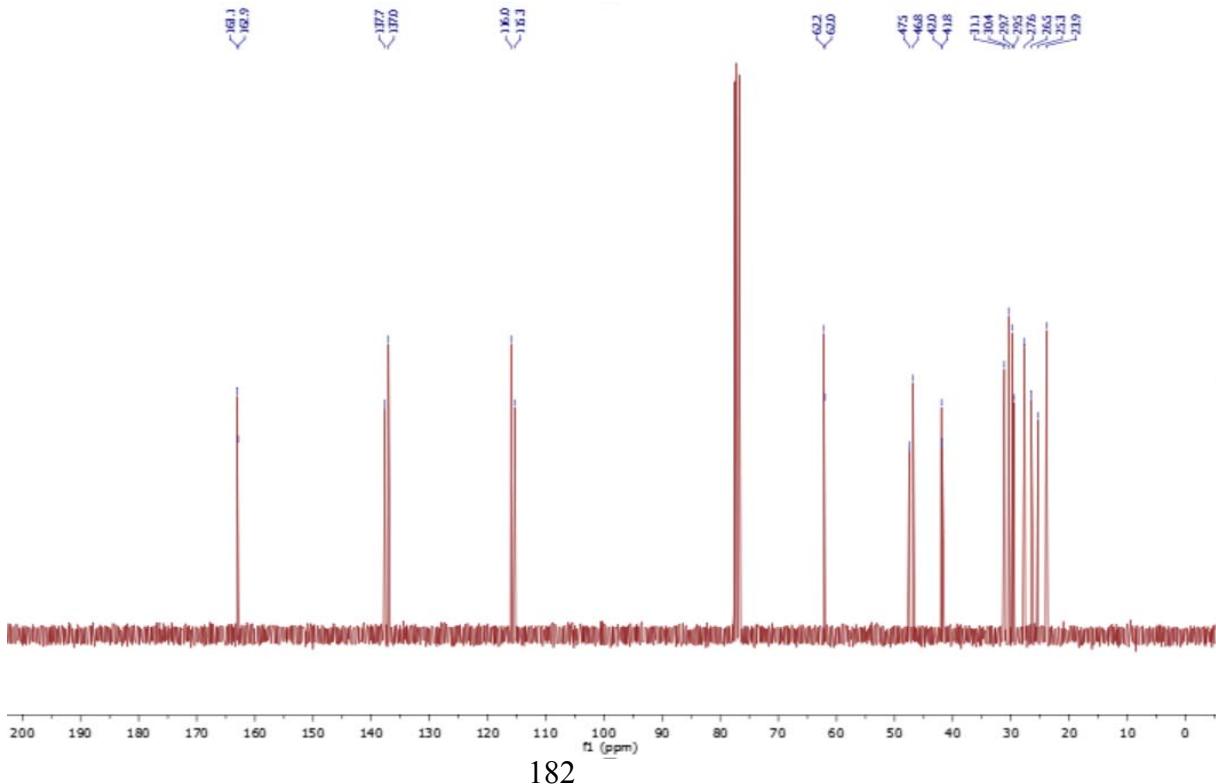
ANNEXE 2: SPECTRES DE RÉSONANCE MAGNÉTIQUE NUCLÉAIRE DES PROTONS

***N*-(4-Hydroxybutyl)-*N*-(pent-4-en-1-yl)formamide (1-9)**

¹H RMN spectrum

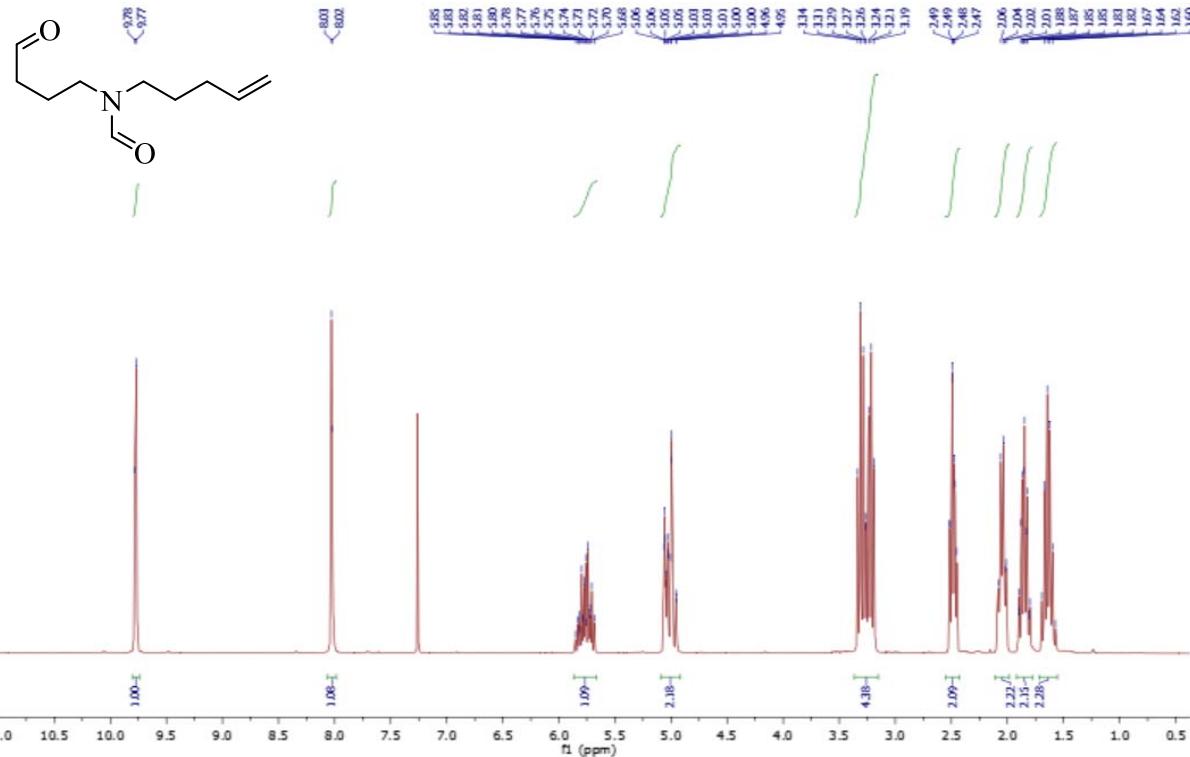


¹³C RMN spectrum

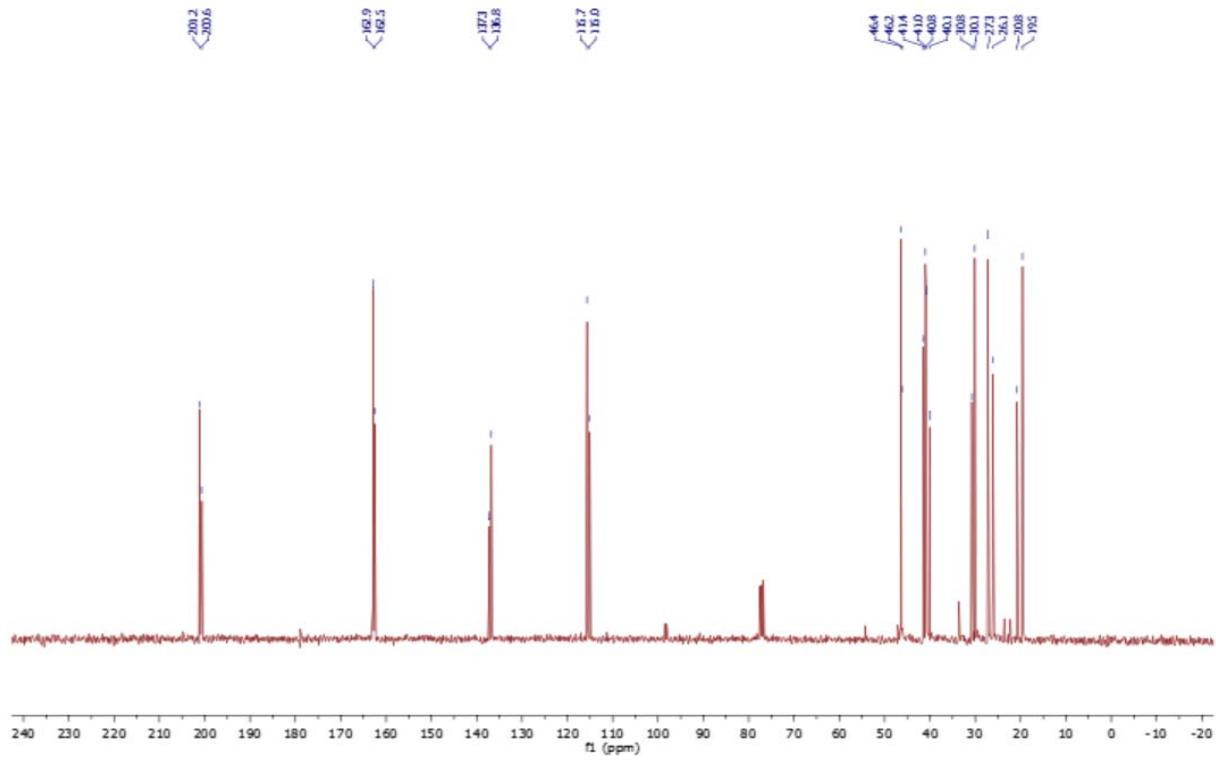


***N*-(4-Oxobutyl)-*N*-(pent-4-en-1-yl)formamide (1-10)**

¹H RMN spectrum

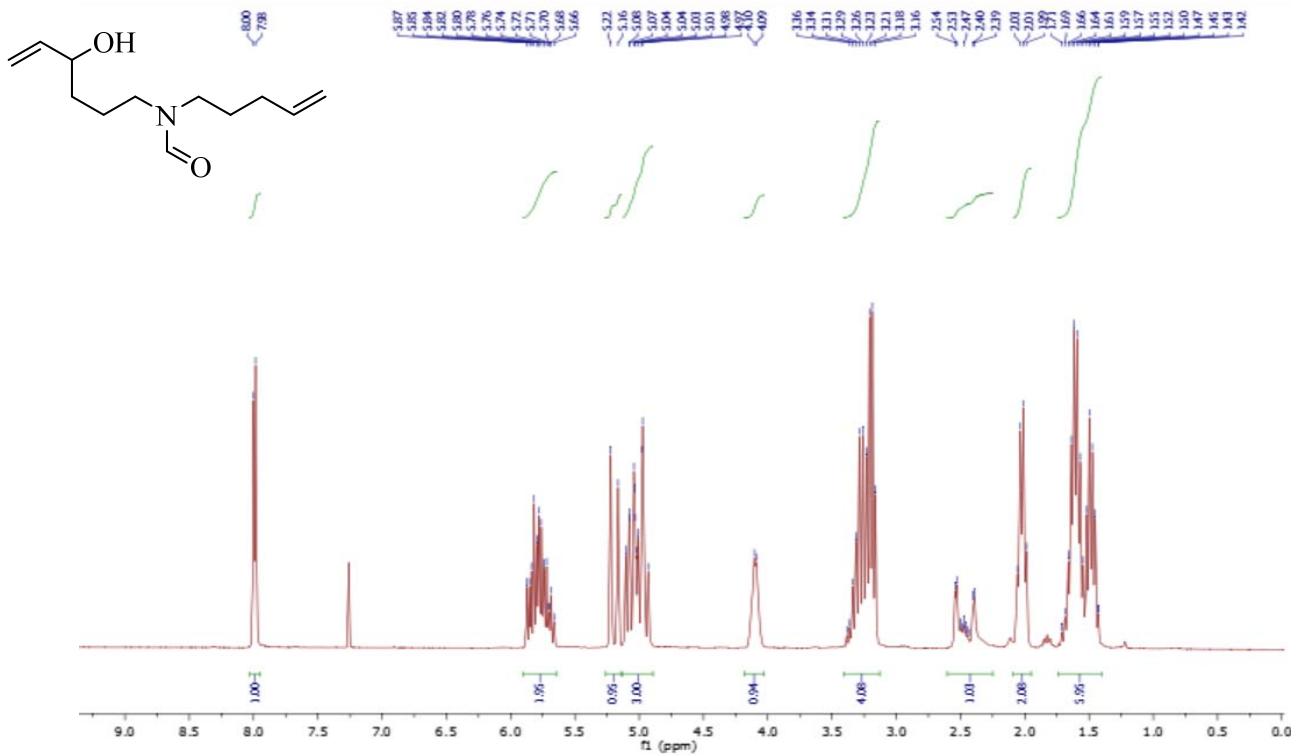


¹³C RMN spectrum

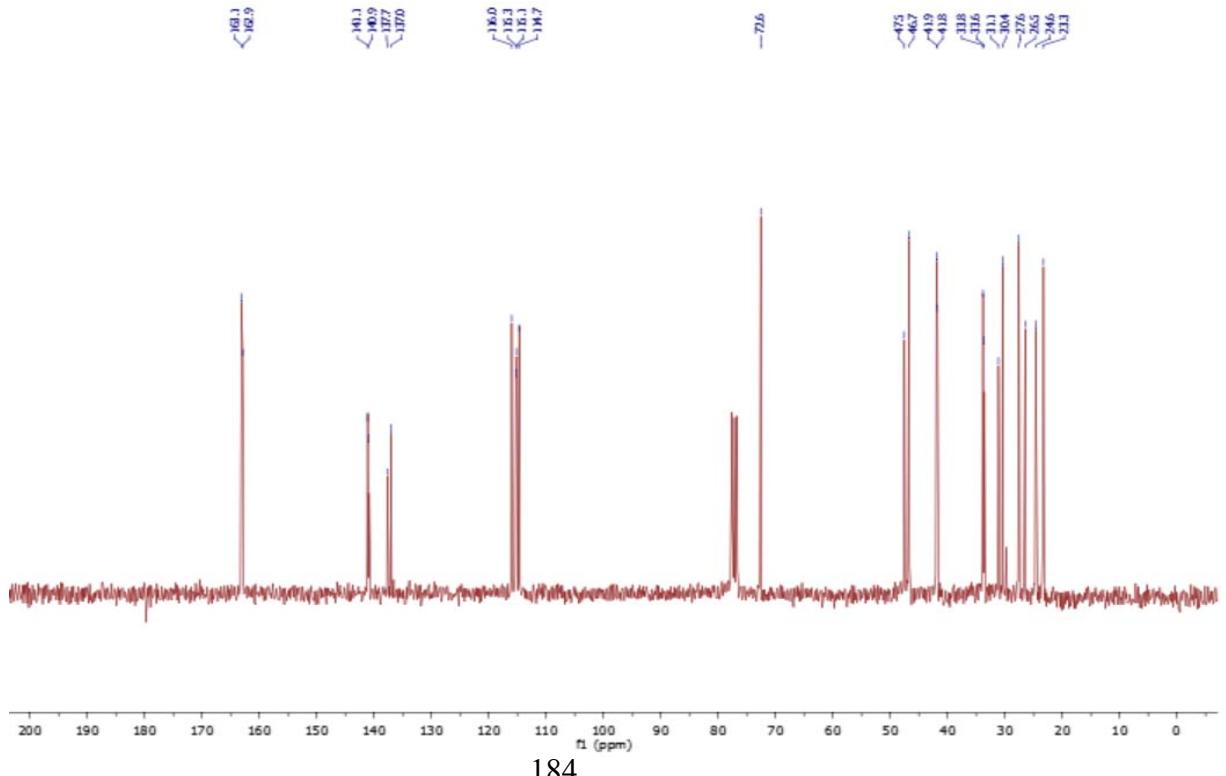


***N*-(4-Hydroxyhex-5-en-1-yl)-*N*-(pent-4-en-1-yl)formamide (1-11)**

¹H RMN spectrum

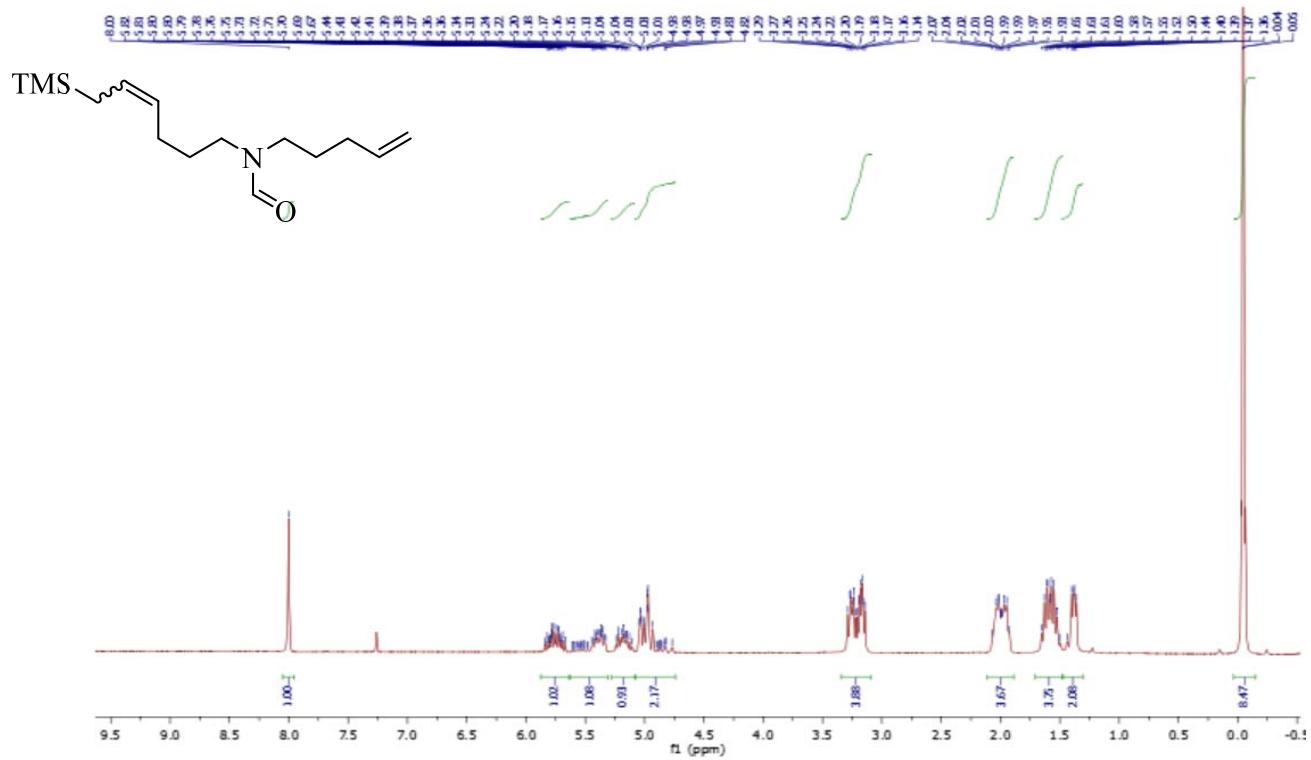


¹³C RMN spectrum

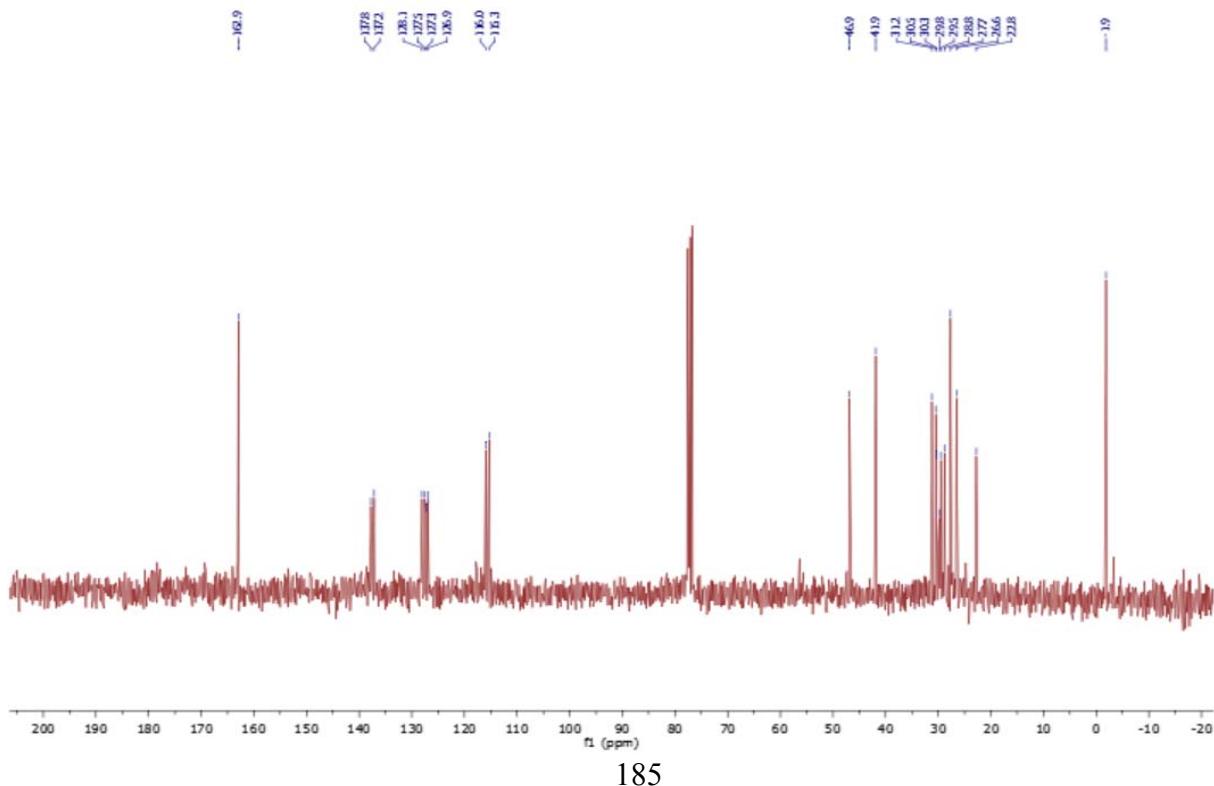


N-(Pent-4-en-1-yl)-N-(6-(trimethylsilyl)hex-4-en-1-yl)formamide (1-12)

^1H RMN spectrum

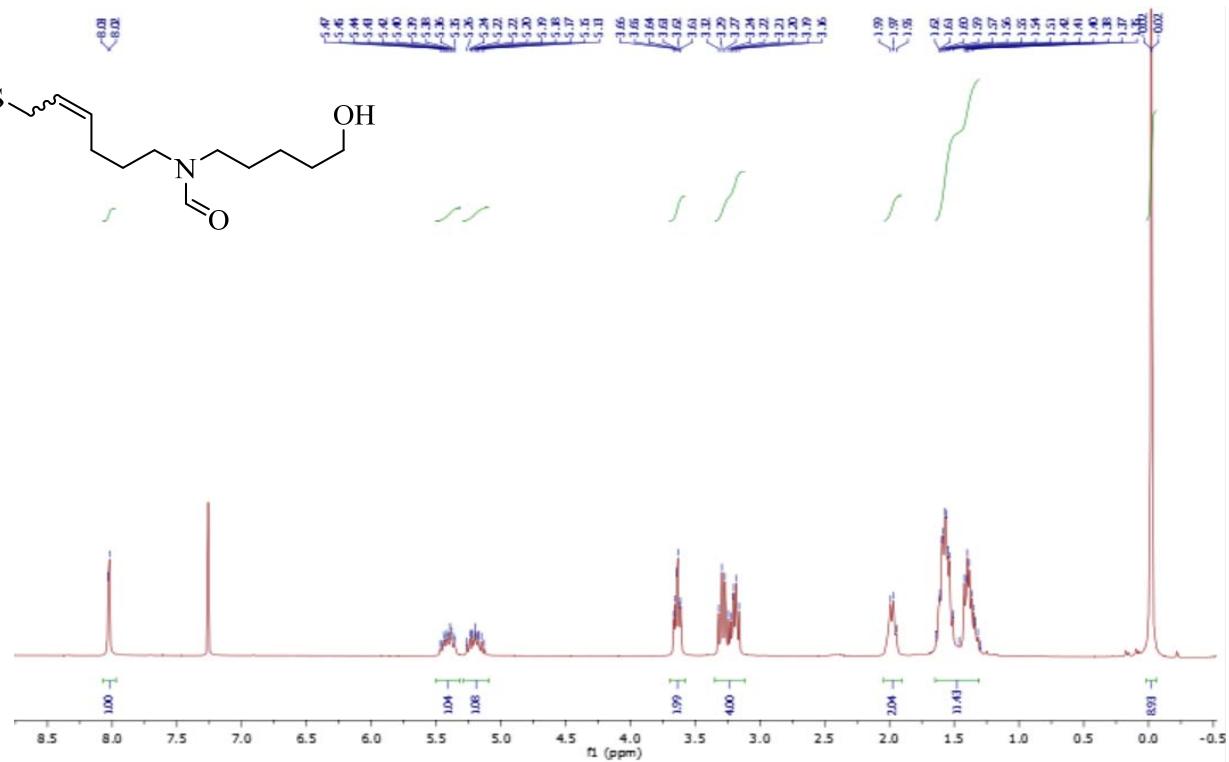
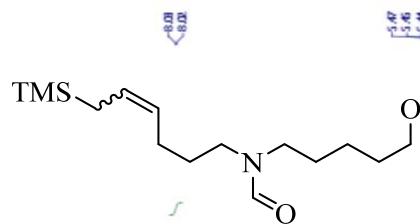


^{13}C RMN spectrum

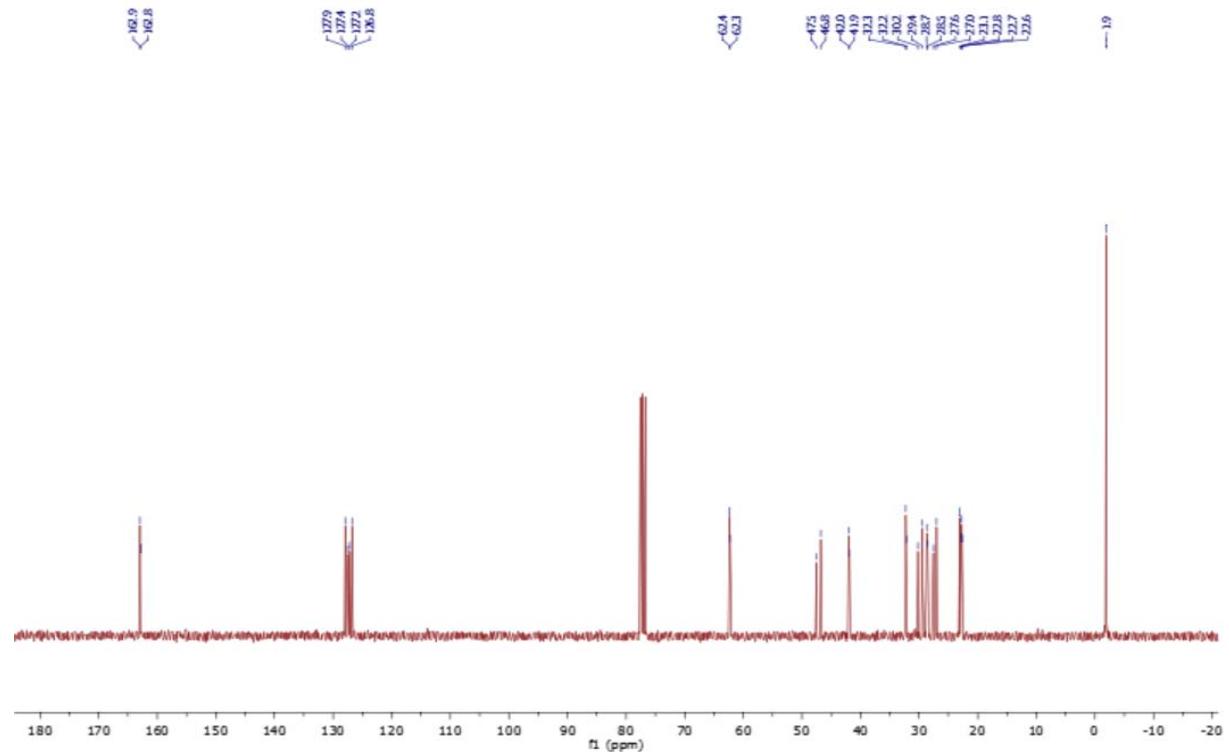


N-(5-Hydroxypentyl)-N-(6-(trimethylsilyl)hex-4-en-1-yl)formamide (1-13)

¹H RMN spectrum

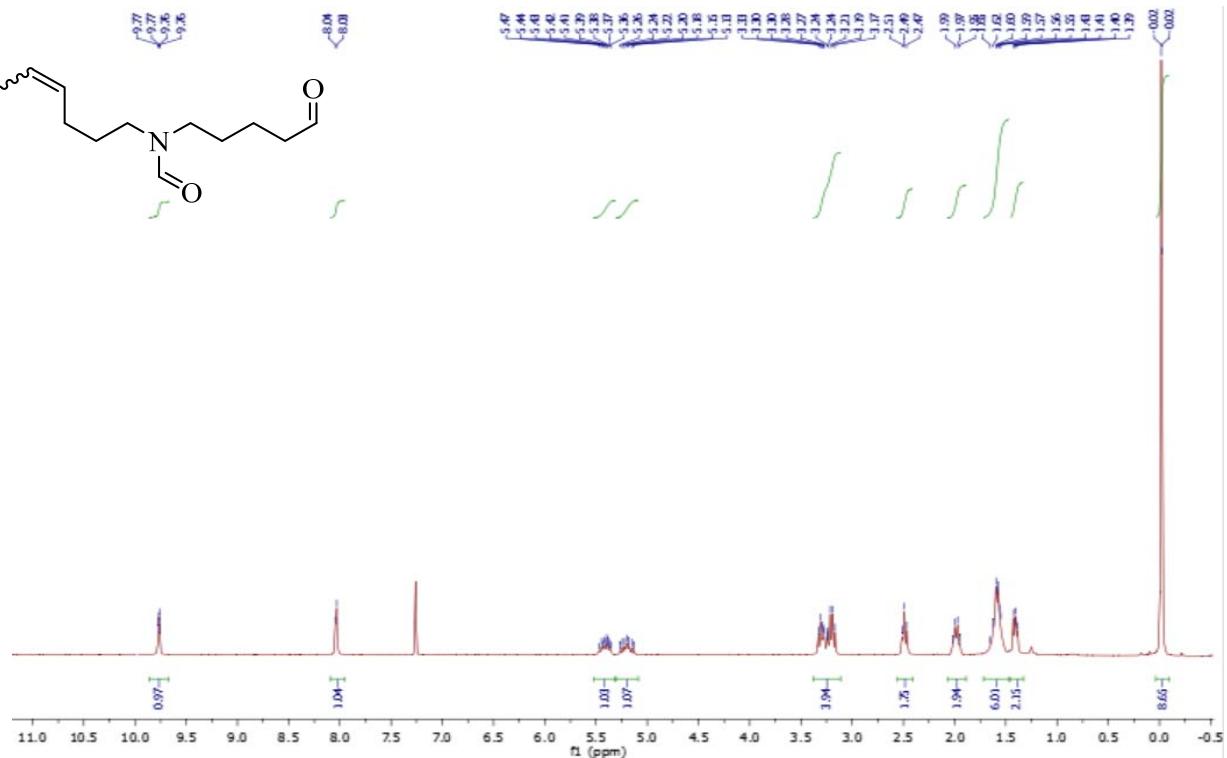
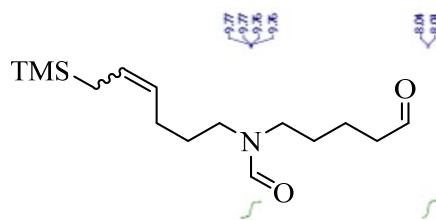


¹³C RMN spectrum

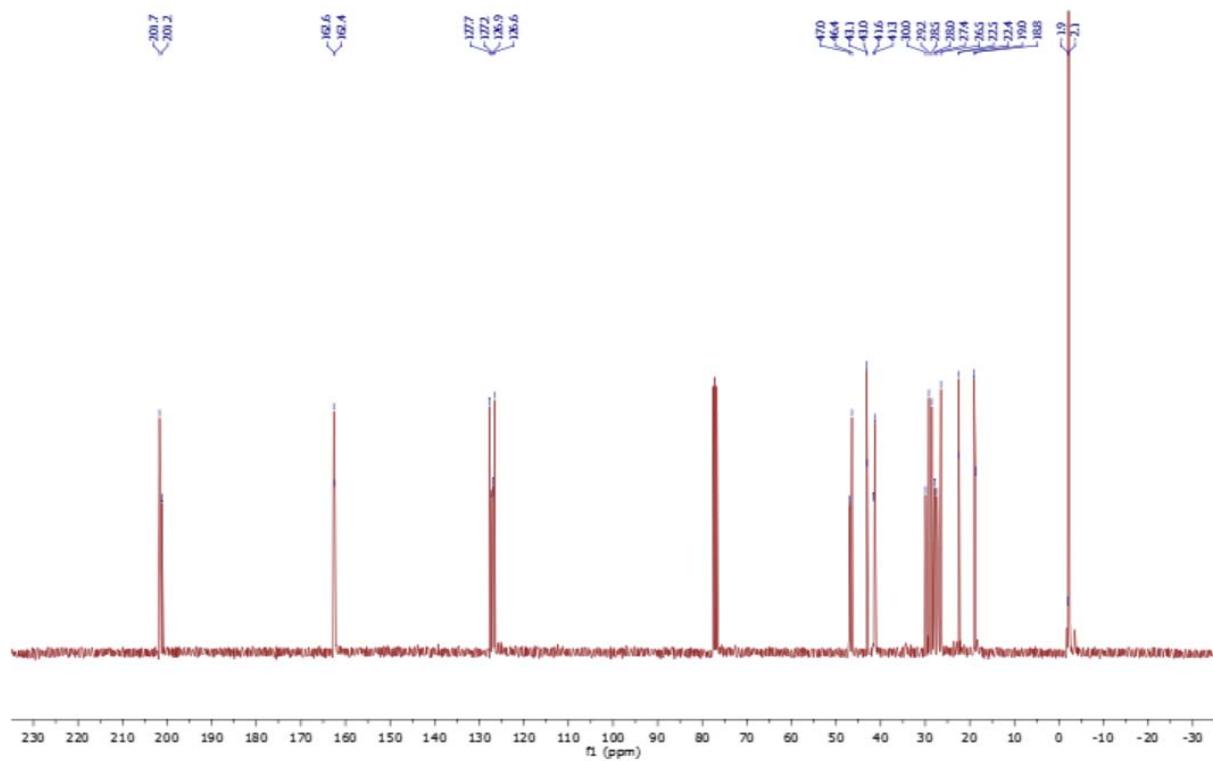


N-(5-Oxopentyl)-N-(6-(trimethylsilyl)hex-4-en-1-yl)formamide (1-14)

¹H RMN spectrum

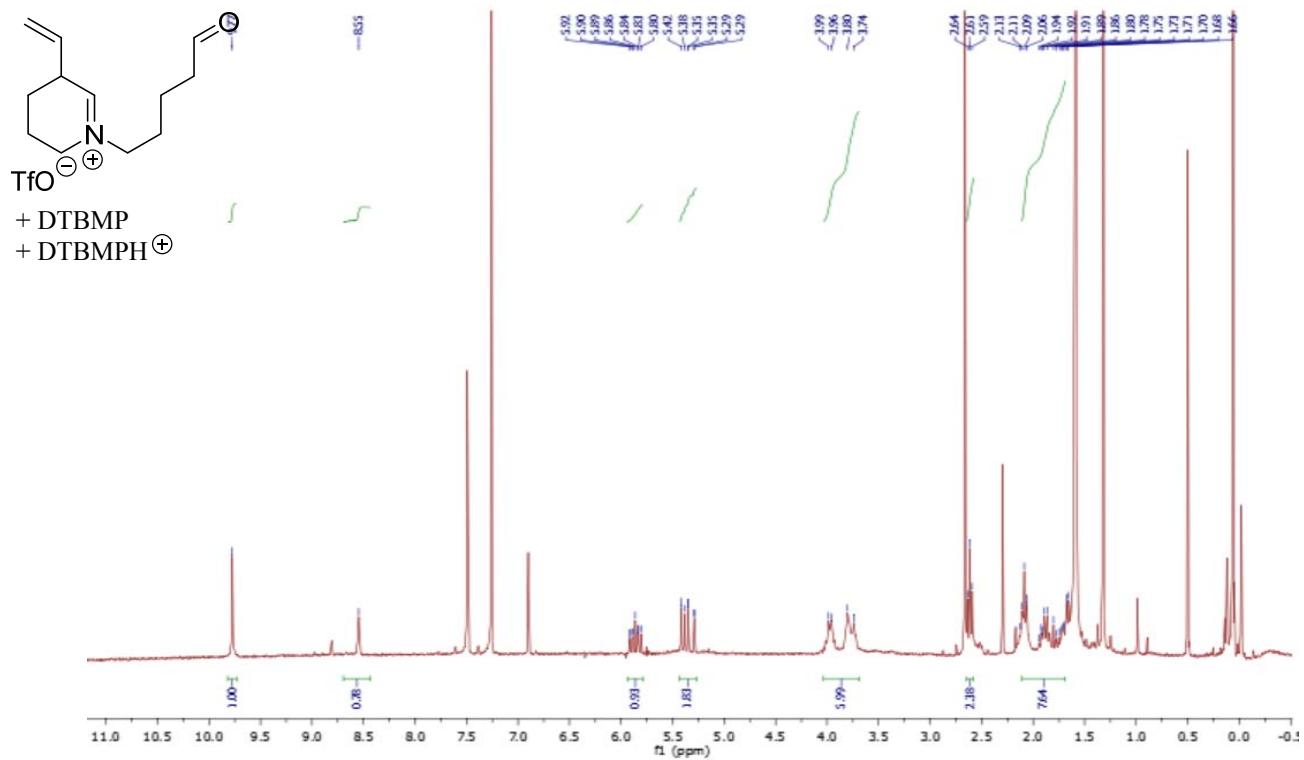


¹³C RMN spectrum



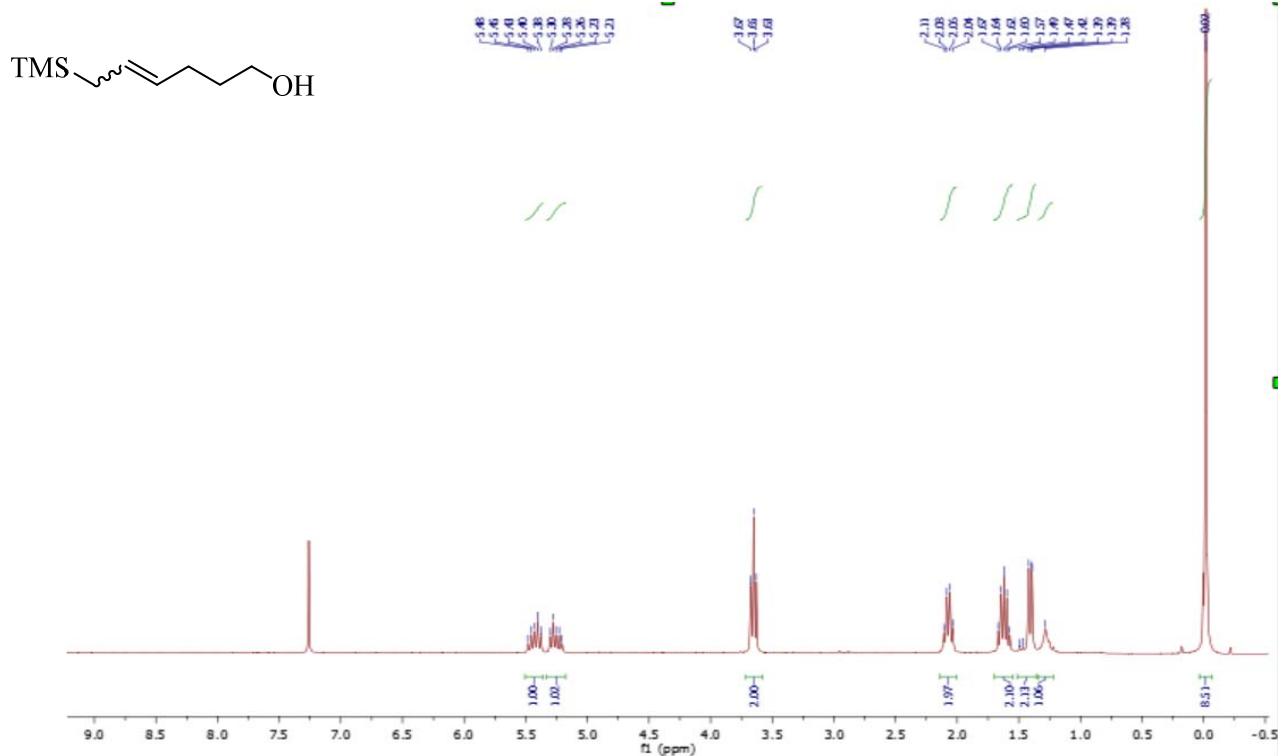
Iminium ion (1-14a)

^1H RMN spectrum

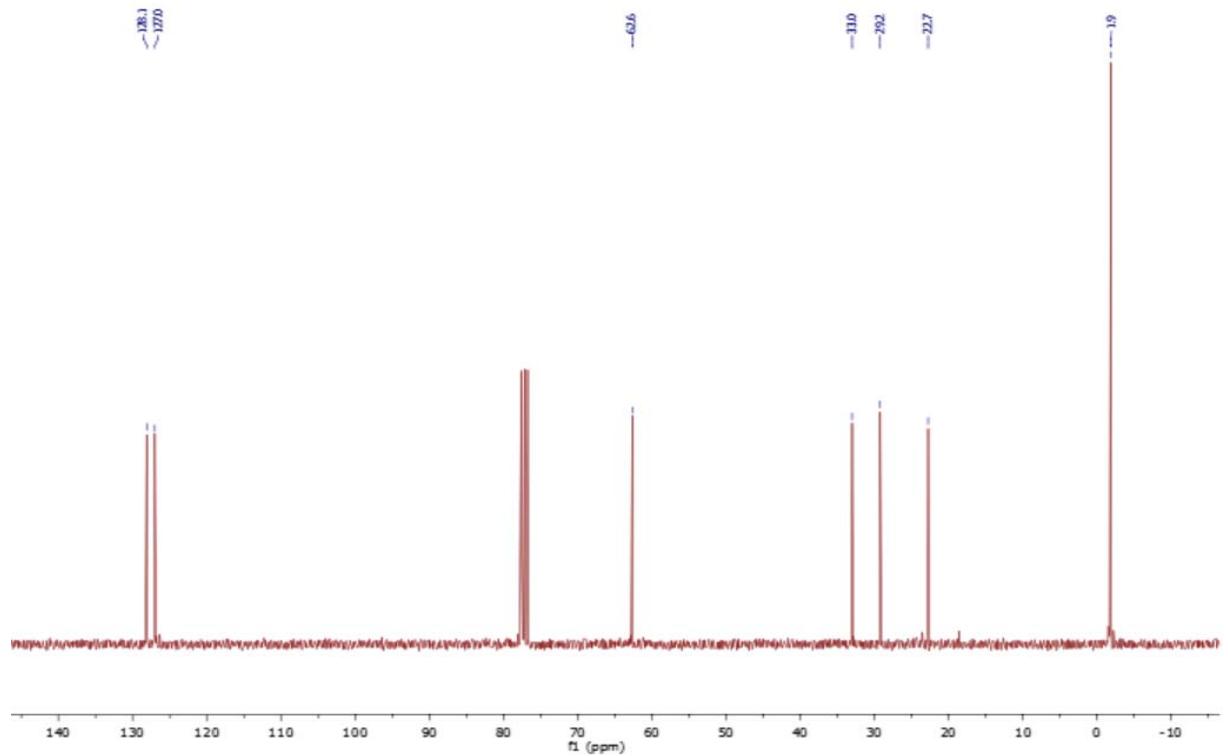


6-(Trimethylsilyl)hex-4-en-1-ol (1-17)

^1H RMN spectrum

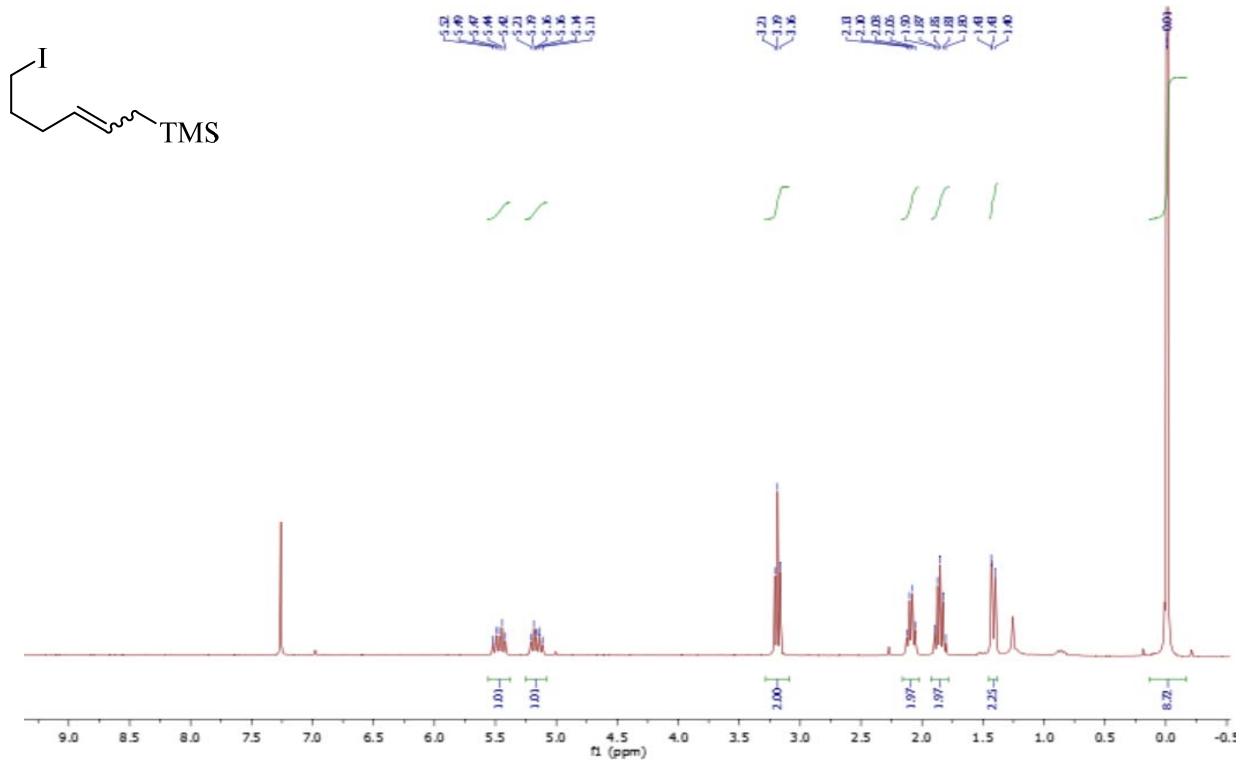


^{13}C RMN spectrum



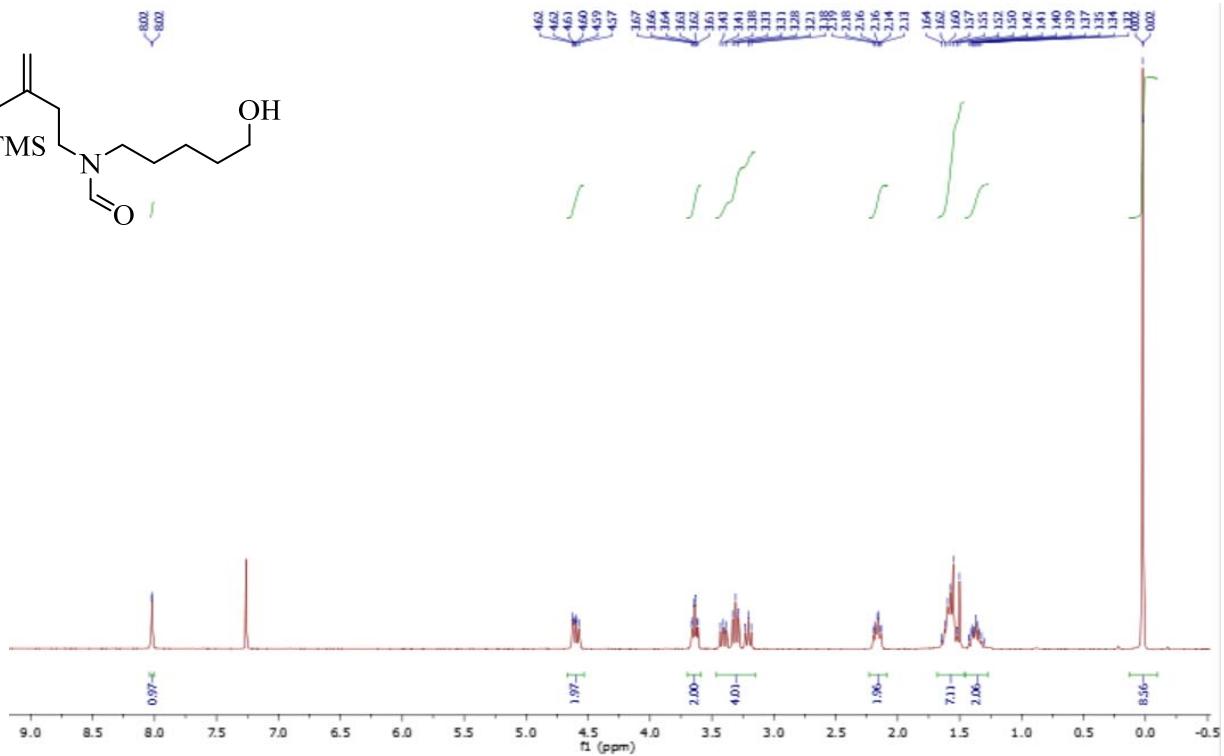
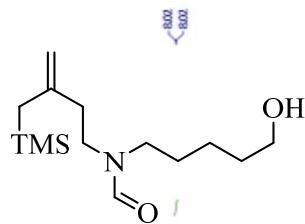
(6-Iodohex-2-en-1-yl)trimethylsilane (1-18)

^1H RMN spectrum

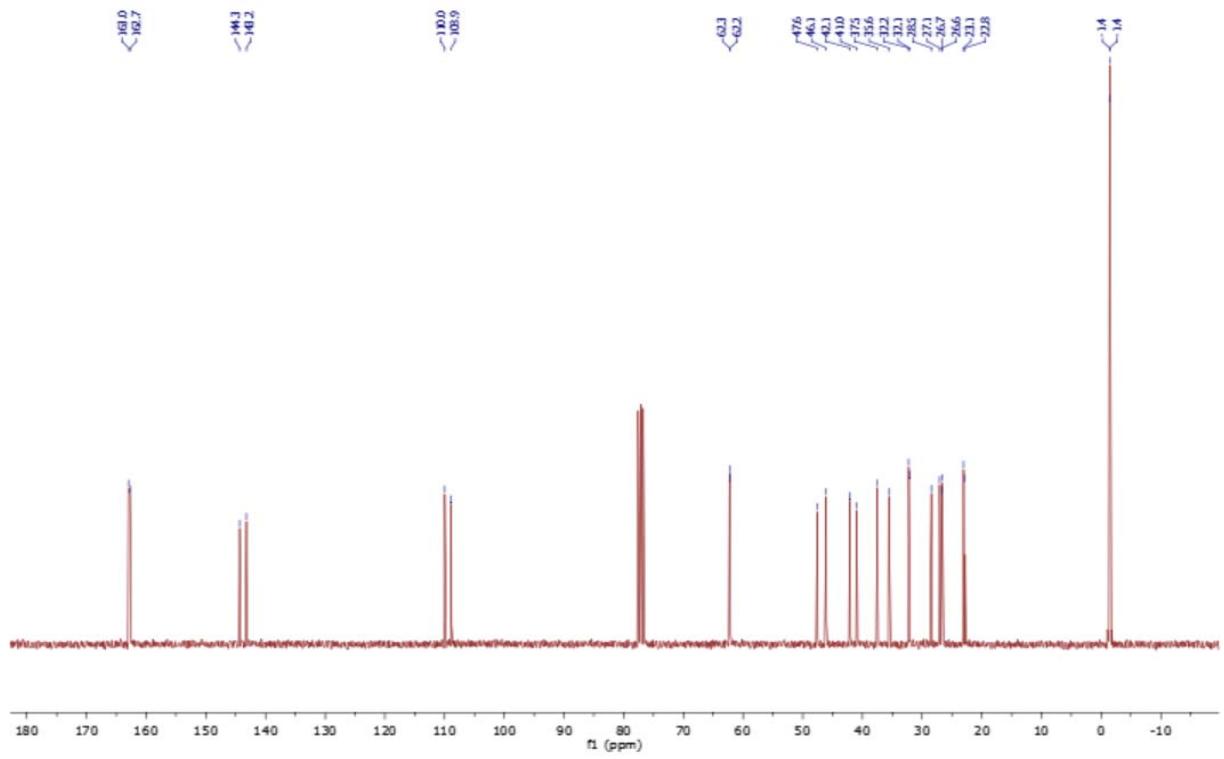


N-(5-Hydroxypentyl)-N-(3-((trimethylsilyl)methyl)but-3-en-1-yl)formamide (1-20)

¹H RMN spectrum

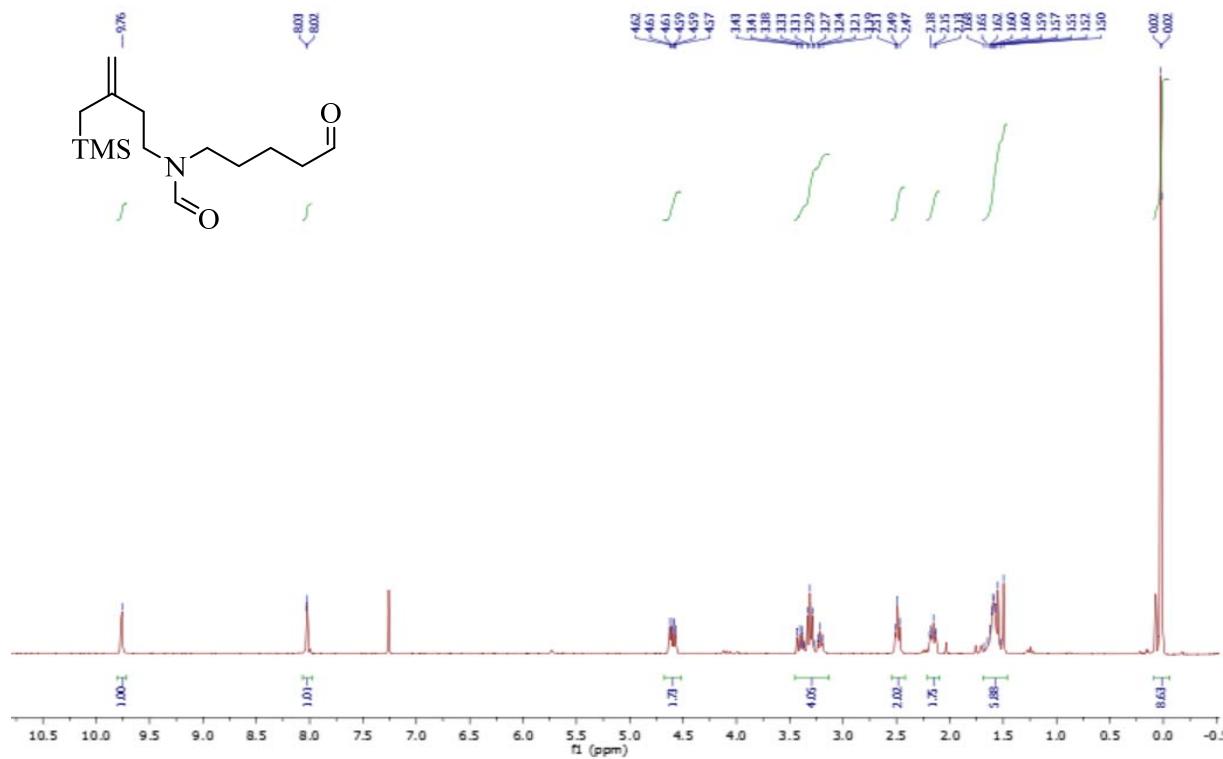


¹³C RMN spectrum



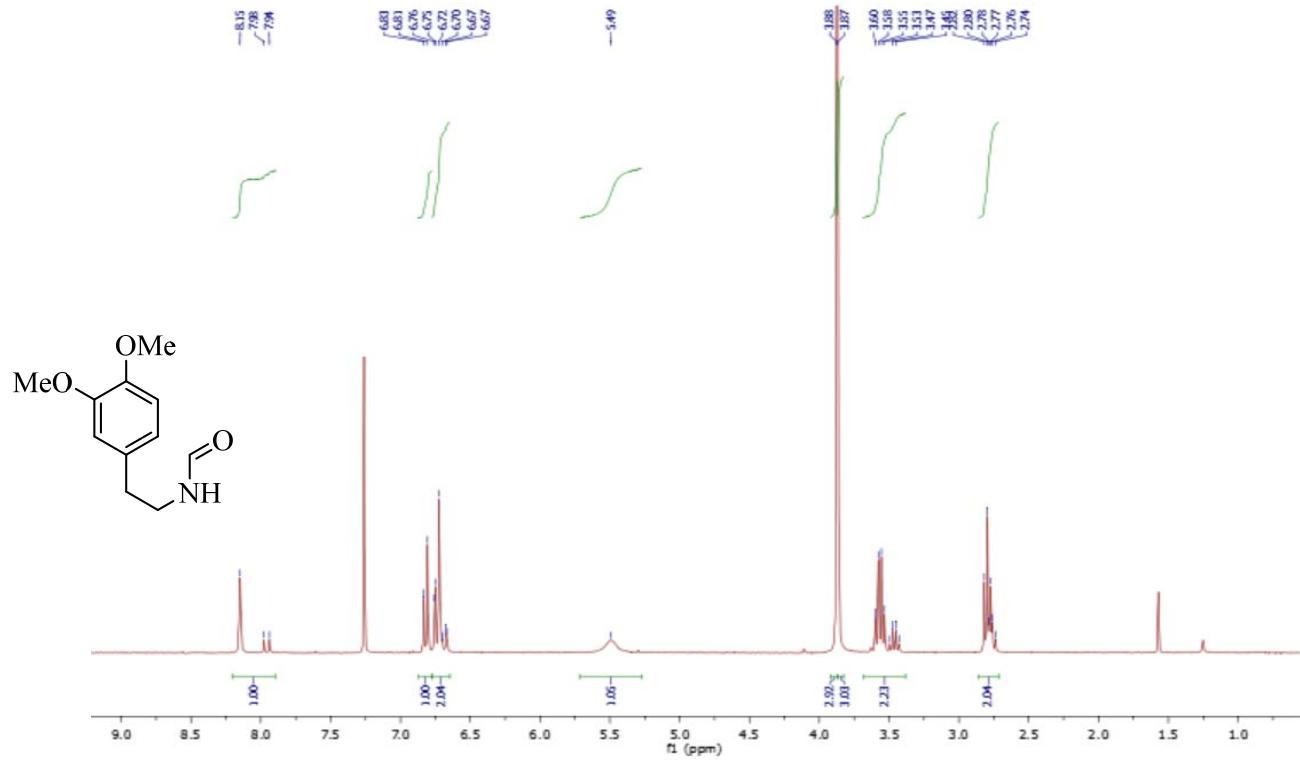
N- (5-oxopentyl)-N-((trimethylsilyl)methyl)but-3-en-1-yl)formamide (1-21)

¹H RMN spectrum

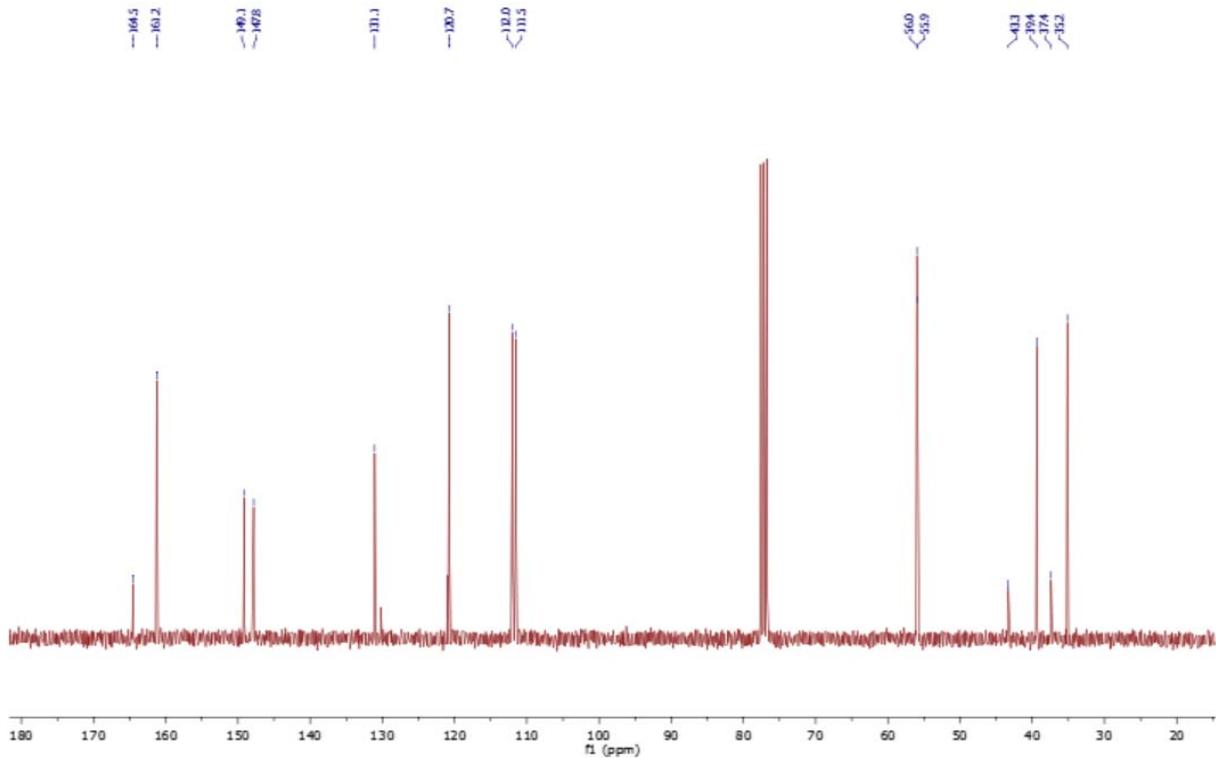


N-(3,4-dimethoxyphenethyl)formamide (1-22)

^1H RMN spectrum

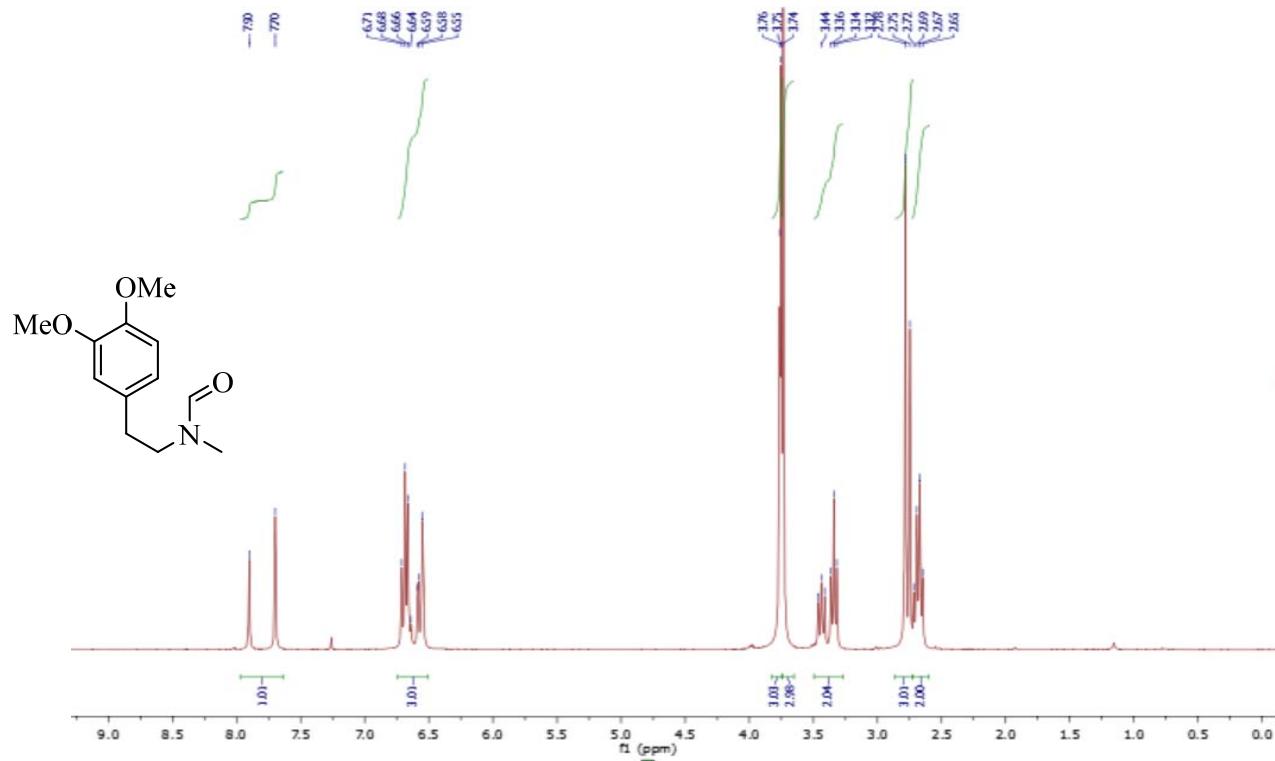


^{13}C RMN spectrum

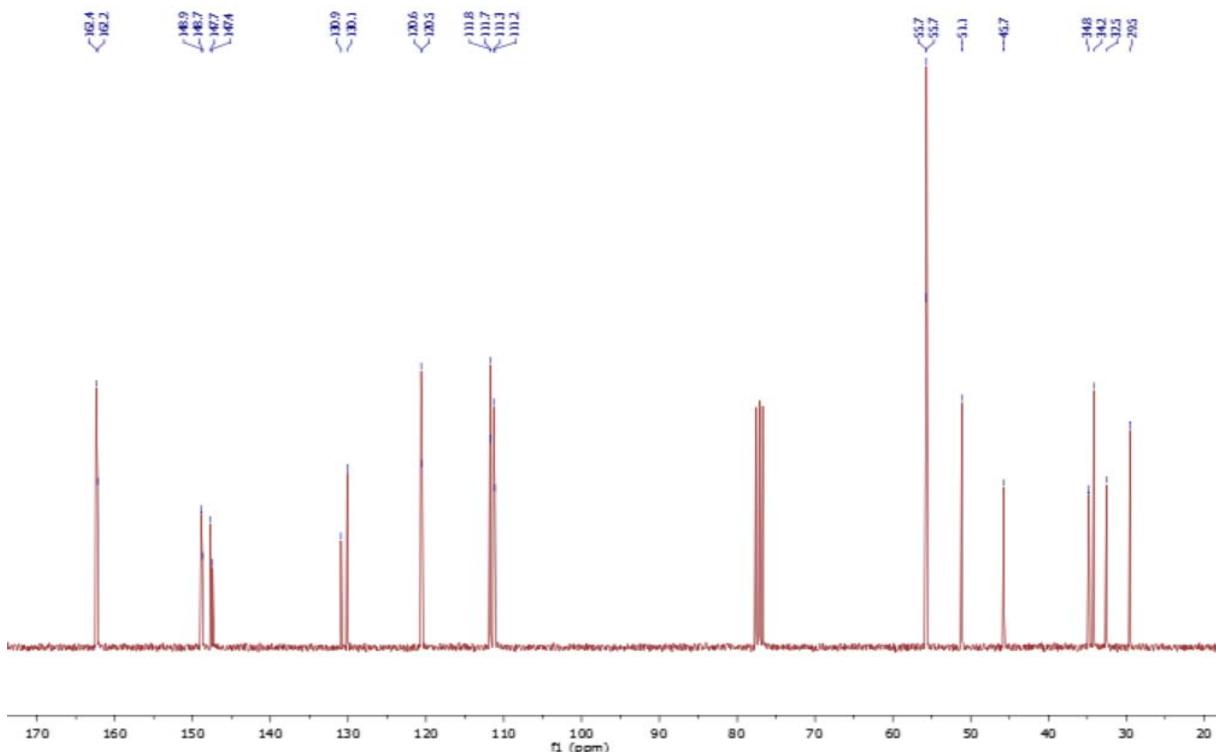


N-(3,4-Dimethoxyphenethyl)-N-methylformamide (1-23)

¹H RMN spectrum

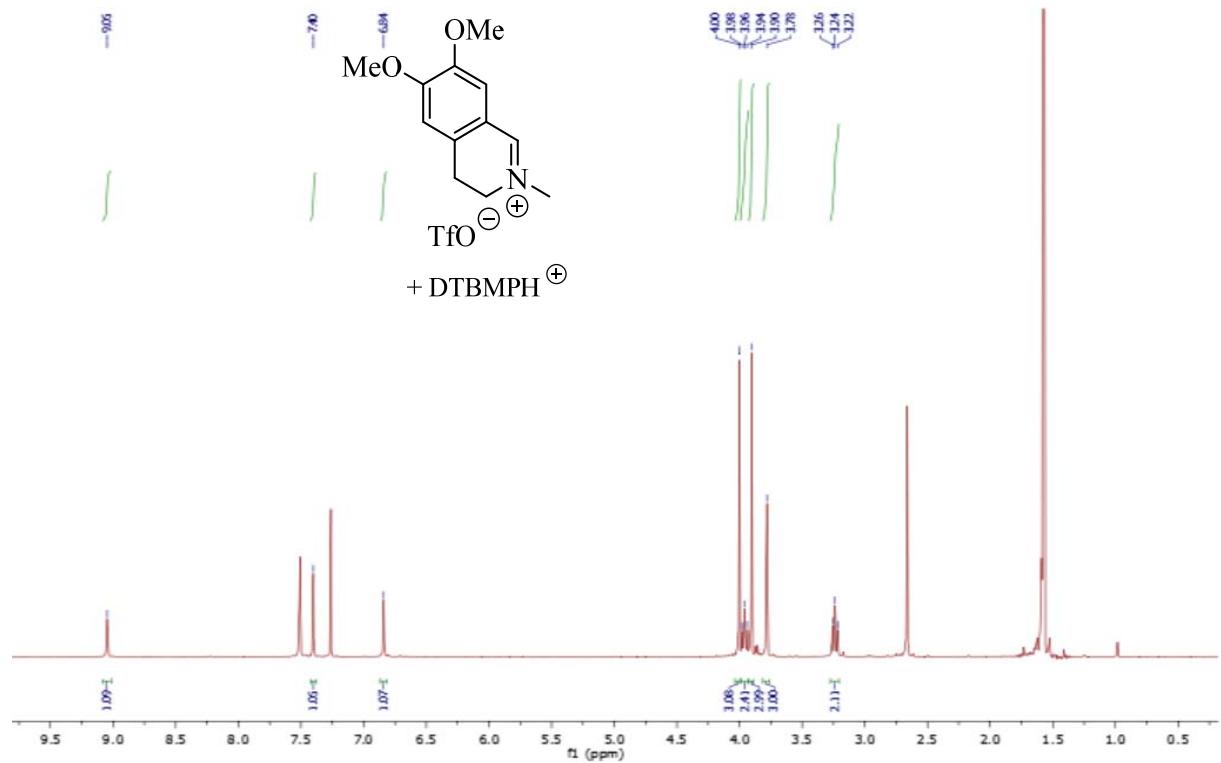


¹³C RMN spectrum



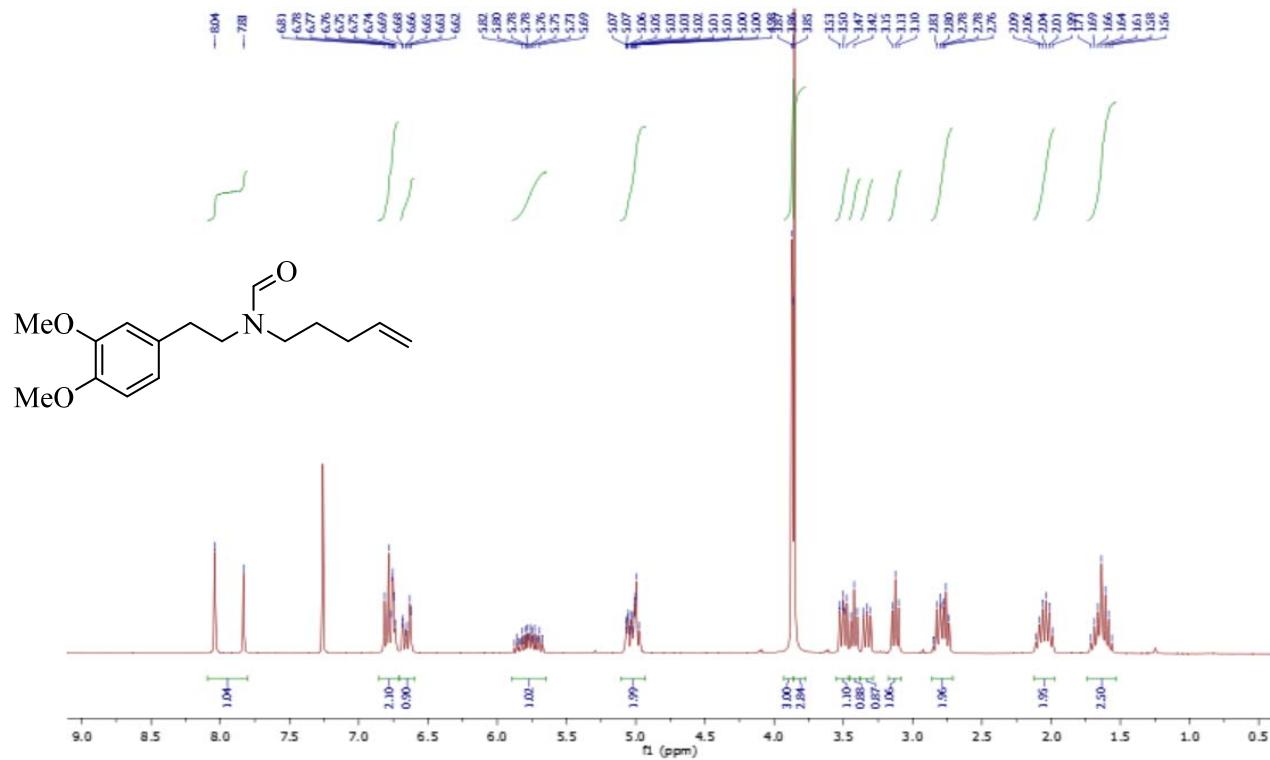
Iminium ion (1-23b)

¹H RMN spectrum

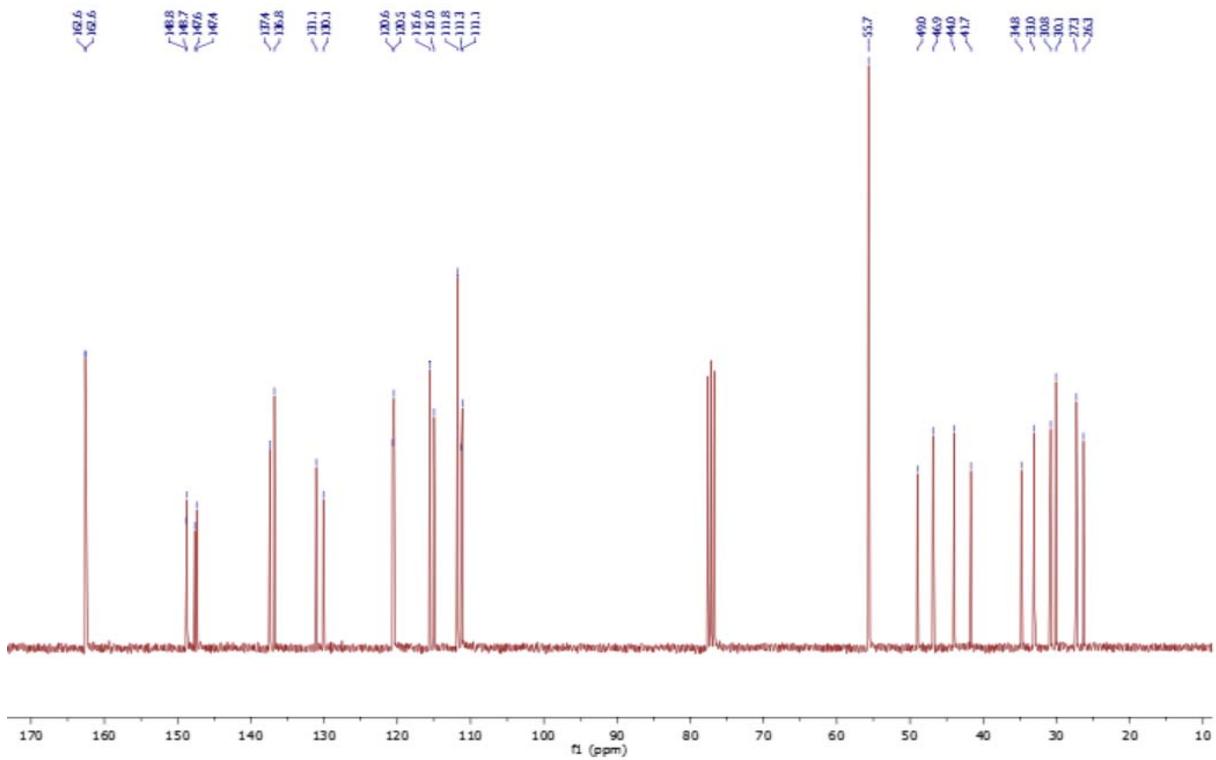


N-(3,4-Dimethoxyphenethyl)-N-(pent-4-en-1-yl)formamide (1-24)

¹H RMN spectrum

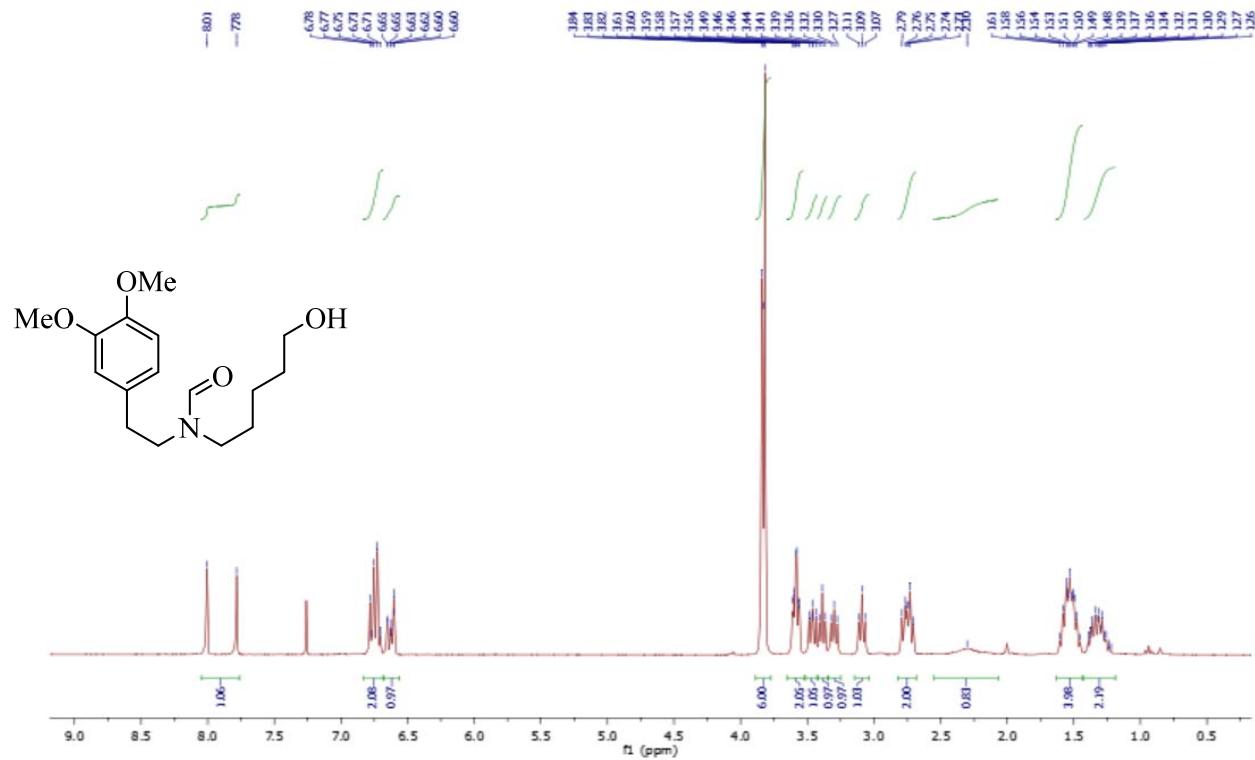


¹³C RMN spectrum

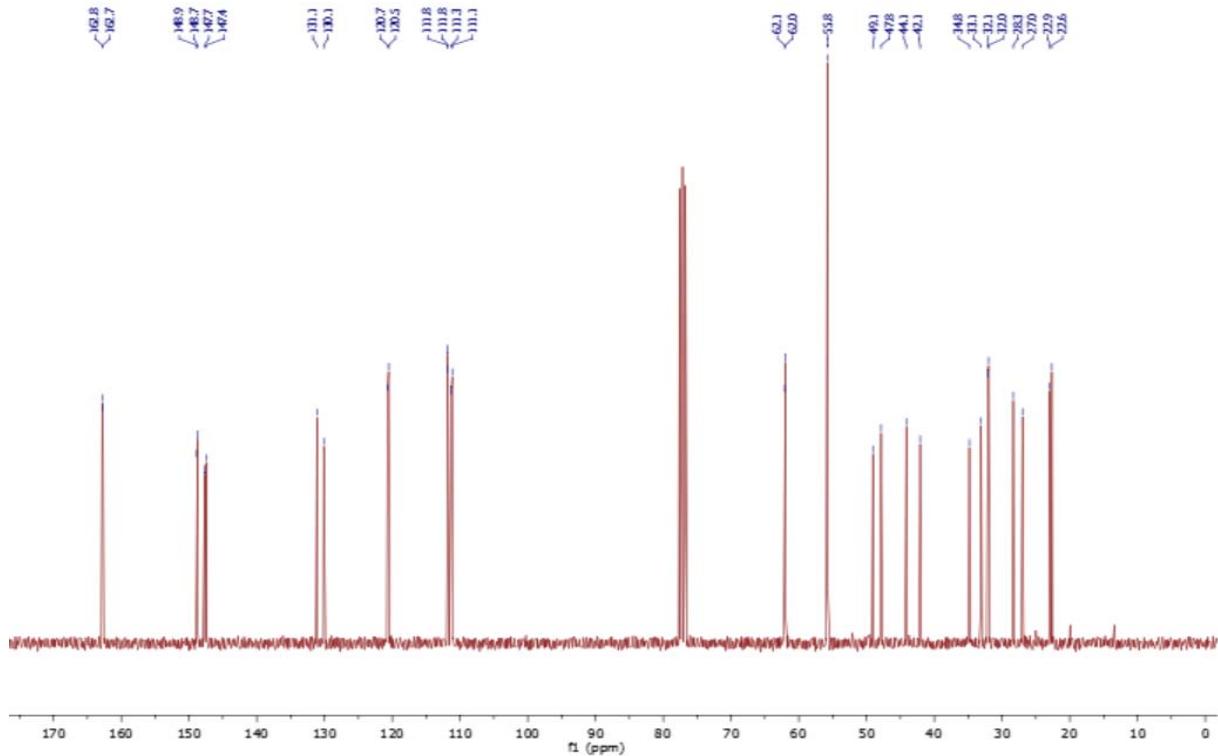


N-(3,4-Dimethoxyphenethyl)-N-(5-hydroxypentyl)formamide (1-25)

¹H RMN spectrum

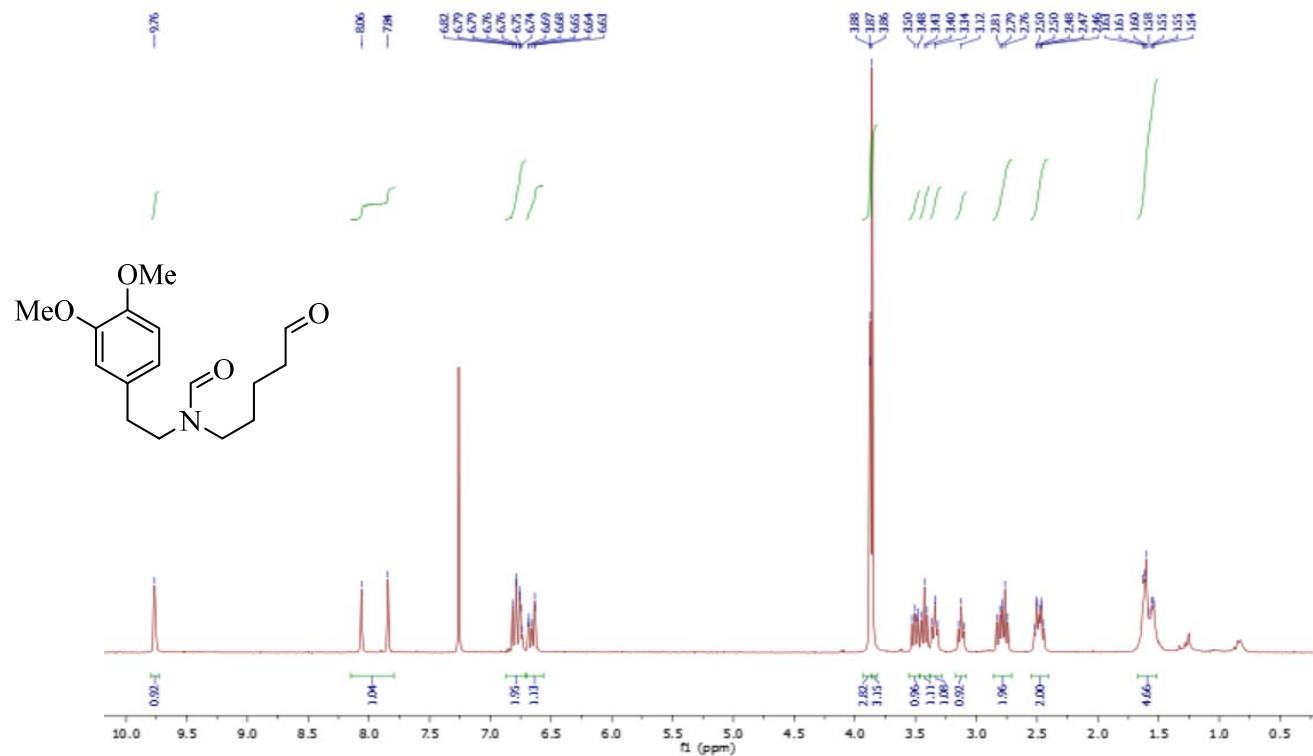


¹³C RMN spectrum

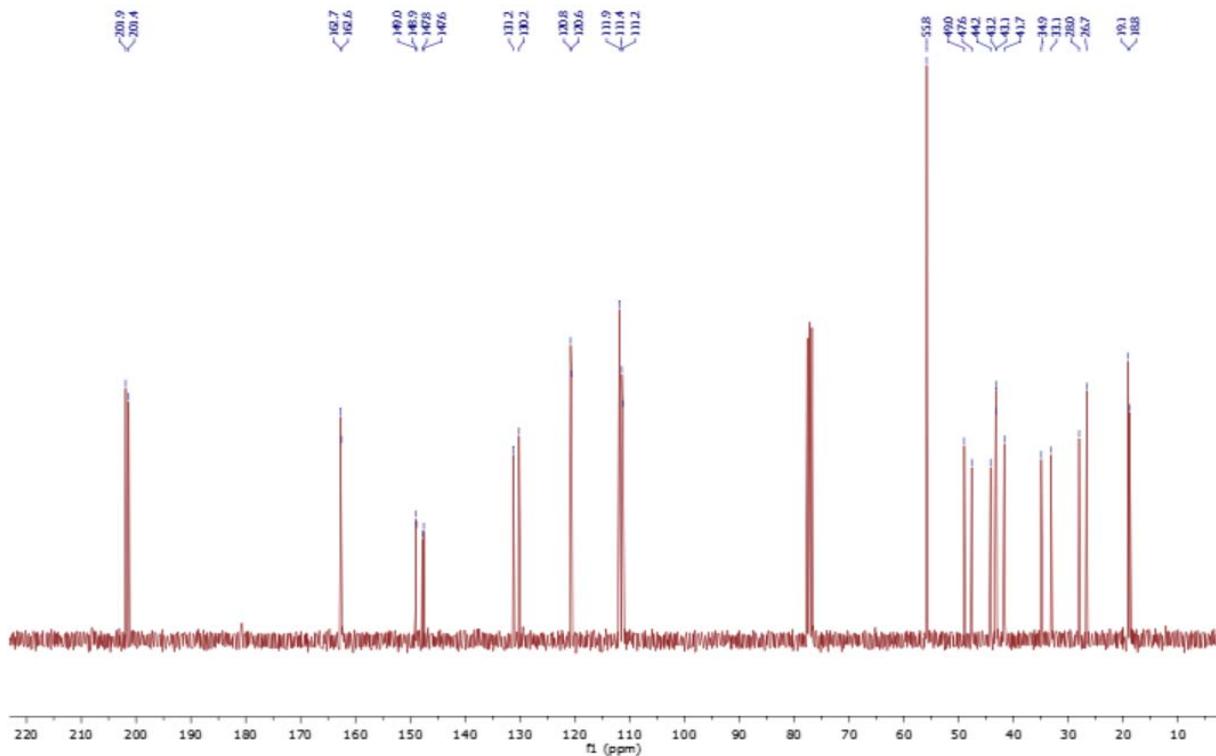


N-(3,4-Dimethoxyphenethyl)-N-(5-oxopentyl)formamide (1-26)

¹H RMN spectrum

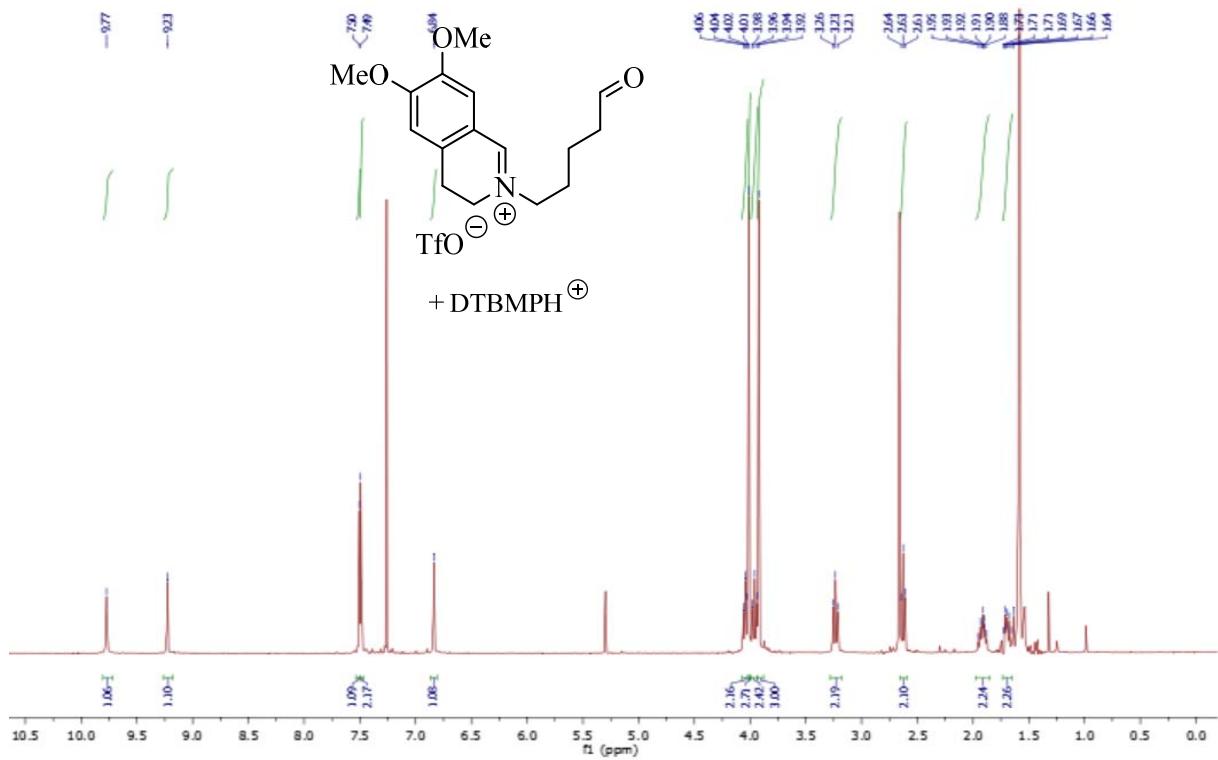


¹³C RMN spectrum



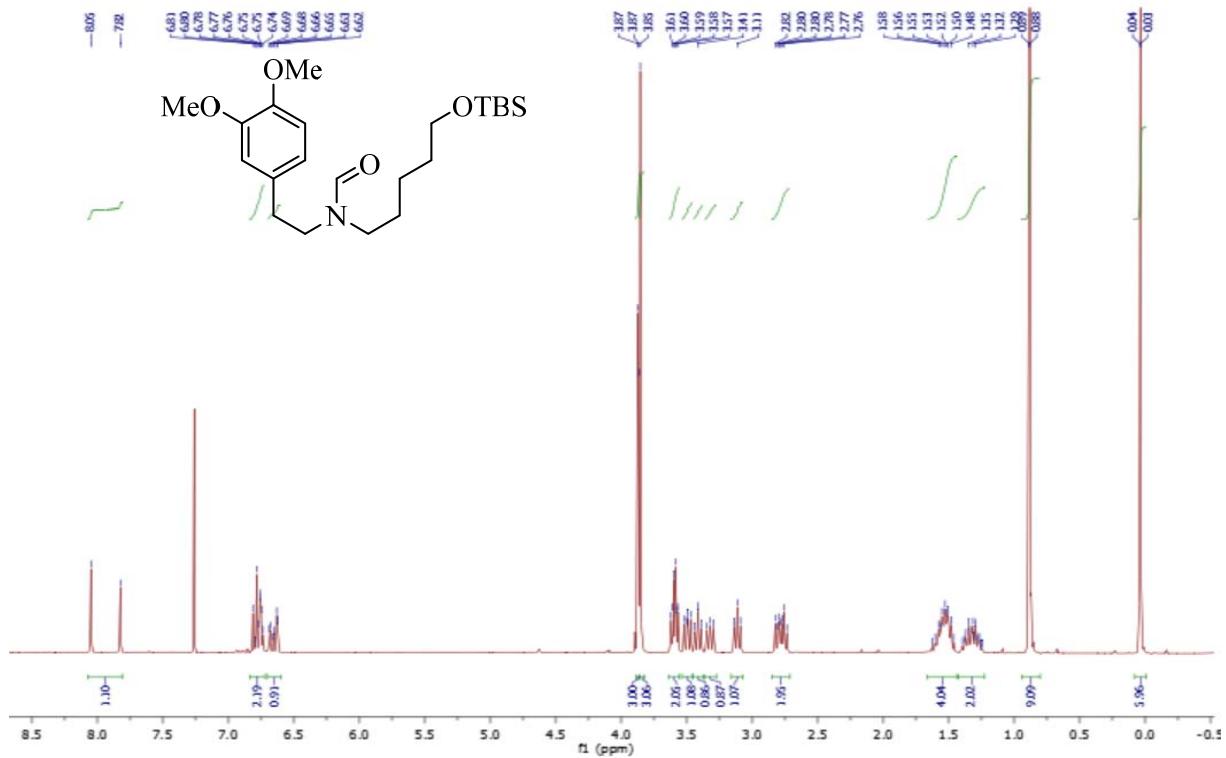
Iminium ion (1-26a)

^1H RMN spectrum

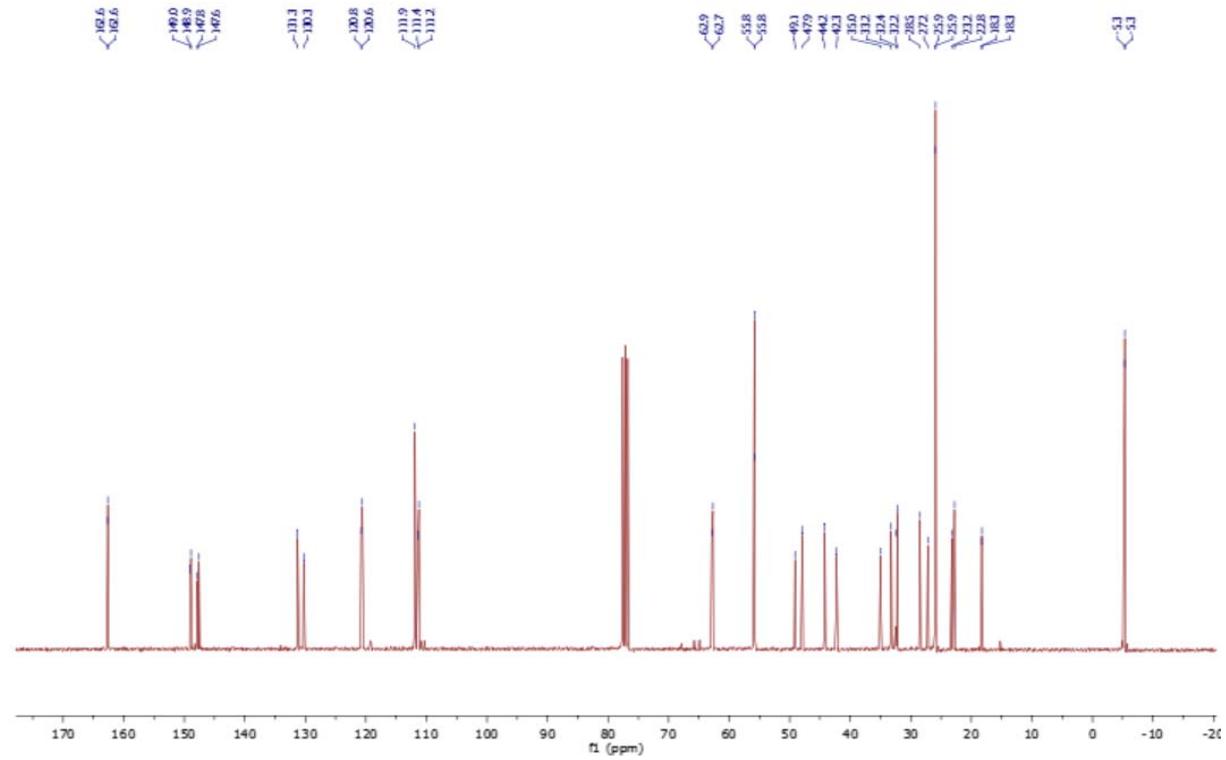


N-(5-((tert-Butyldimethylsilyl)oxy)pentyl)-N-(3,4-dimethoxyphenethyl)formamide (1-28)

¹H RMN spectrum

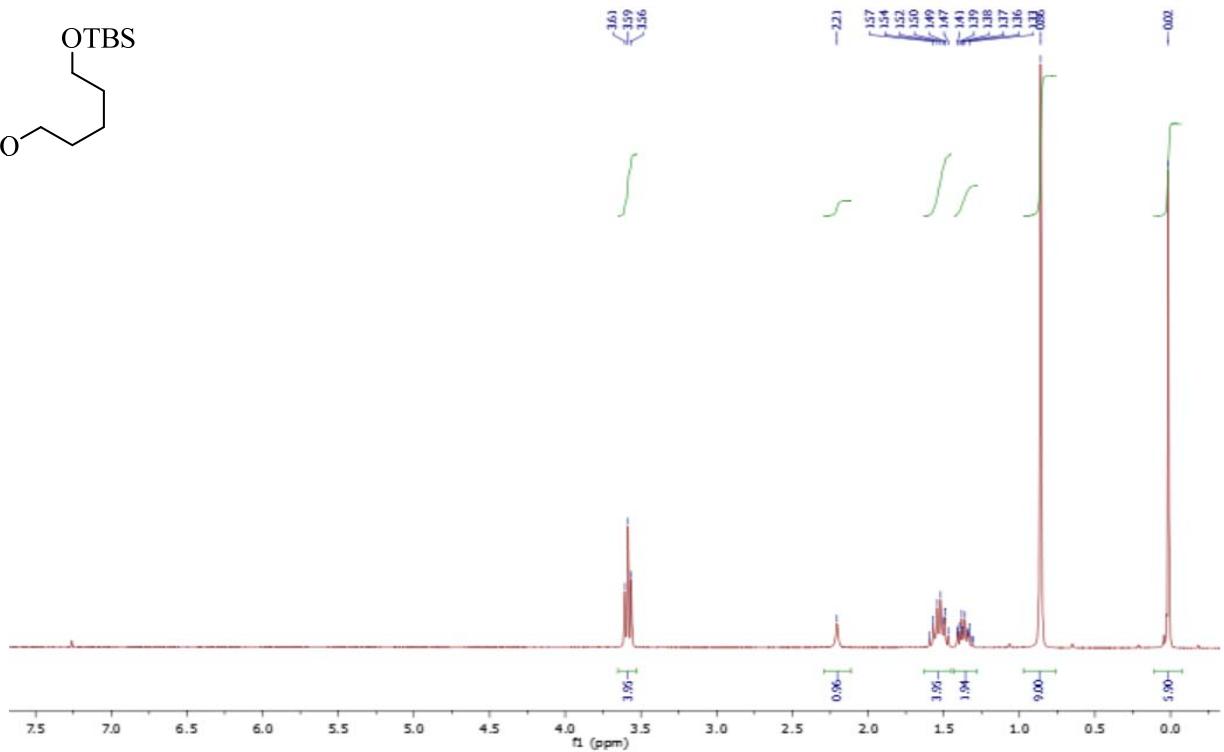
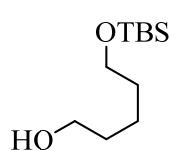


¹³C RMN spectrum

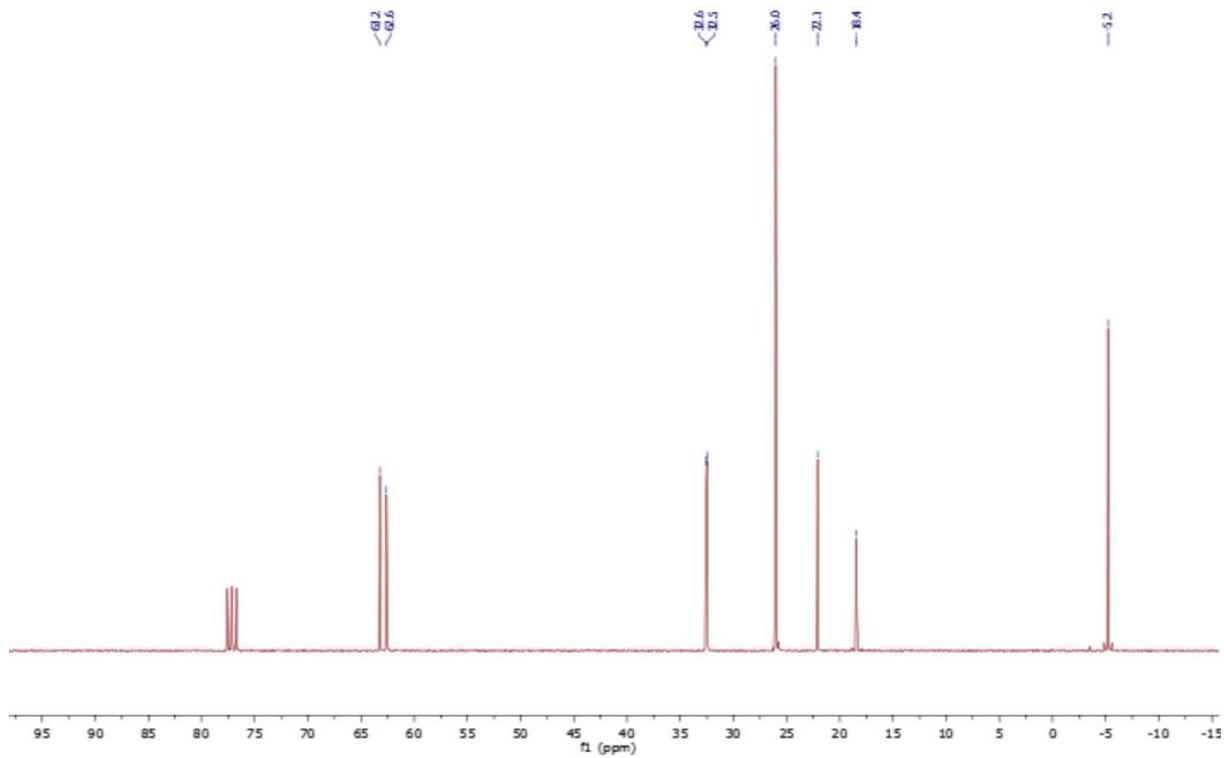


5-((*tert*-Butyldimethylsilyl)oxy)pentan-1-ol (1-29)

¹H RMN spectrum

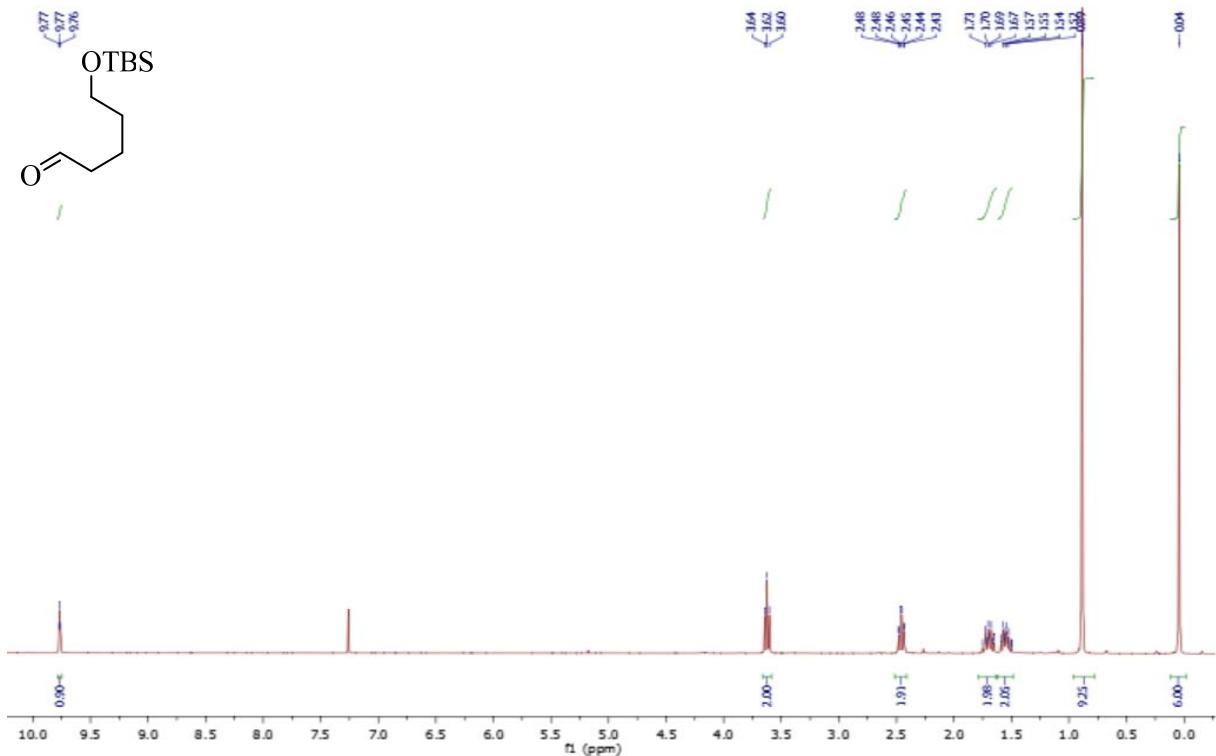


¹³C RMN spectrum

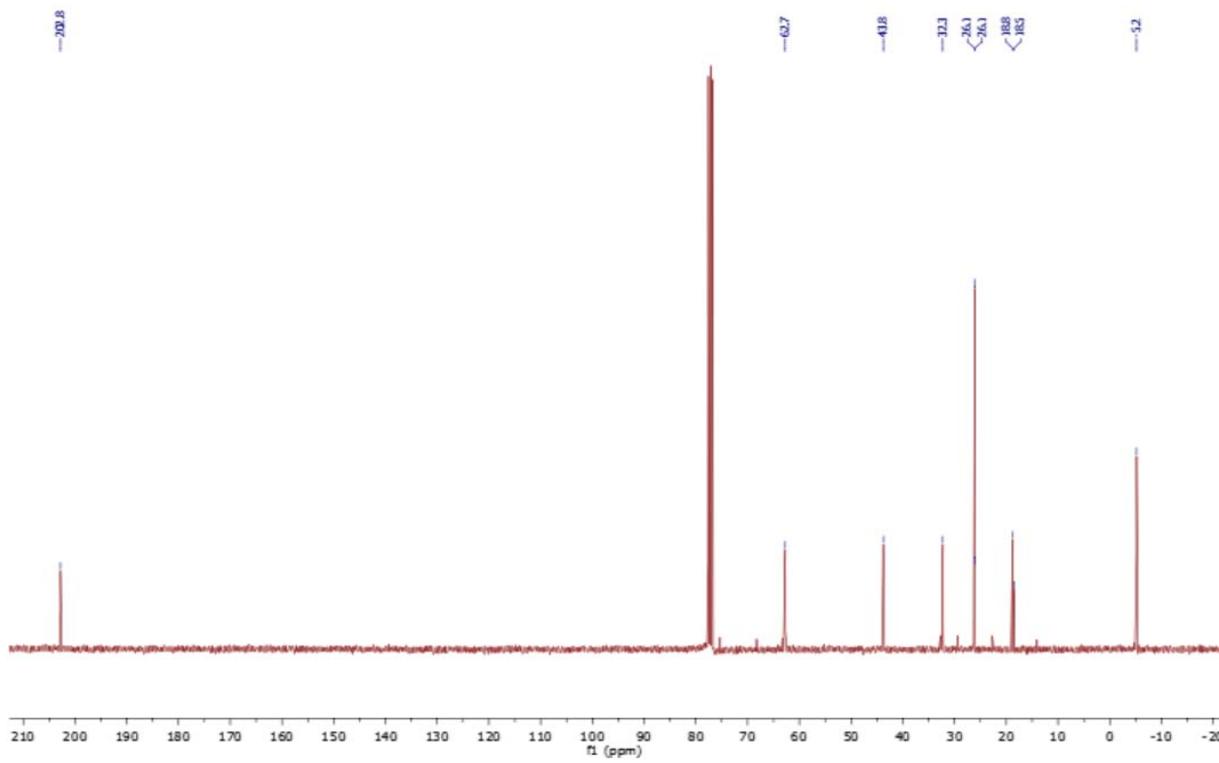


5-((*tert*-Butyldimethylsilyl)oxy)pentanal (1-30)

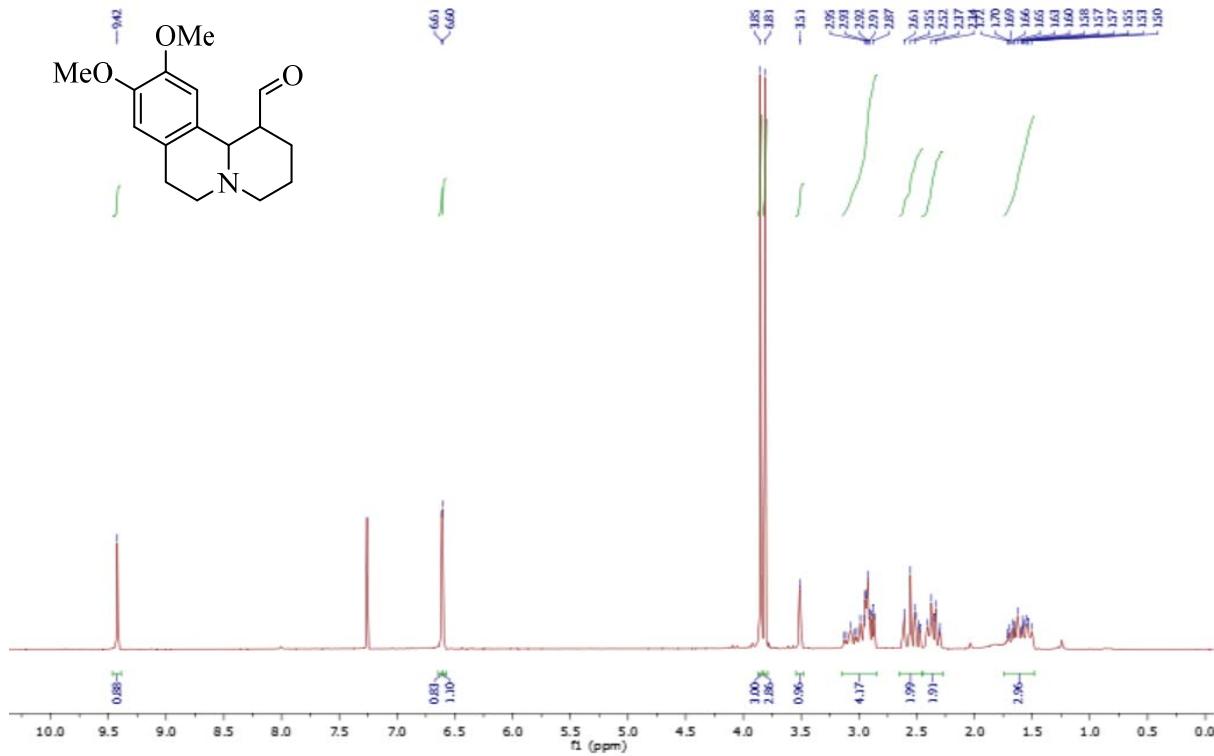
^1H RMN spectrum



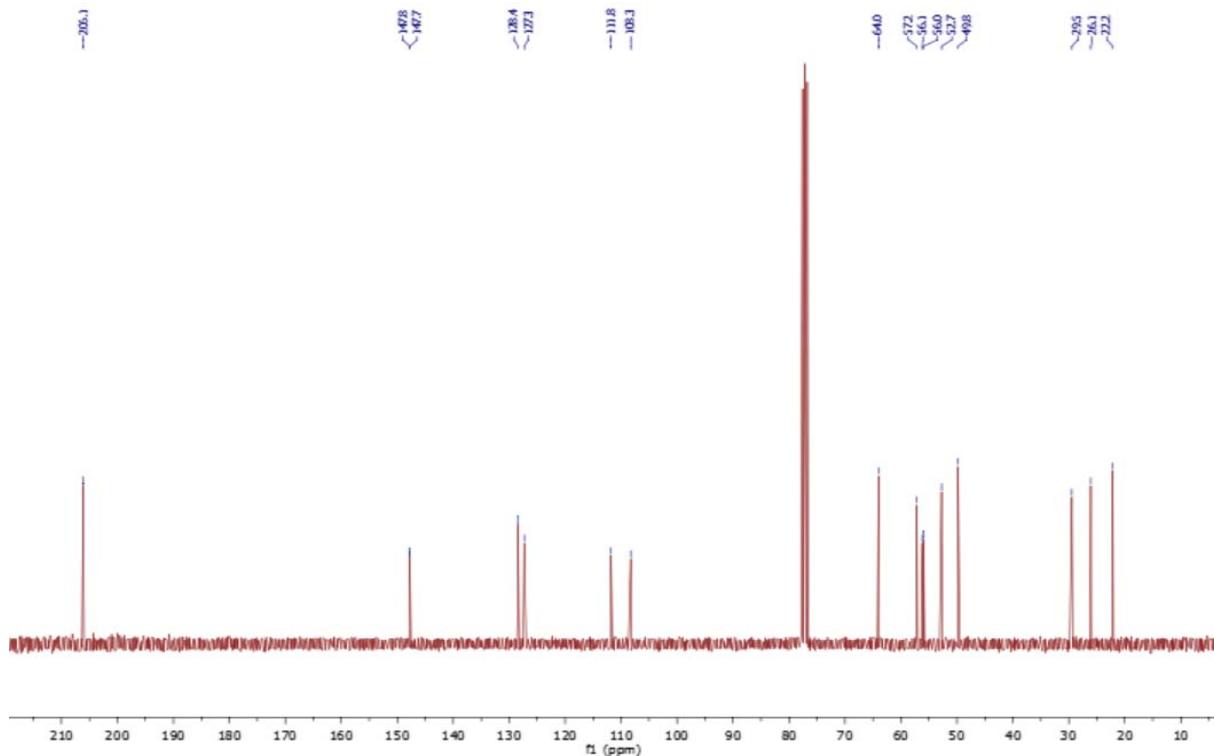
^{13}C RMN spectrum



9,10-Dimethoxy-1,3,4,6,7,11b-hexahydro-2*H*-pyrido[2,1-*a*]isoquinoline-1-carbaldehyde (1-31)
¹H RMN spectrum

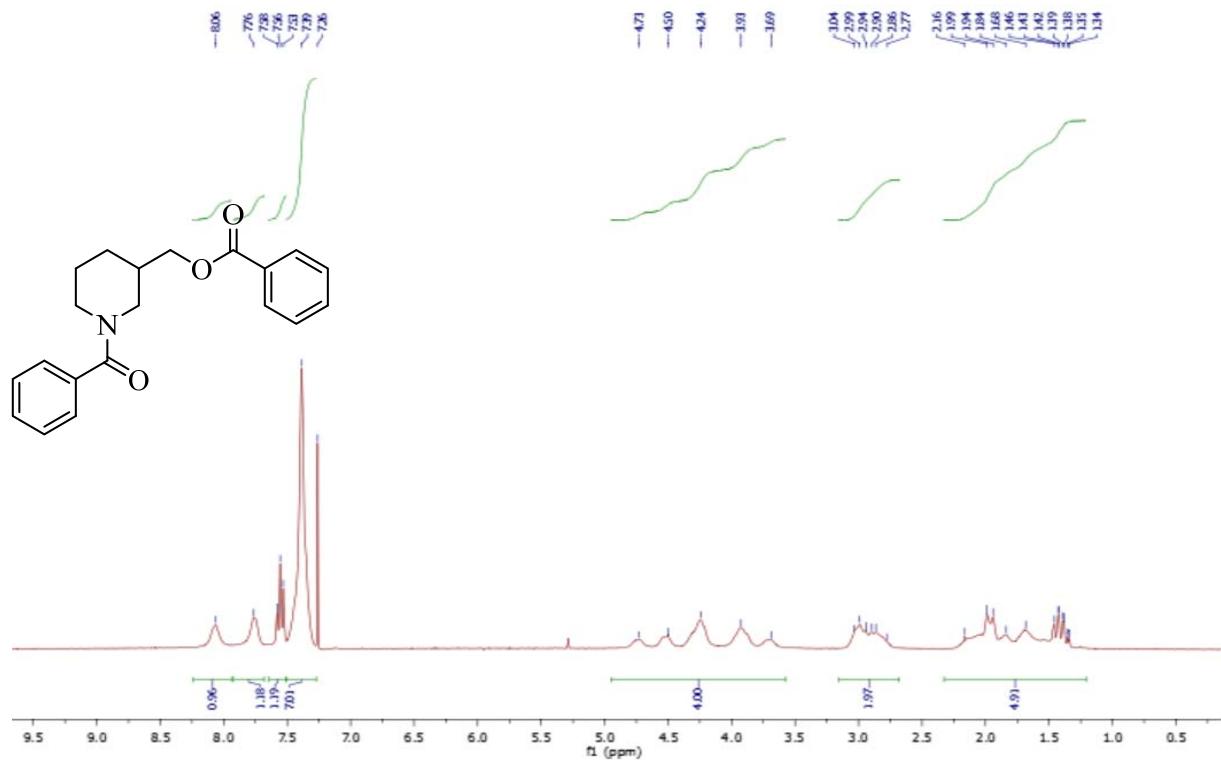


¹³C RMN spectrum

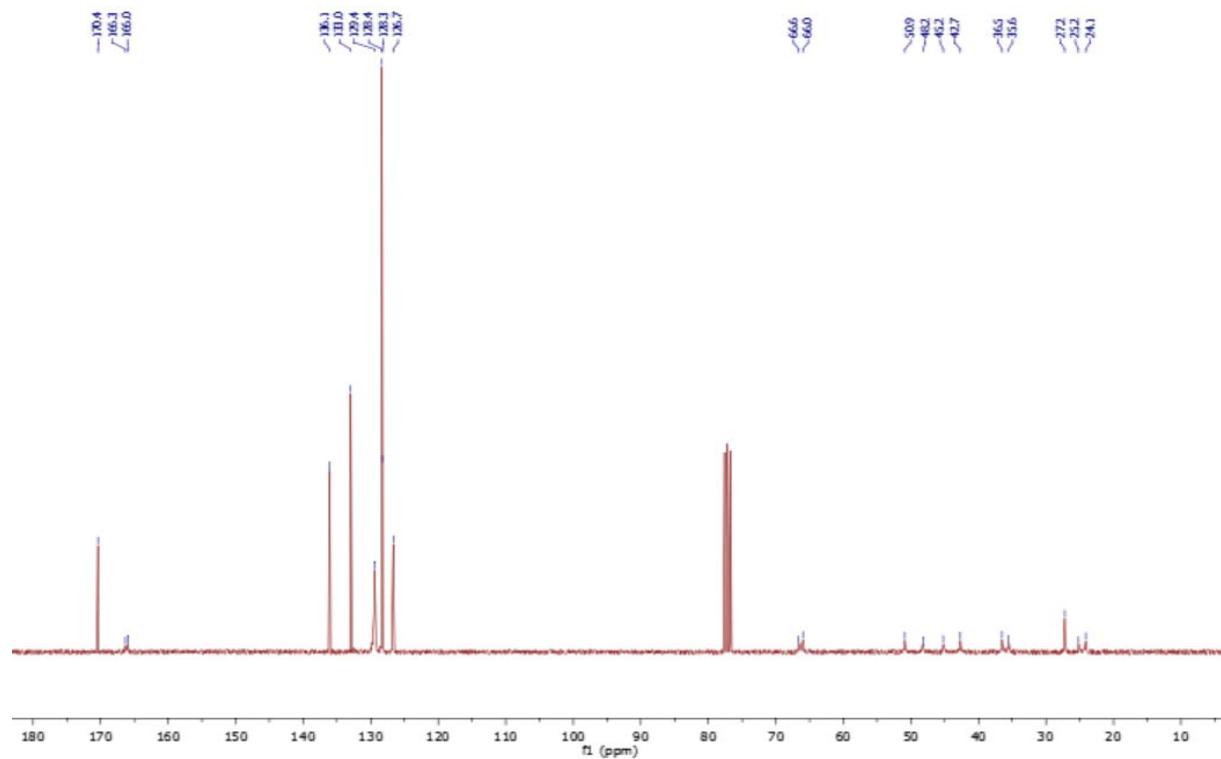


(1-Benzoylpiperidin-3-yl)methyl benzoate (1-33)

¹H RMN spectrum

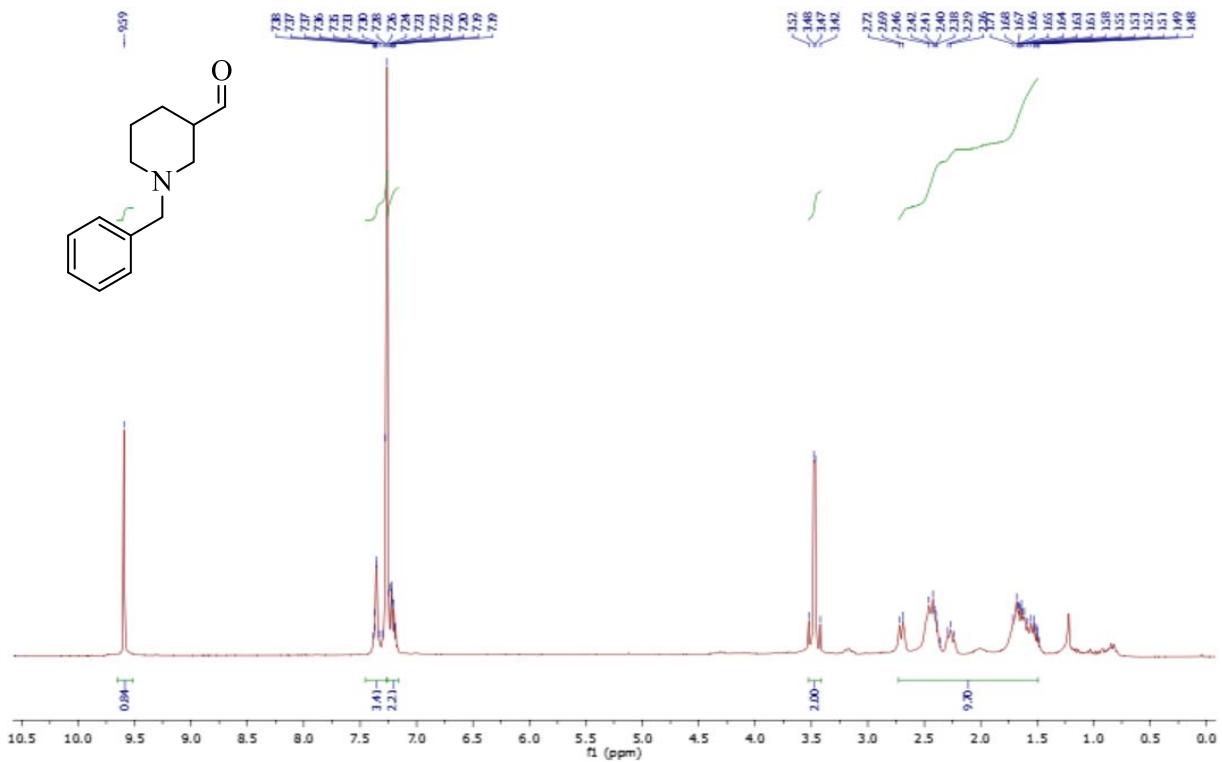


¹³C RMN spectrum

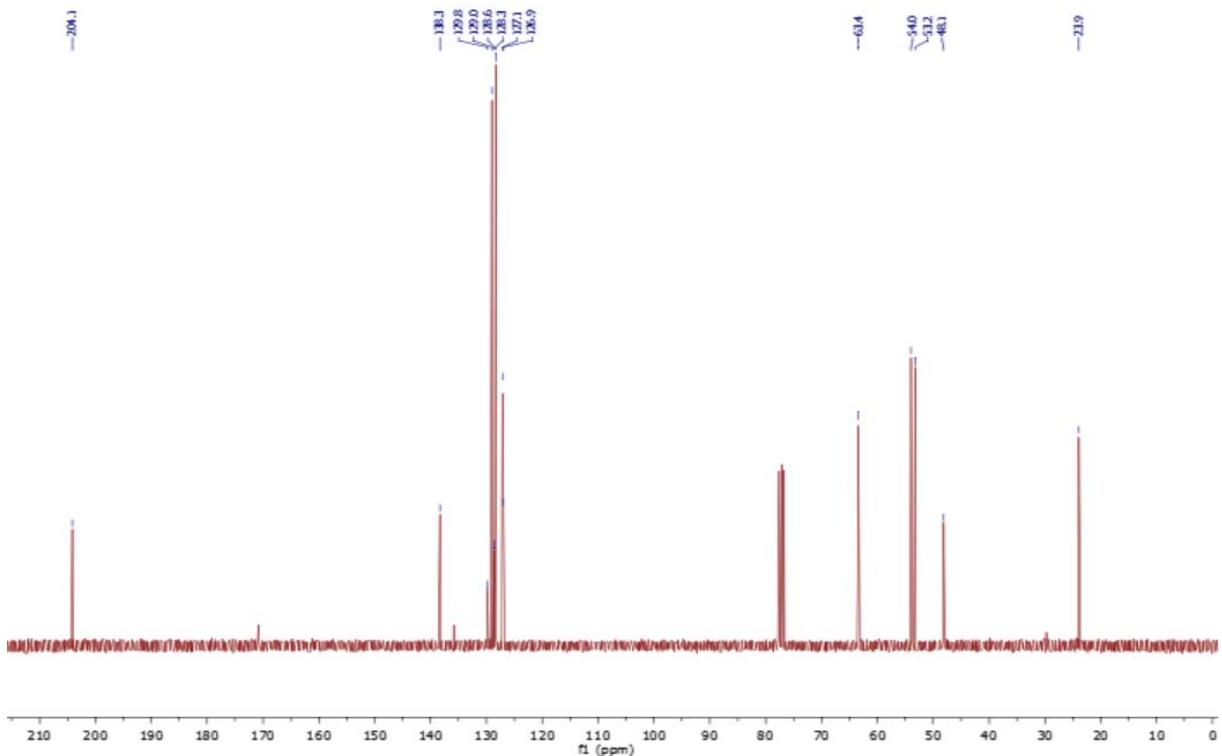


1-Benzylpiperidine-3-carbaldehyde (1-35)

^1H RMN spectrum

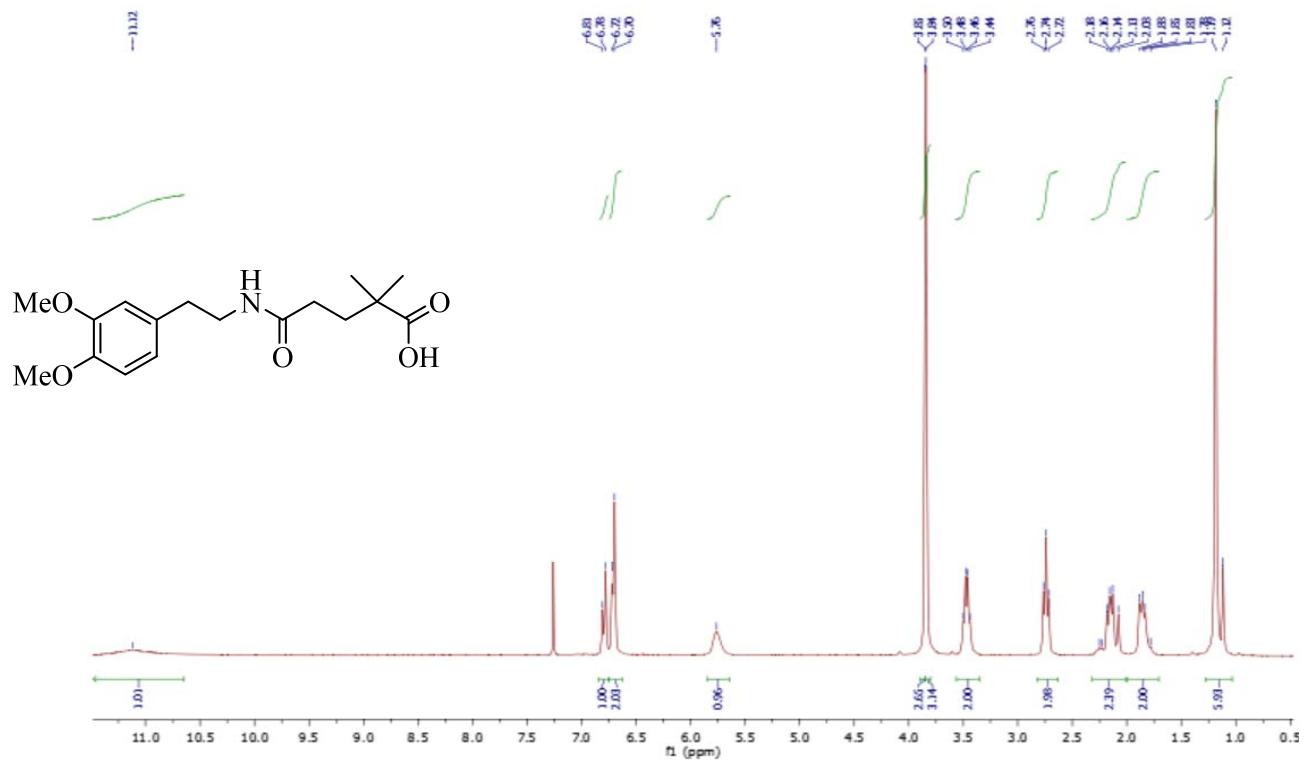


^{13}C RMN spectrum

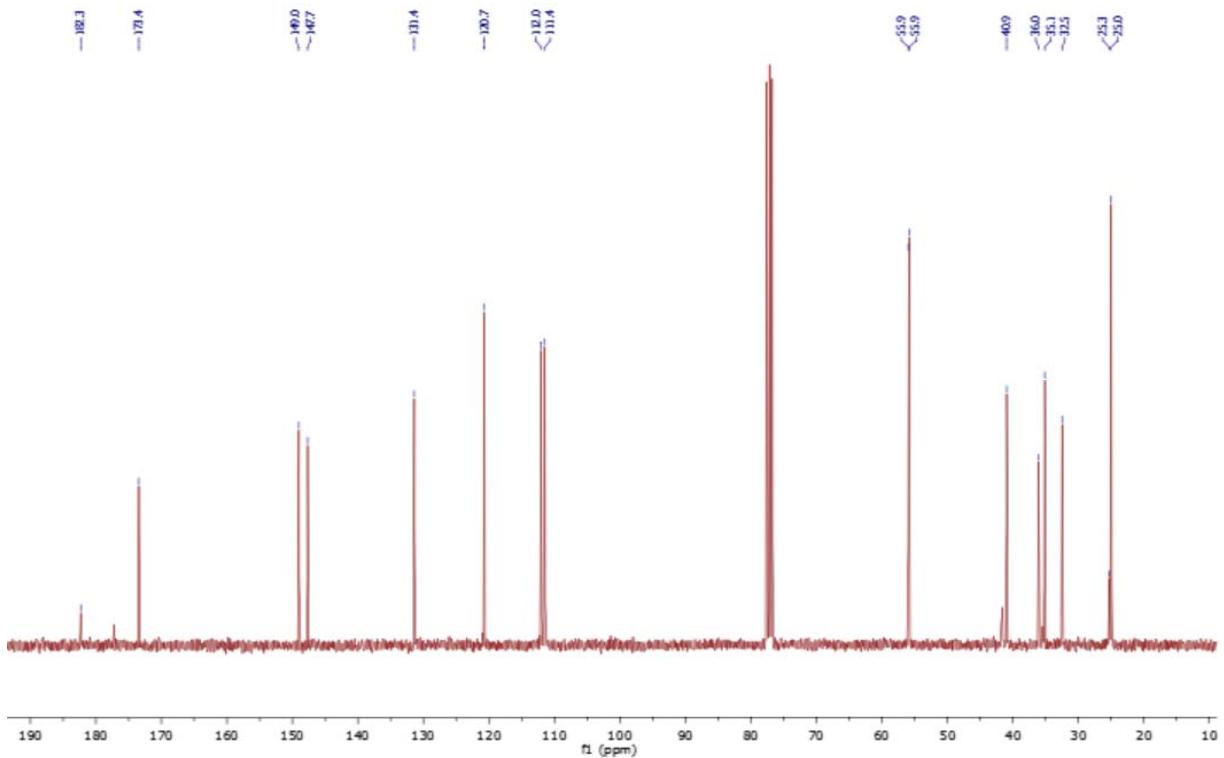


5-((3,4-Dimethoxyphenethyl)amino)-2,2-dimethyl-5-oxopentanoic acid (1-39a)

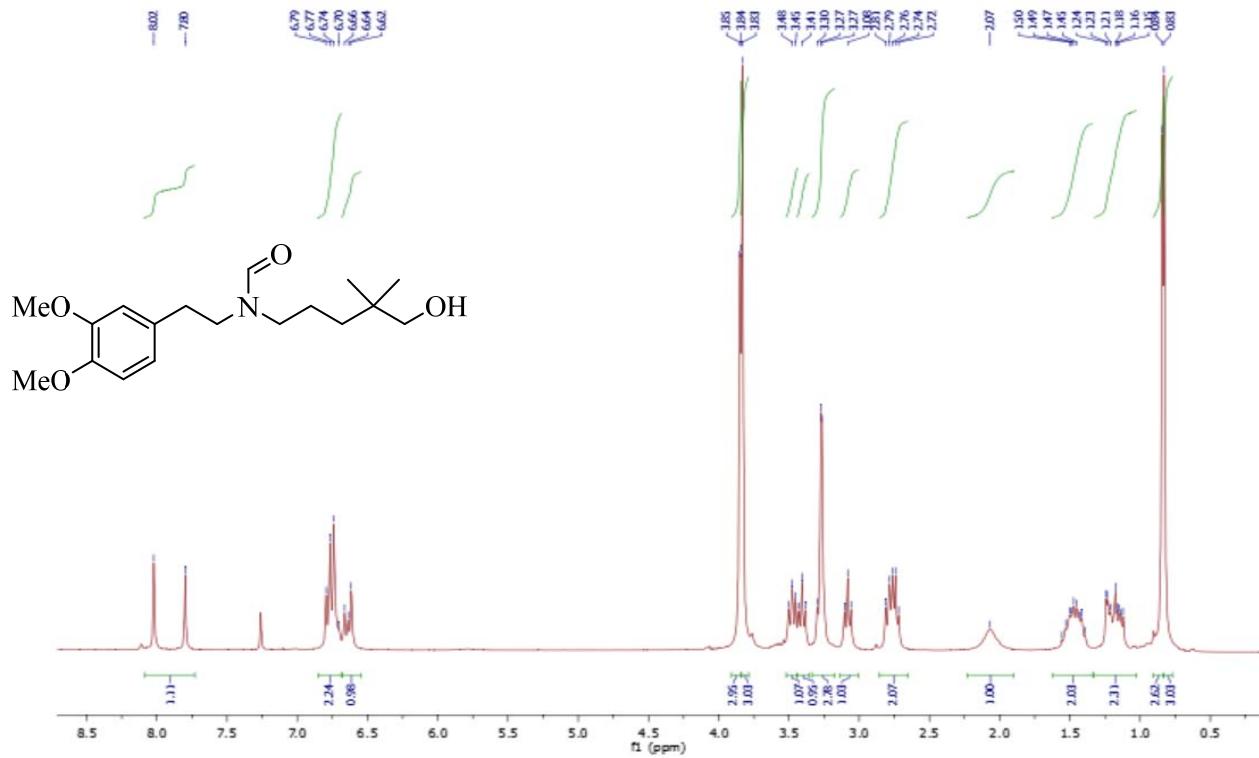
^1H RMN spectrum



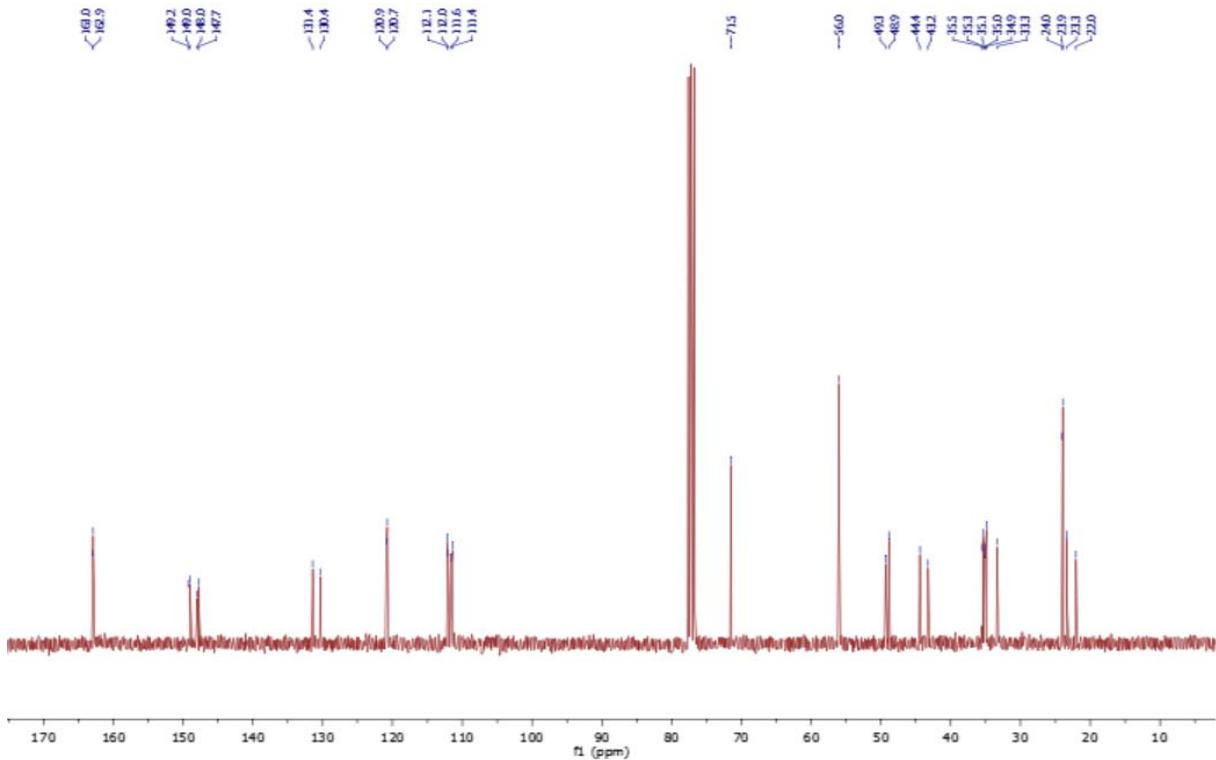
^{13}C RMN spectrum



N-(3,4-Dimethoxyphenethyl)-N-(5-hydroxy-4,4-dimethylpentyl)formamide (1-40a)
 ^1H RMN spectrum

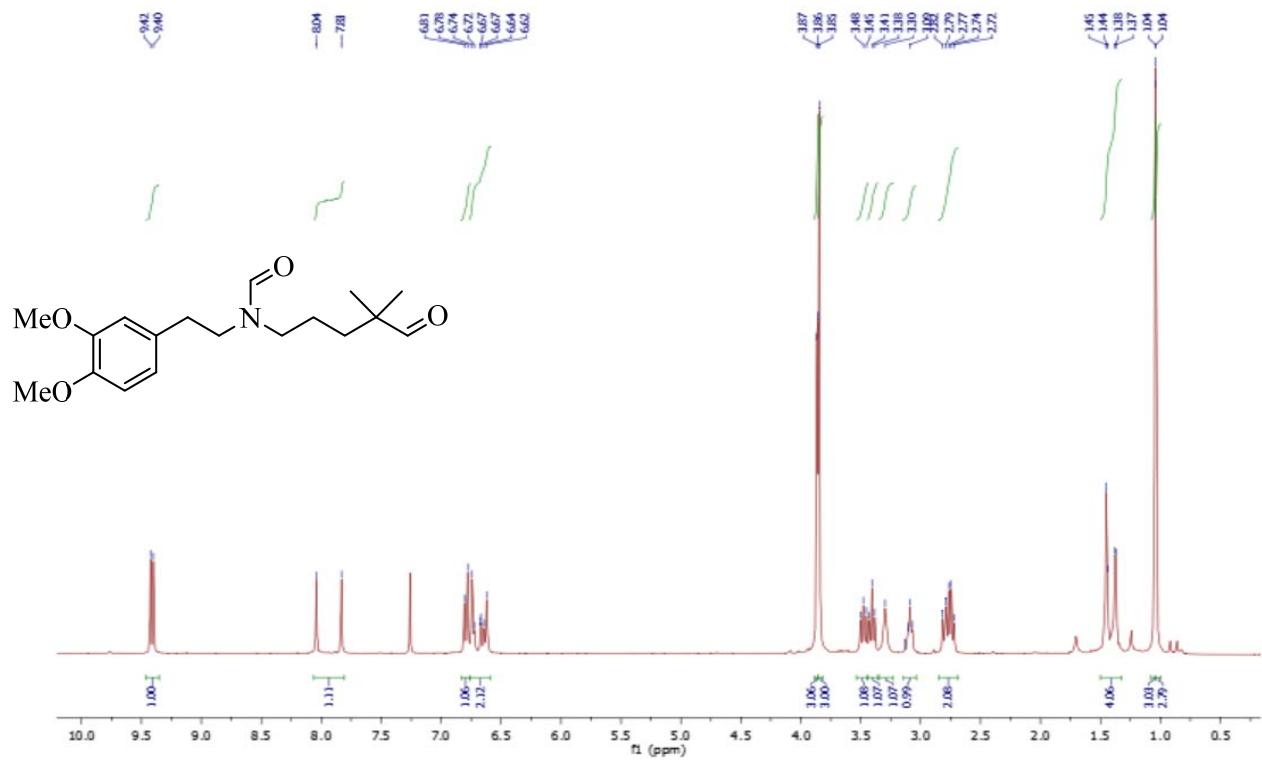


¹³C RMN spectrum

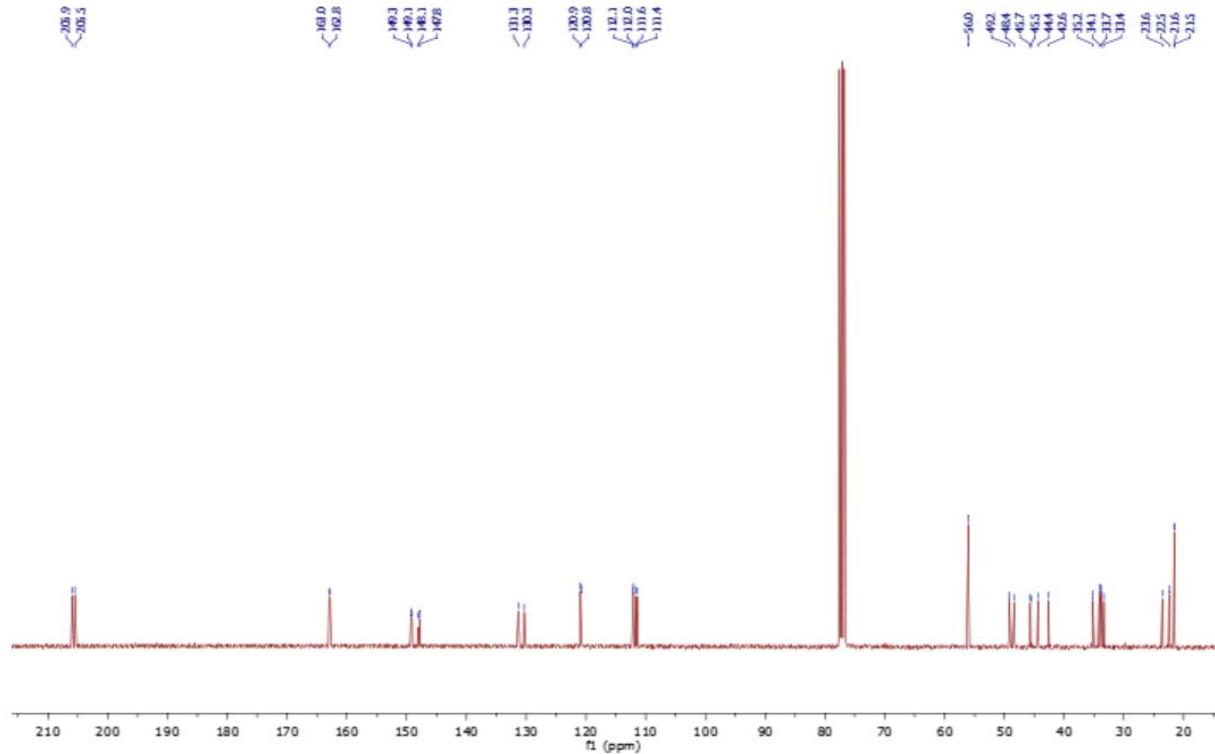


N-(3,4-Dimethoxyphenethyl)-N-(4,4-dimethyl-5-oxopentyl)formamide (1-41a)

¹H RMN spectrum

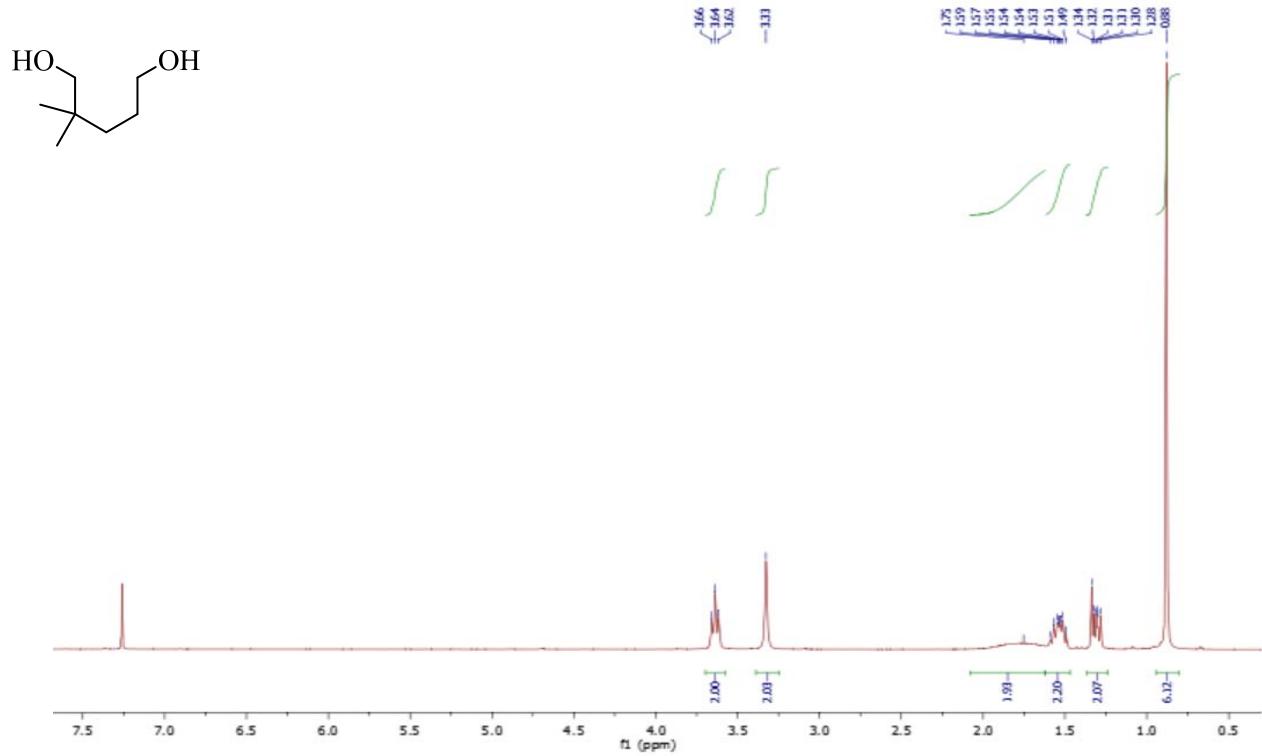


¹³C RMN spectrum

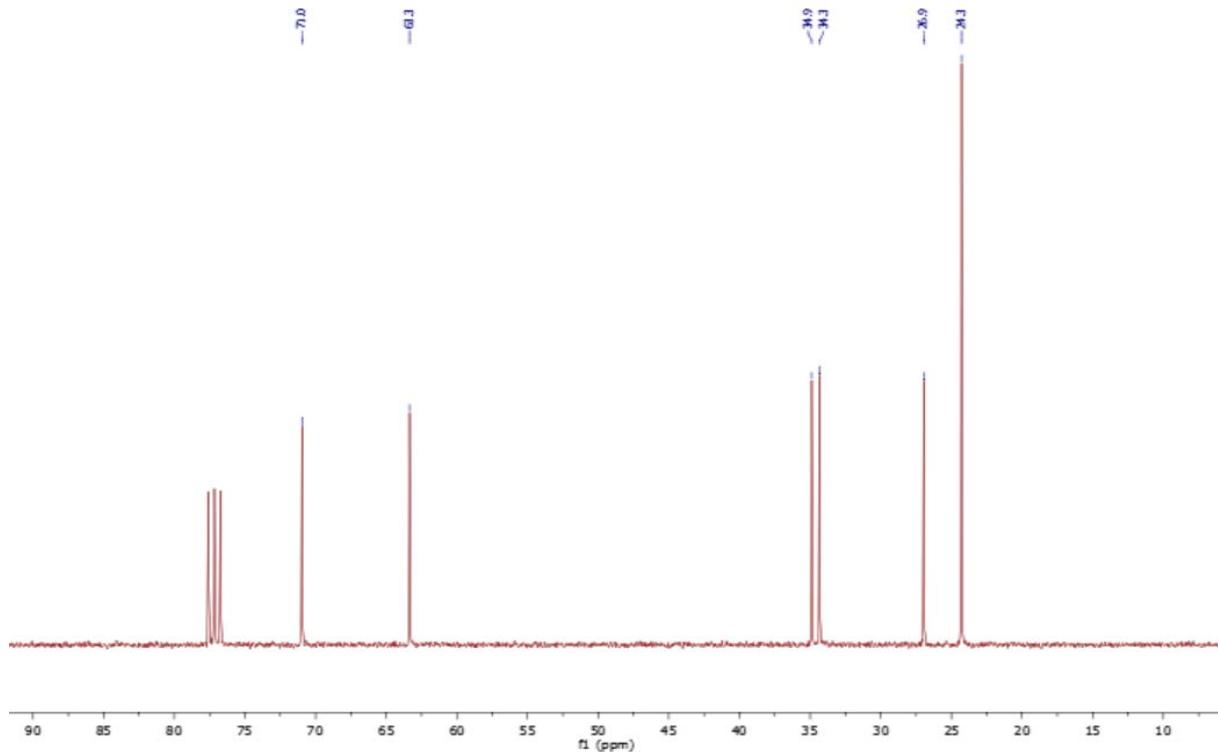


2,2-Dimethylpentane-1,5-diol (1-42)

^1H RMN spectrum

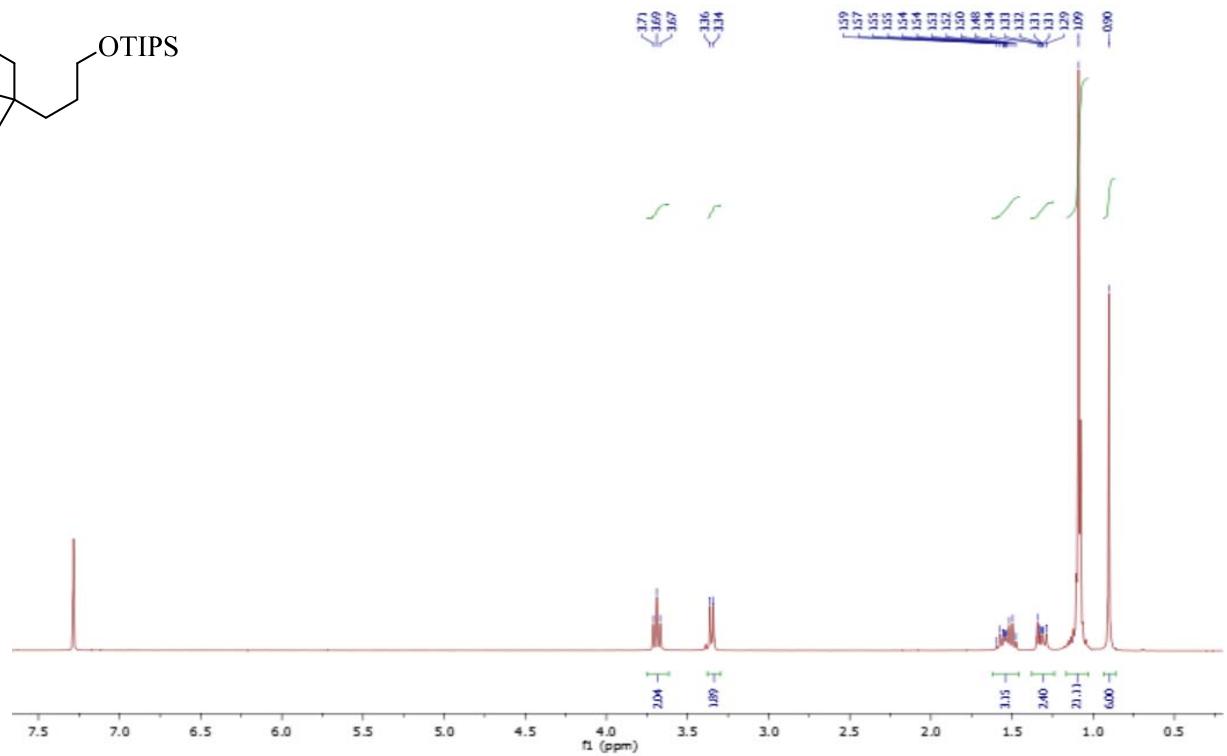
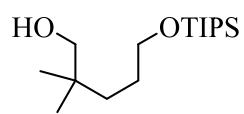


^{13}C RMN spectrum

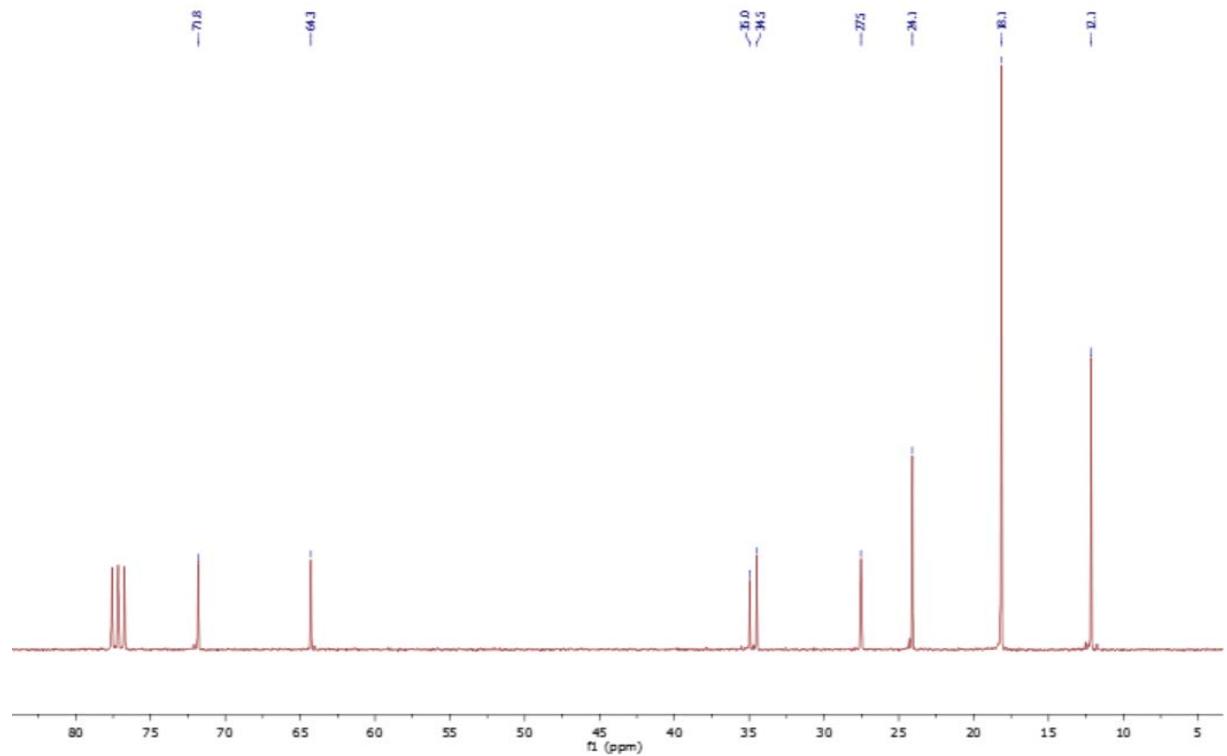


2,2-Dimethyl-5-((triisopropylsilyl)oxy)pentan-1-ol (1-43)

¹H RMN spectrum

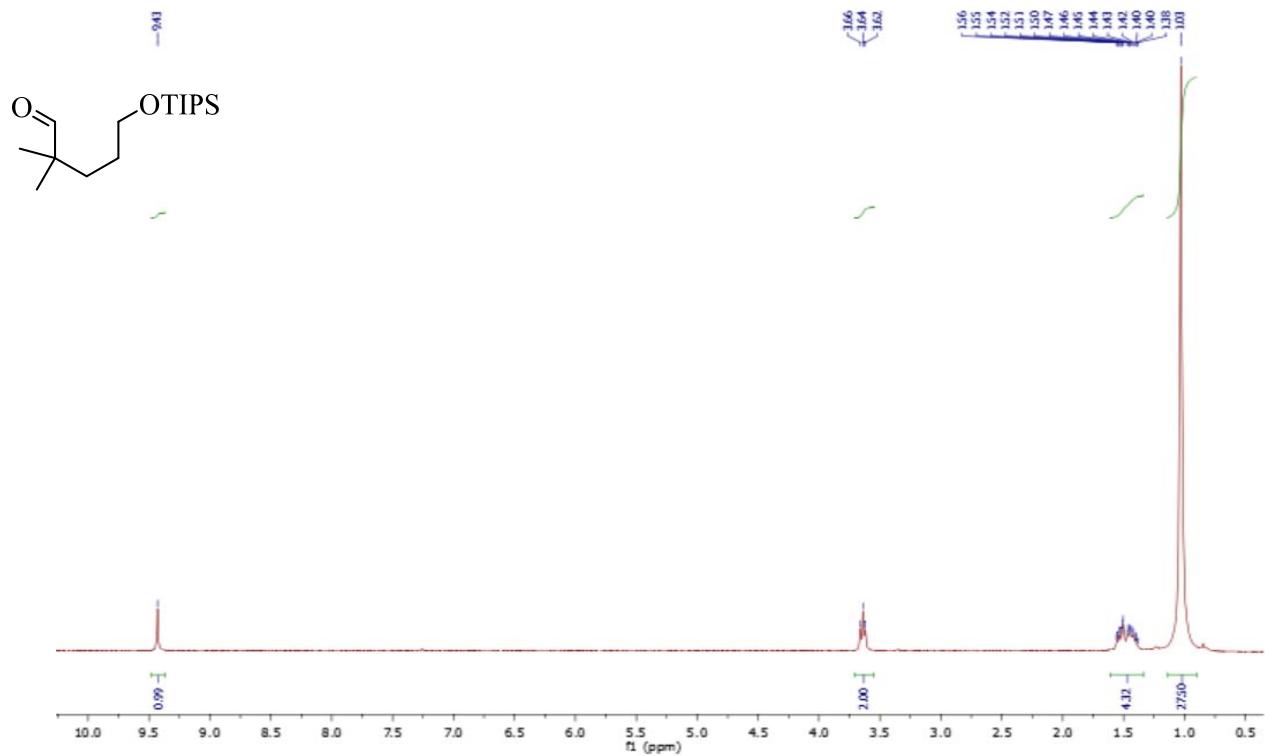


¹³C RMN spectrum

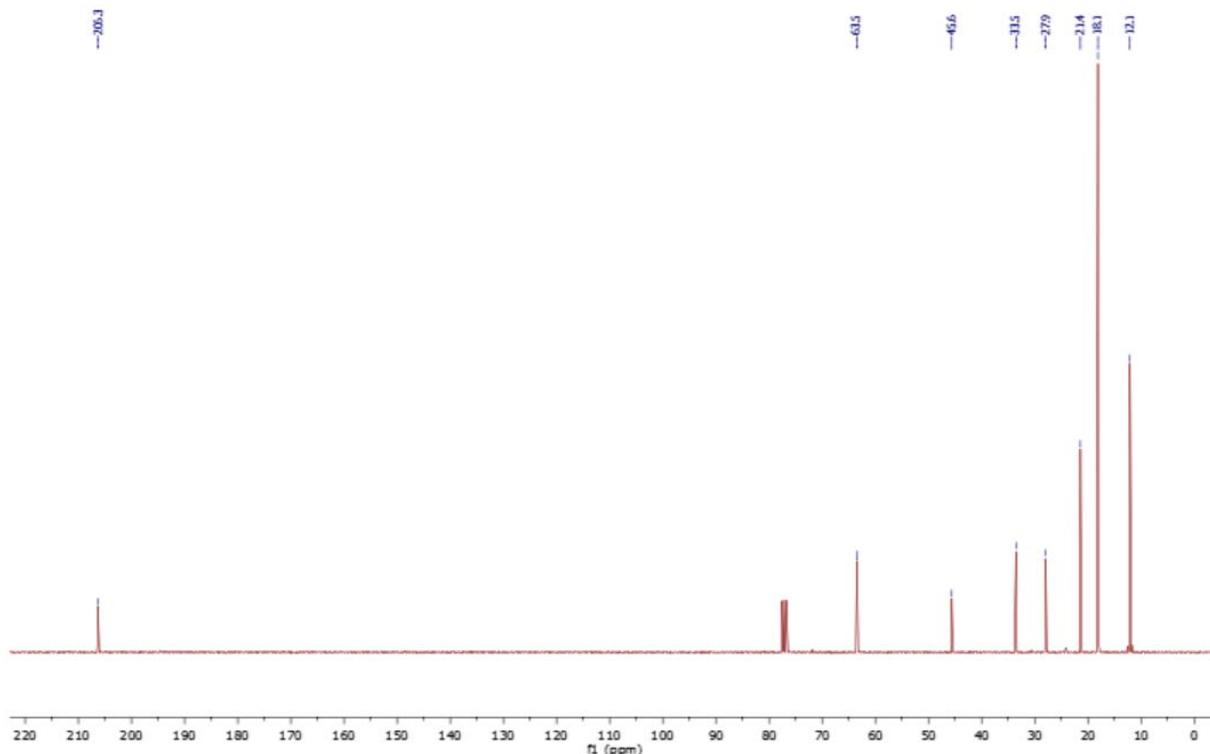


2,2-Dimethyl-5-((triisopropylsilyl)oxy)pentanal (1-44)

^1H RMN spectrum

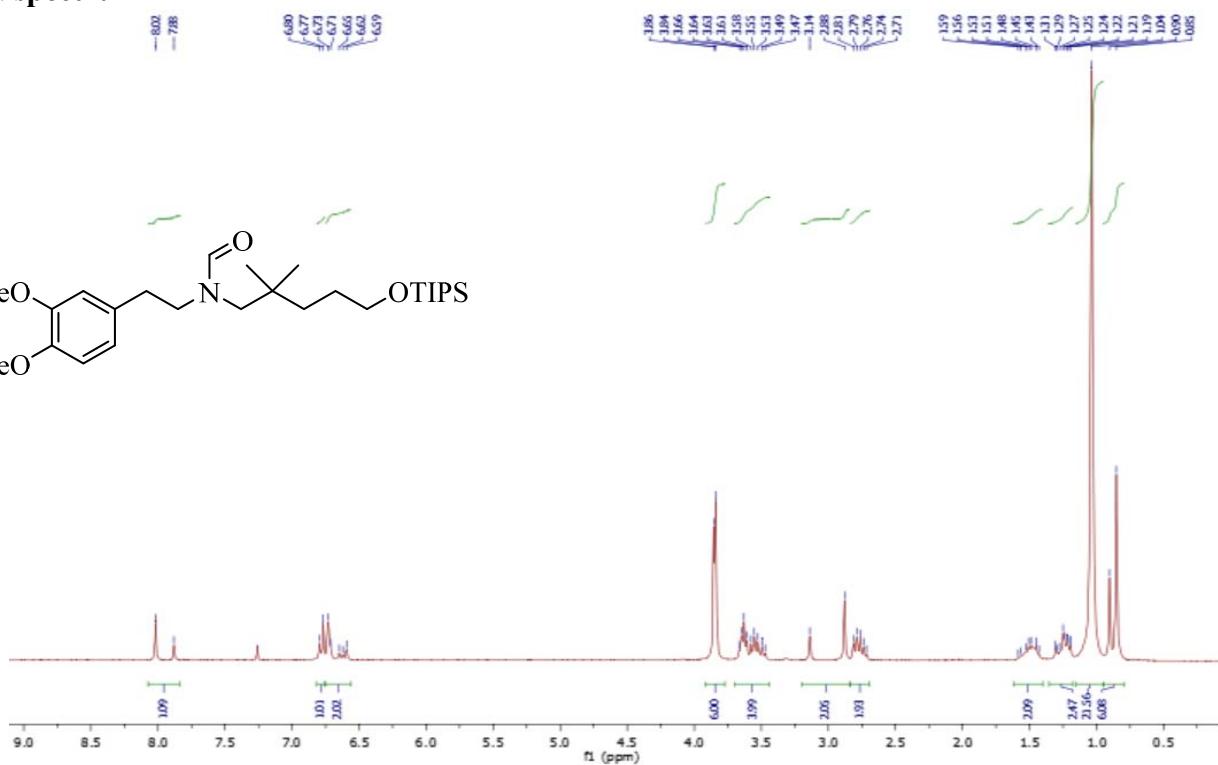
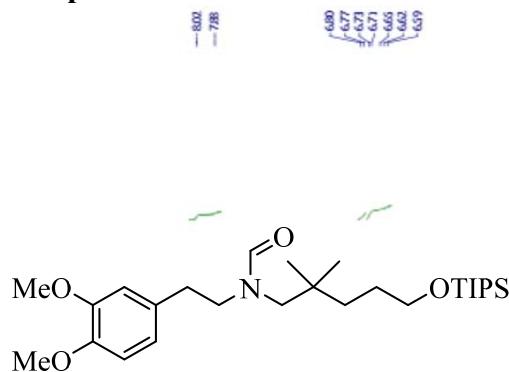


^{13}C RMN spectrum

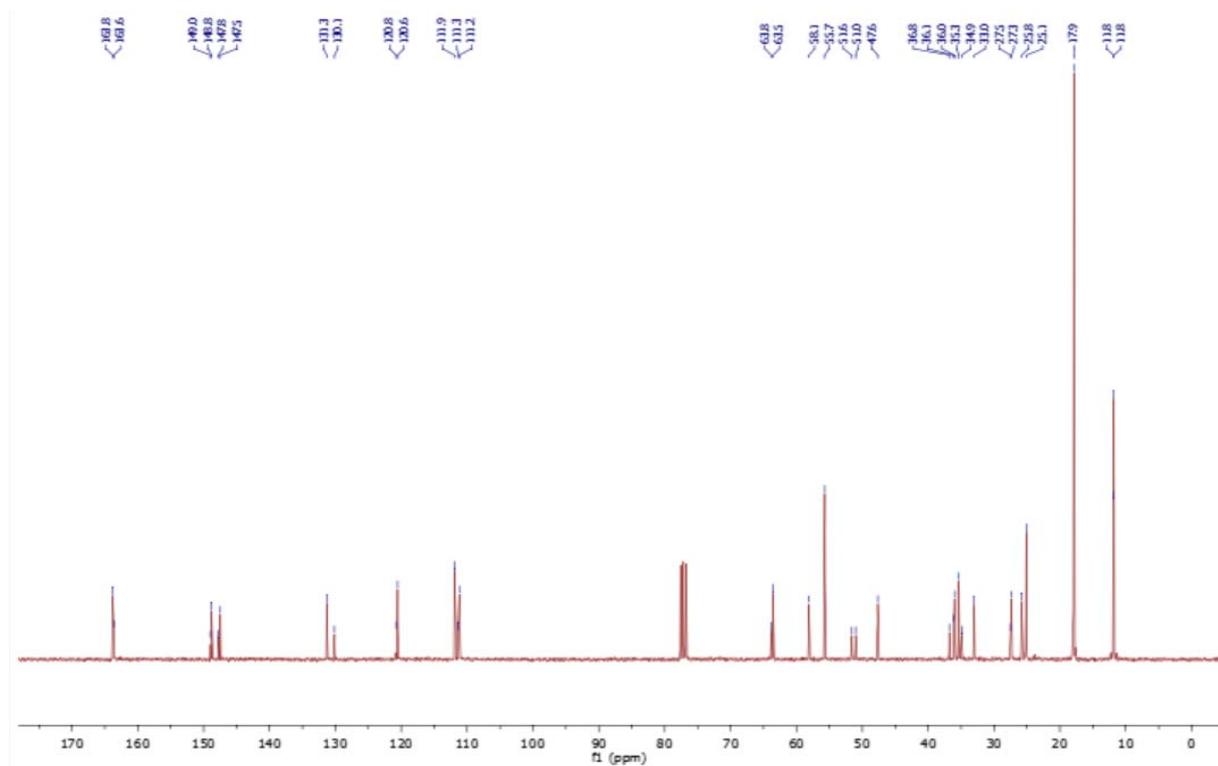


**N-(3,4-Dimethoxyphenethyl)-N-(2,2-dimethyl-5-((triisopropylsilyl)oxy)pentyl)formamide
(1-45)**

¹H RMN spectrum

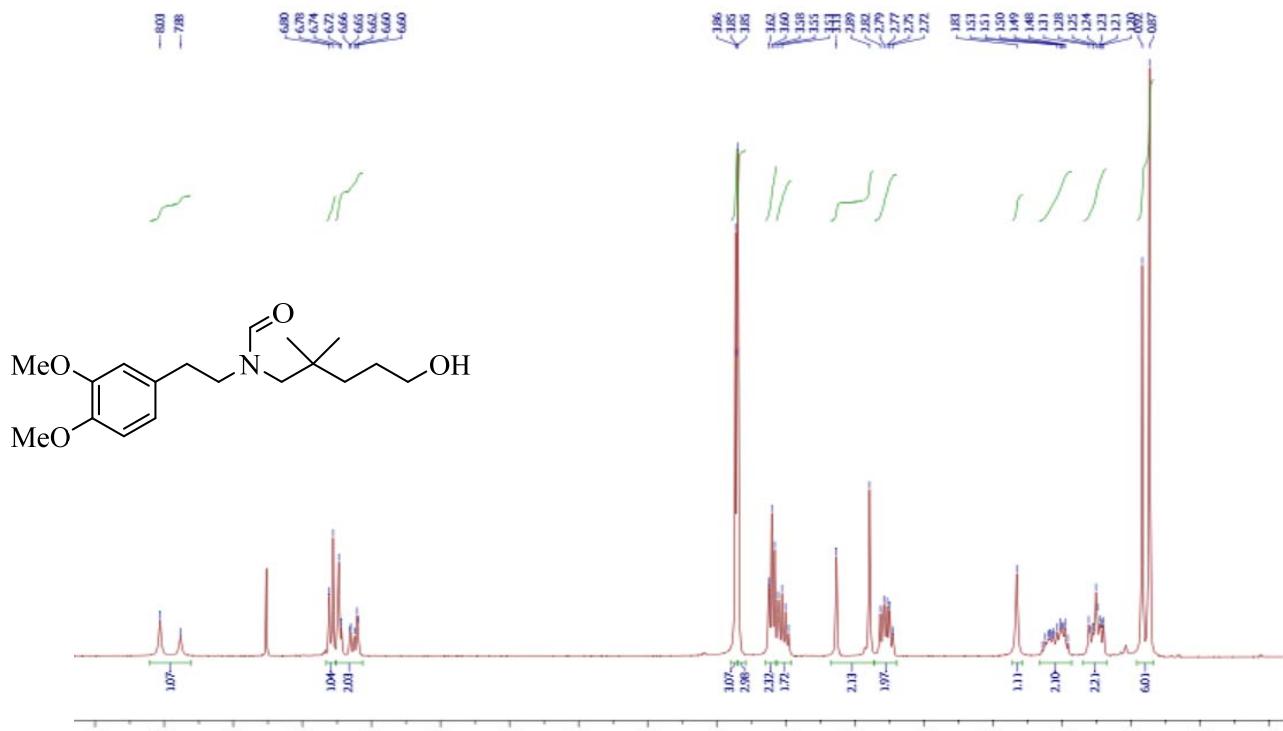


¹³C RMN spectrum

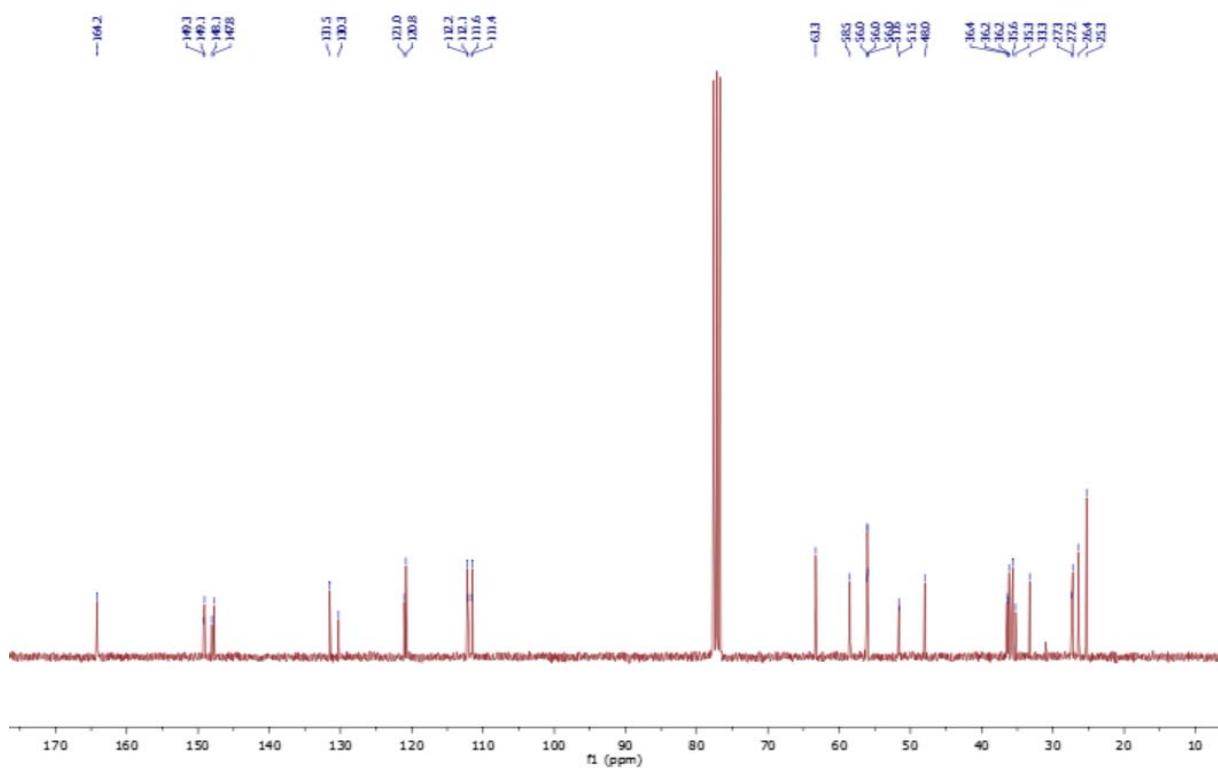


N-(3,4-Dimethoxyphenethyl)-N-(5-hydroxy-2,2-dimethylpentyl)formamide (1-46)

¹H RMN spectrum

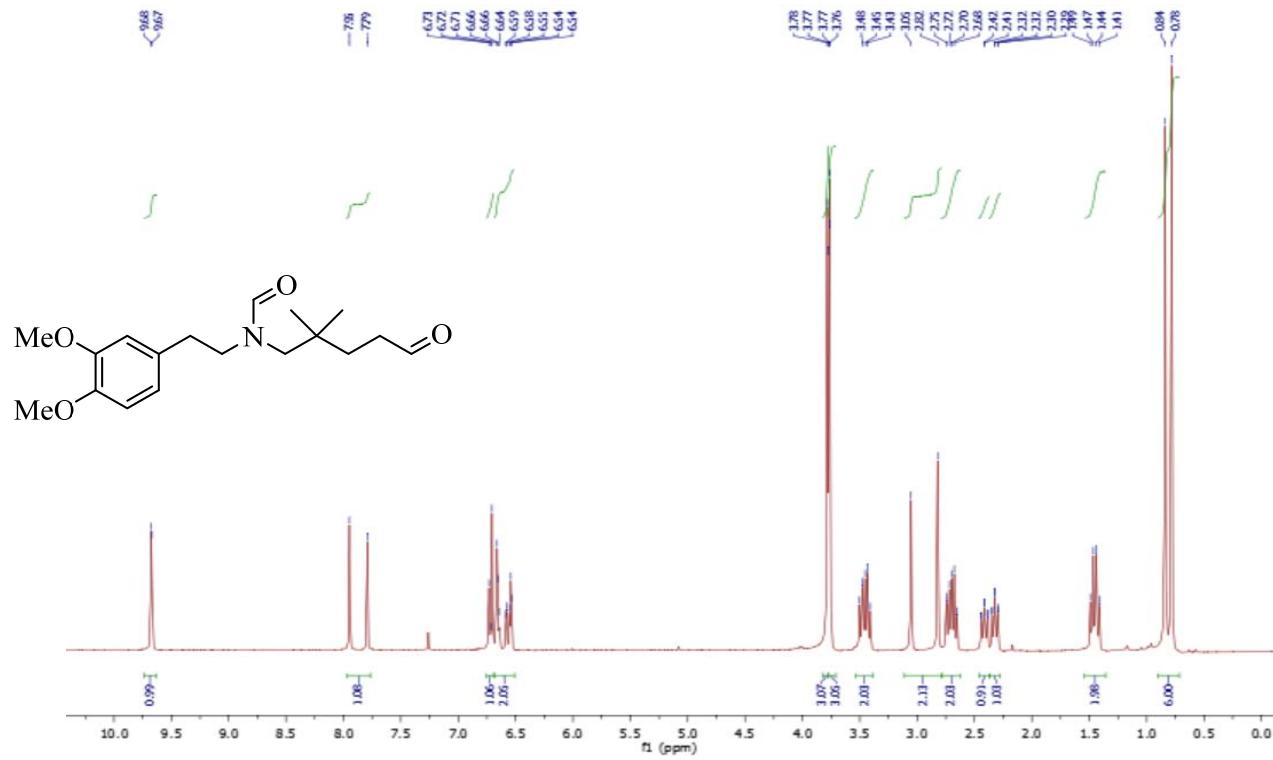


¹³C RMN spectrum

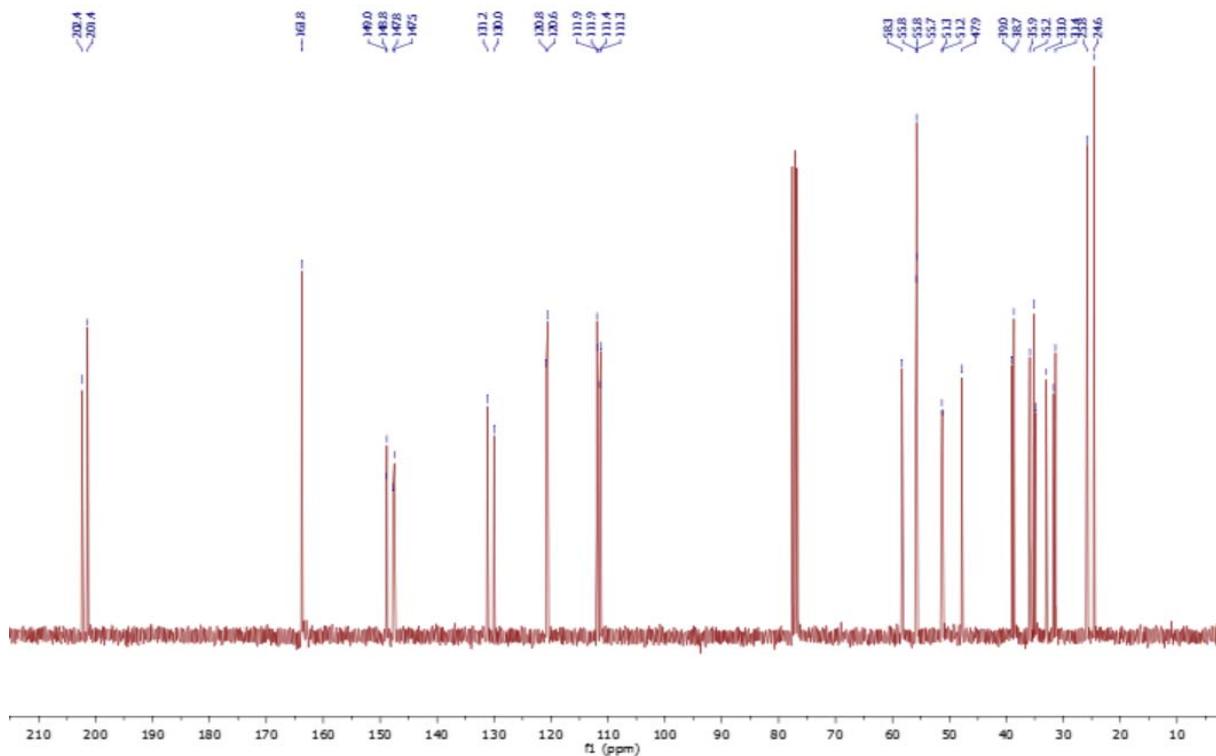


N-(3,4-Dimethoxyphenethyl)-N-(2,2-dimethyl-5-oxopentyl)formamide (1-47)

¹H RMN spectrum

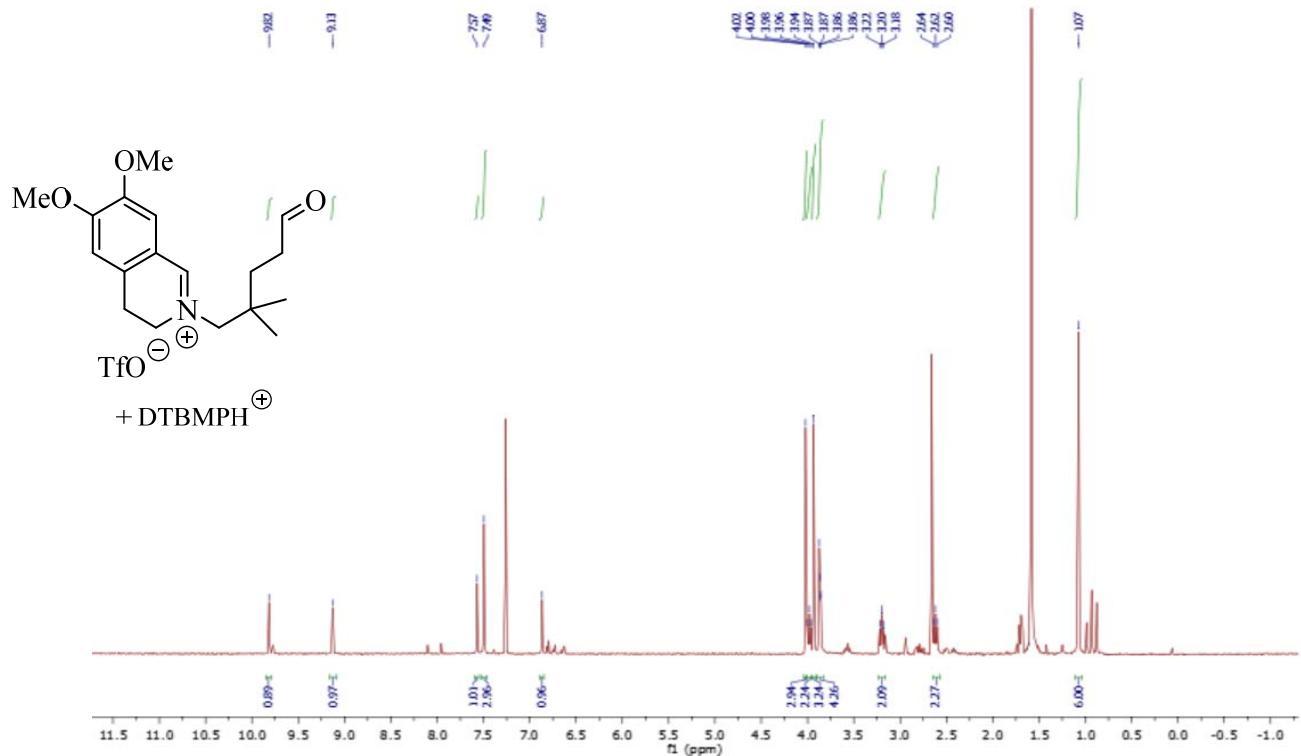


¹³C RMN spectrum



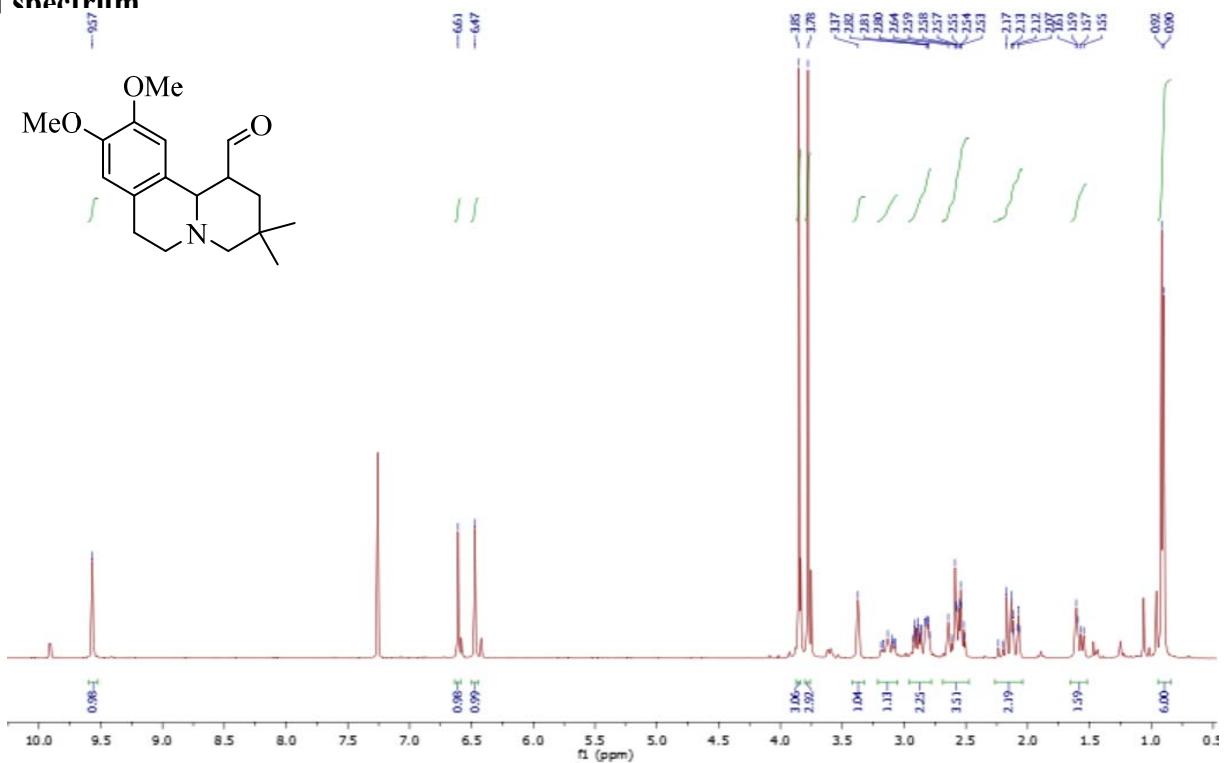
Iminium ion (1-47a)

^1H RMN spectrum

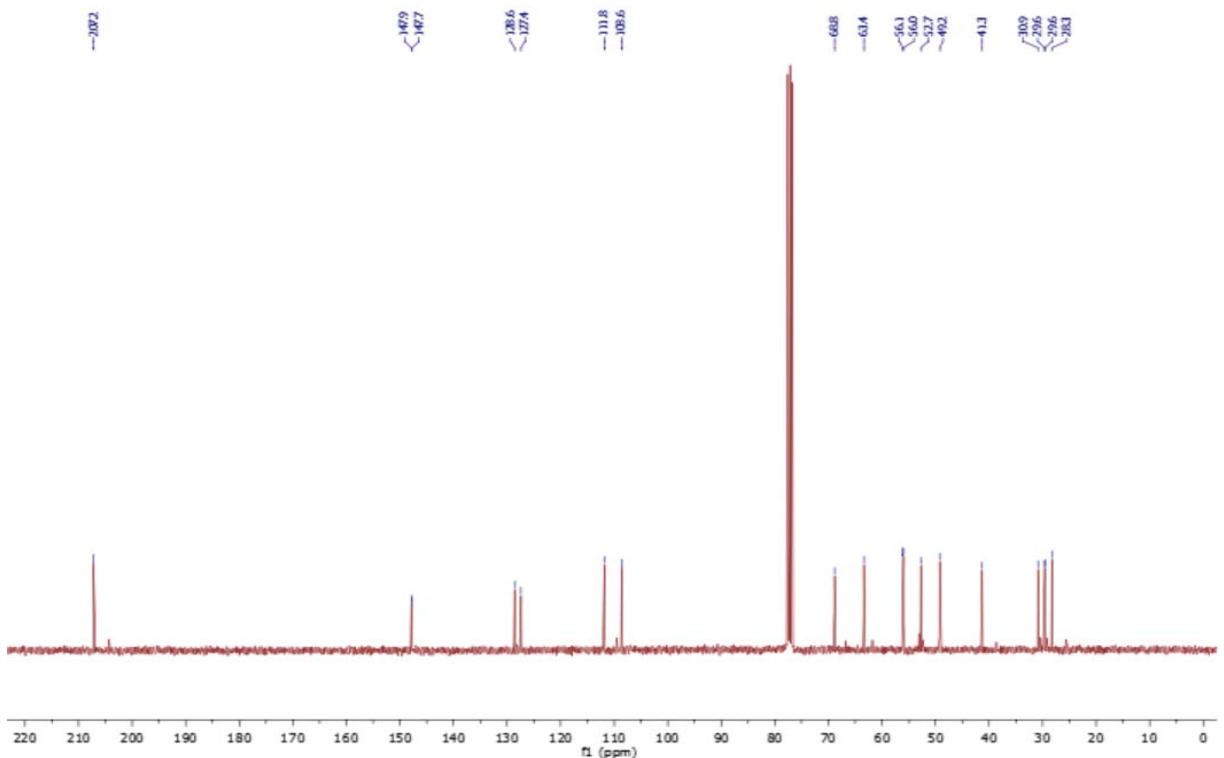


9,10-Dimethoxy-3,3-dimethyl-1,3,4,6,7,11b-hexahydro-2H-pyrido[2,1-a]isoquinoline-1-carbaldehyde (1-48)

^1H RMN spectrum

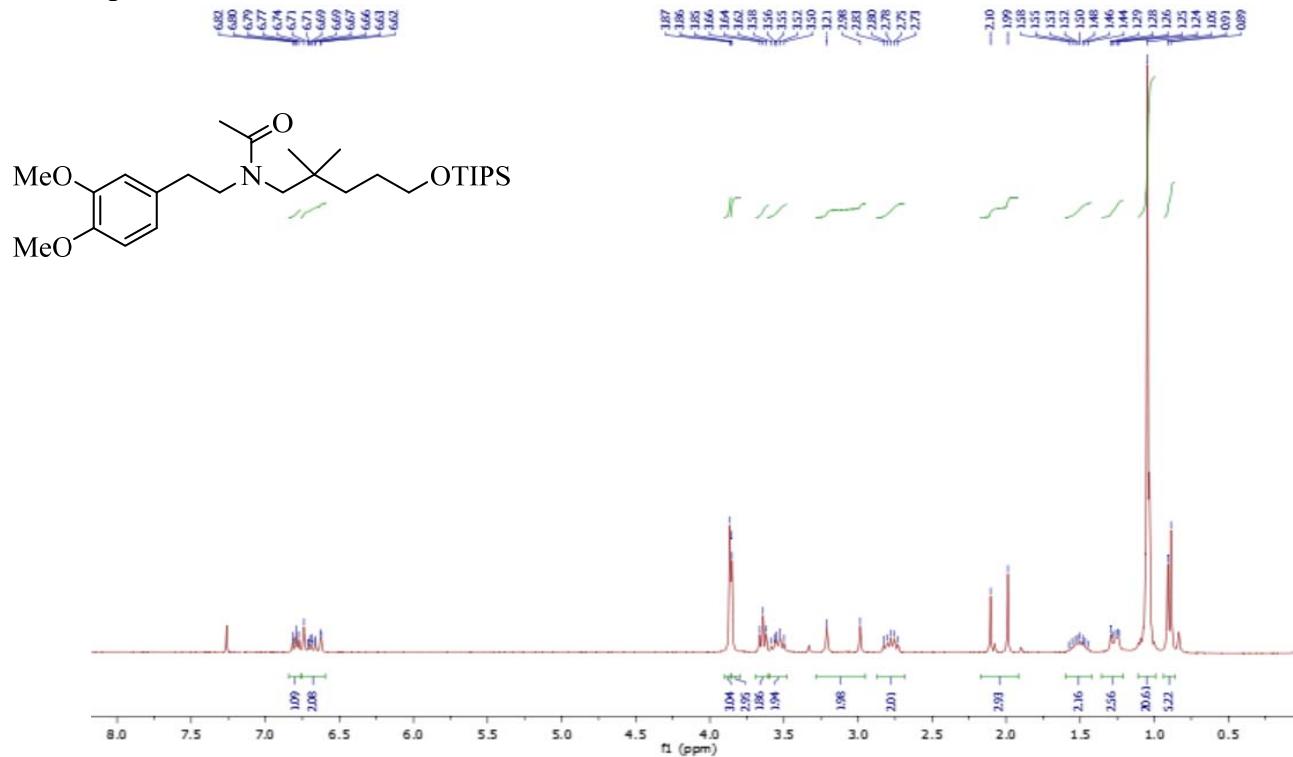


^{13}C RMN spectrum

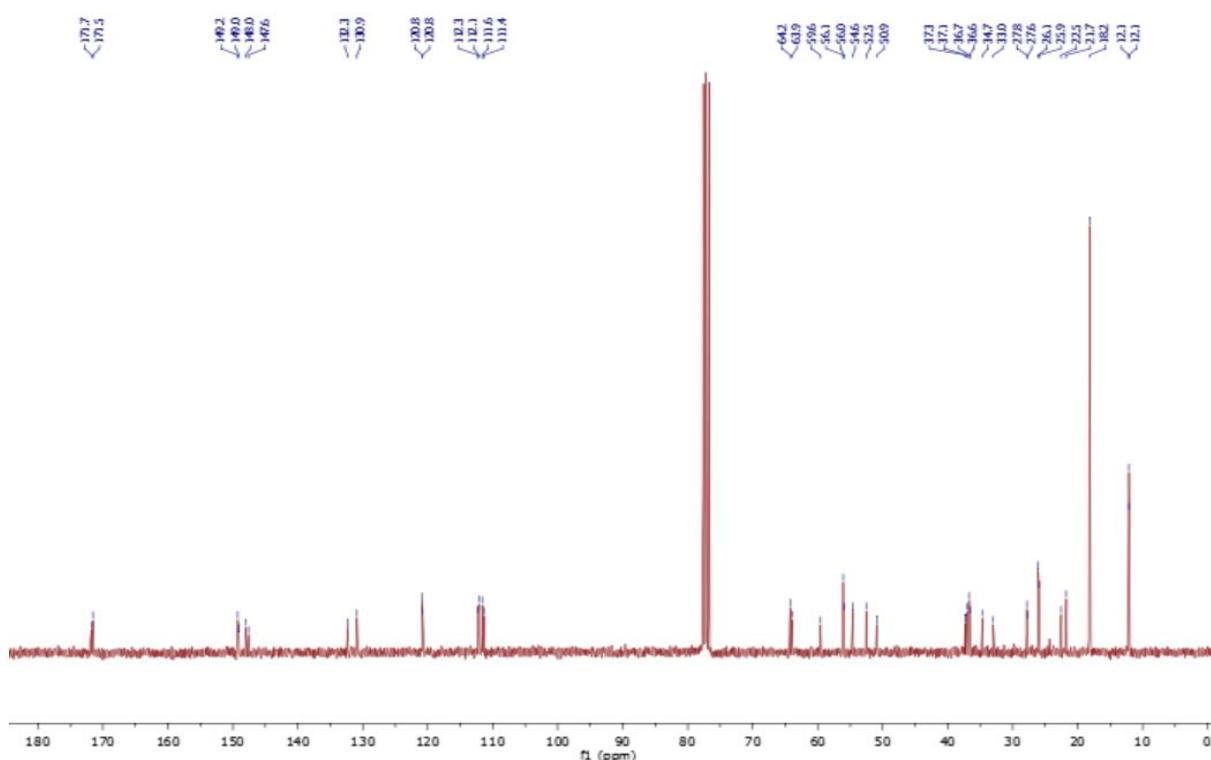


**N-(3,4-Dimethoxyphenethyl)-N-(2,2-dimethyl-5-((triisopropylsilyl)oxy)pentyl)acetamide
(1-49)**

¹H RMN spectrum

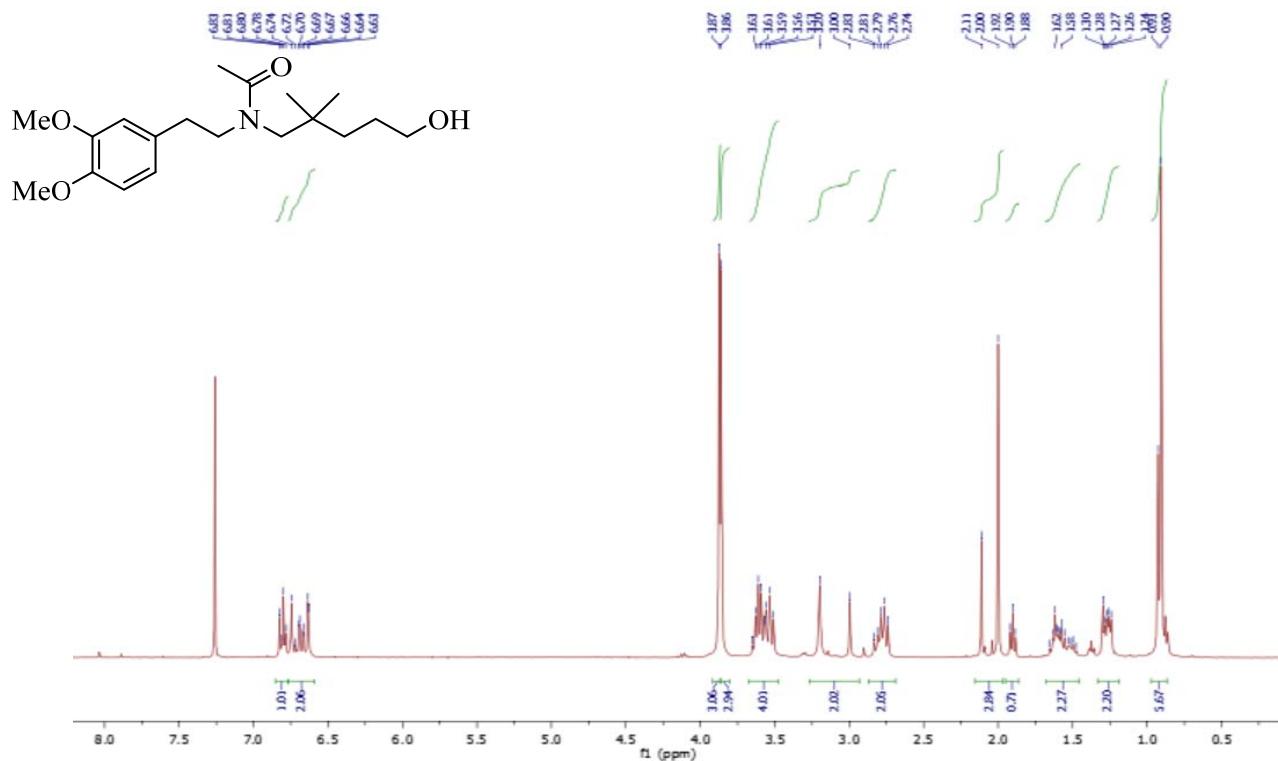


¹³C RMN spectrum

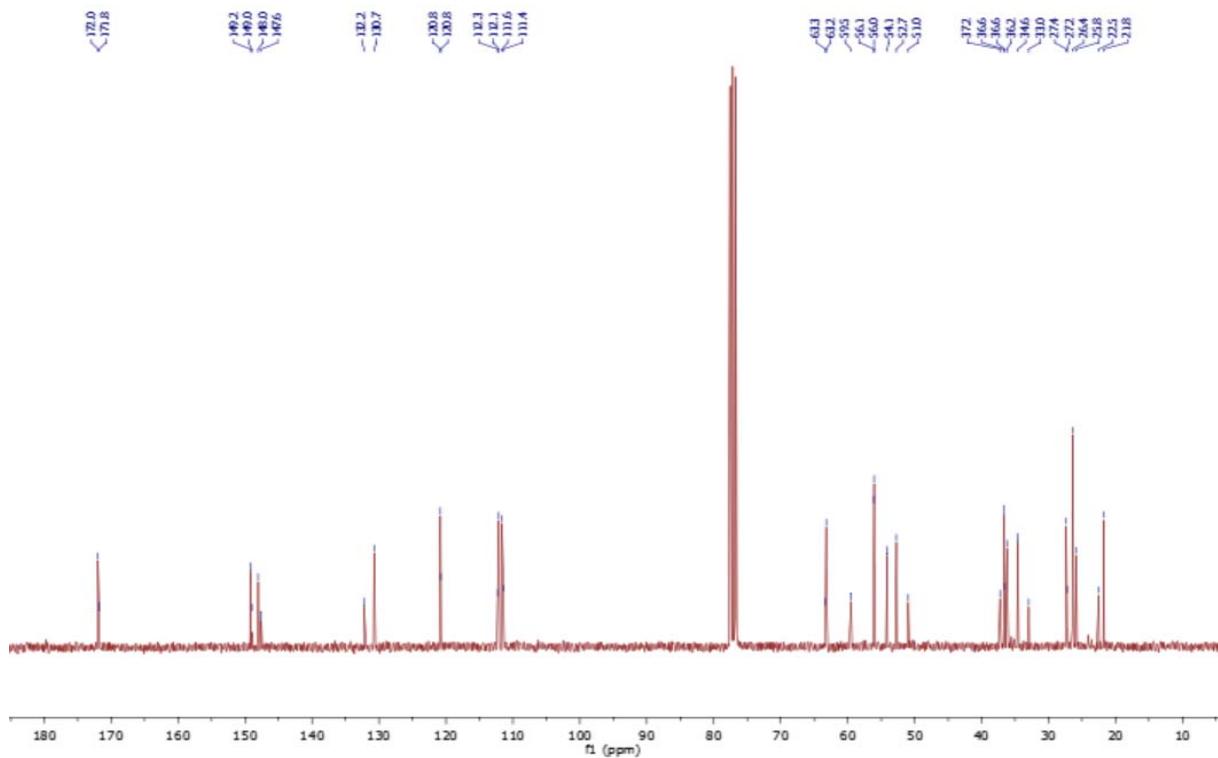


N-(3,4-dimethoxyphenethyl)-N-(5-hydroxy-2,2-dimethylpentyl)acetamide (1-50)

¹H RMN spectrum

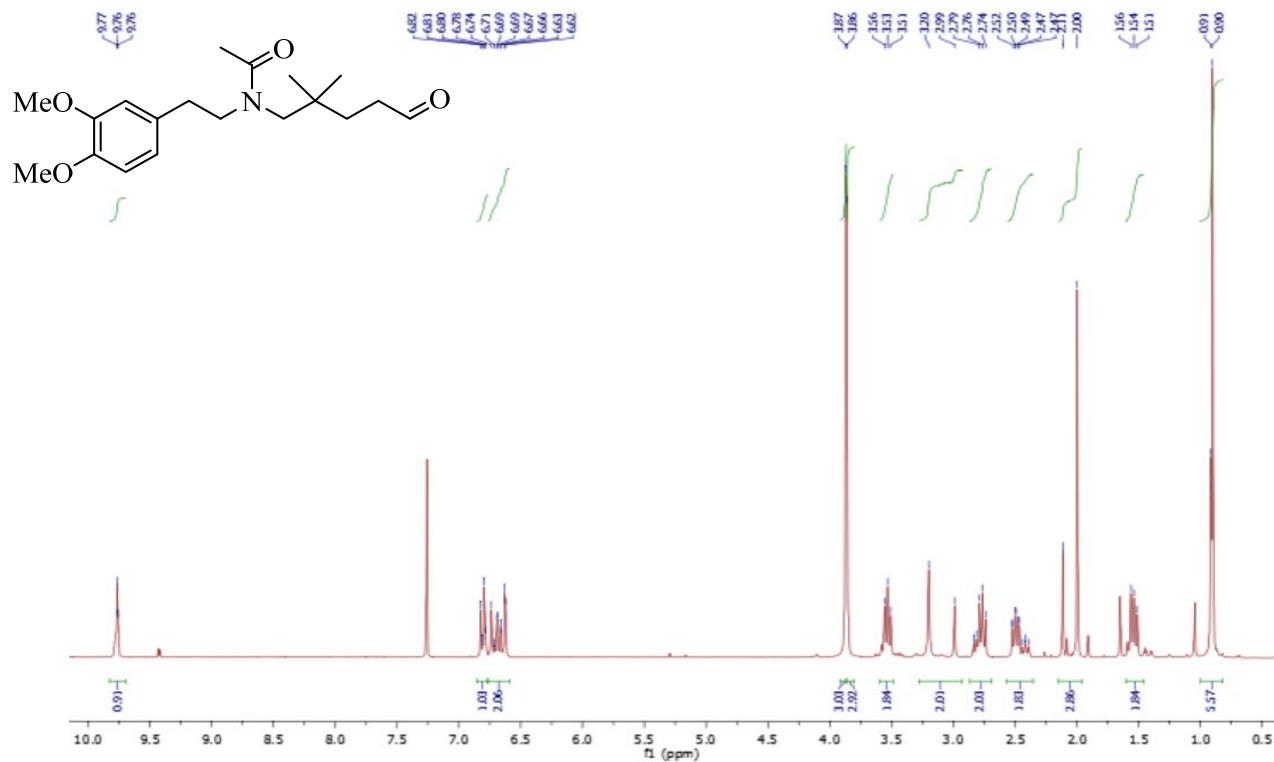


¹³C RMN spectrum

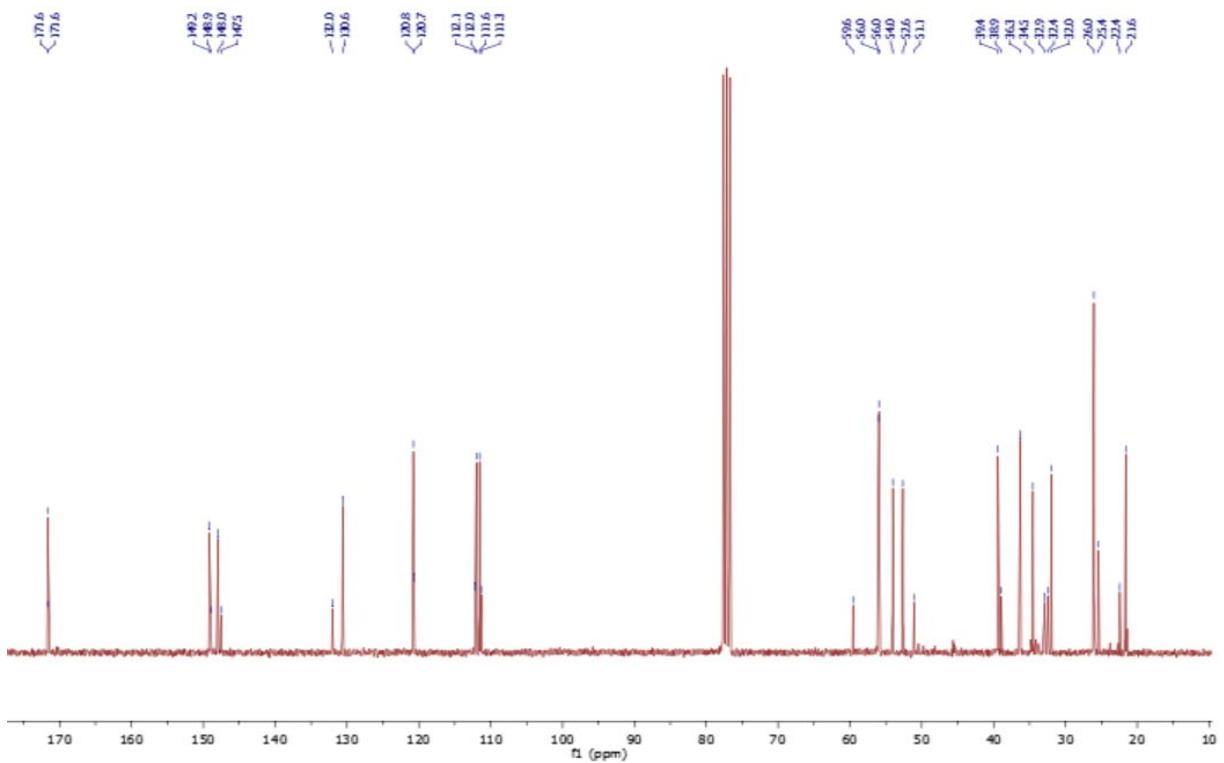


N-(3,4-Dimethoxyphenethyl)-N-(2,2-dimethyl-5-oxopentyl)acetamide (1-51)

¹H RMN spectrum

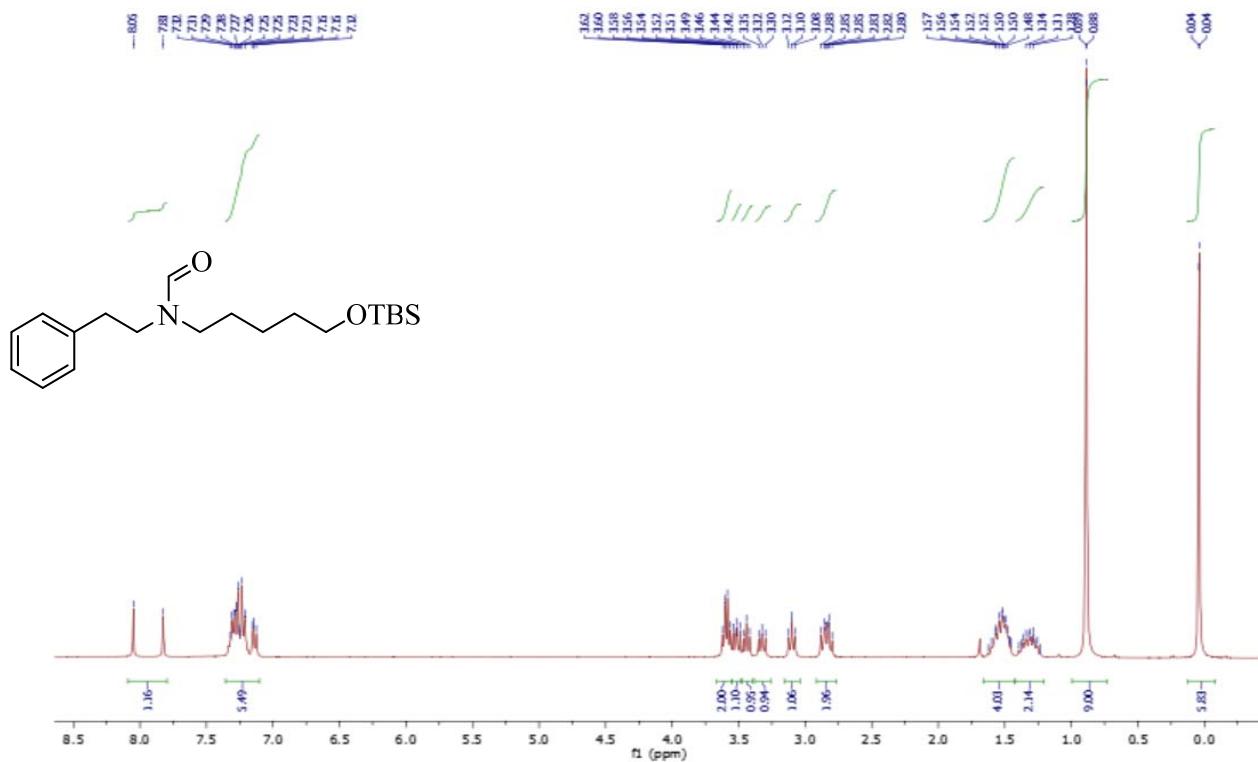


¹³C RMN spectrum

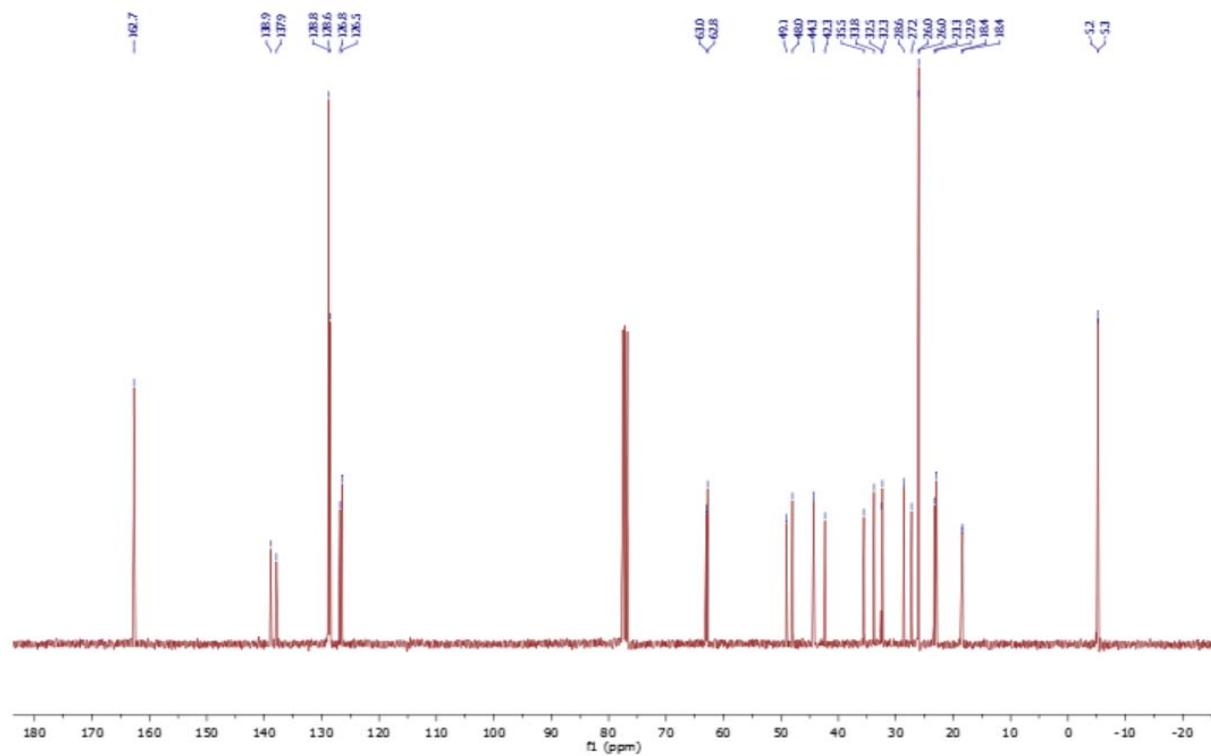


N-(5-((*tert*-Butyldimethylsilyl)oxy)pentyl)-N-phenethylformamide (1-53)

^1H RMN spectrum

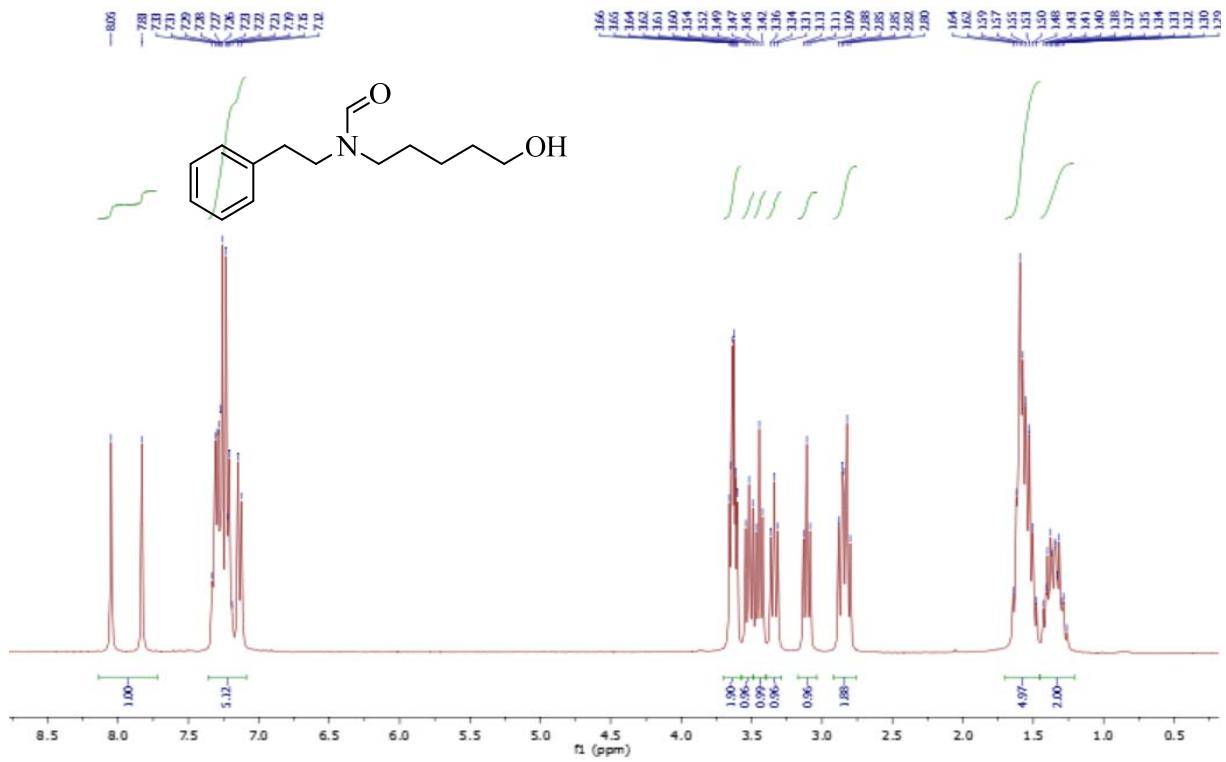


^{13}C RMN spectrum

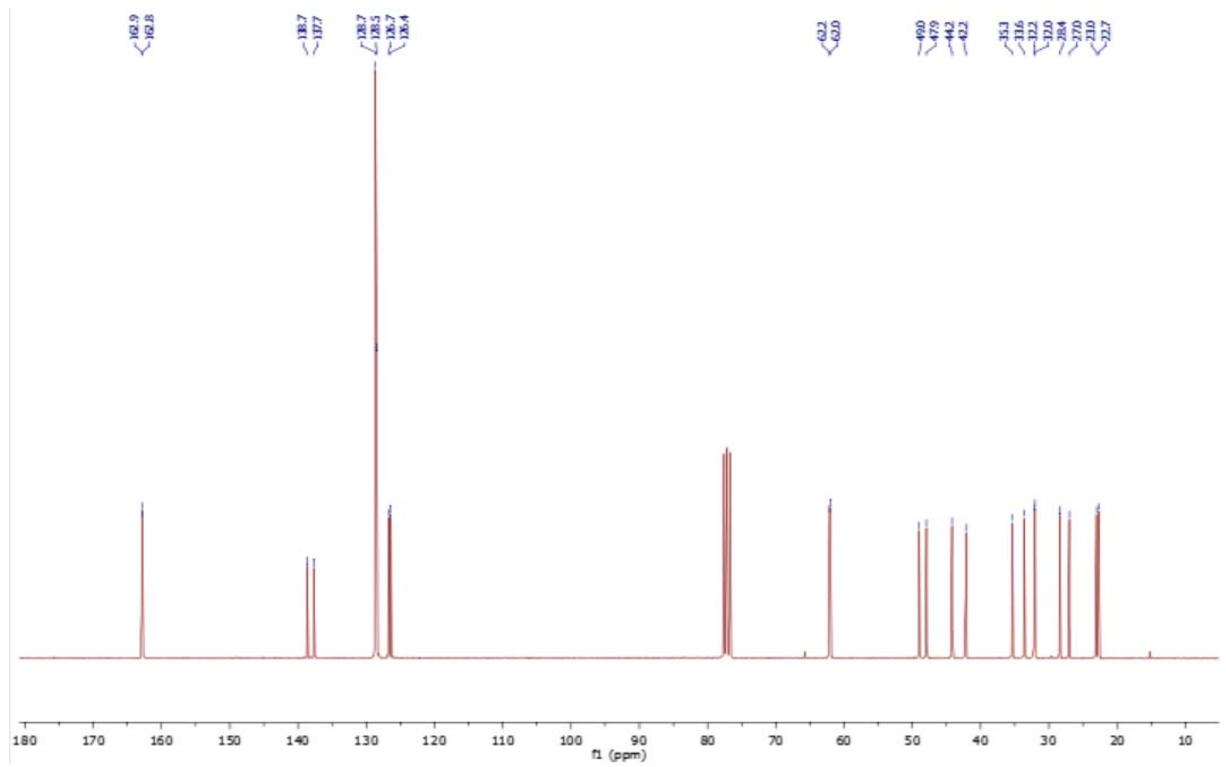


N-(5-Hydroxypentyl)-N-phenethylformamide (1-54)

¹H RMN spectrum

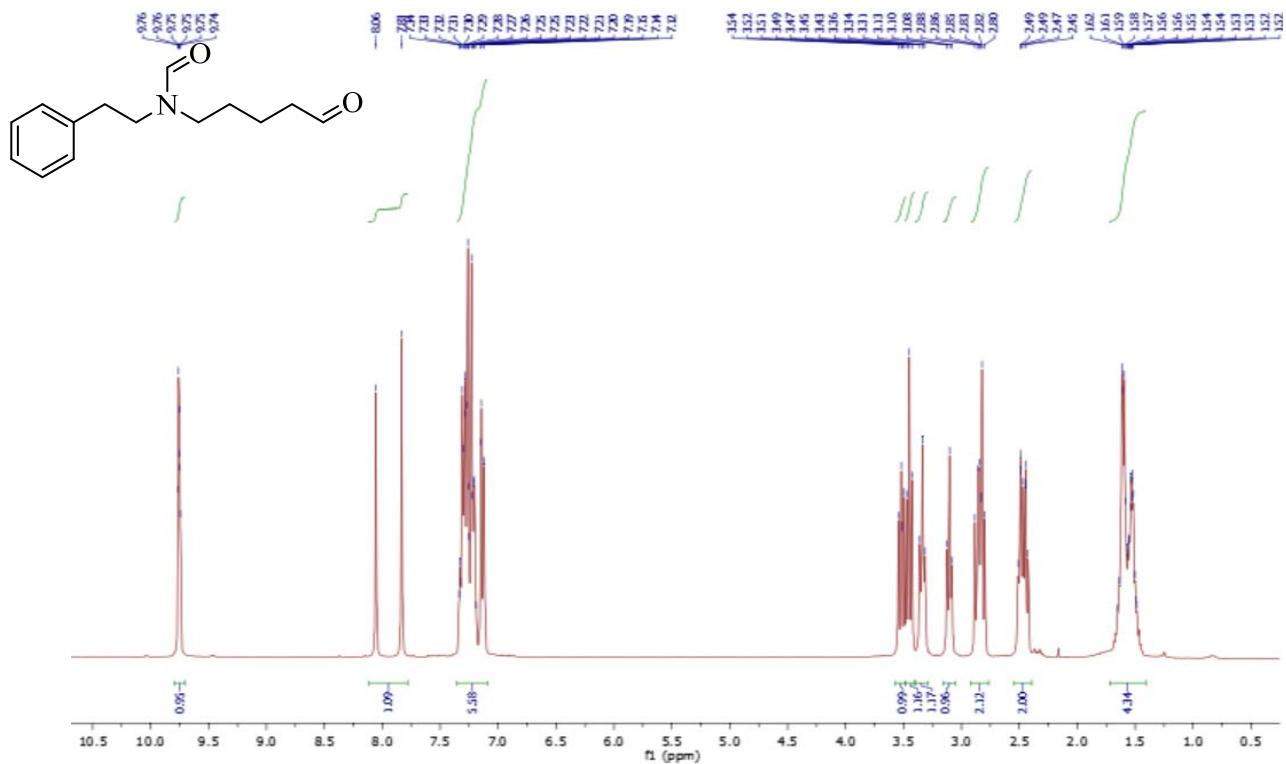


¹³C RMN spectrum

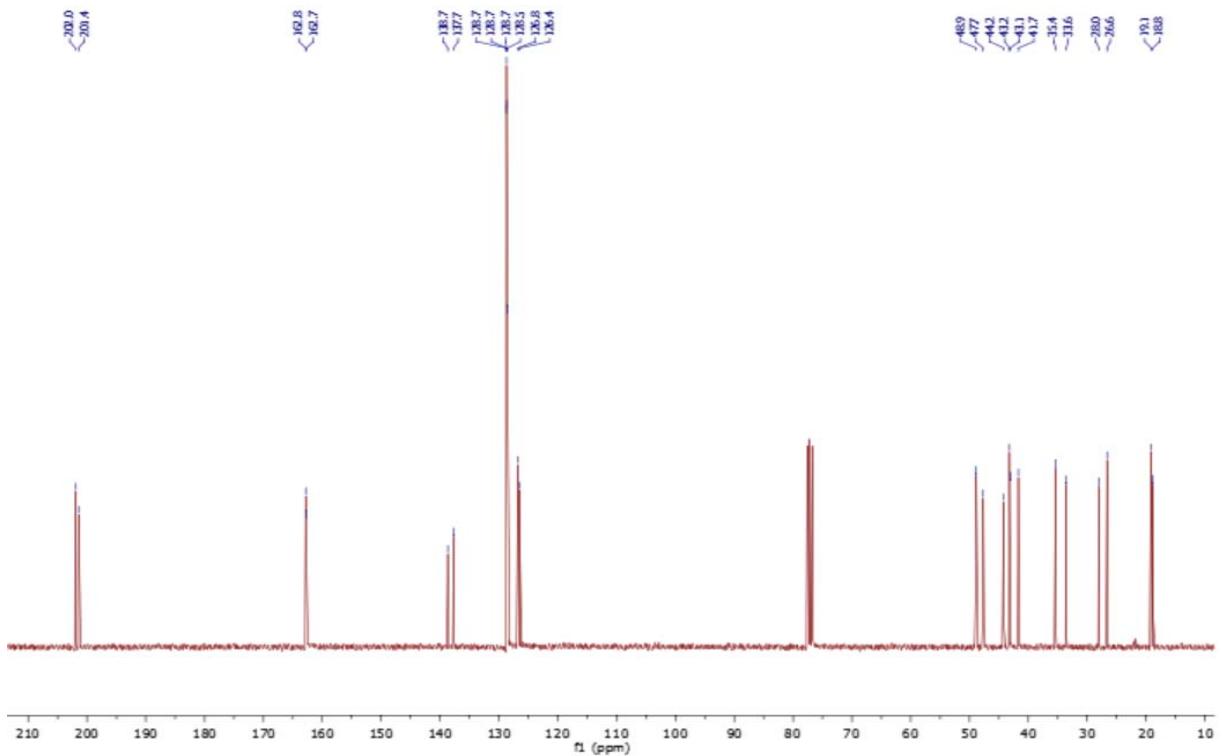


N-(5-Oxopentyl)-N-phenethylformamide (1-55)

¹H RMN spectrum

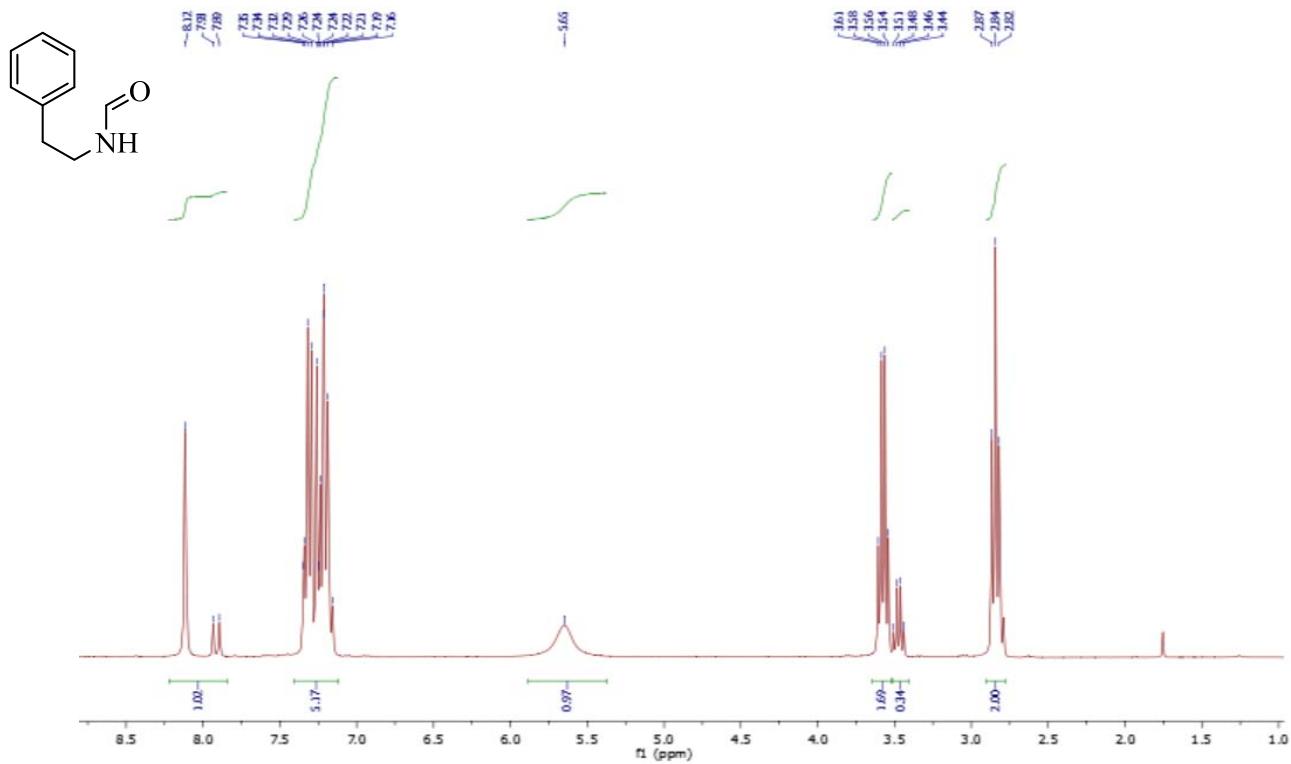


¹³C RMN spectrum

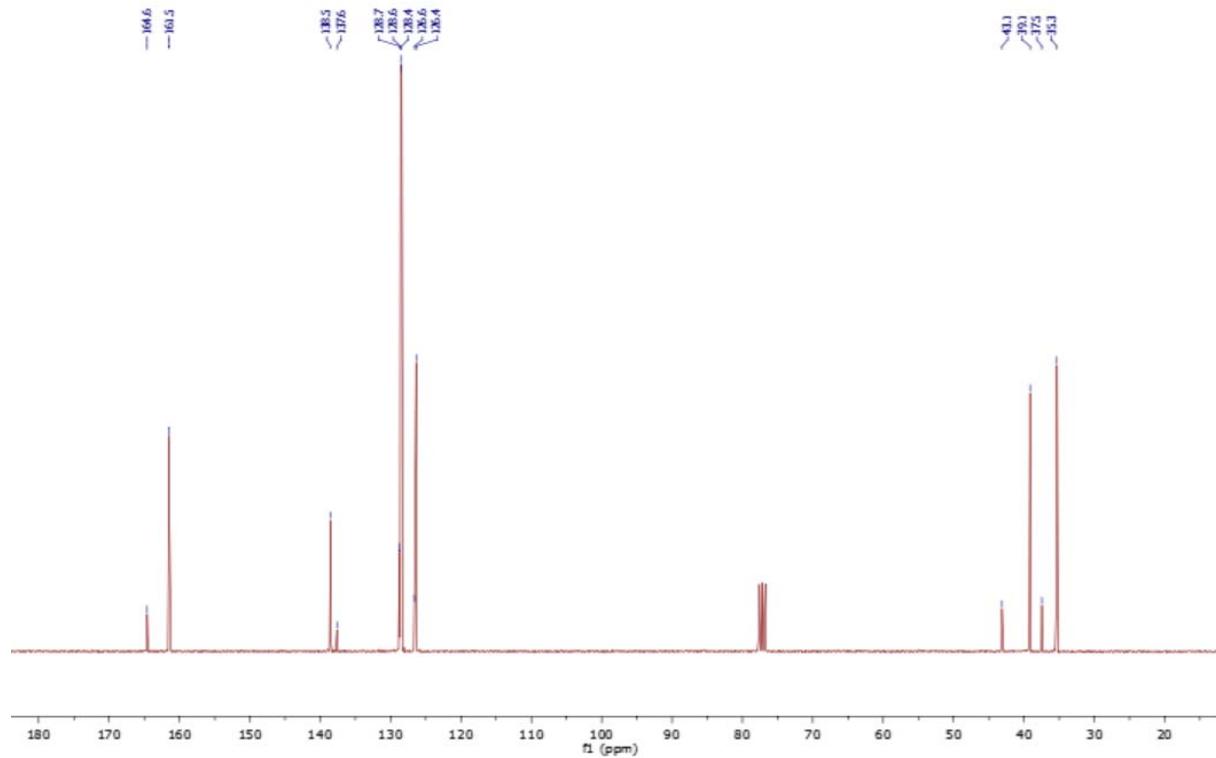


N-Phenethylformamide (1-57)

¹H RMN spectrum

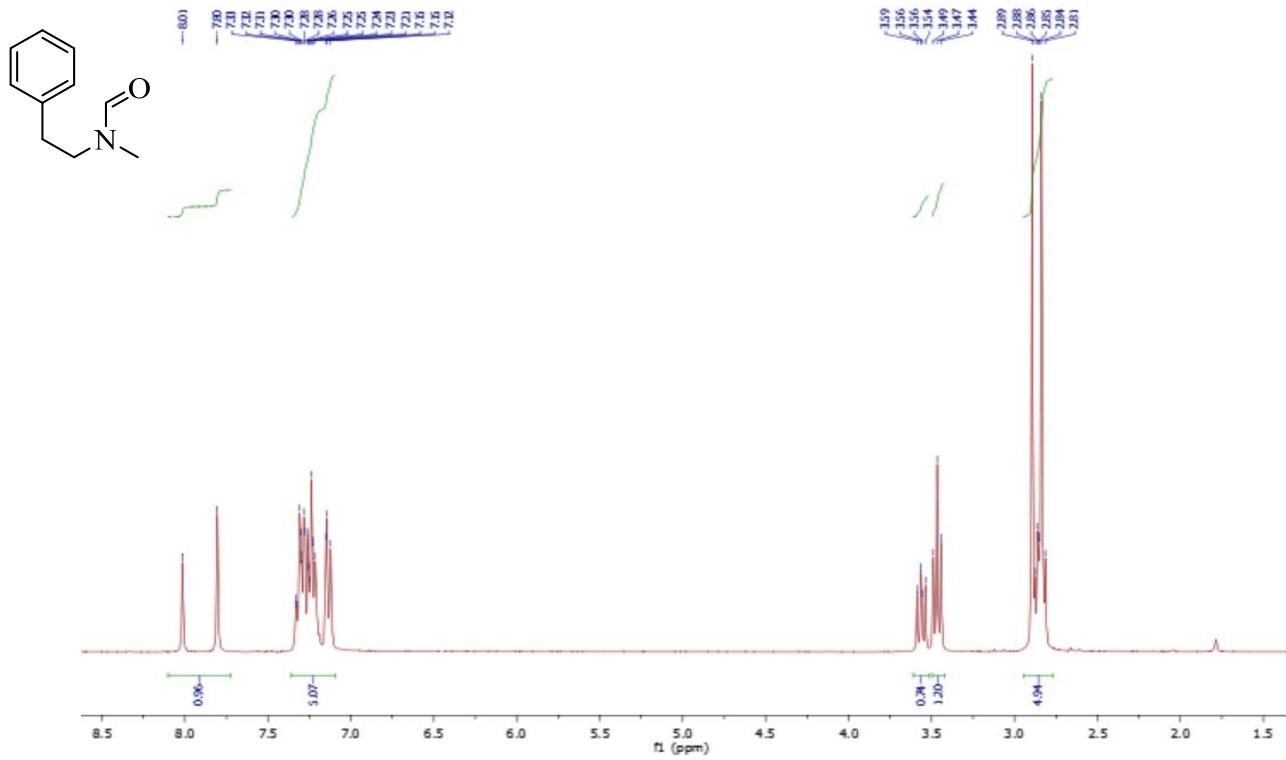


¹³C RMN spectrum

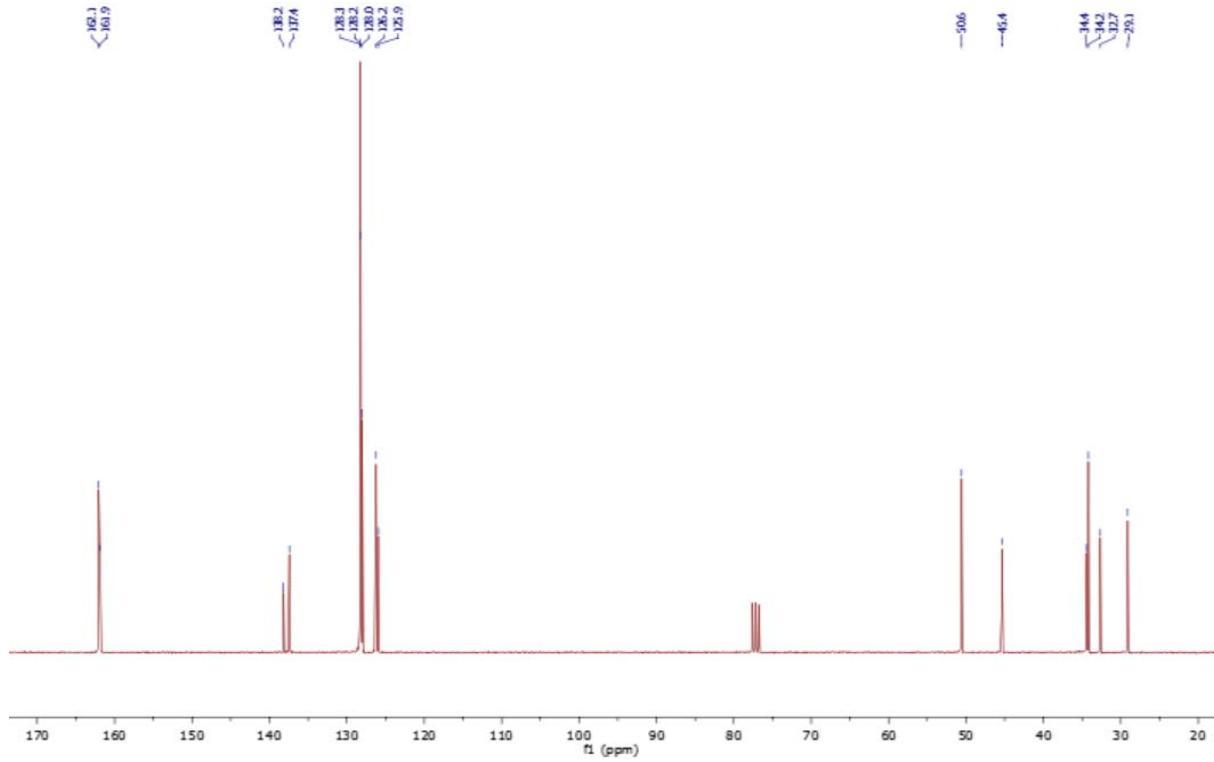


N-Methyl-N-phenethylformamide (1-58)

^1H RMN spectrum

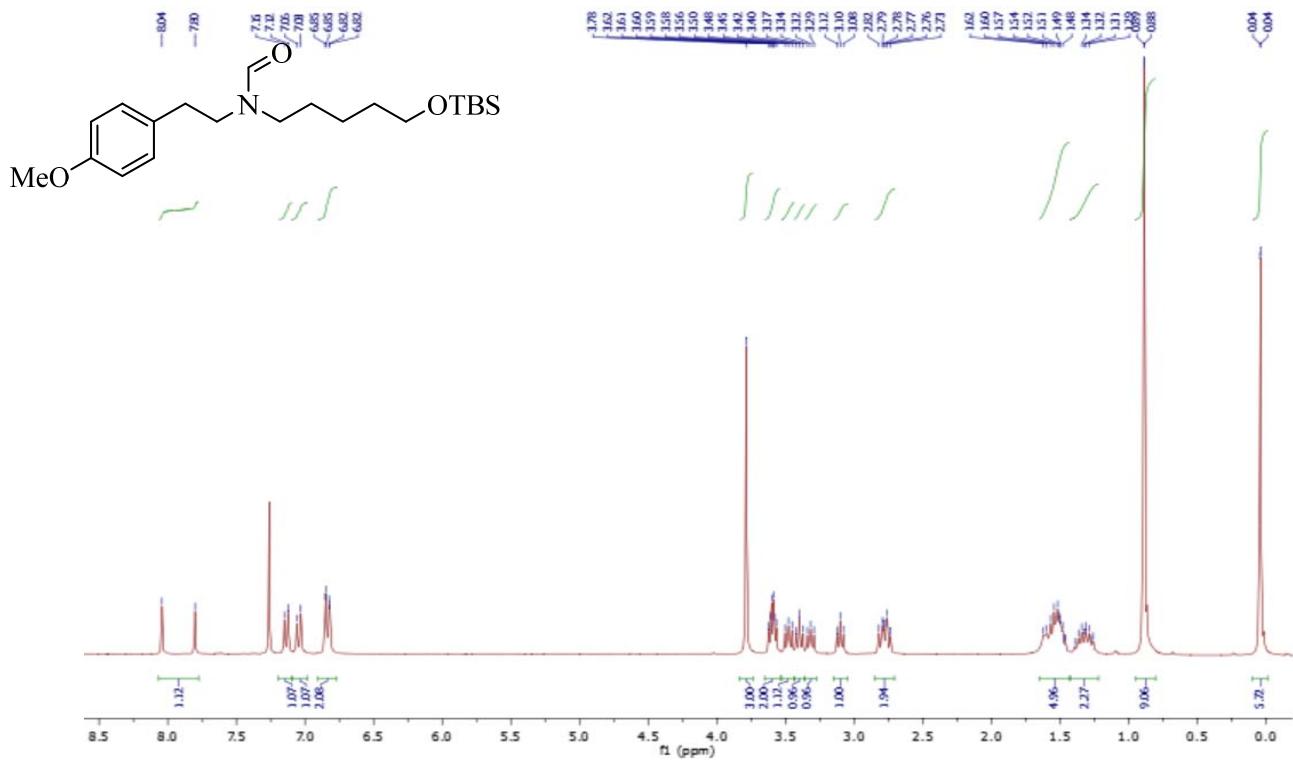


^{13}C RMN spectrum

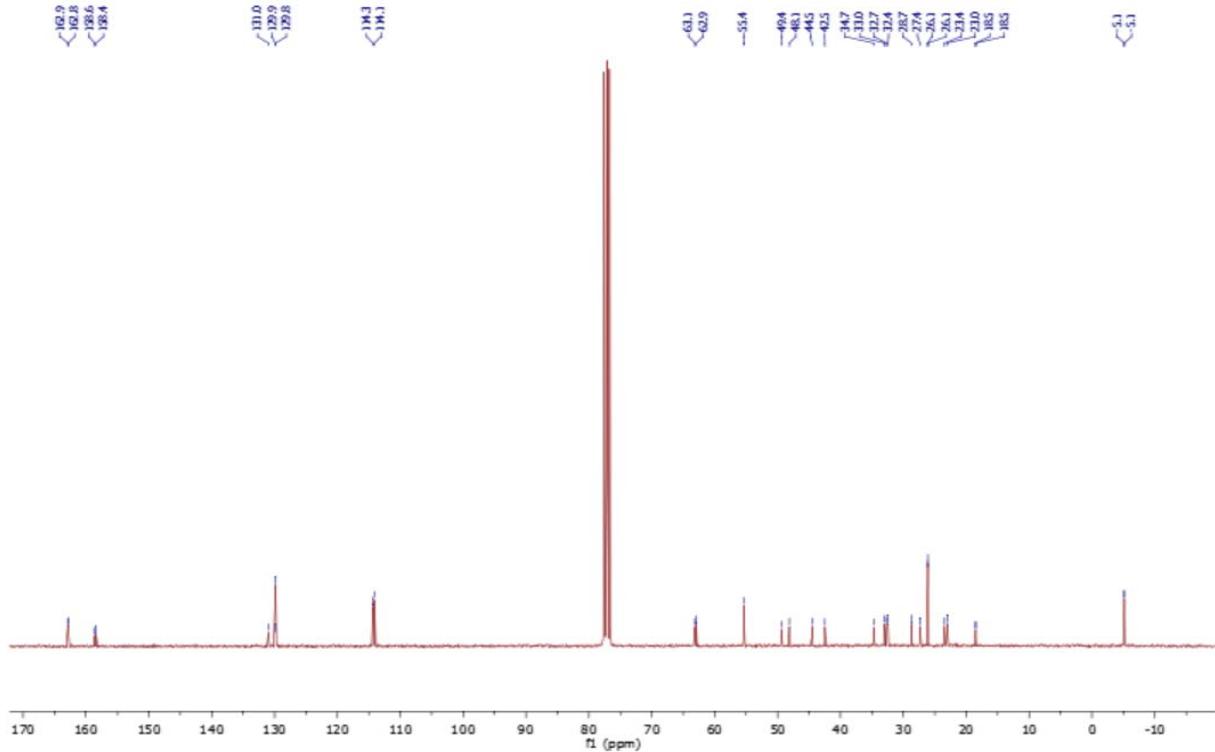


N-((tert-Butyldimethylsilyl)oxy)pentyl)-N-(4-methoxyphenethyl)formamide (1-60)

^1H RMN spectrum

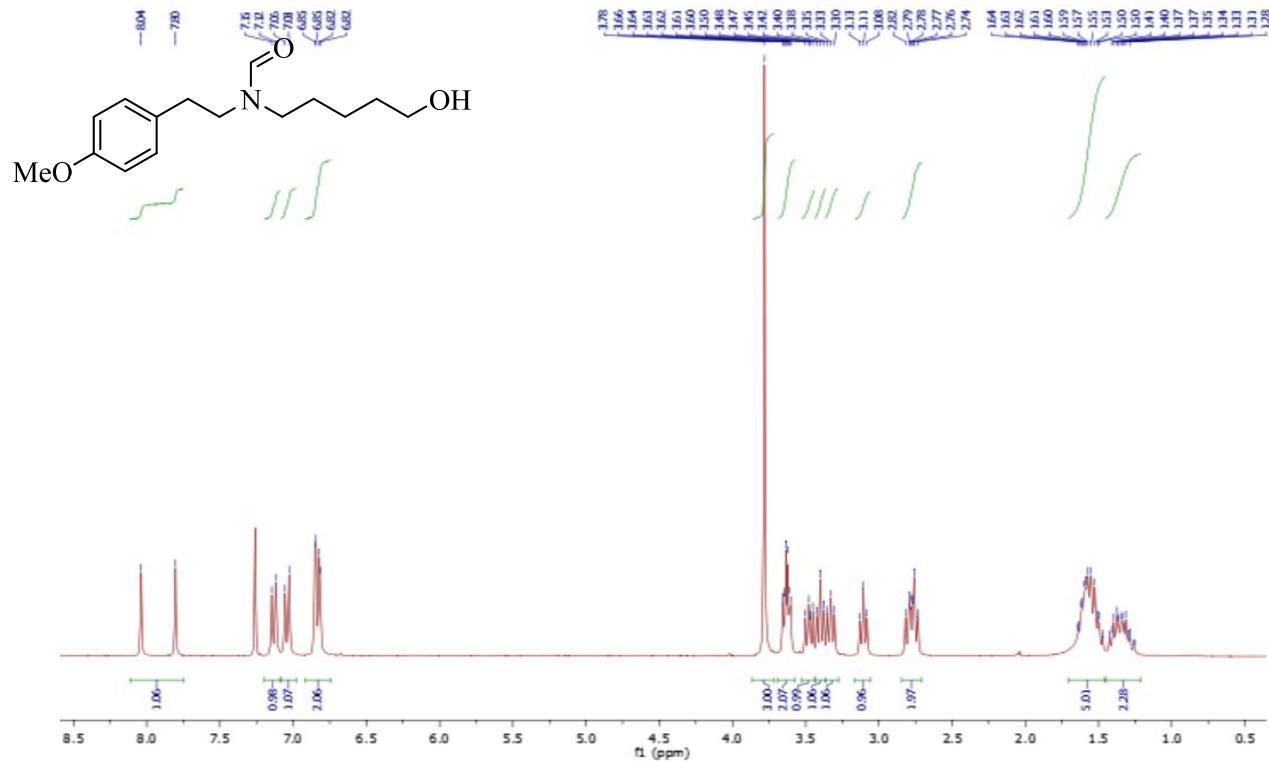


^{13}C RMN spectrum

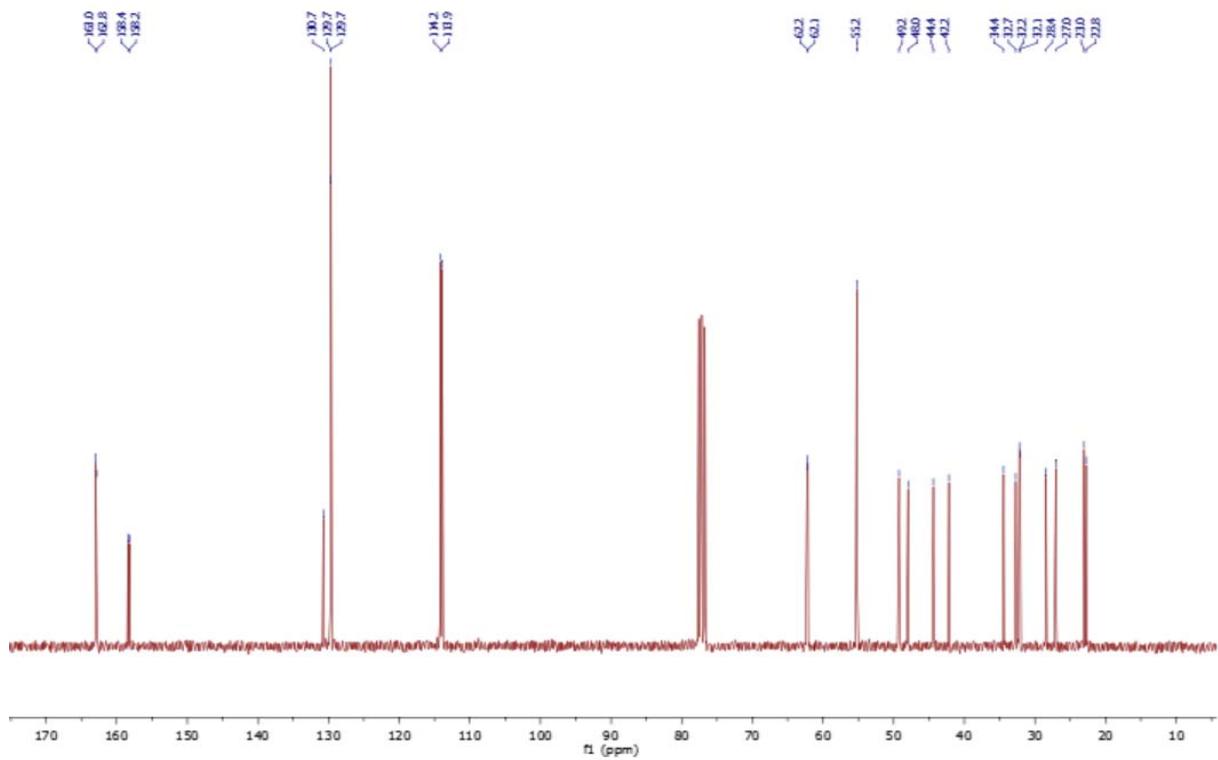


N-(5-Hydroxypentyl)-N-(4-methoxyphenethyl)formamide (1-61)

^1H RMN spectrum

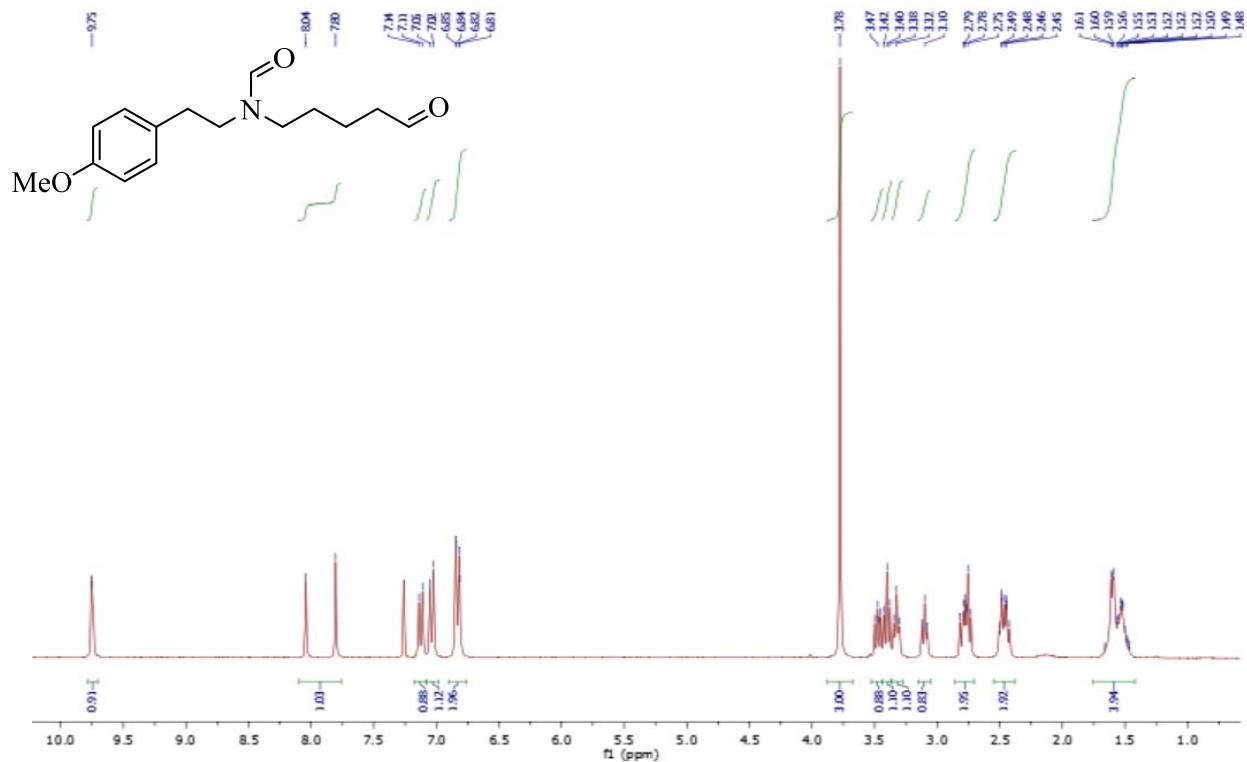


^{13}C RMN spectrum

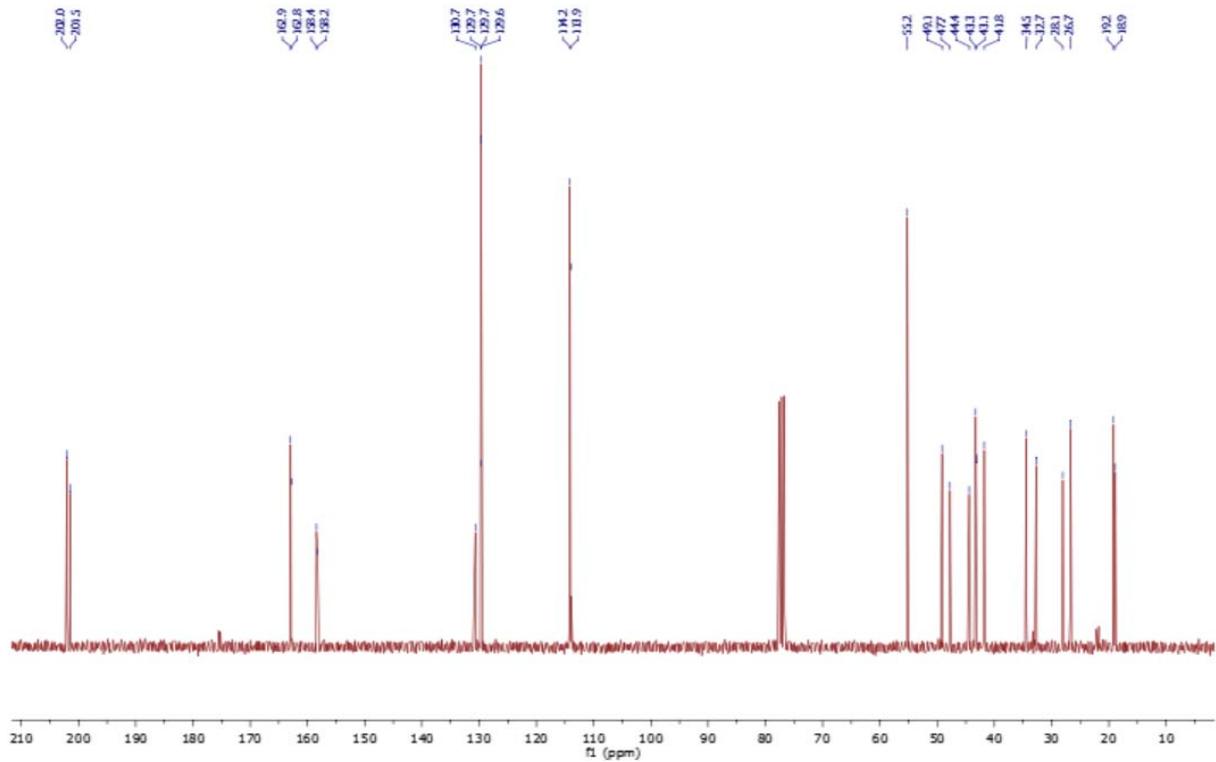


N-(4-Methoxyphenethyl)-N-(5-oxopentyl)formamide (1-62)

¹H RMN spectrum

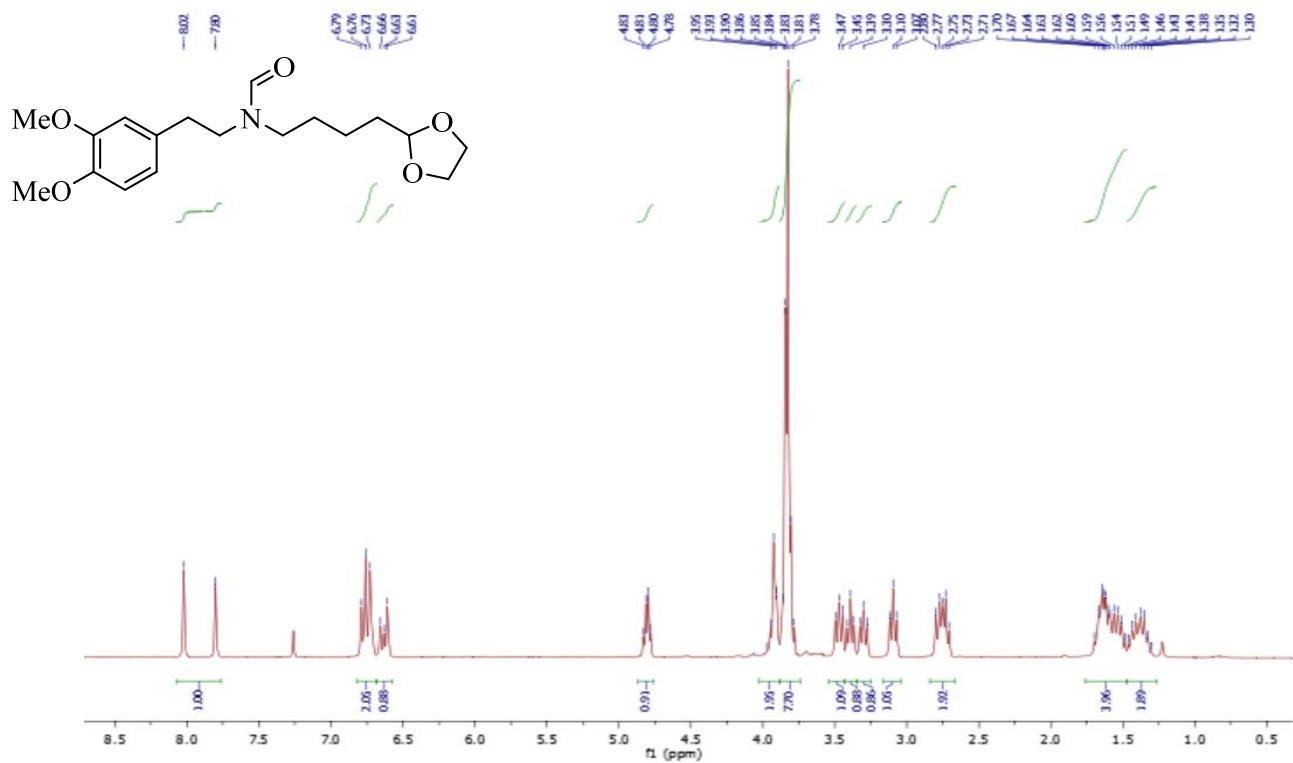


¹³C RMN spectrum

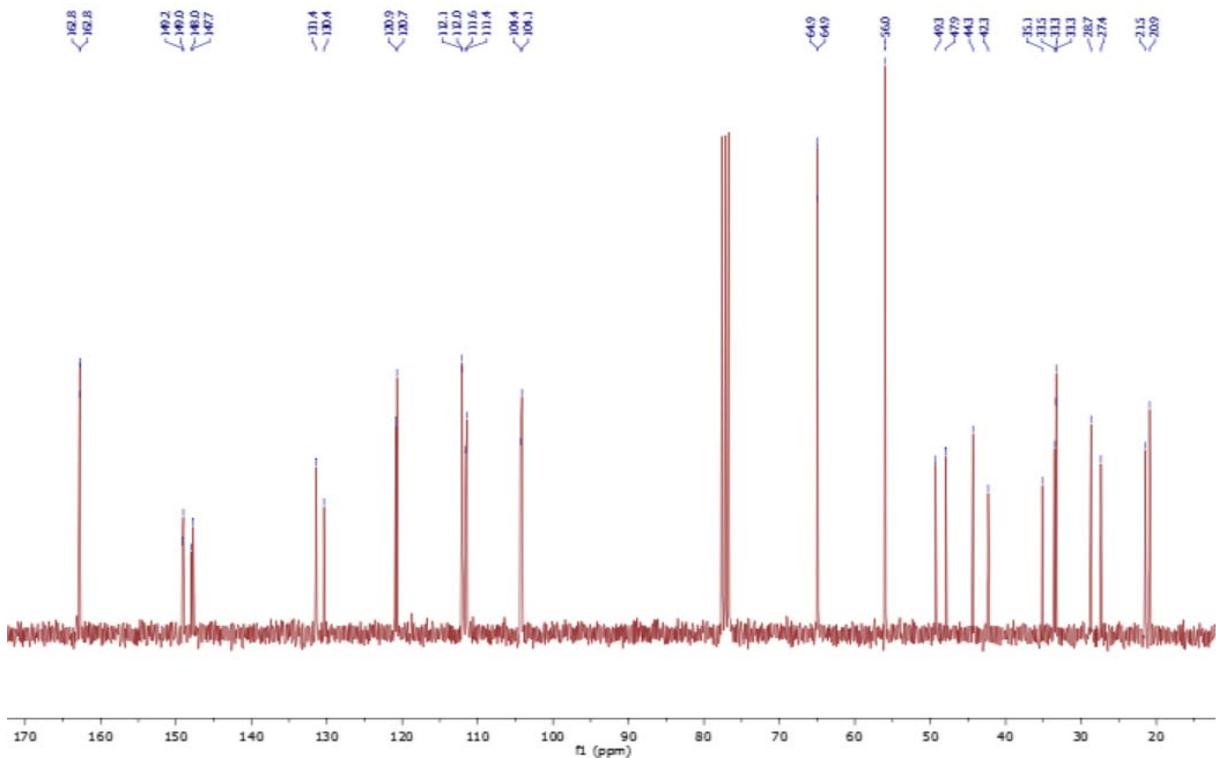


N-(4-(1,3-Dioxolan-2-yl)butyl)-N-(3,4-dimethoxyphenethyl)formamide (1-68)

¹H RMN spectrum

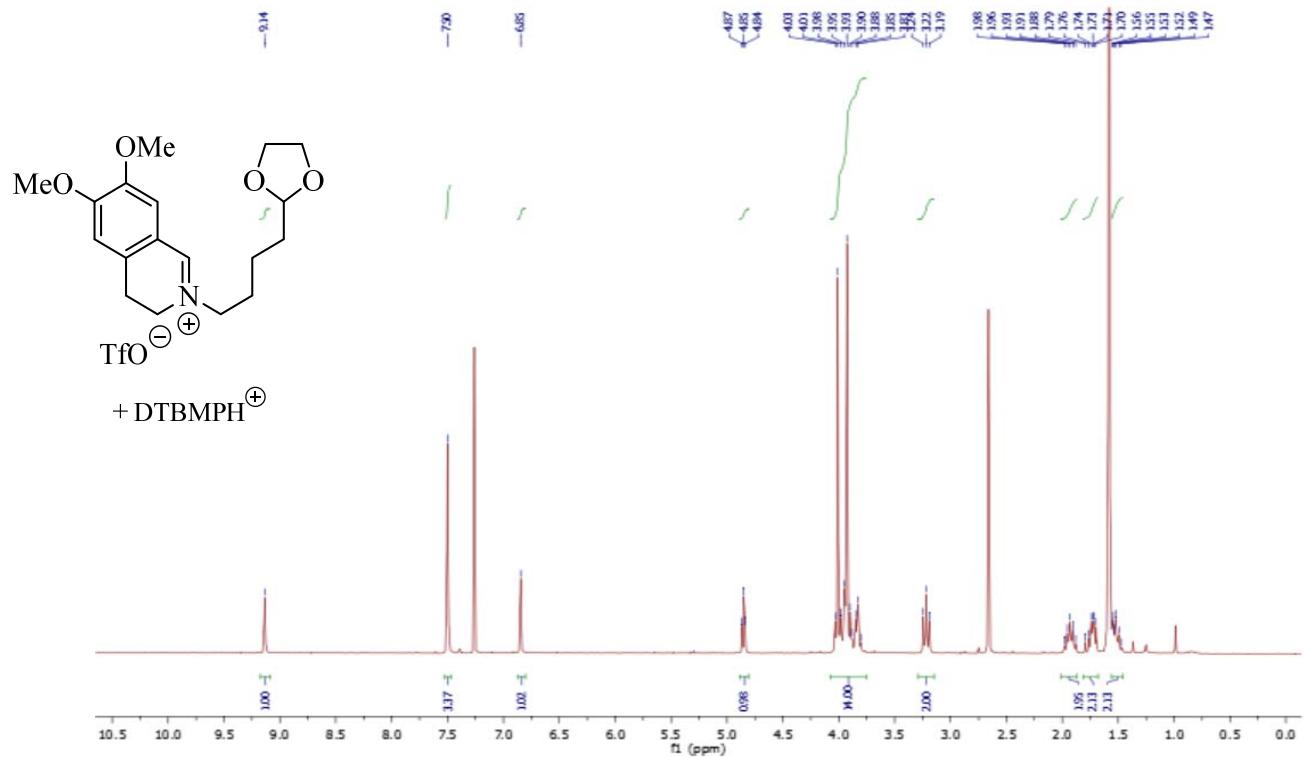


¹³C RMN spectrum



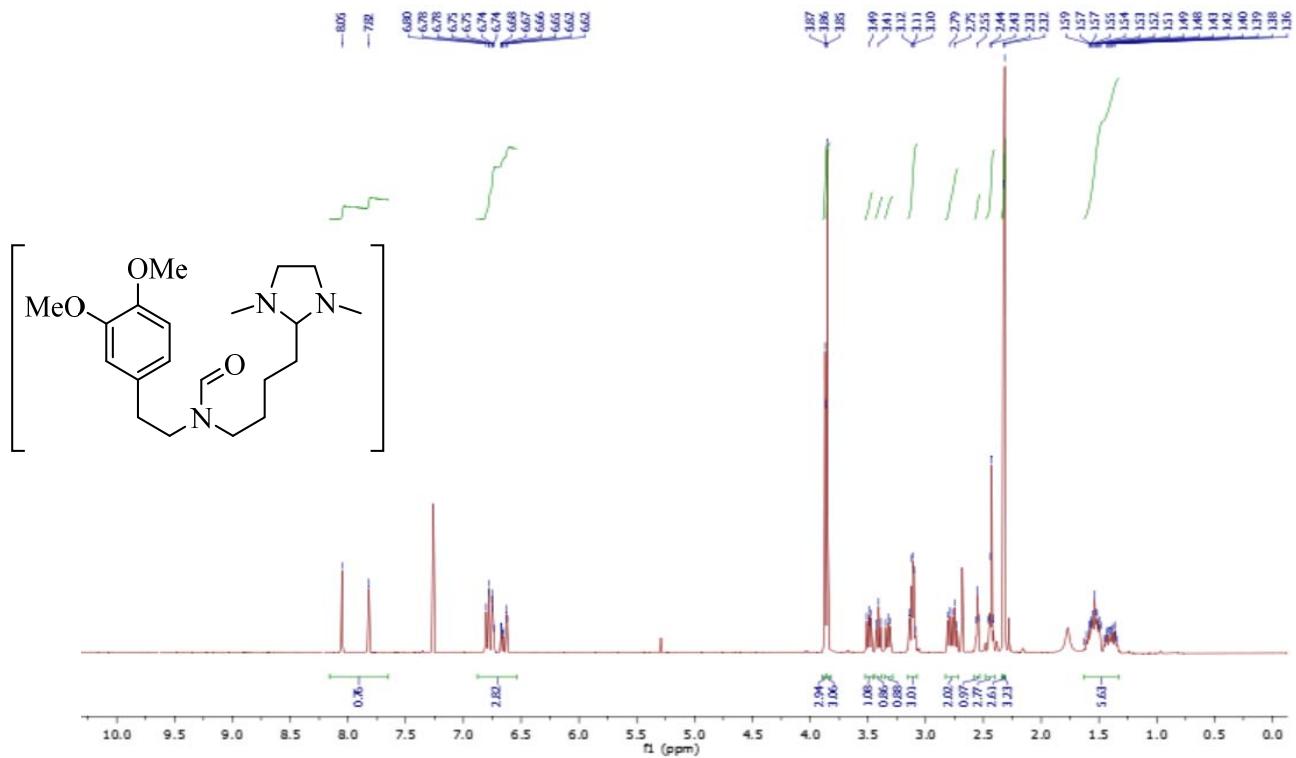
Iminium ion (1-68a)

^1H RMN spectrum



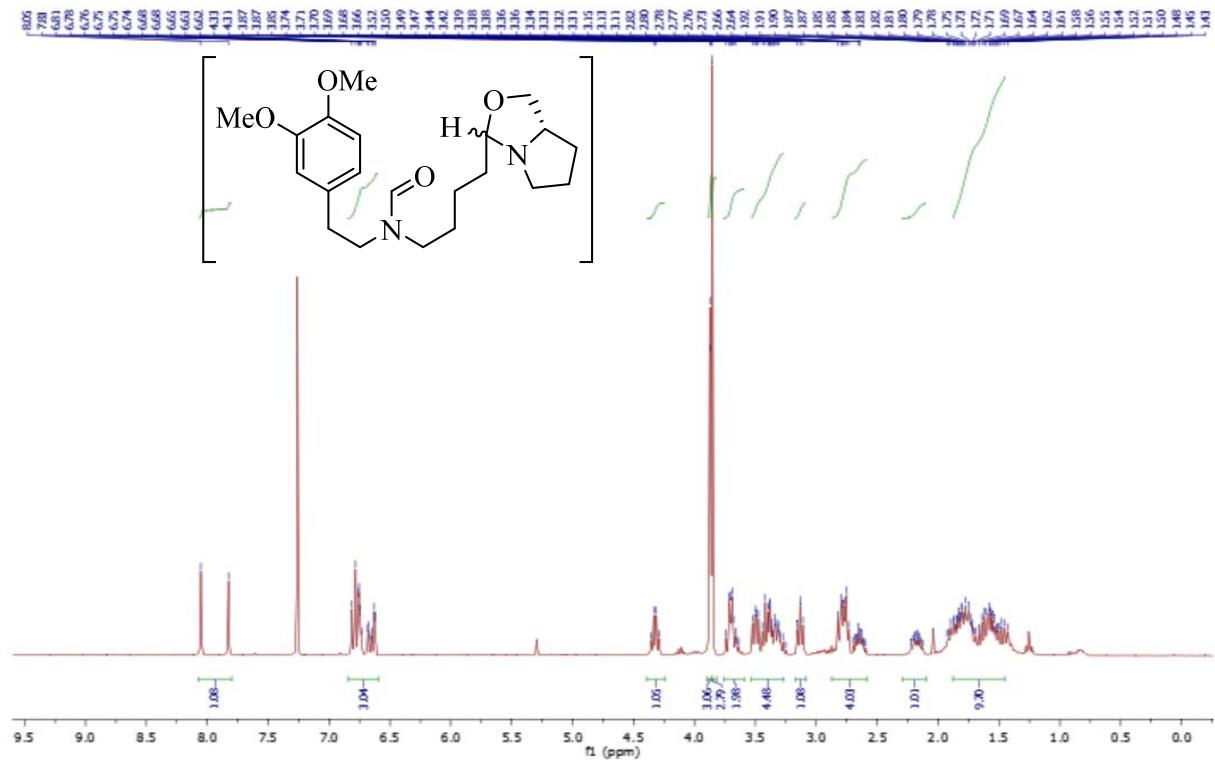
N-(3,4-Dimethoxyphenethyl)-N-(4-(1,3-dimethylimidazolidin-2-yl)butyl)formamide (1-70)

¹H RMN spectrum



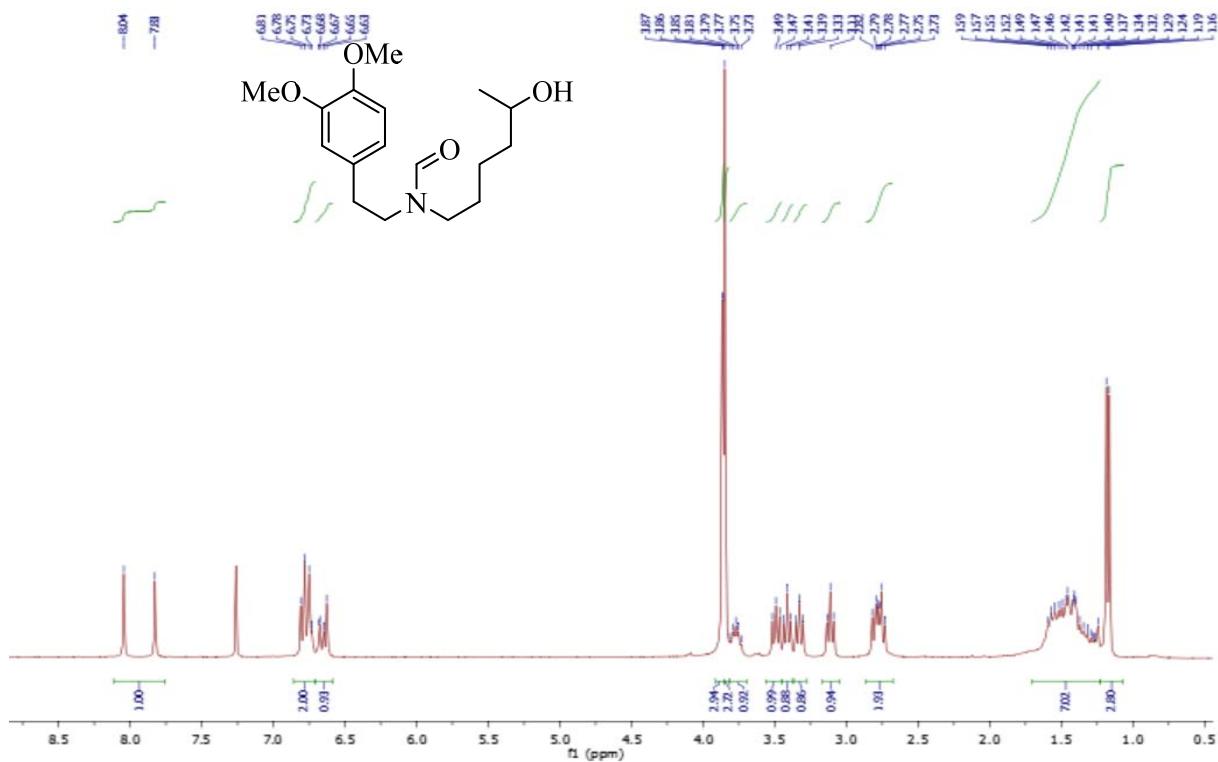
N-(3,4-Dimethoxyphenethyl)-N-(4-((7aS)-tetrahydro-1*H*,3*H*-pyrrolo[1,2-*c*]oxazol-3-yl)butyl)formamide (1-72)

¹H RMN spectrum

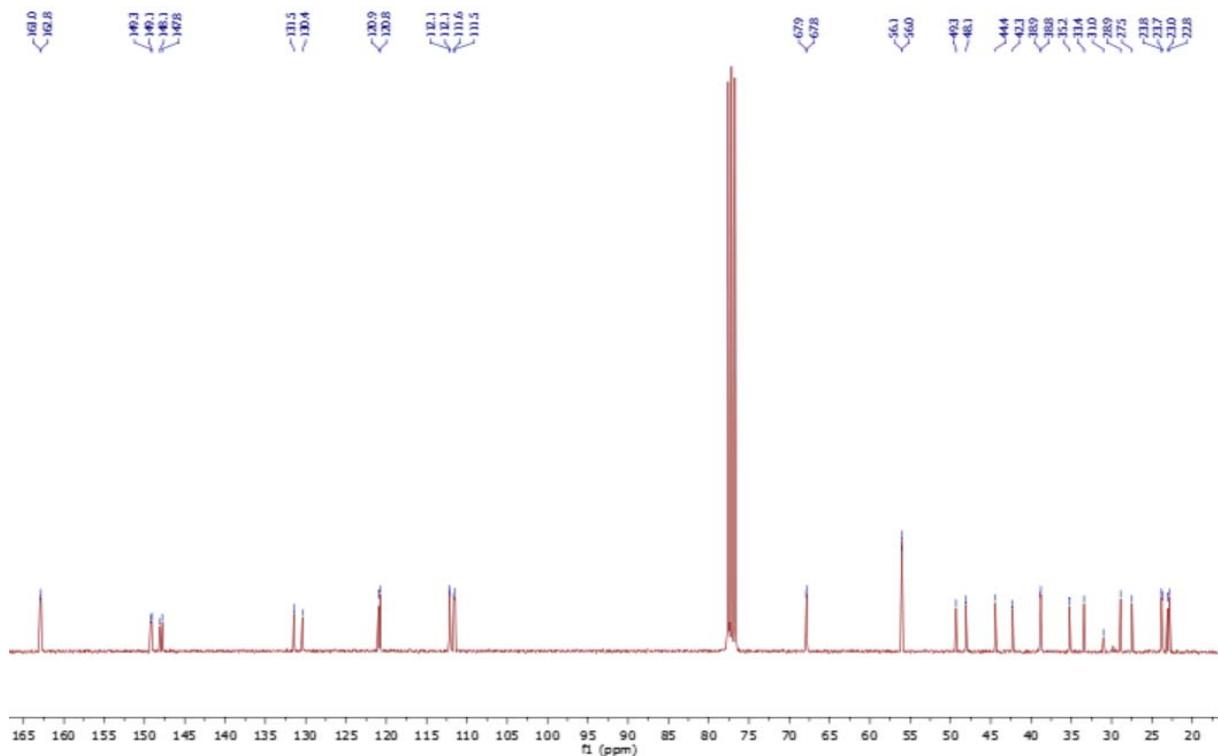


***N*-(3,4-Dimethoxyphenethyl)-*N*-(5-hydroxyhexyl)formamide (2-1)**

¹H RMN spectrum

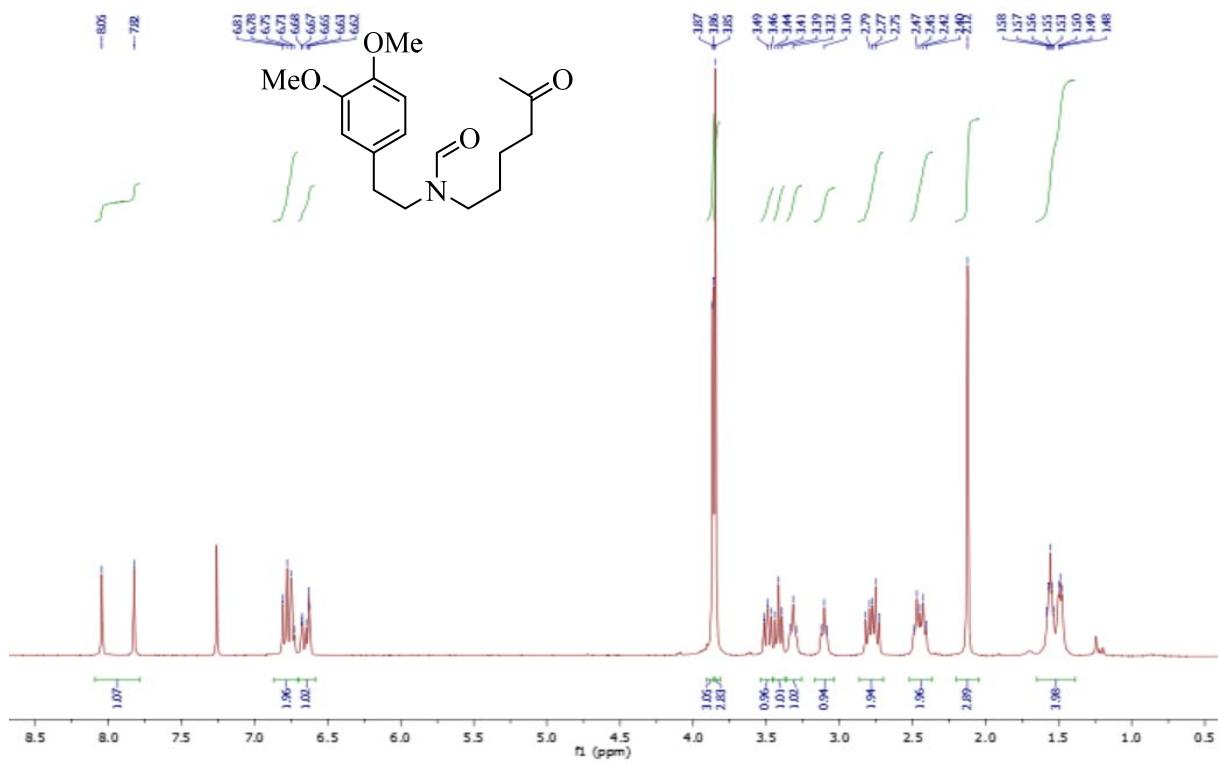


¹³C RMN spectrum

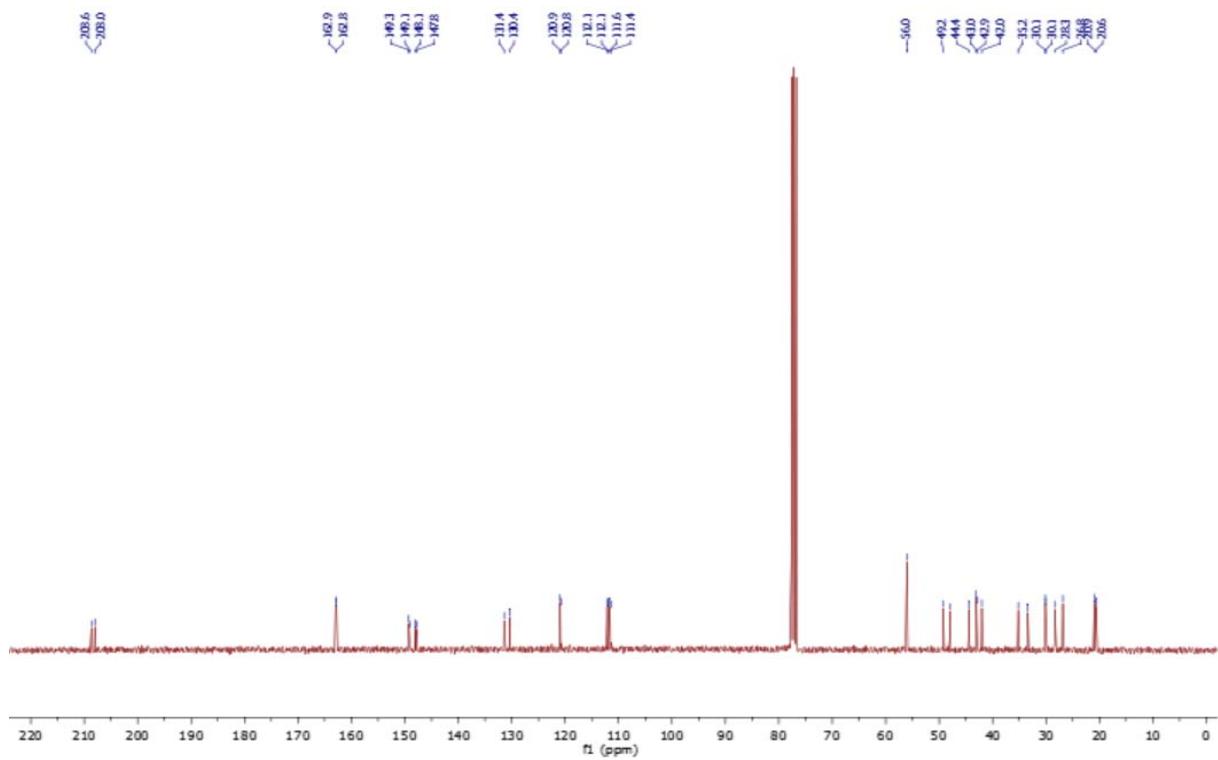


N-(3,4-Dimethoxyphenethyl)-N-(5-oxohexyl)formamide (2-2)

¹H RMN spectrum

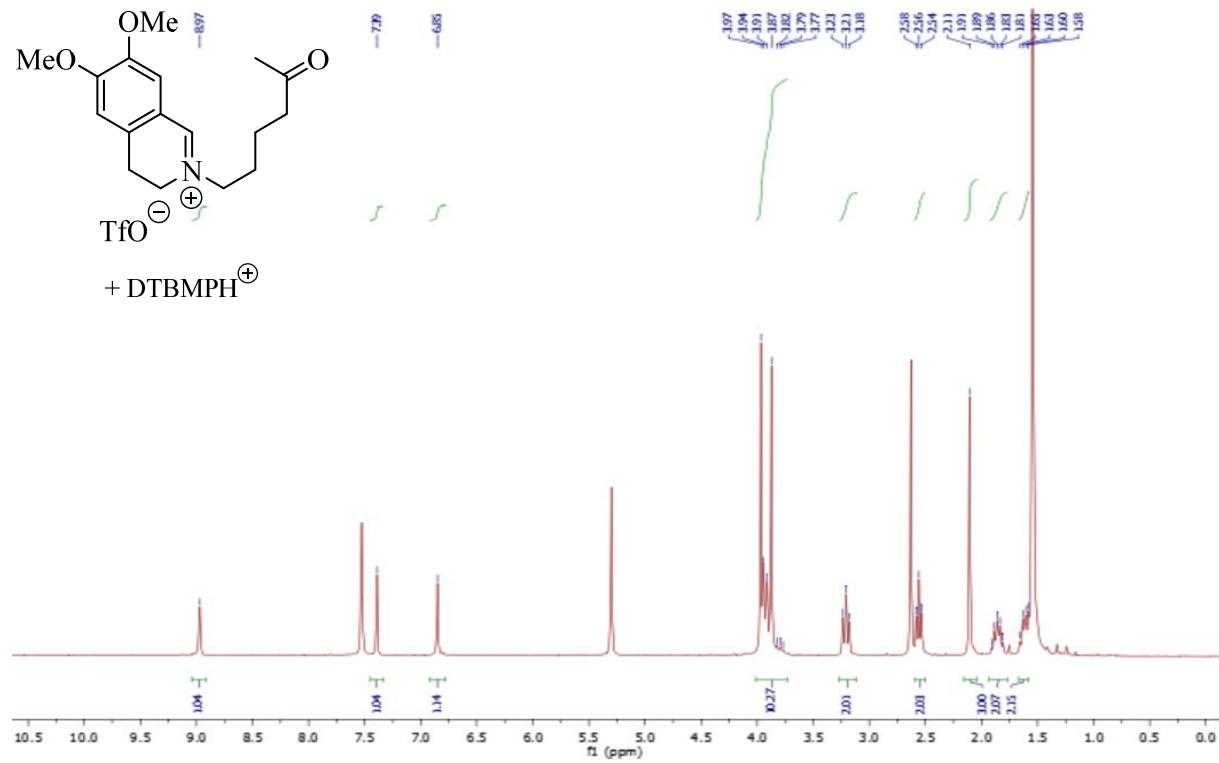


¹³C RMN spectrum



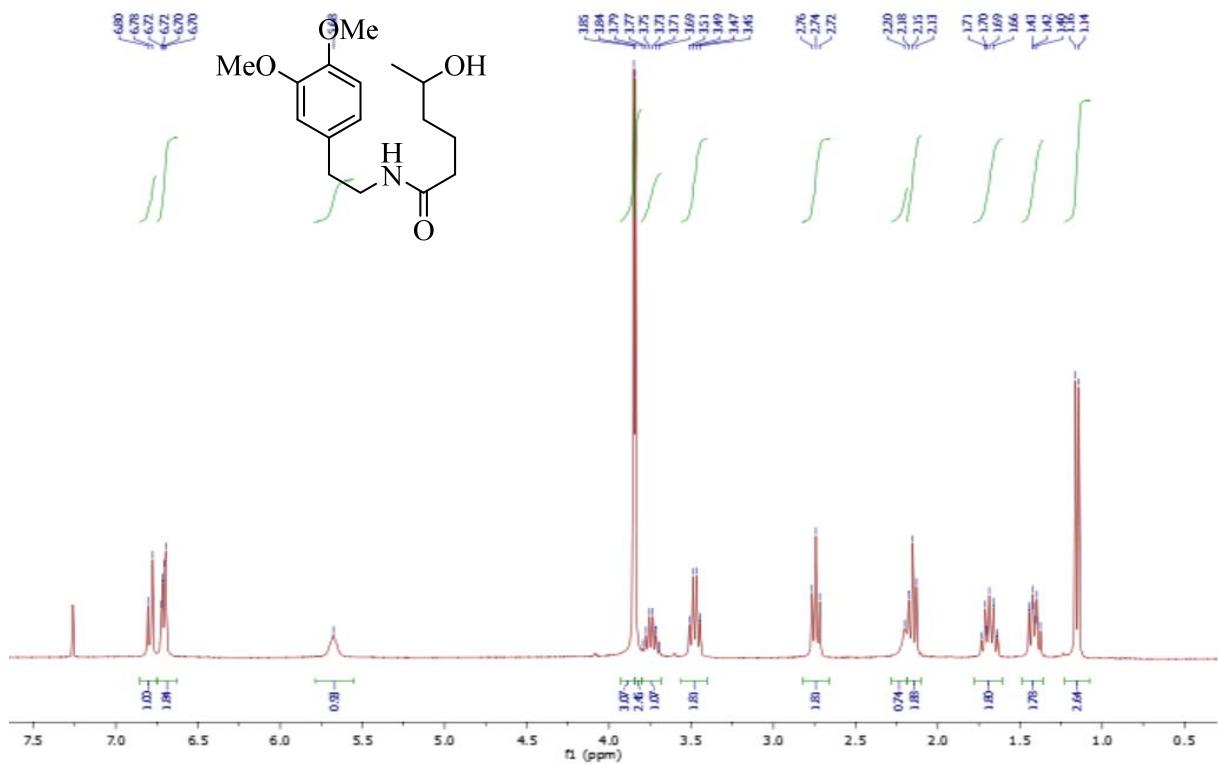
Iminium ion (2-2a)

^1H RMN spectrum

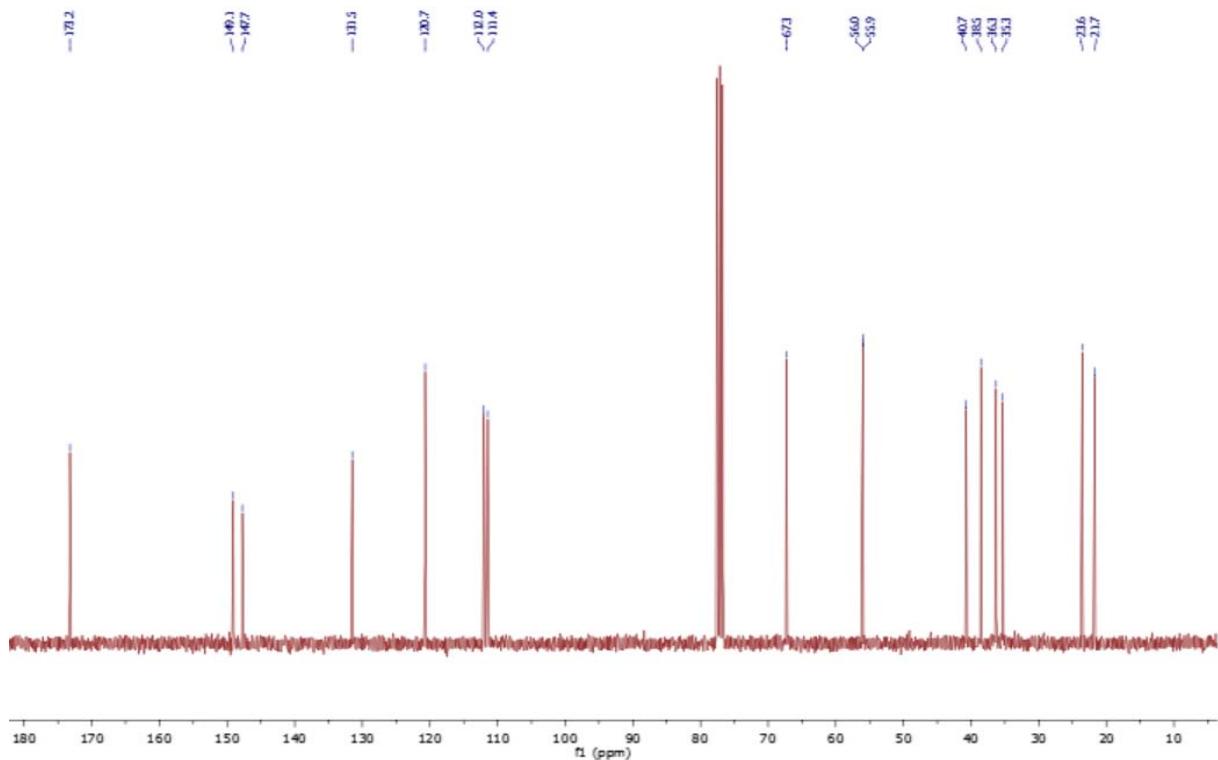


N-(3,4-Dimethoxyphenethyl)-5-hydroxyhexanamide (2-3)

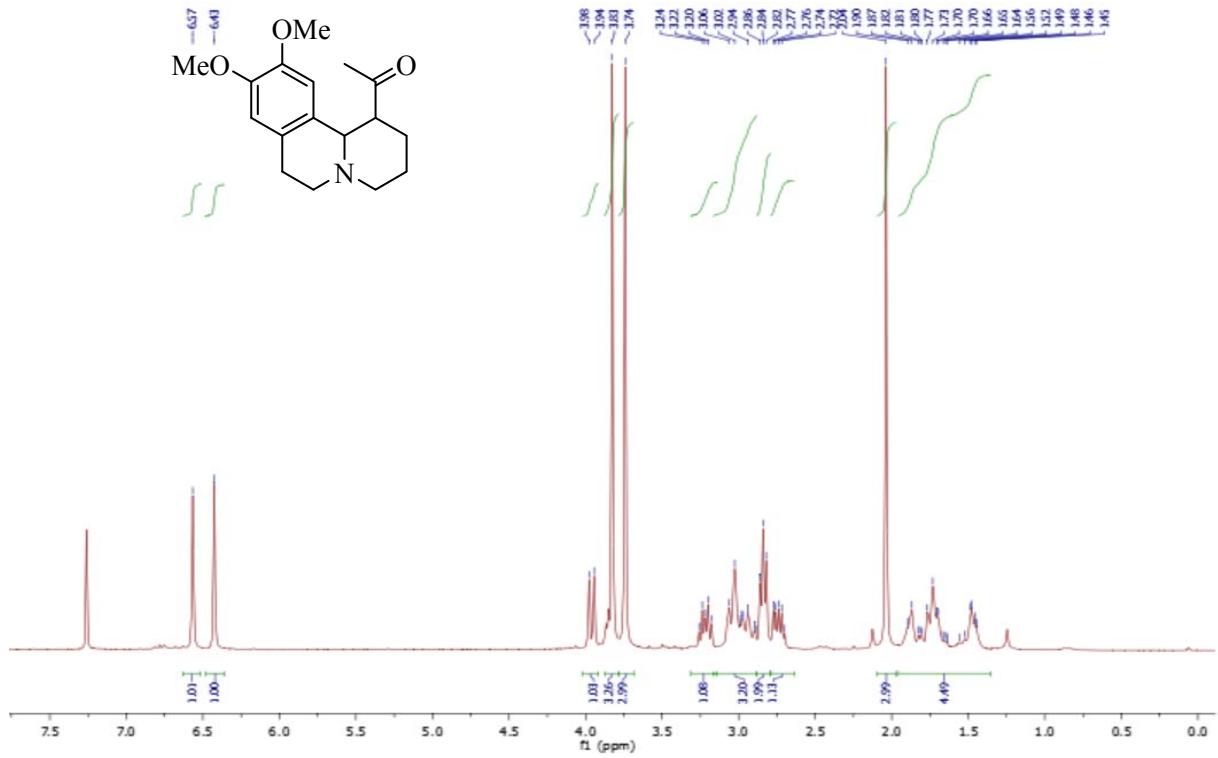
¹H RMN spectrum



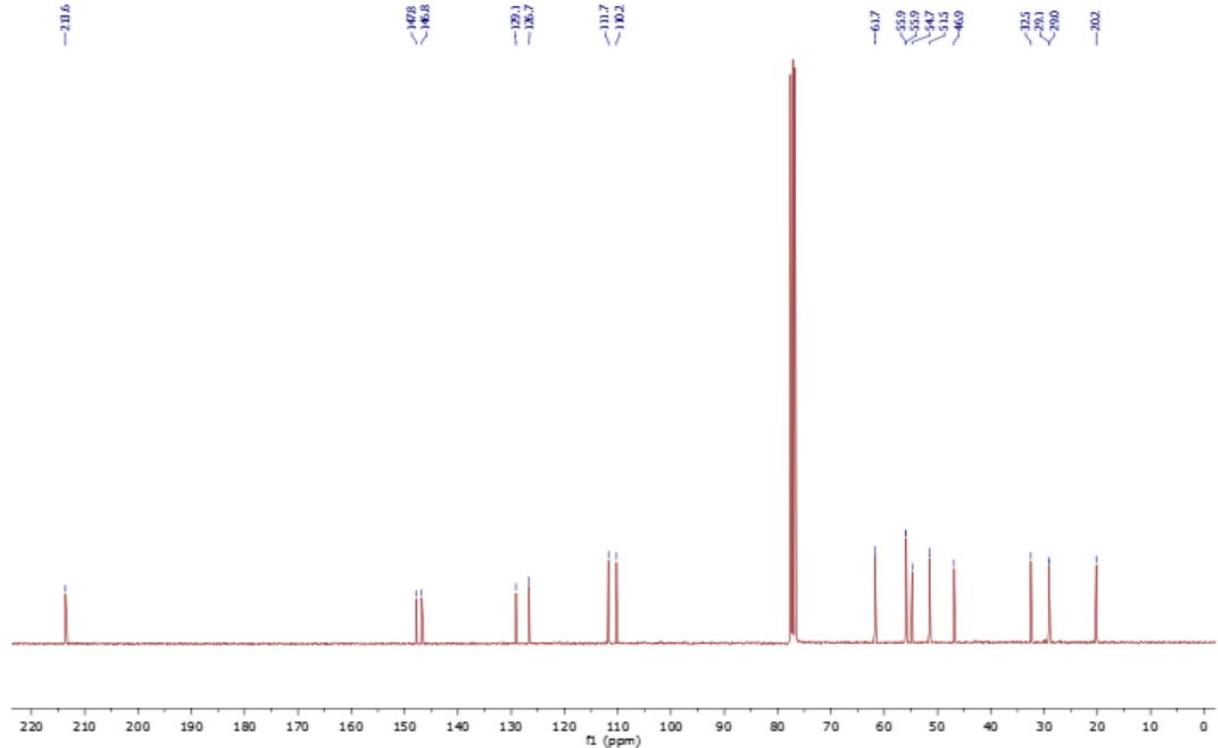
¹³C RMN spectrum



1-(9,10-Dimethoxy-1,3,4,6,7,11b-hexahydro-2H-pyrido[2,1-a]isoquinolin-1-yl)ethan-1-one (2-4)
¹H RMN spectrum

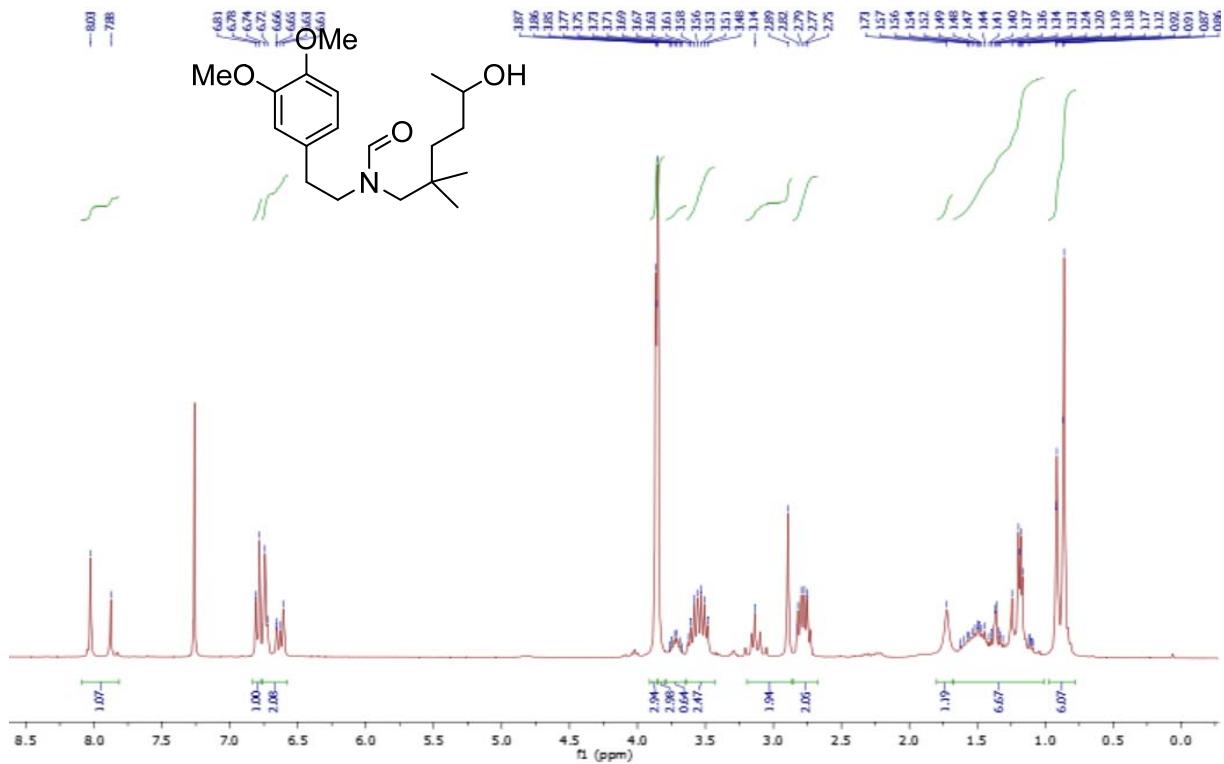


¹³C RMN spectrum

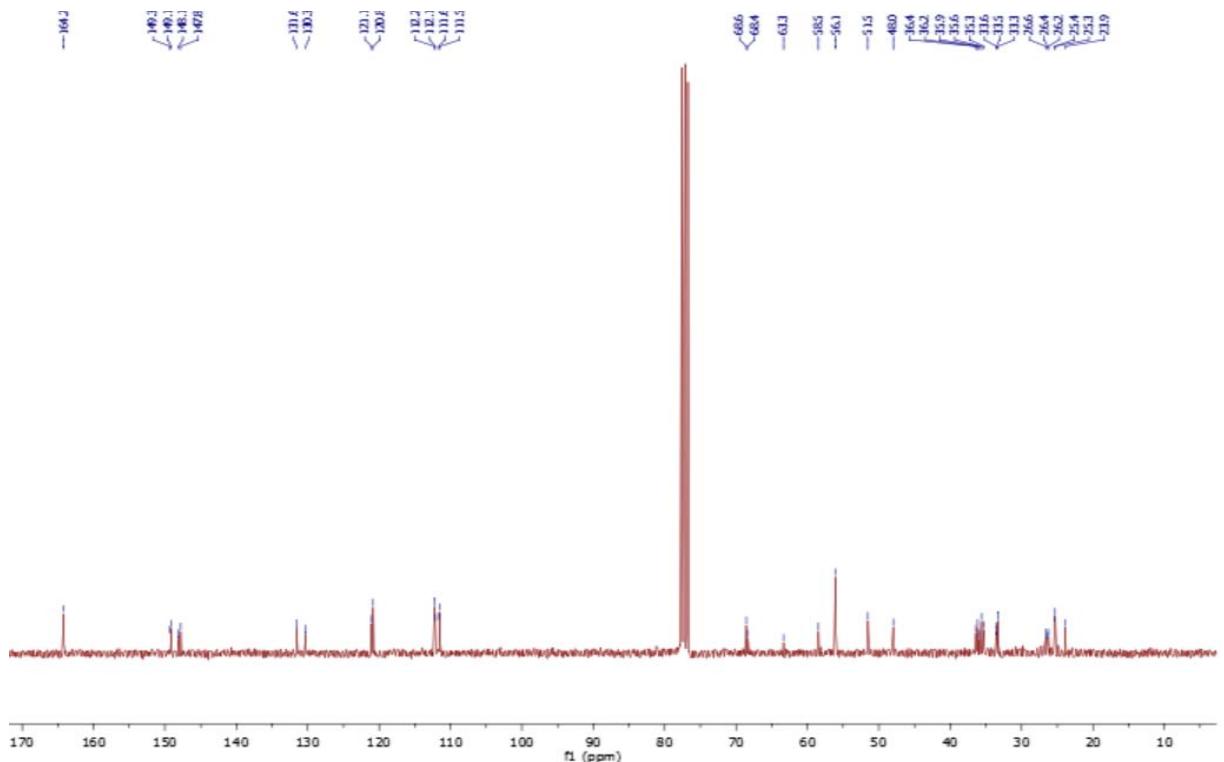


N-(3,4-Dimethoxyphenethyl)-N-(5-hydroxy-2,2-dimethylhexyl)formamide (2-5)

^1H RMN spectrum

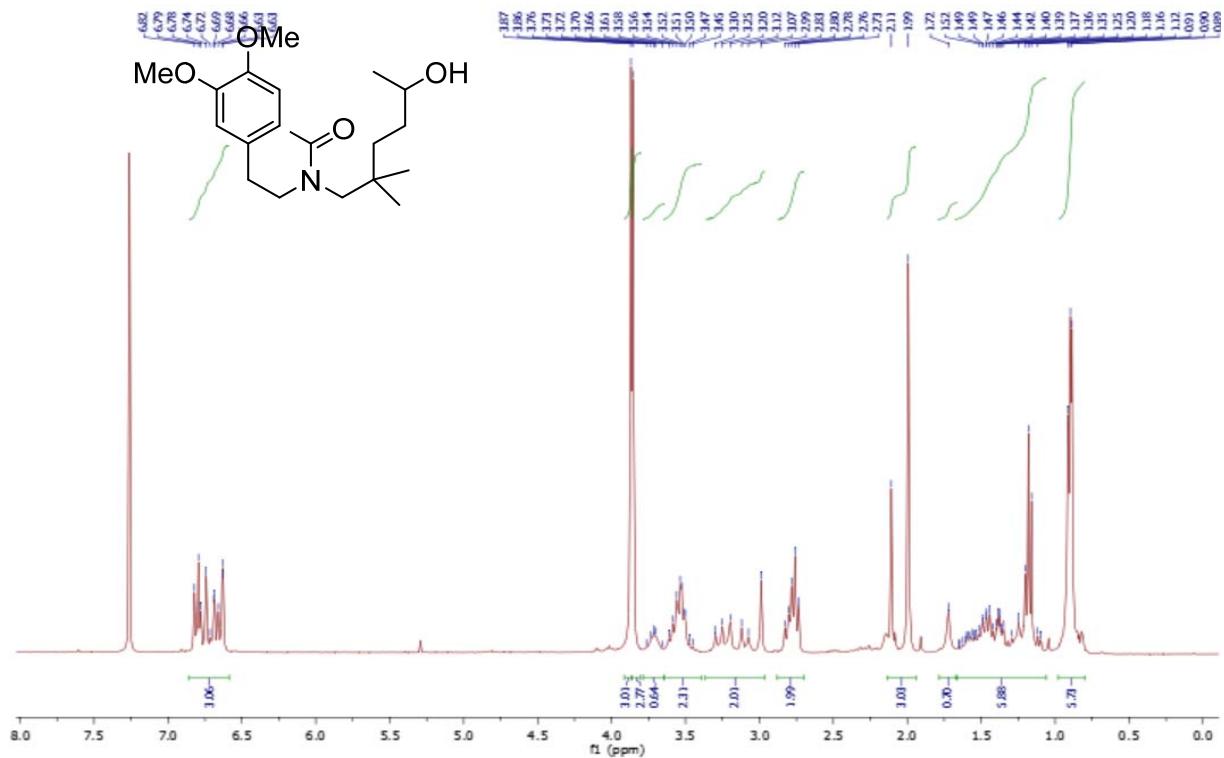


^{13}C RMN spectrum

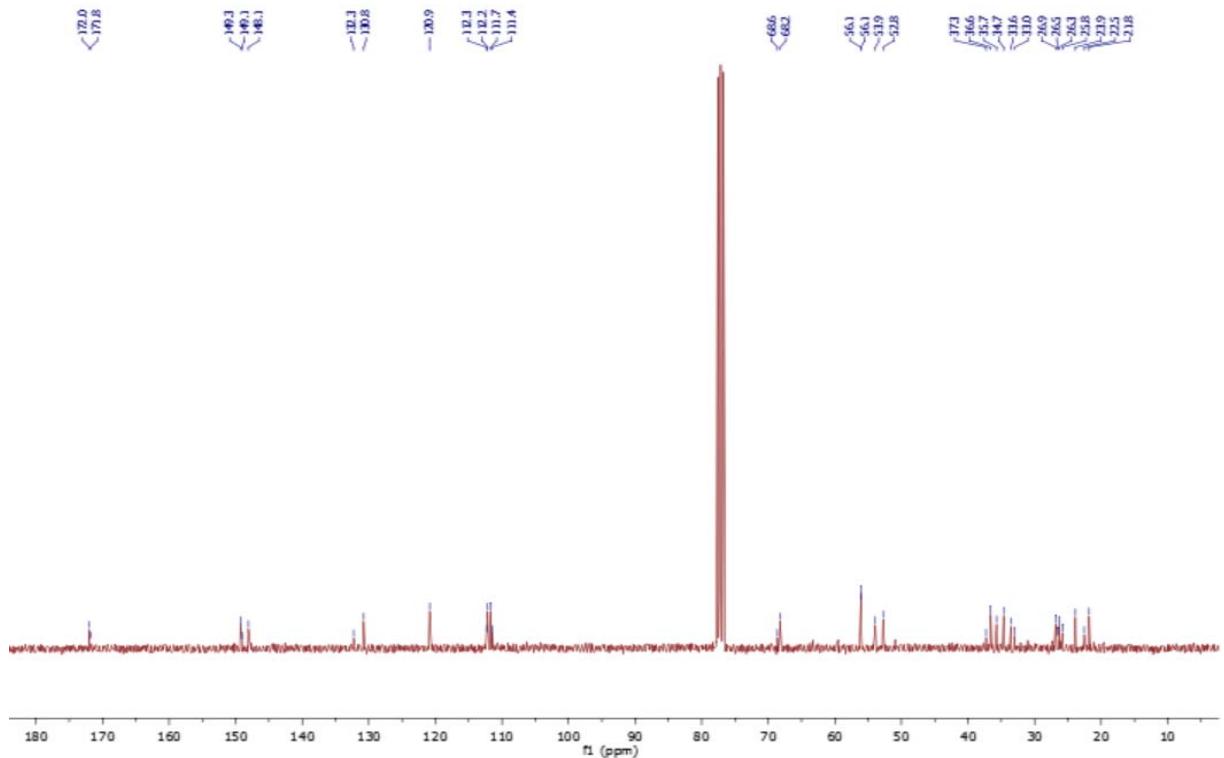


N-(3,4-Dimethoxyphenethyl)-N-(5-hydroxy-2,2-dimethylhexyl)acetamide (2-6)

^1H RMN spectrum

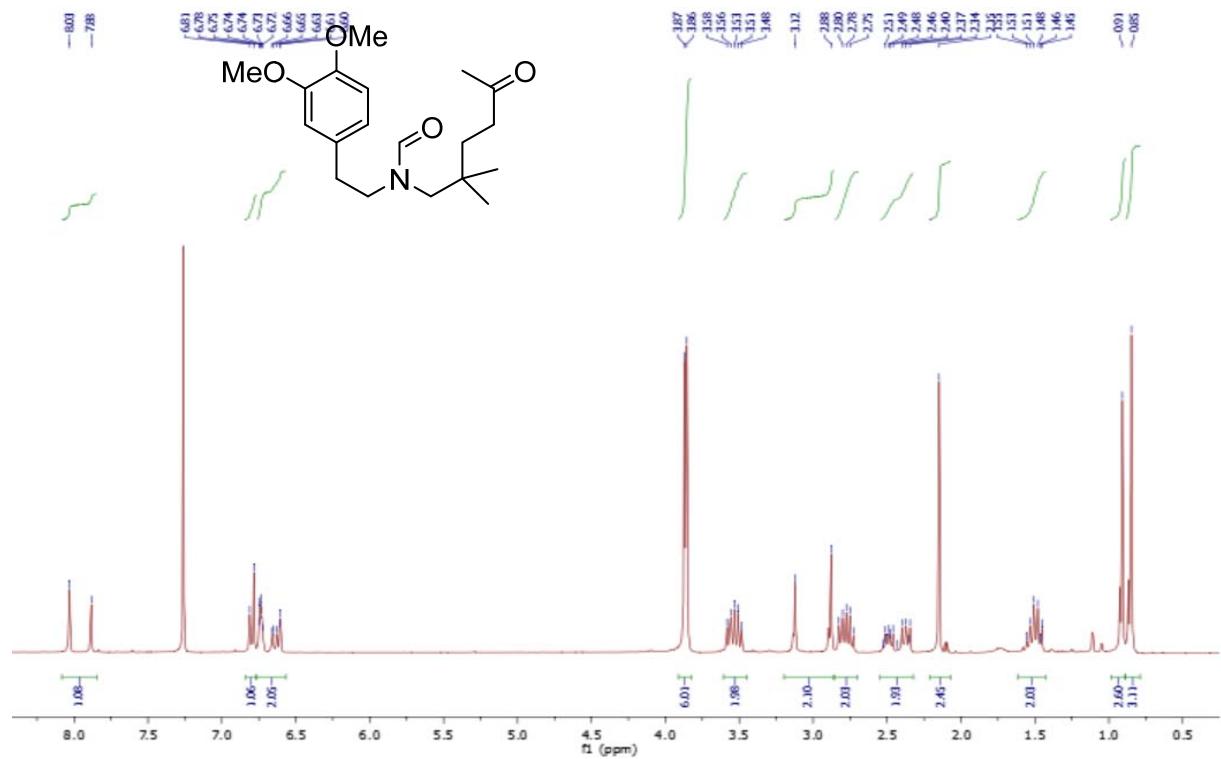


^{13}C RMN spectrum

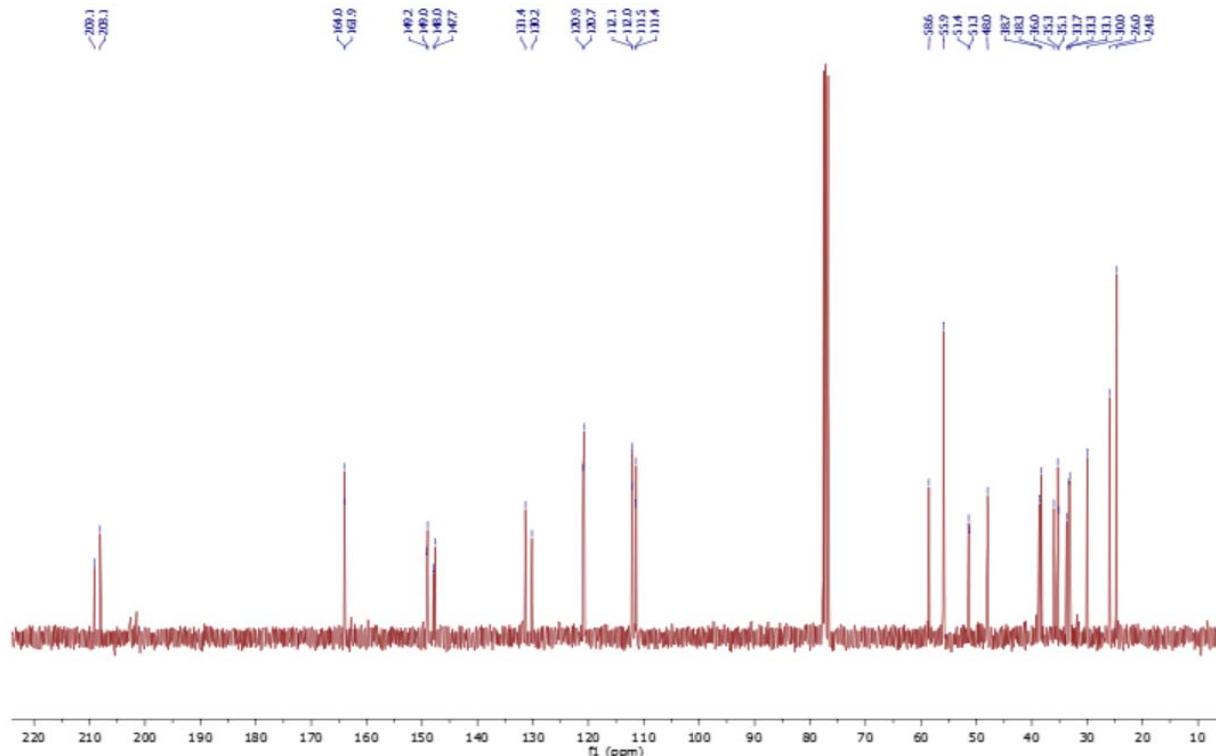


***N*-(3,4-Dimethoxyphenethyl)-*N*-(2,2-dimethyl-5-oxohexyl)formamide (2-7)**

¹H RMN spectrum

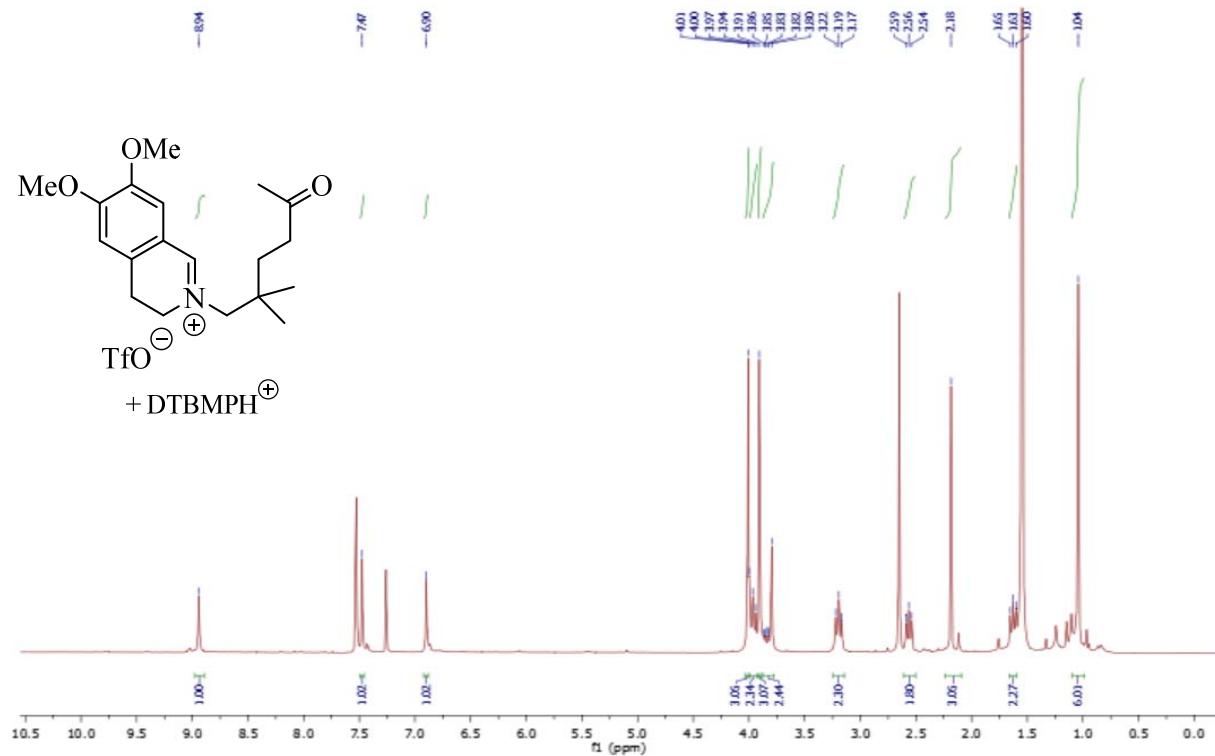


¹³C RMN spectrum



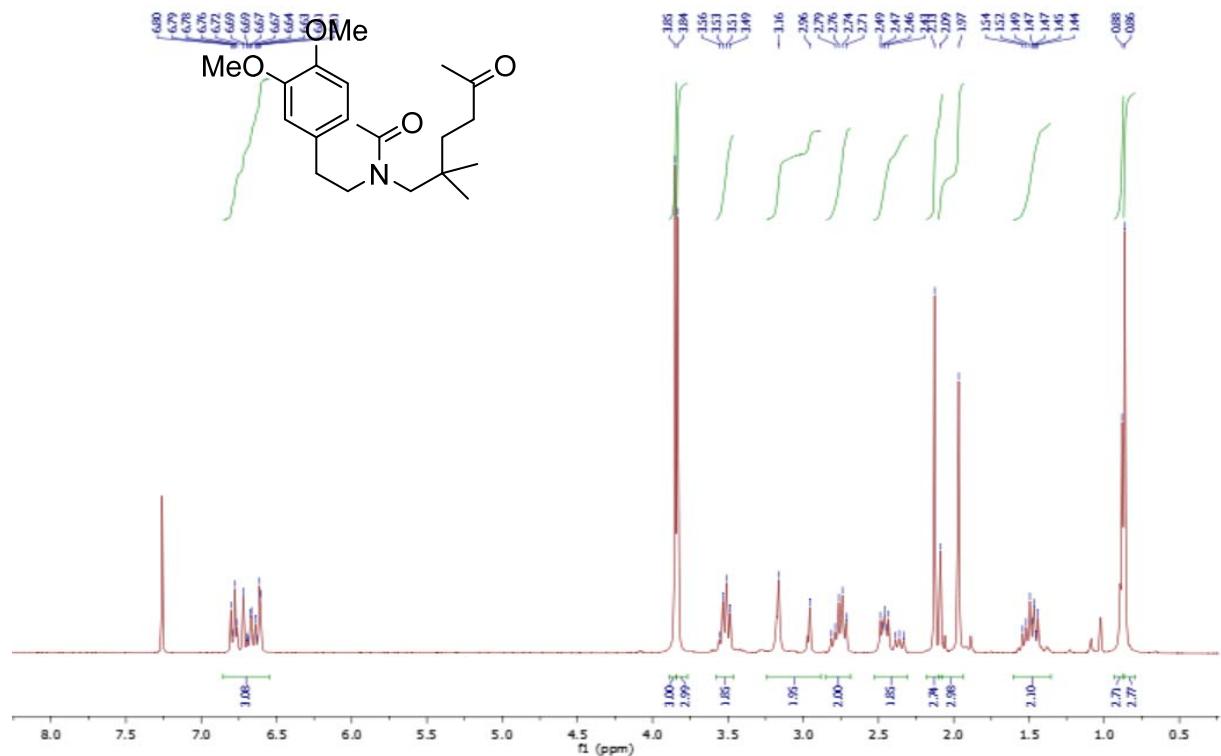
Iminium ion (2-7a)

^1H RMN spectrum

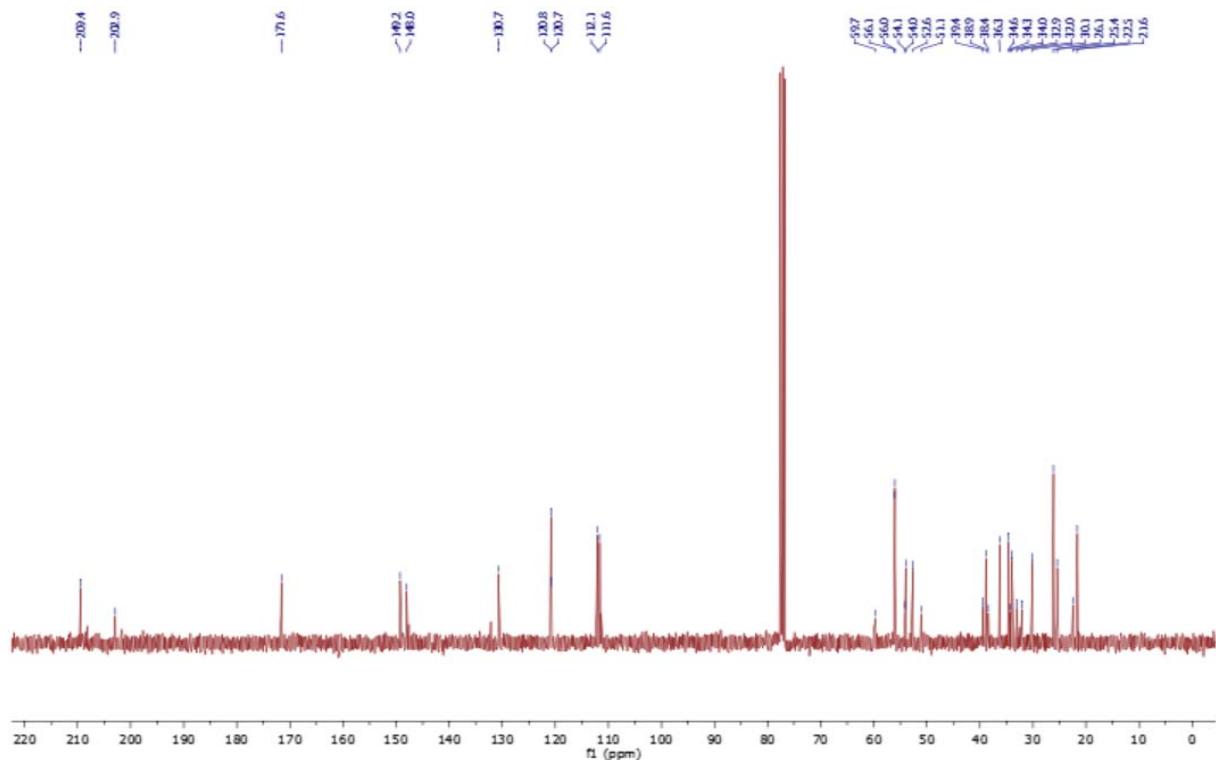


N-(3,4-dimethoxyphenethyl)-*N*-(2,2-dimethyl-5-oxohexyl)acetamide (2-8)

¹H RMN spectrum

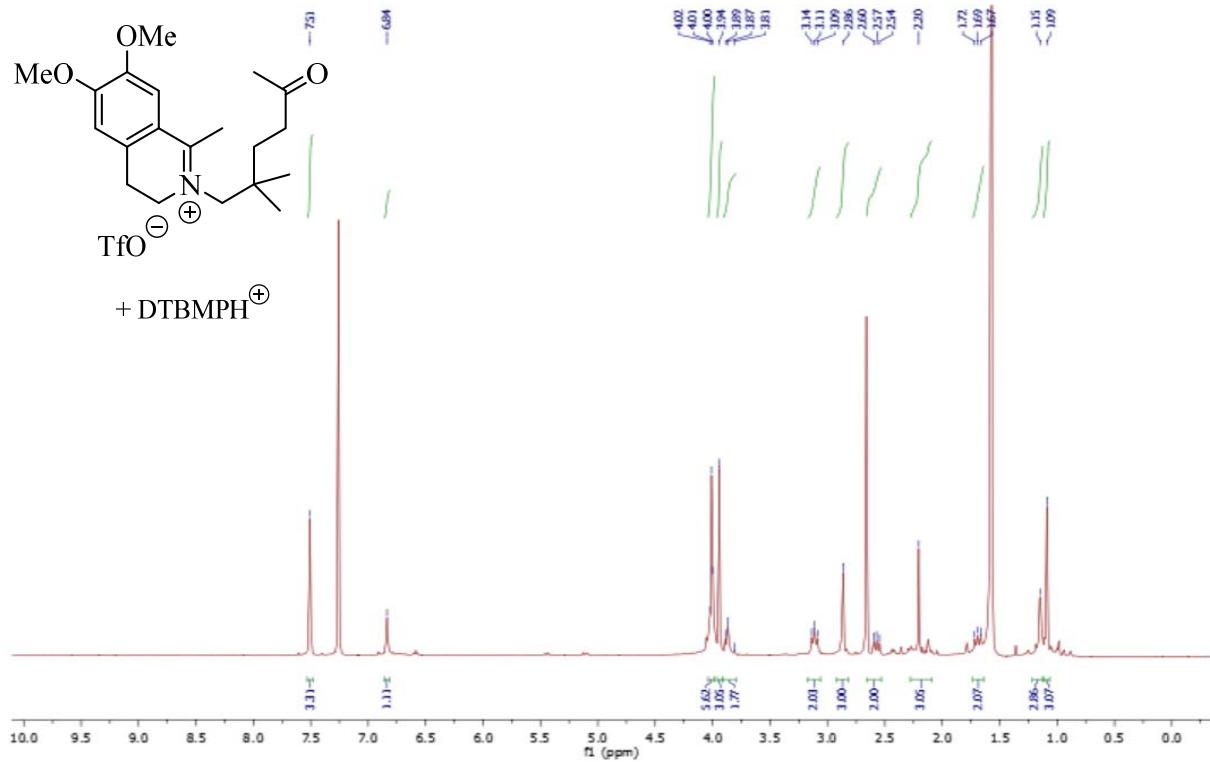


¹³C RMN spectrum



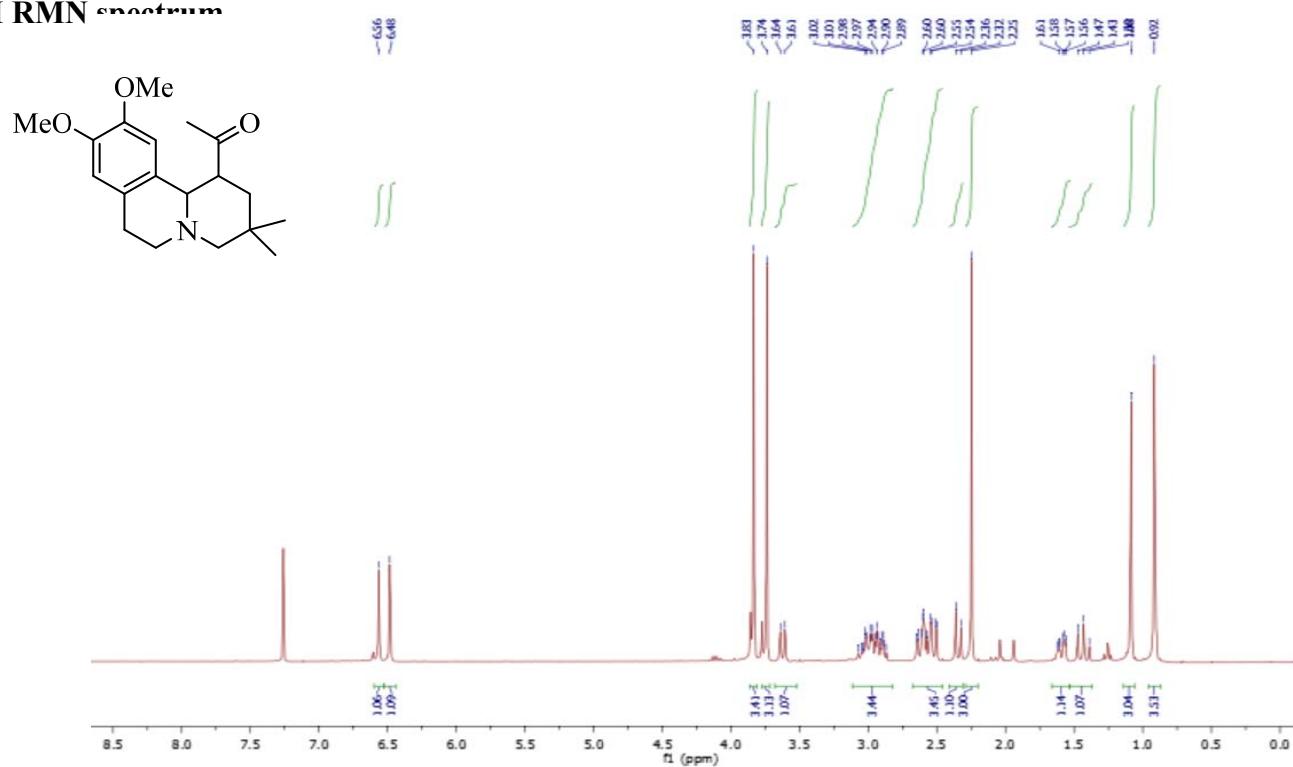
Iminium ion (2-8a)

¹H RMN spectrum

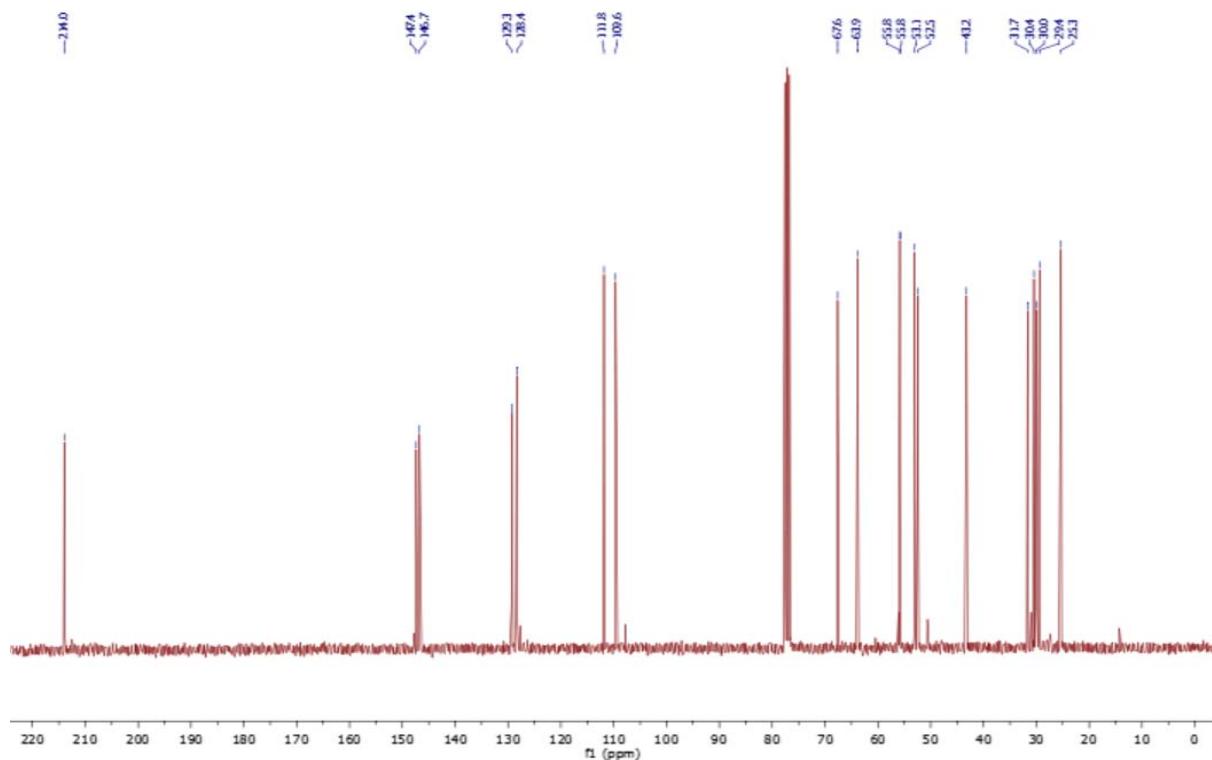


1-(9,10-dimethoxy-3,3-dimethyl-1,3,4,6,7,11b-hexahydro-2H-pyrido[2,1-a]isoquinolin-1-yl)ethan-1-one (2-9)

^1H RMN spectrum

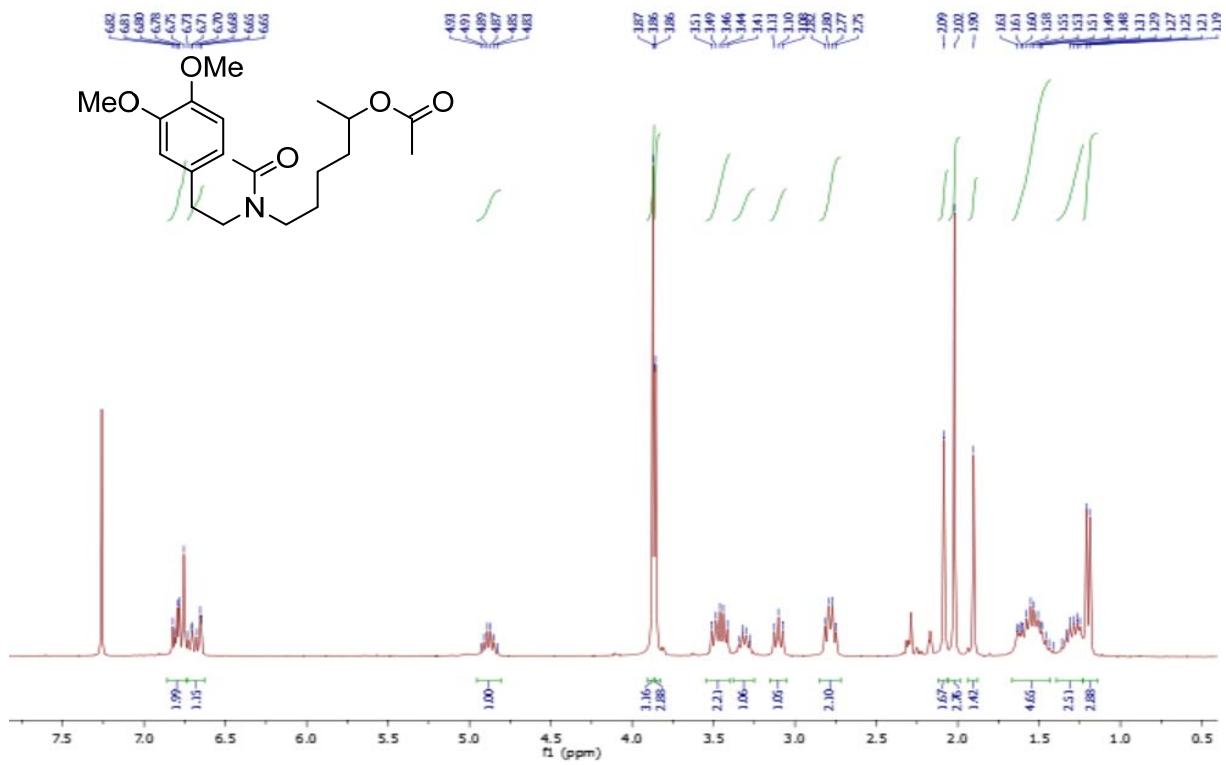


^{13}C RMN spectrum

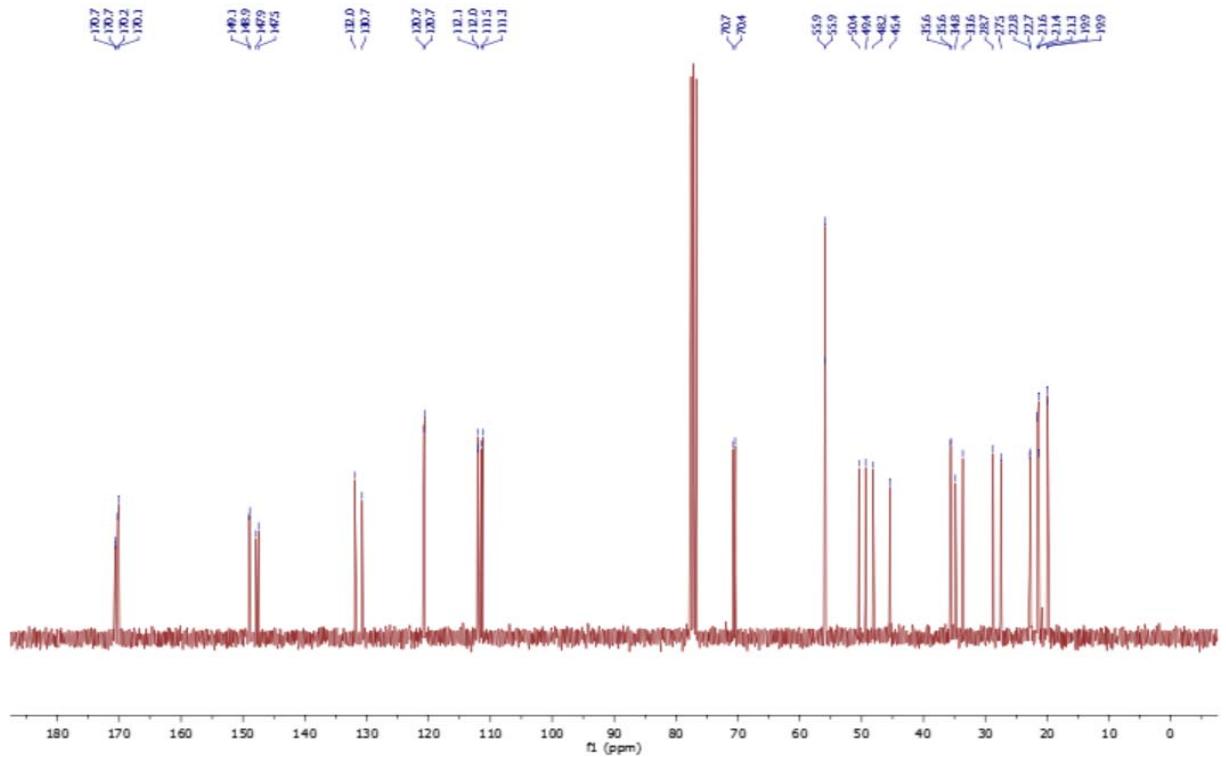


6-(N-(3,4-Dimethoxyphenethyl)acetamido)hexan-2-yl acetate (2-10)

^1H RMN spectrum

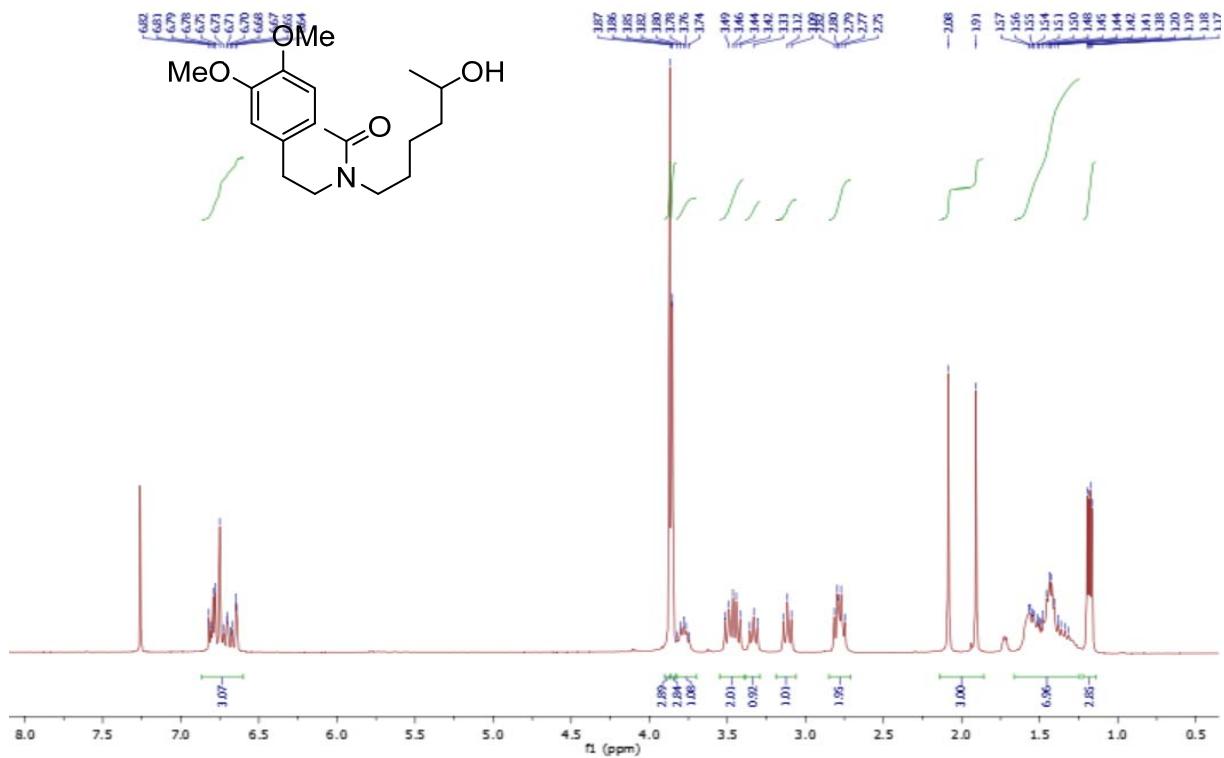


^{13}C RMN spectrum

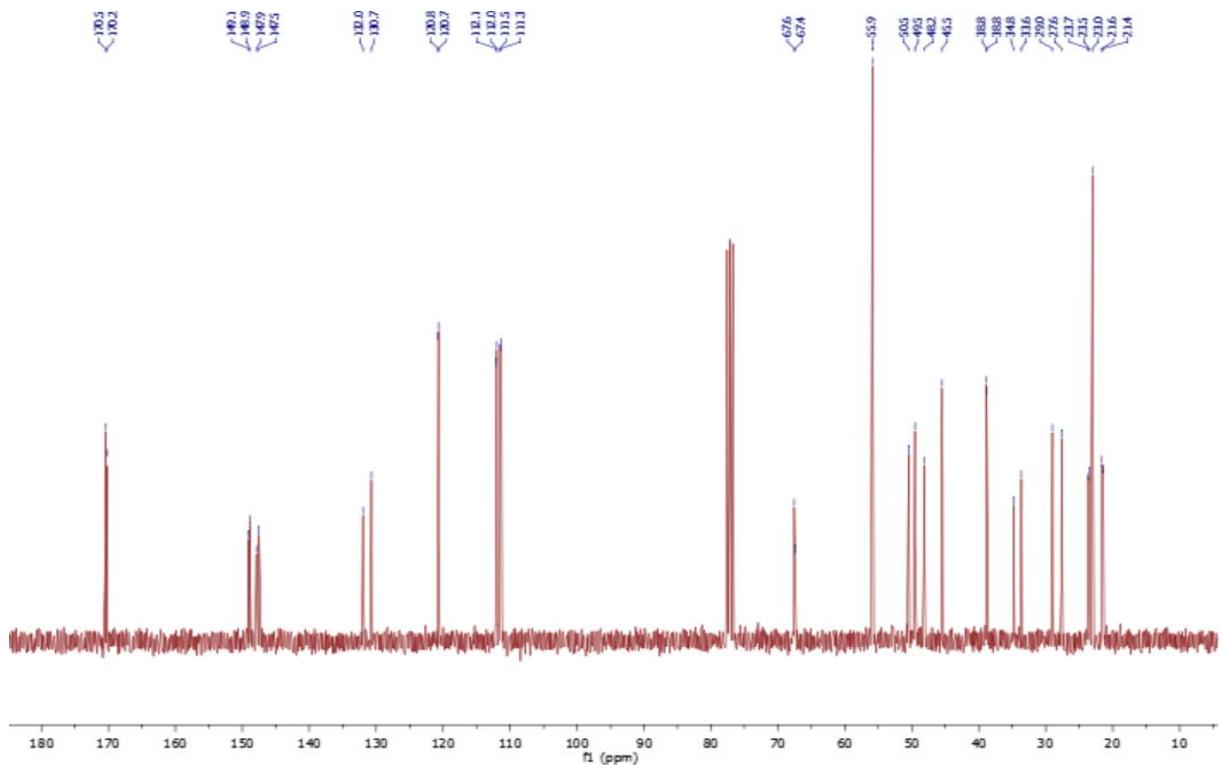


N-(3,4-Dimethoxyphenethyl)-N-(5-hydroxyhexyl)acetamide (2-11)

^1H RMN spectrum

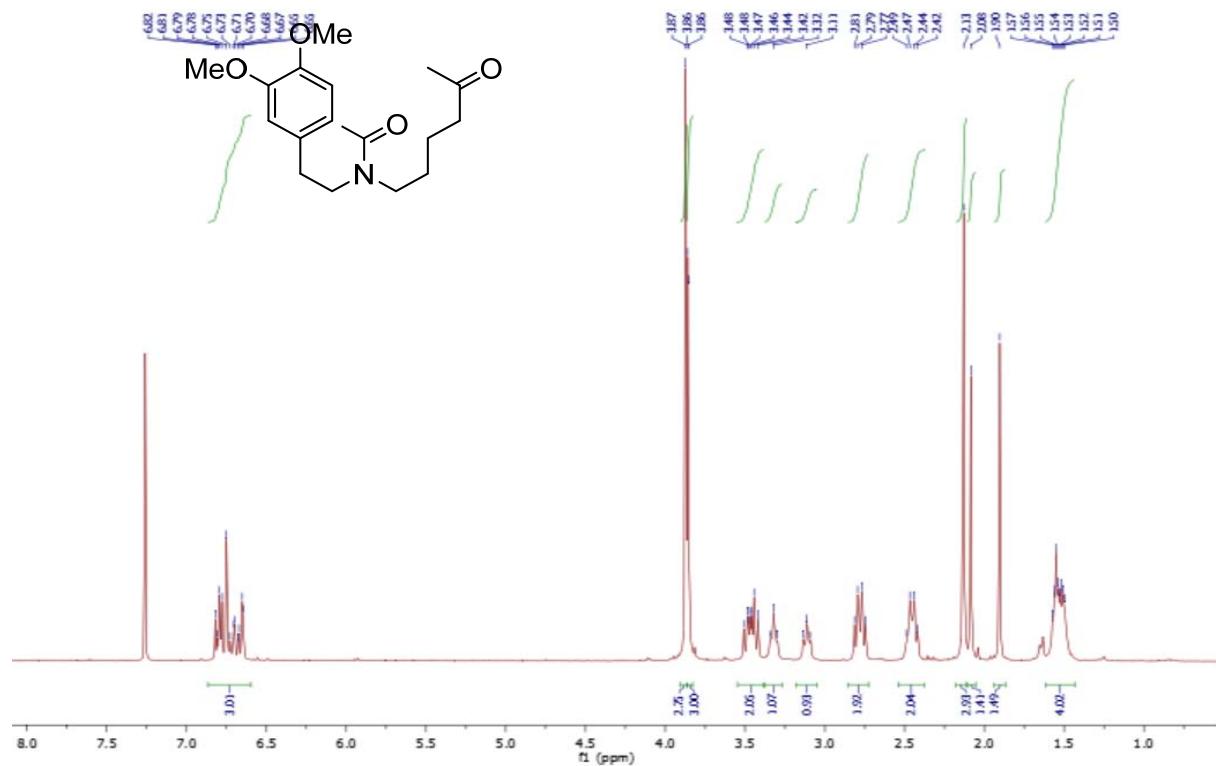


^{13}C RMN spectrum

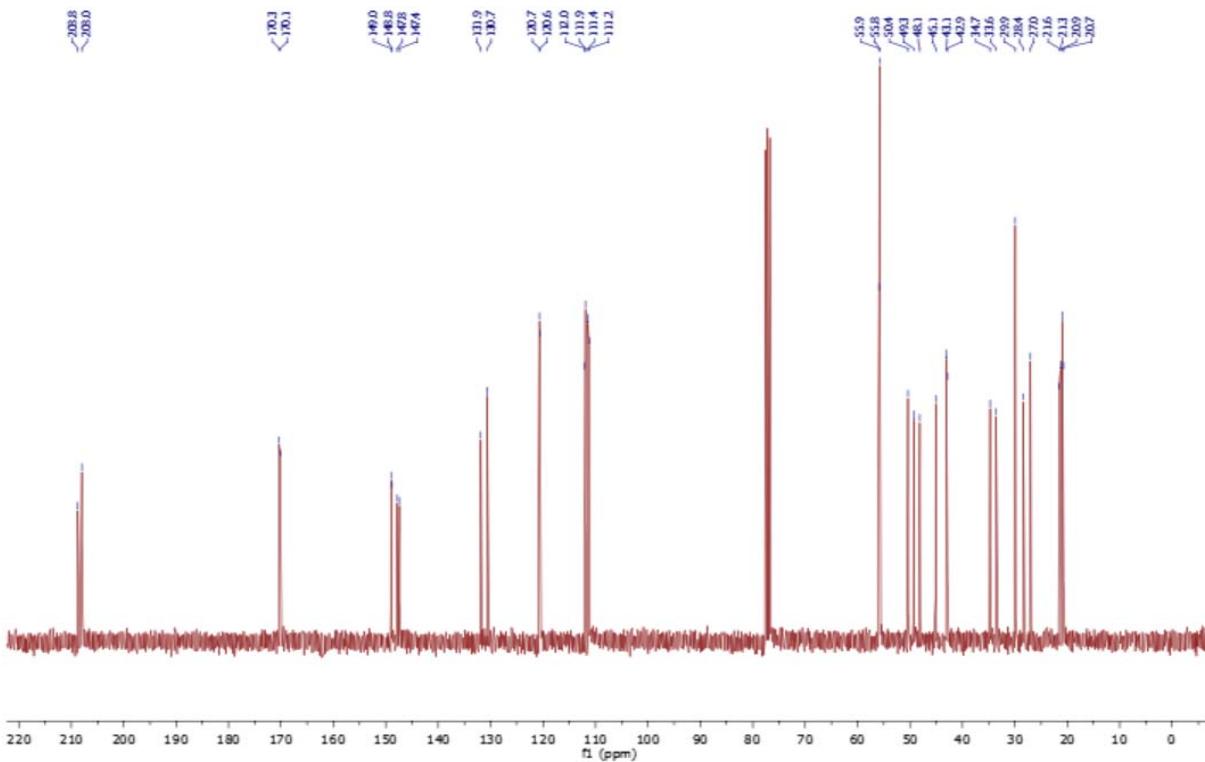


***N*-(3,4-Dimethoxyphenethyl)-*N*-(5-oxohexyl)acetamide (2-12)**

¹H RMN spectrum

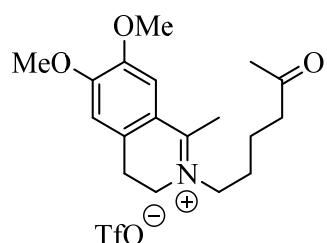


¹³C RMN spectrum

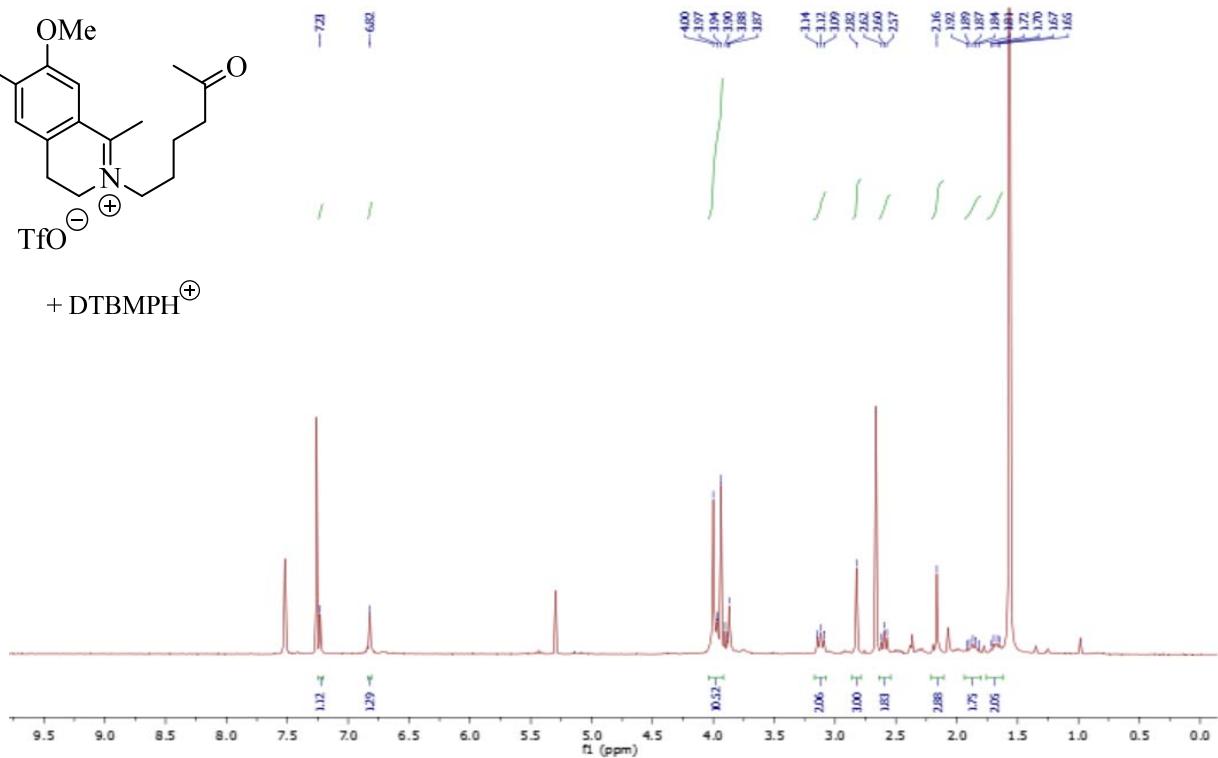


Iminium ion (2-12a)

^1H RMN spectrum

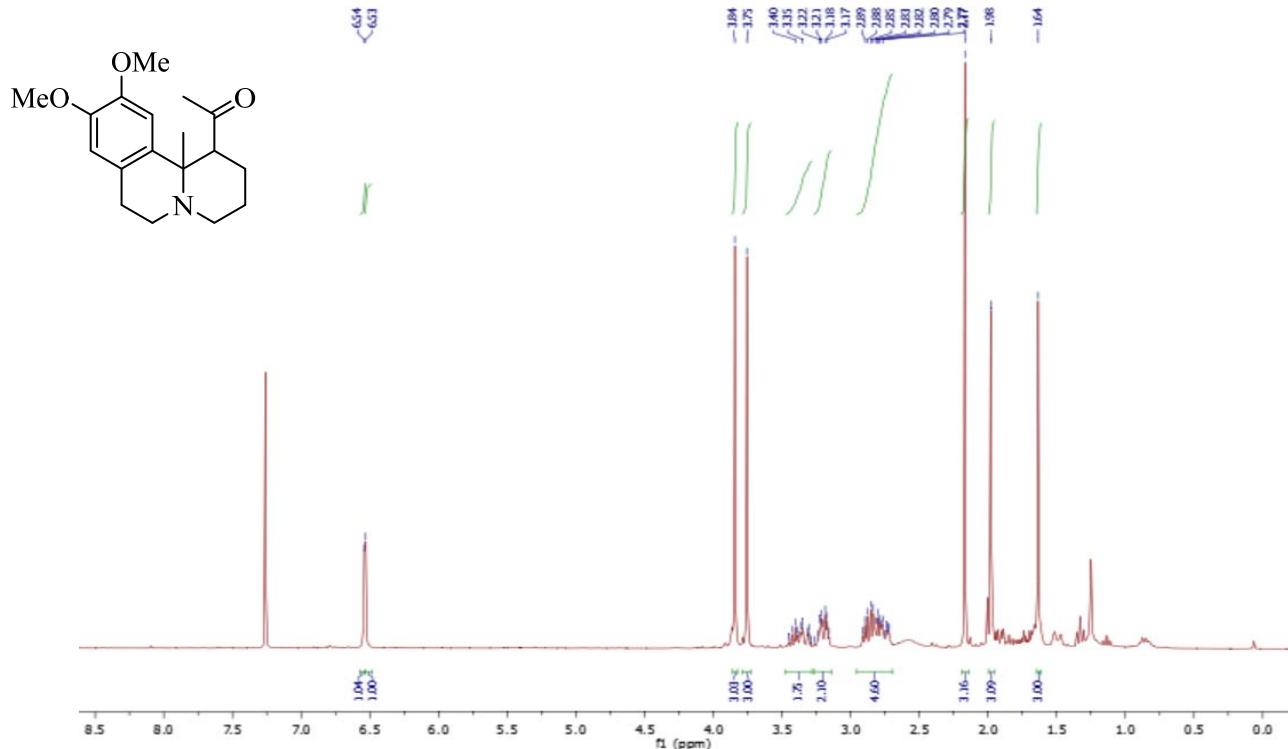


+ DTBMPH $^\oplus$

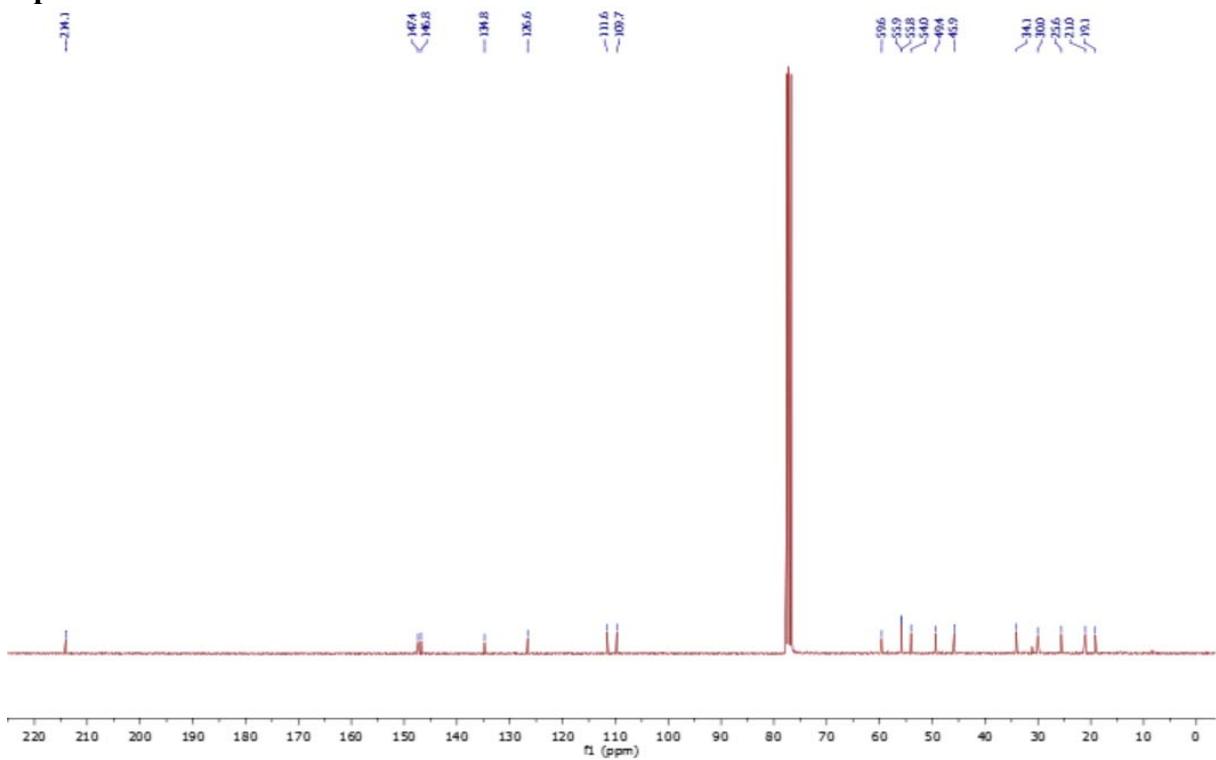


1-(9,10-Dimethoxy-11b-methyl-1,3,4,6,7,11b-hexahydro-2H-pyrido[2,1-a]isoquinolin-1-yl)ethan-1-one (2-13)

^1H RMN spectrum

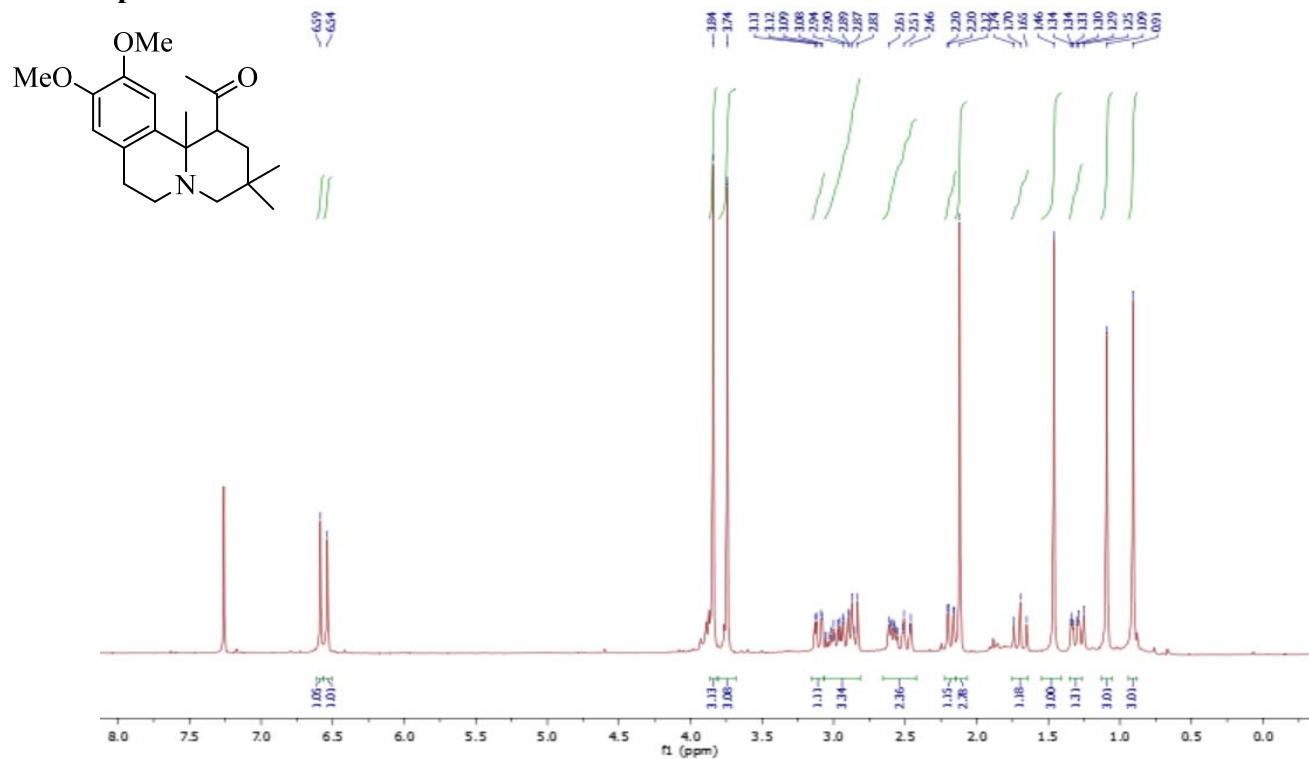


^{13}C RMN spectrum

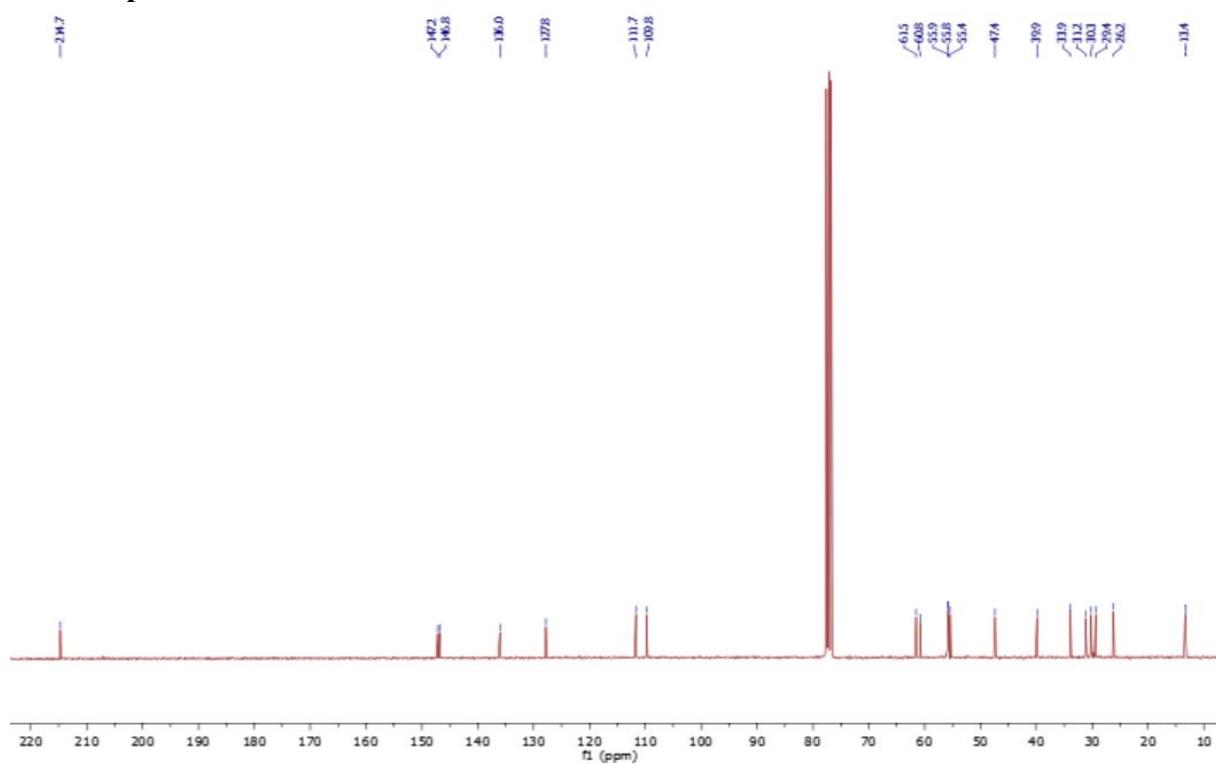


1-(9,10-Dimethoxy-3,3,11b-trimethyl-1,3,4,6,7,11b-hexahydro-2H-pyrido[2,1-a]isoquinolin-1-yl)ethan-1-one (2-14)

^1H RMN spectrum

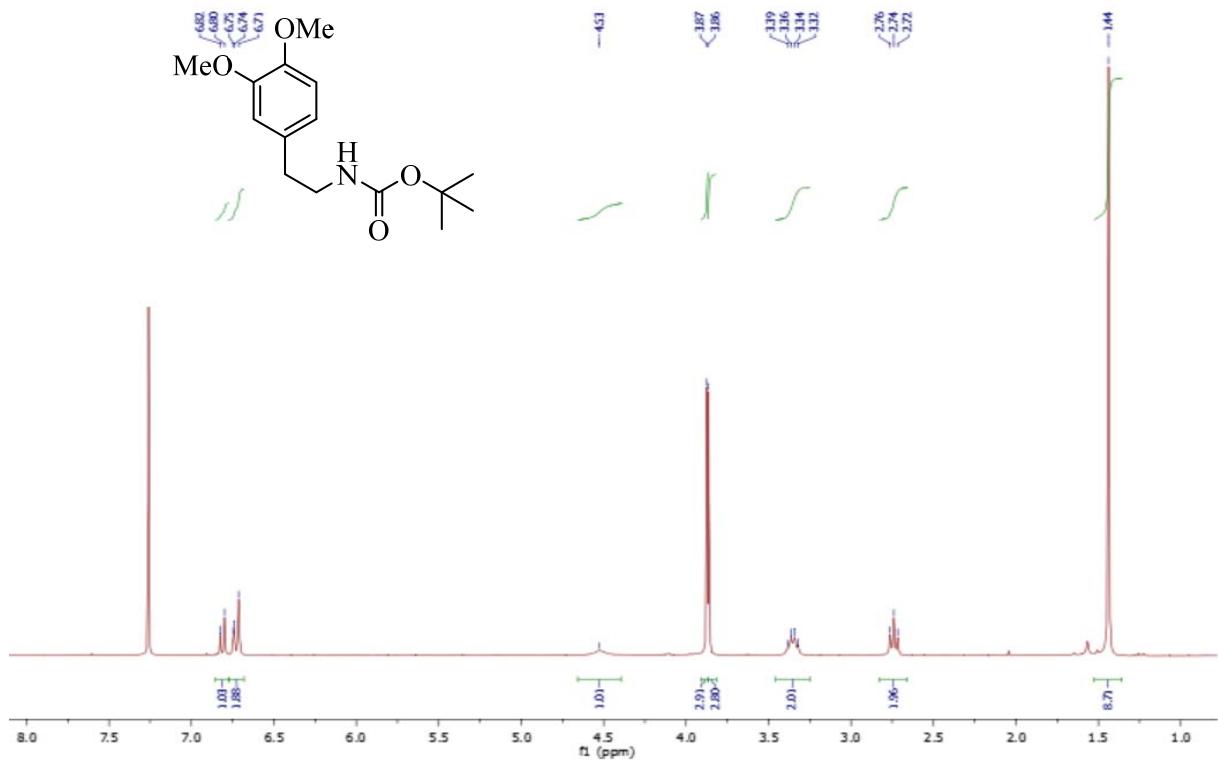


^{13}C RMN spectrum

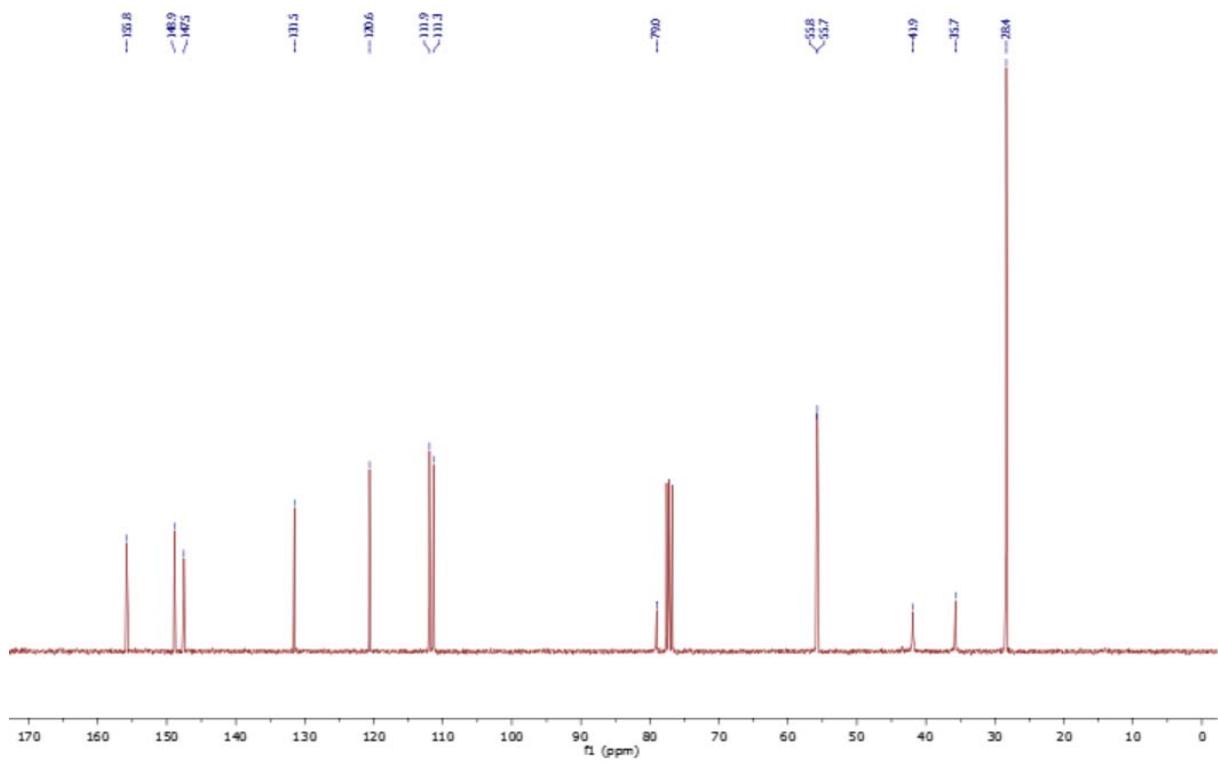


tert-Butyl (3,4-dimethoxyphenethyl)carbamate (2-15)

^1H RMN spectrum

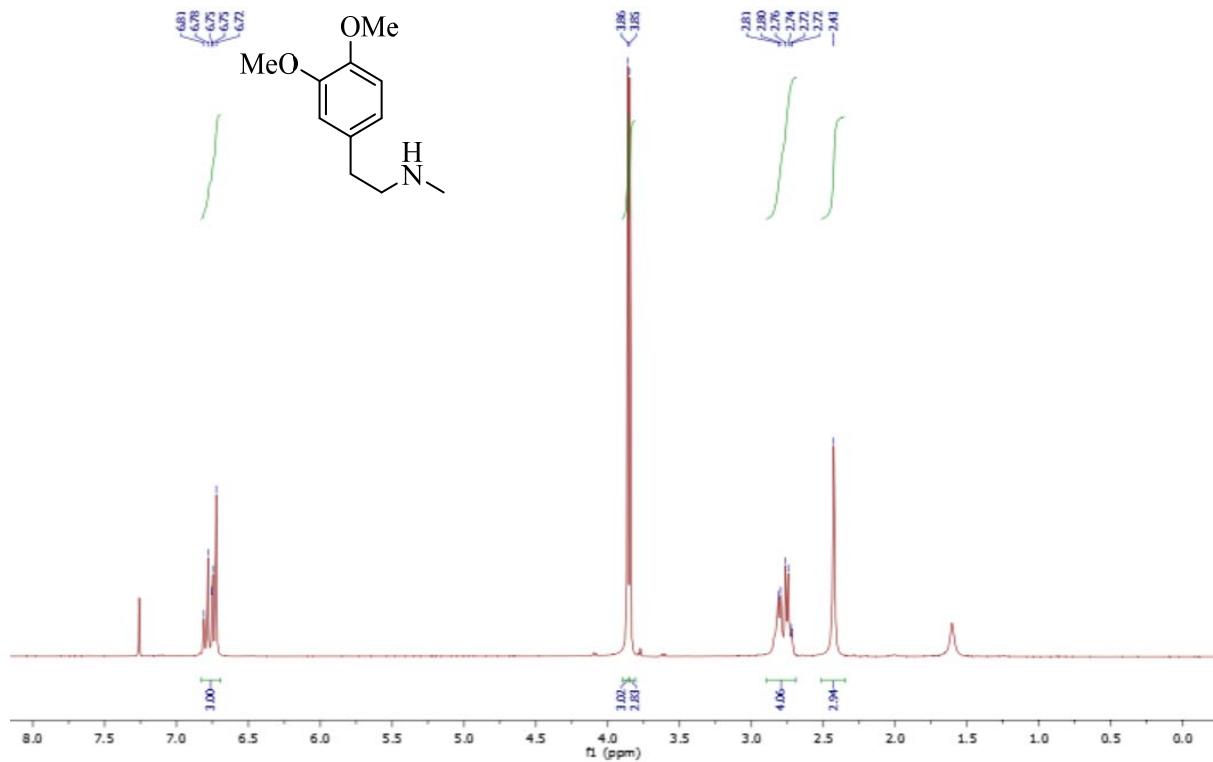


^{13}C RMN spectrum

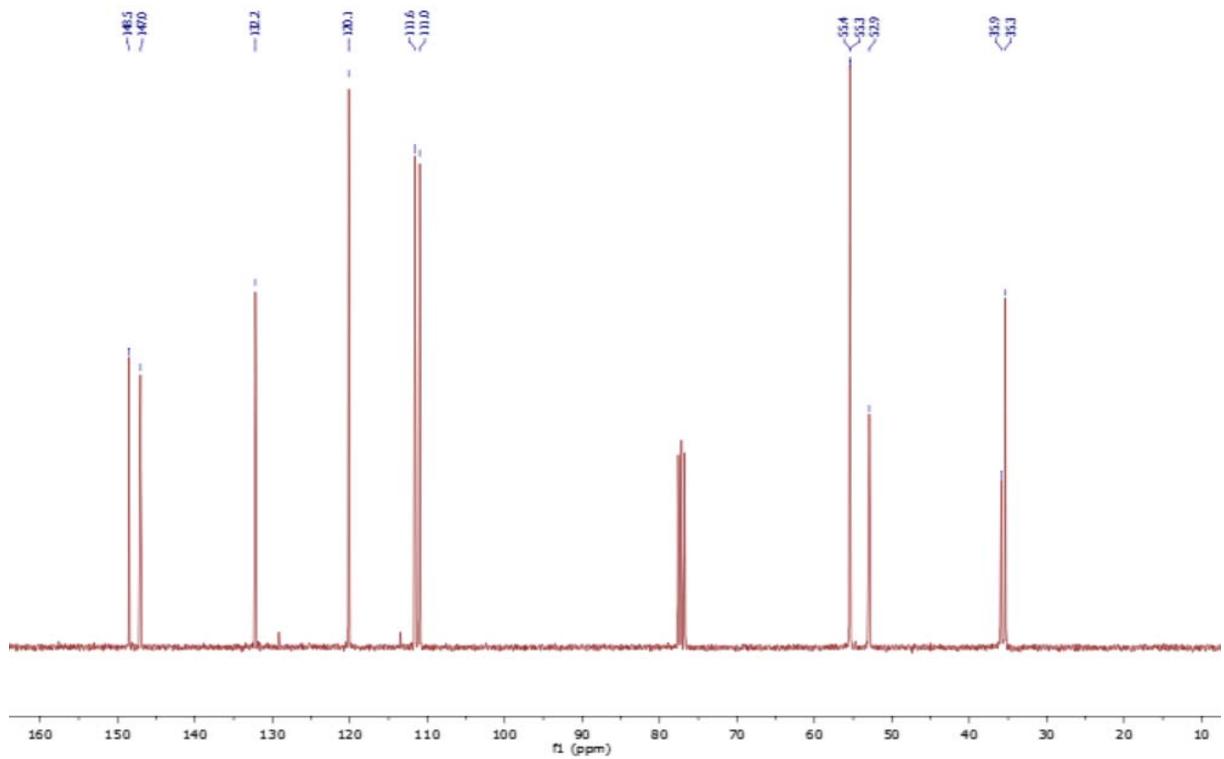


2-(3,4-Dimethoxyphenyl)-N-methylethan-1-amine (2-16)

^1H RMN spectrum

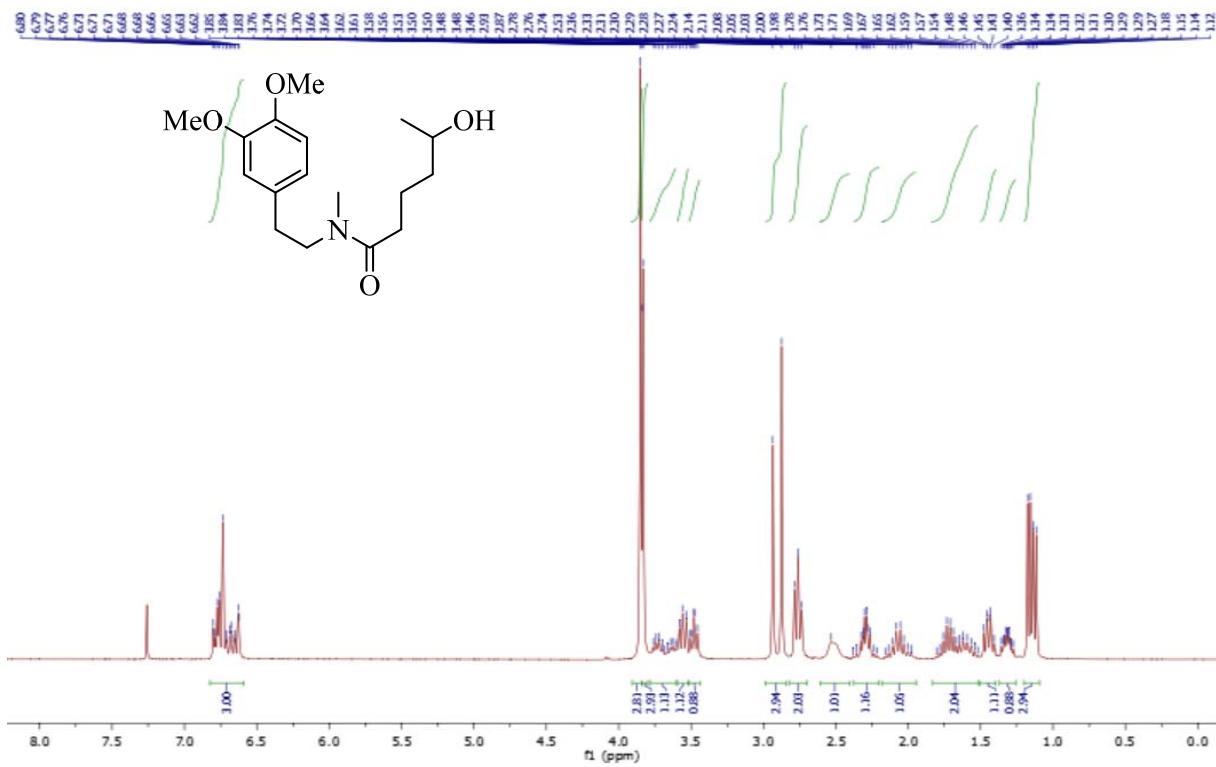


^{13}C RMN spectrum

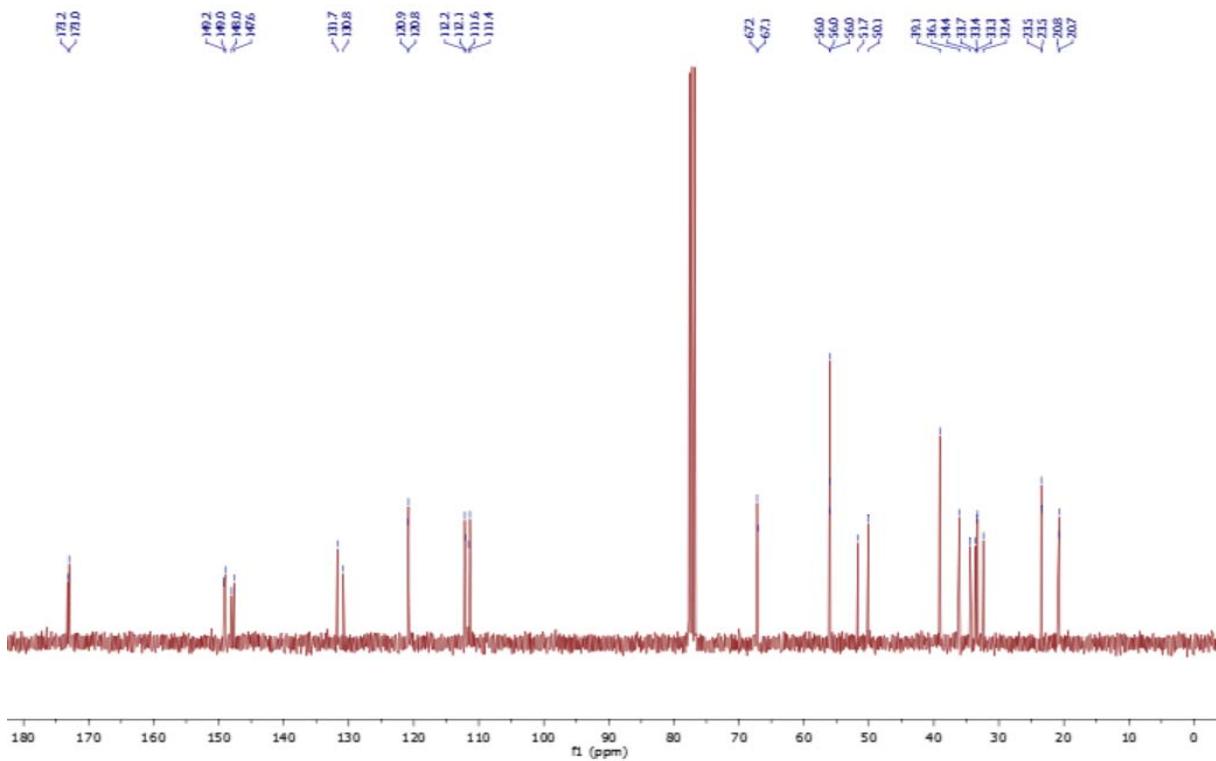


N-(3,4-Dimethoxyphenethyl)-5-hydroxy-N-methylhexanamide (2-17)

¹H RMN spectrum

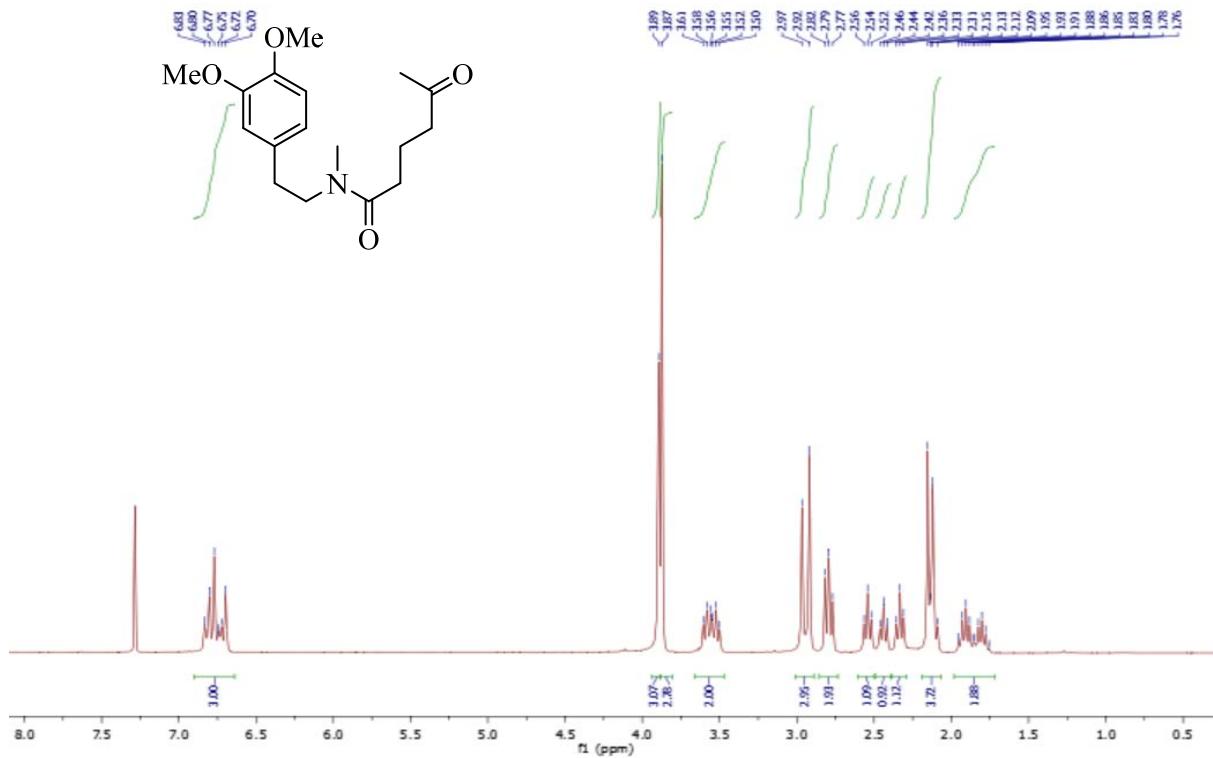


¹³C RMN spectrum

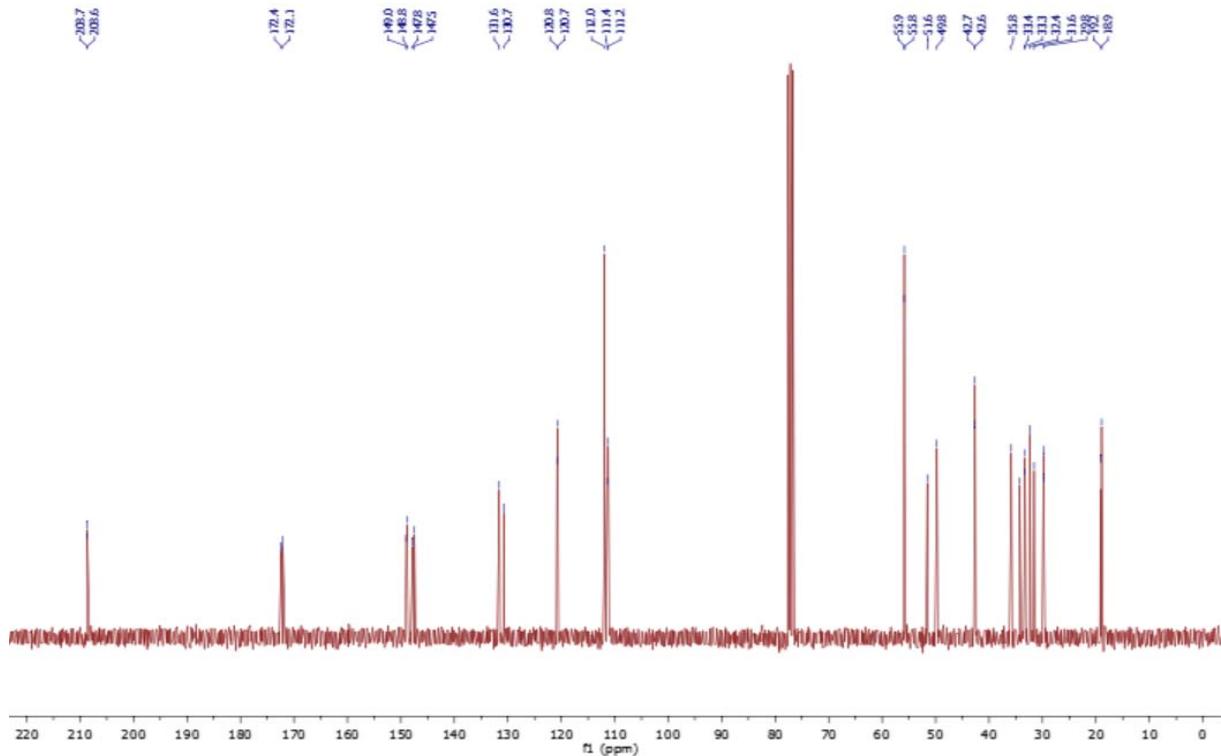


N-(3,4-Dimethoxyphenethyl)-N-methyl-5-oxohexanamide (2-18)

¹H RMN spectrum

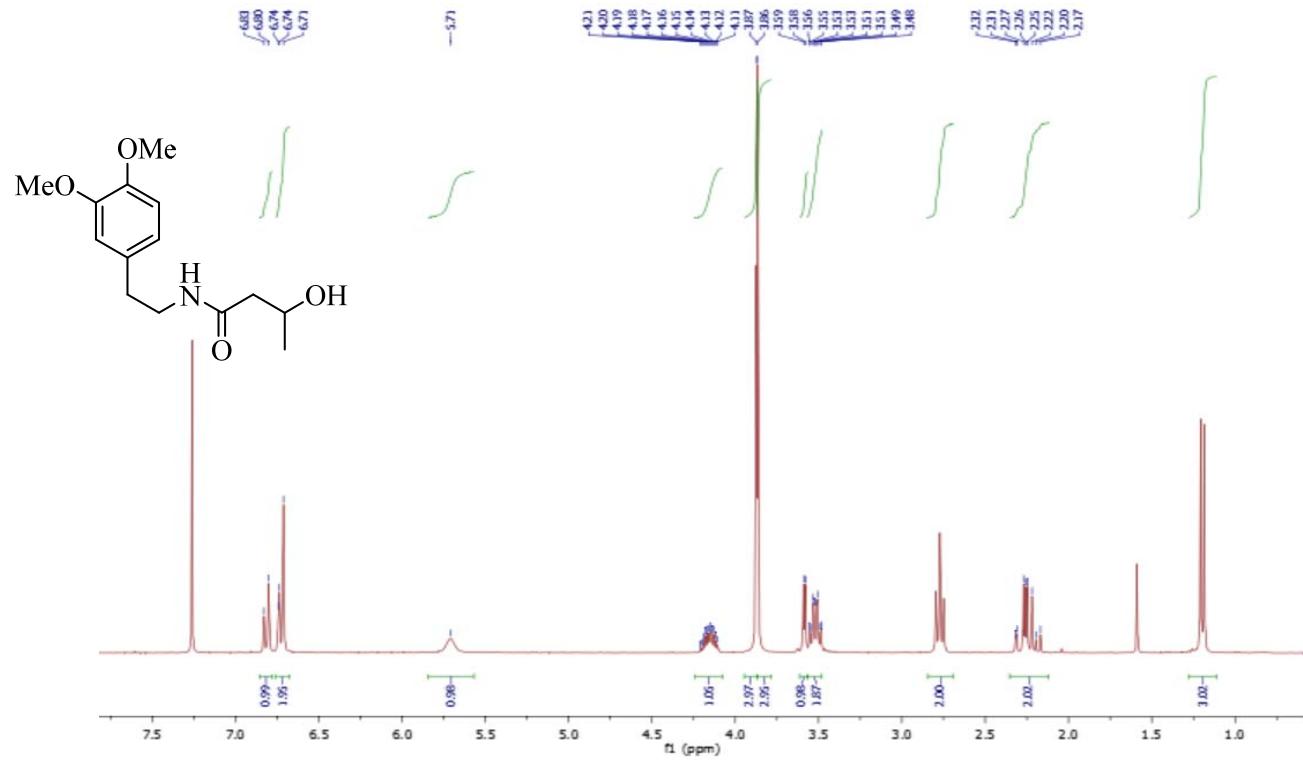


¹³C RMN spectrum

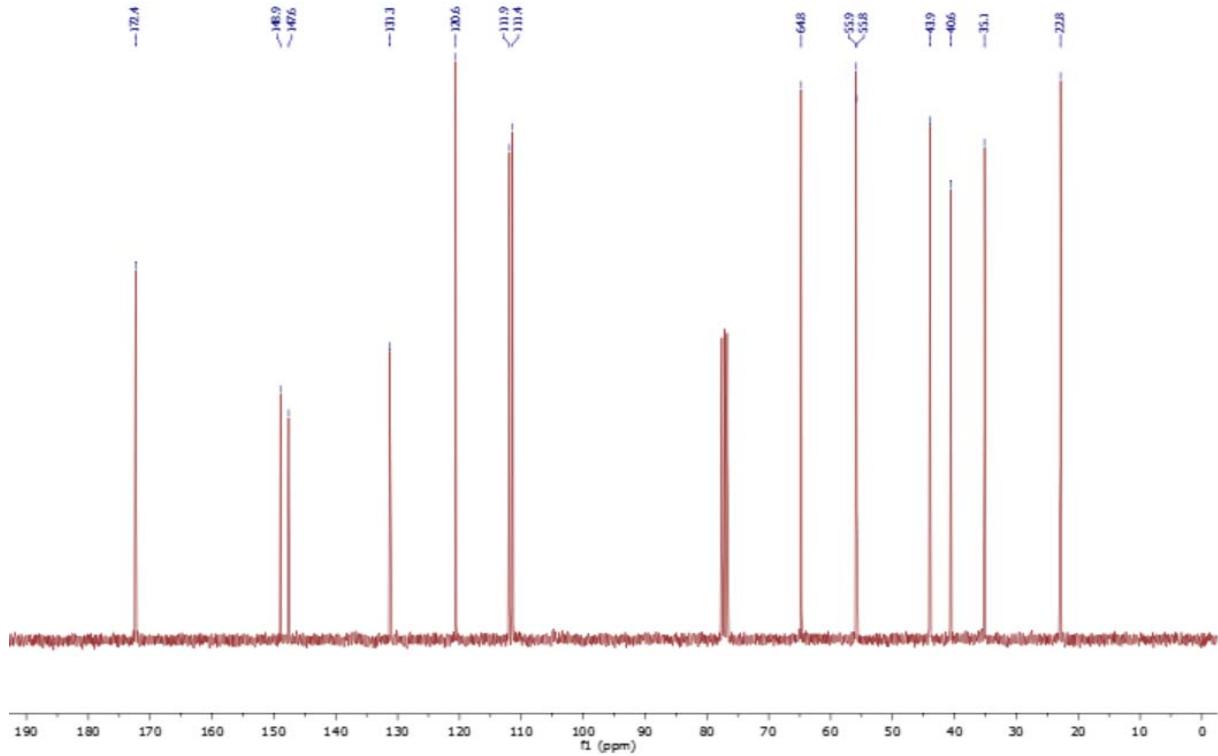


N-(3,4-Dimethoxyphenethyl)-3-hydroxybutanamide (2-20)

^1H RMN spectrum

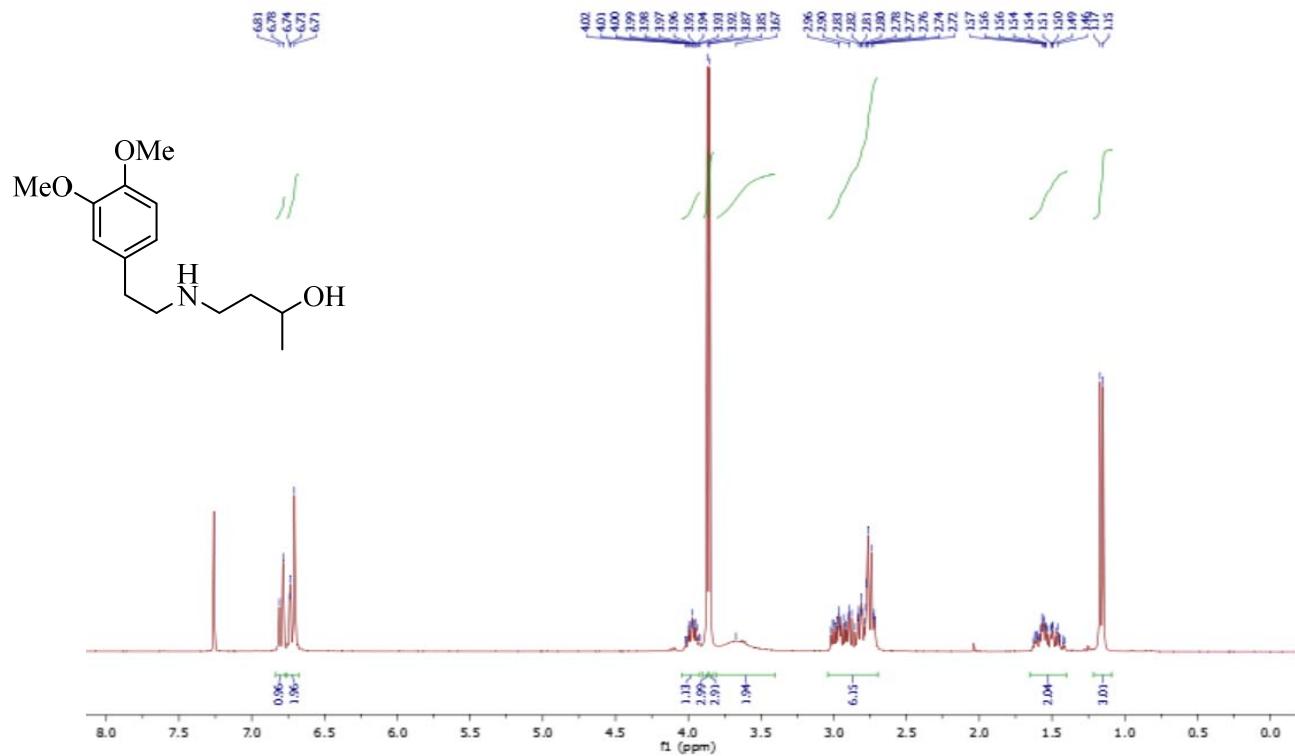


^{13}C RMN spectrum

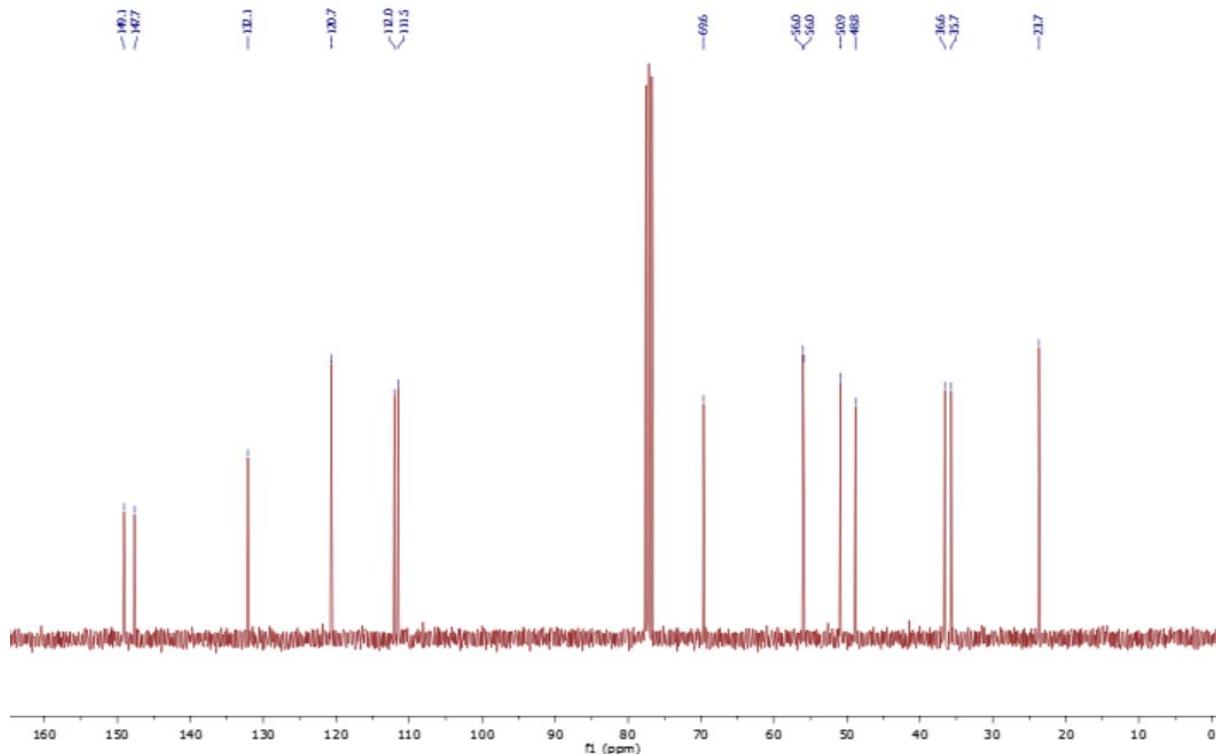


N-(3,4-Dimethoxyphenethyl)-N-methyl-5-oxohexanamide (2-21)

^1H RMN spectrum

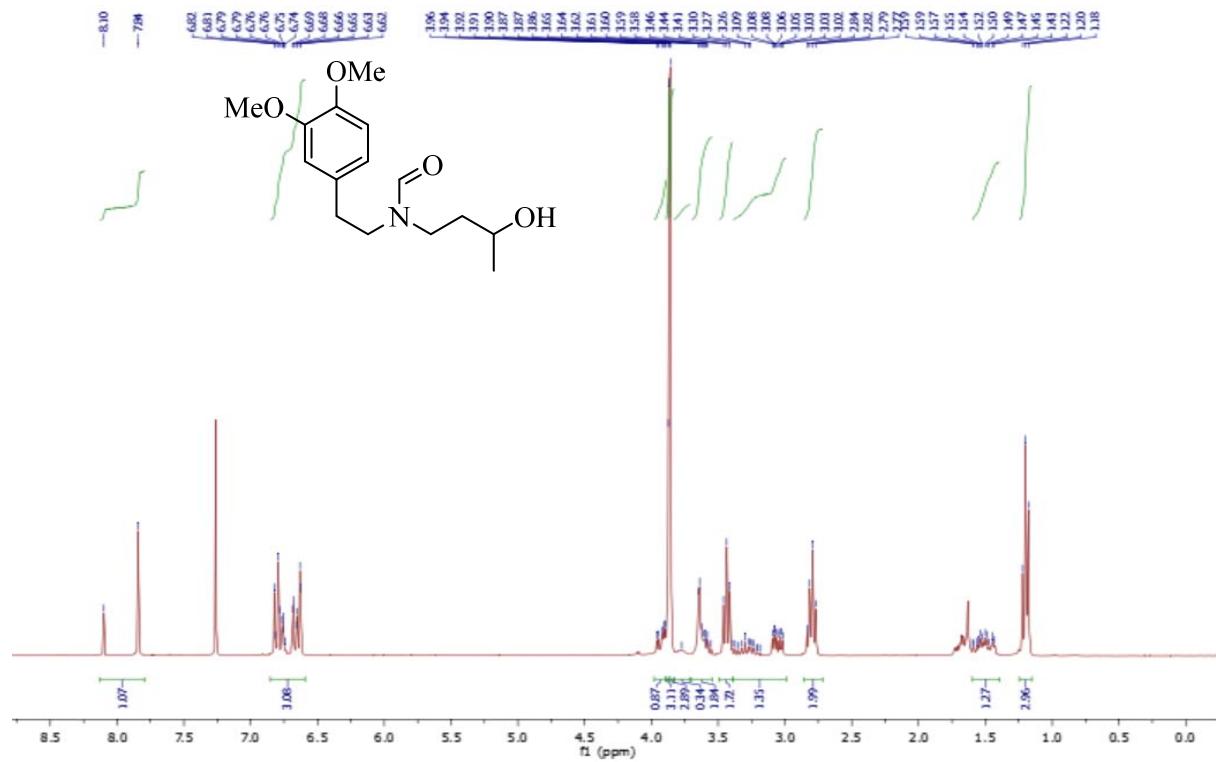


^{13}C RMN spectrum

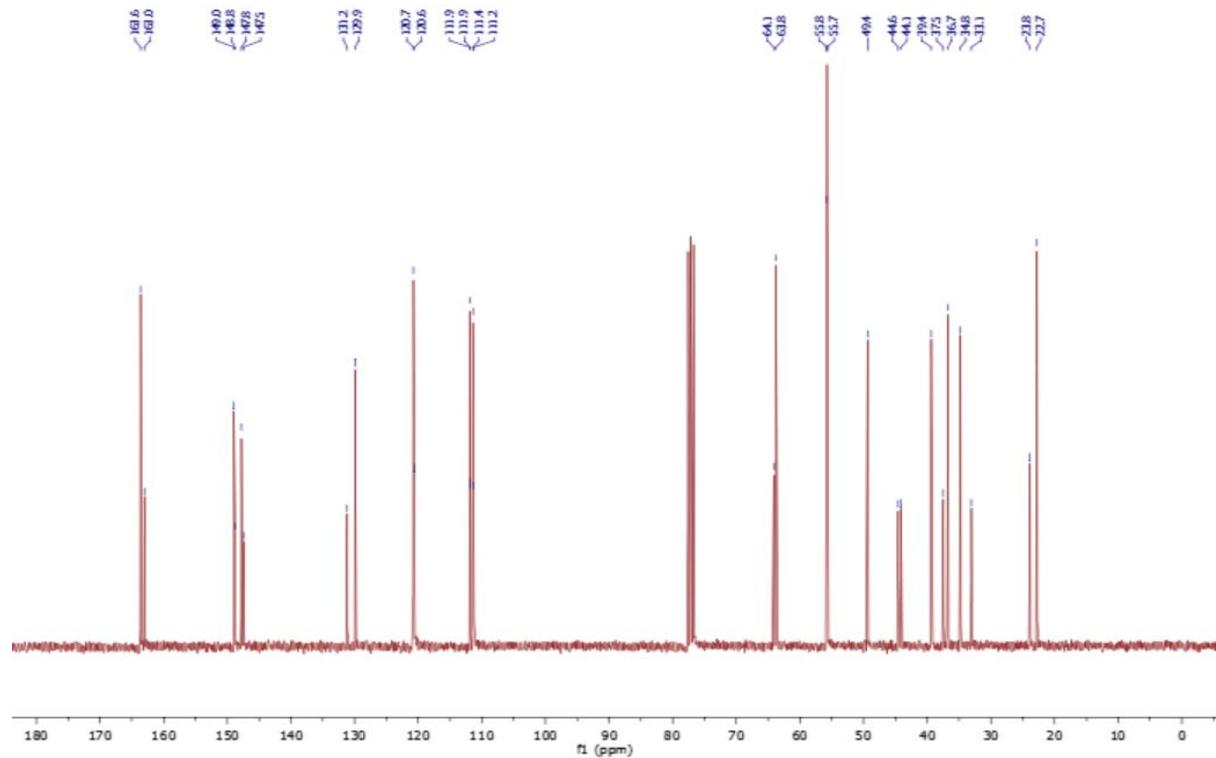


N-(3,4-Dimethoxyphenethyl)-N-(3-hydroxybutyl)formamide (2-22)

¹H RMN spectrum

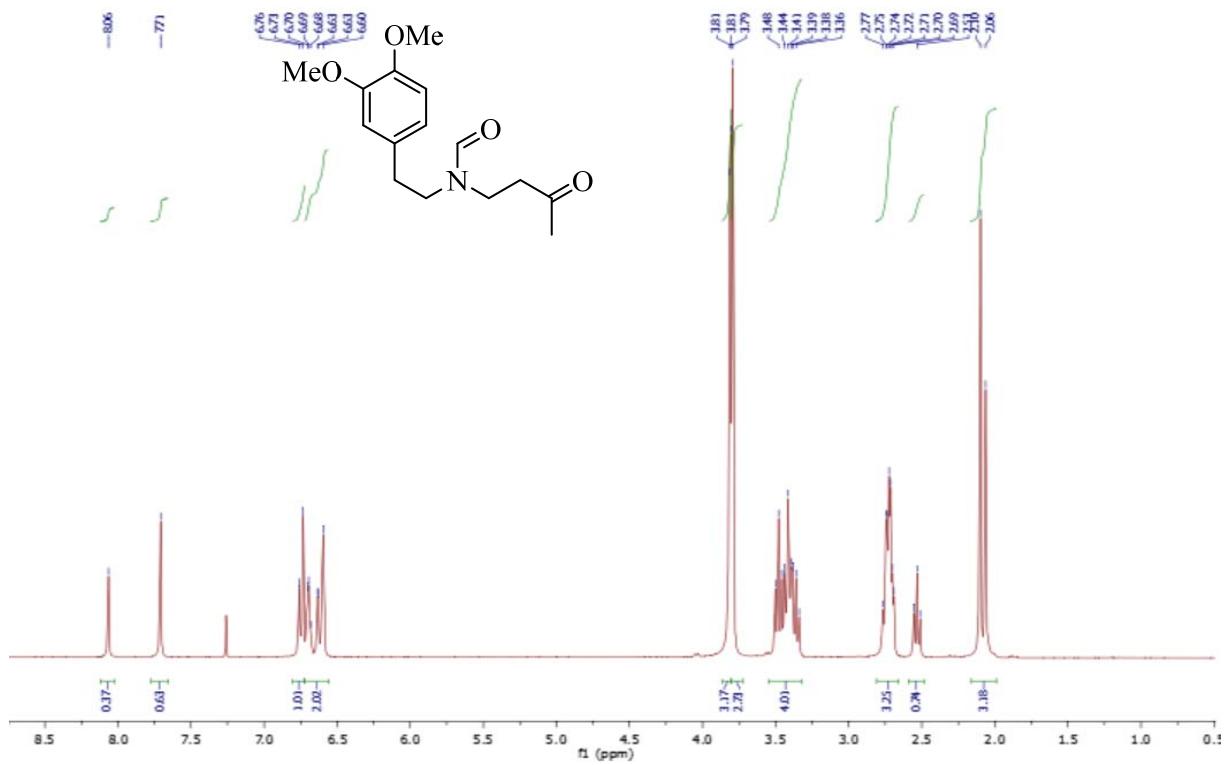


¹³C RMN spectrum

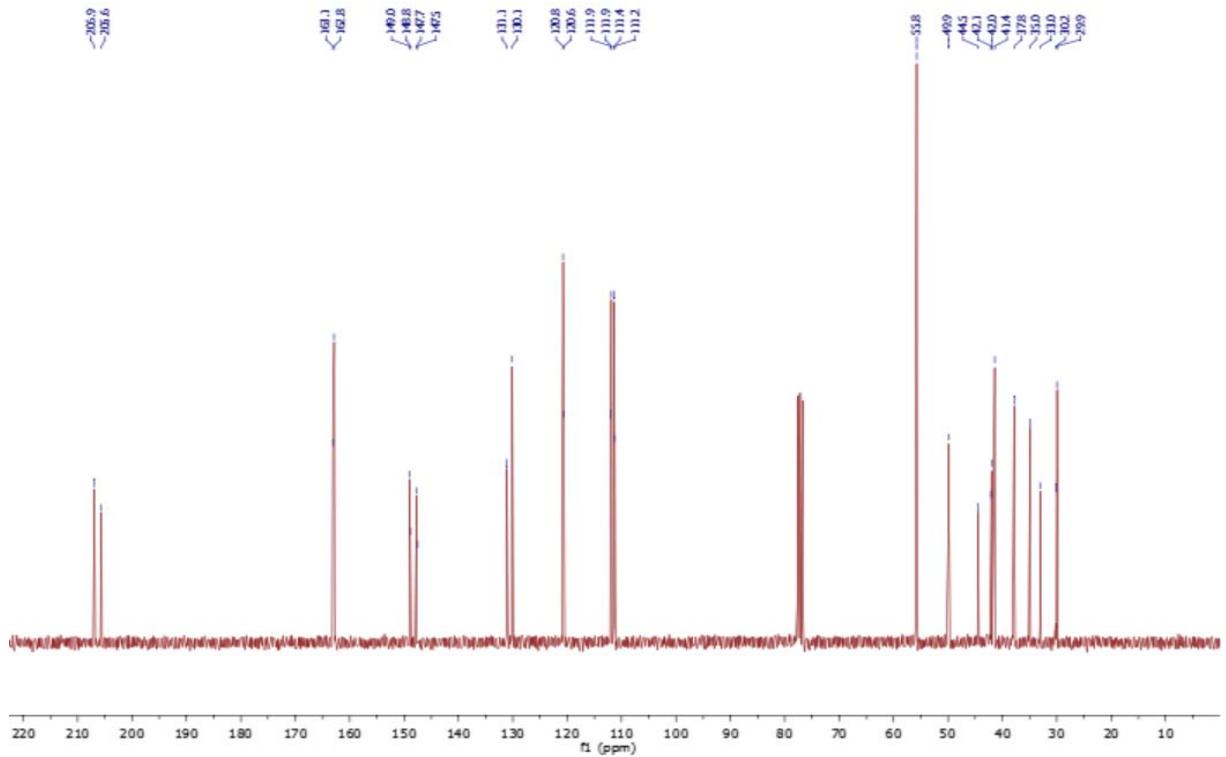


N-(3,4-Dimethoxyphenethyl)-N-(3-oxobutyl)formamide (2-23)

^1H RMN spectrum

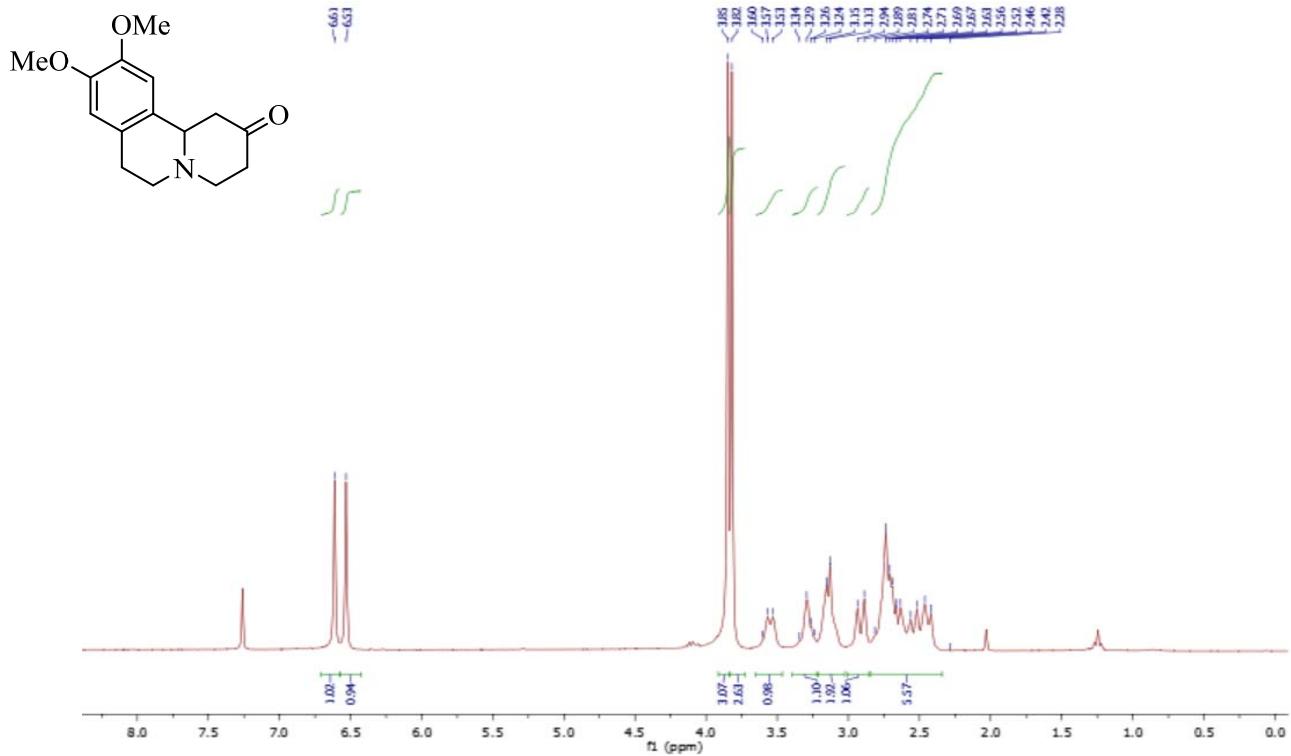


^{13}C RMN spectrum

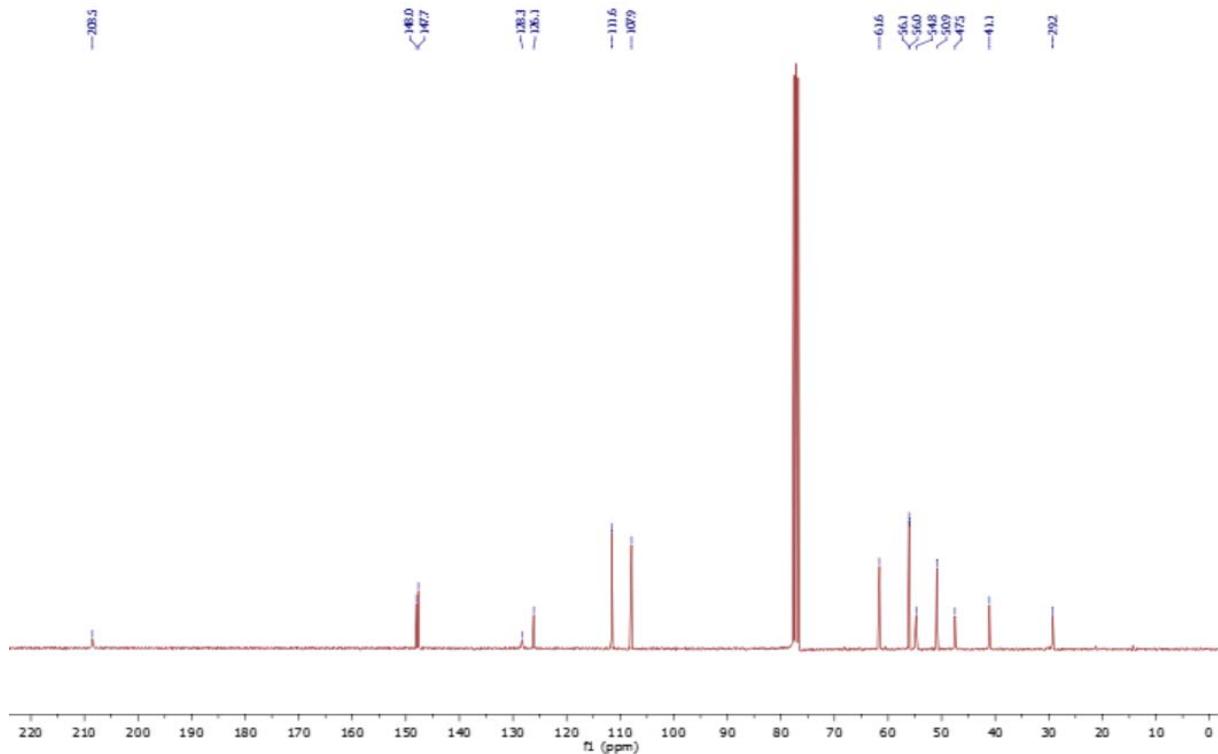


9,10-Dimethoxy-1,3,4,6,7,11b-hexahydro-2H-pyrido[2,1-a]isoquinolin-2-one (2-24)

¹H RMN spectrum

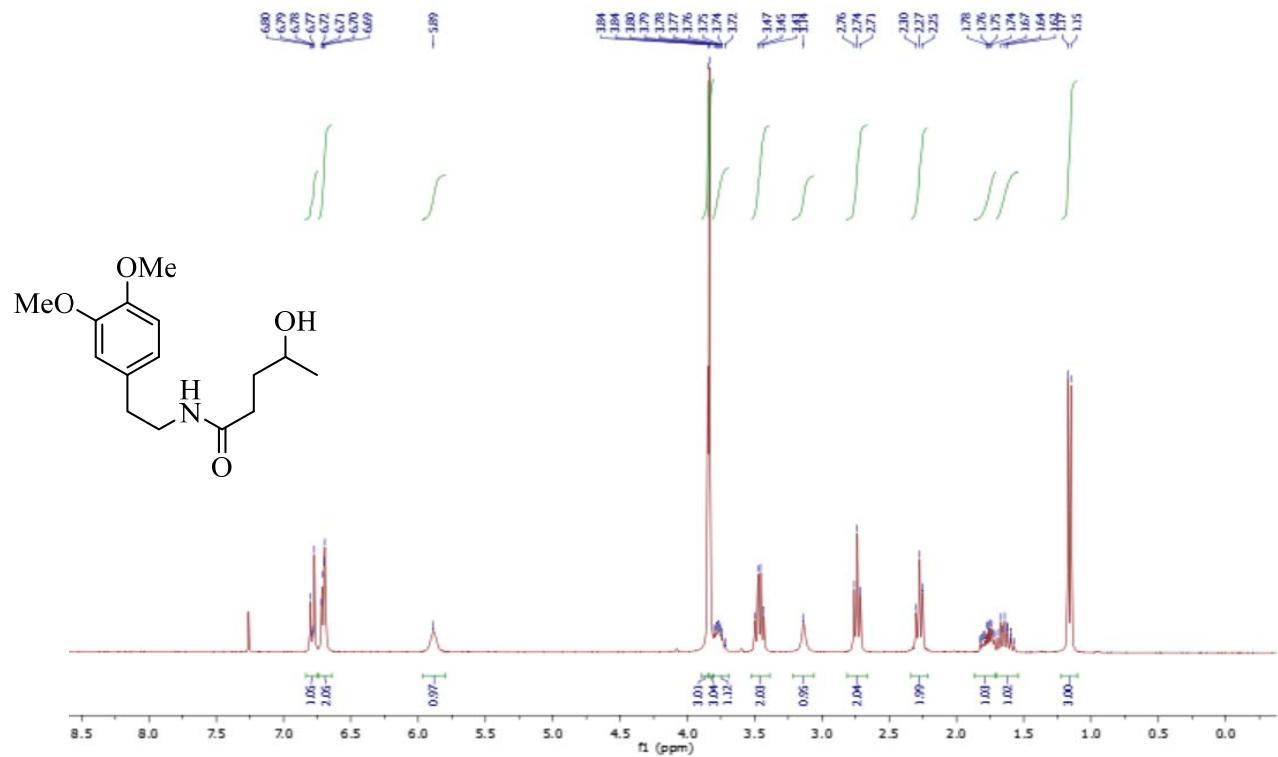


¹³C RMN spectrum

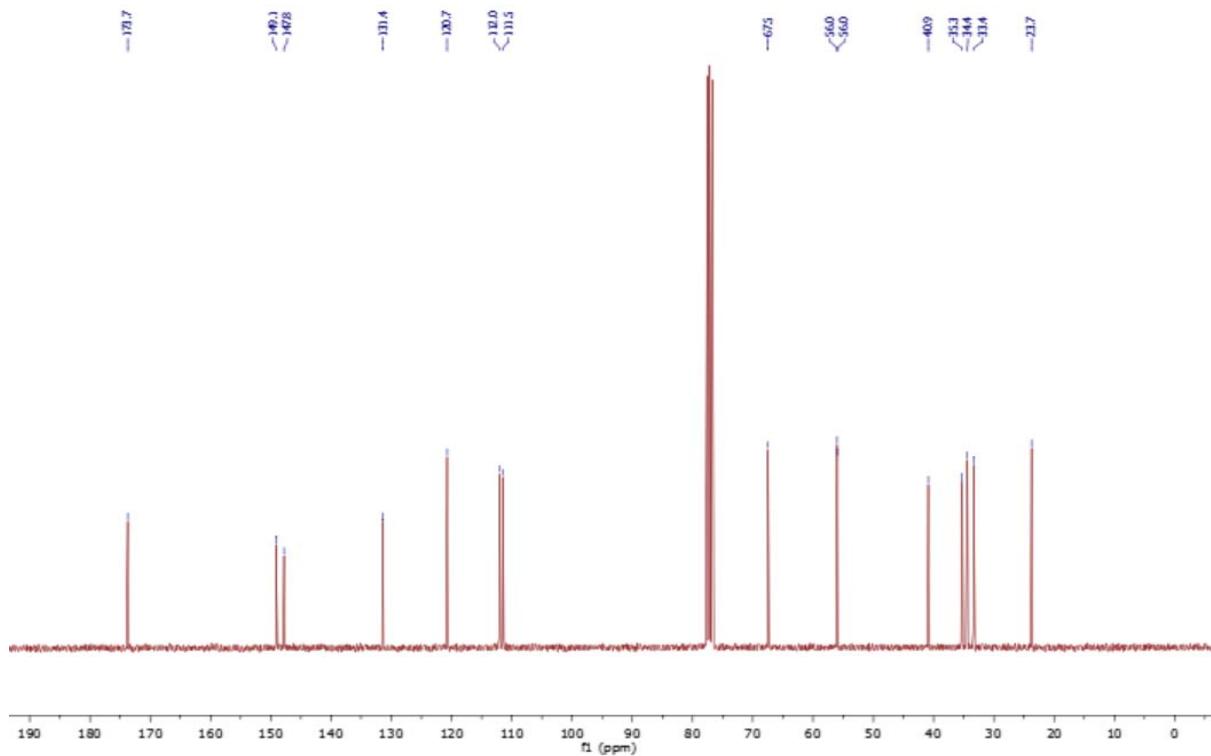


N-(3,4-Dimethoxyphenethyl)-N-(5-oxohexyl)acetamide (2-25)

¹H RMN spectrum

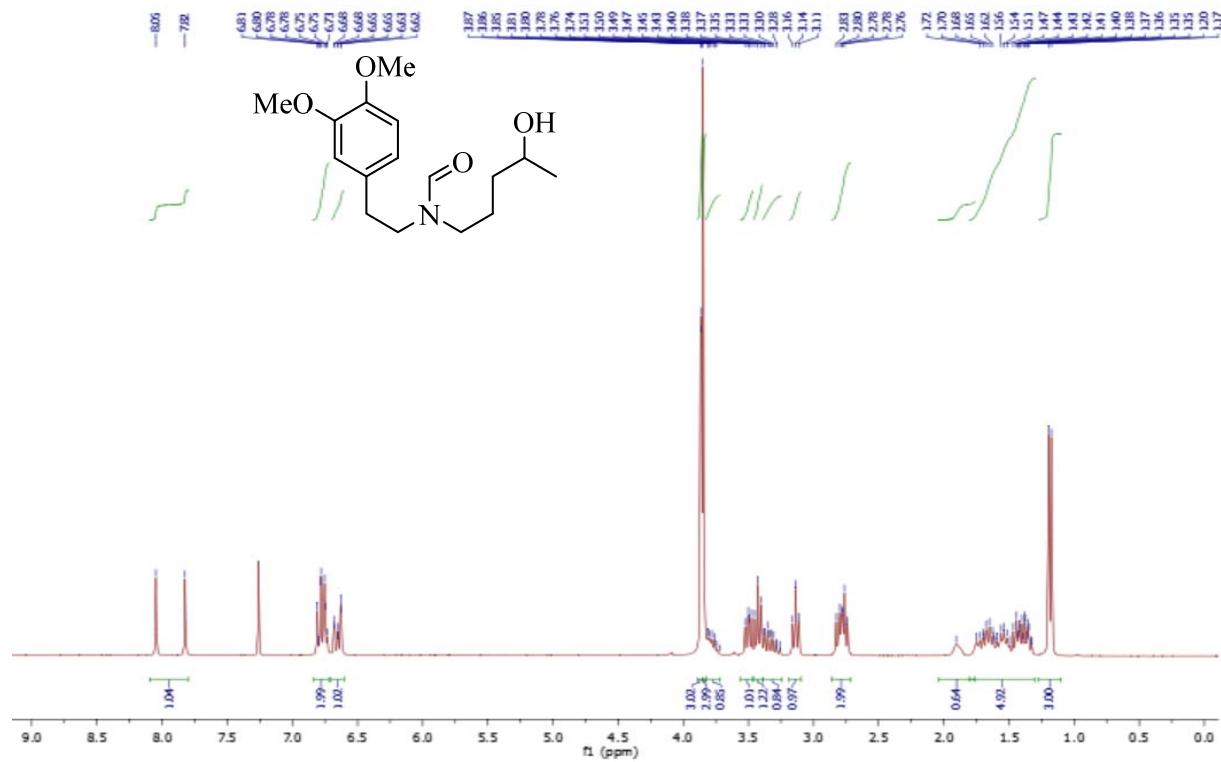


¹³C RMN spectrum

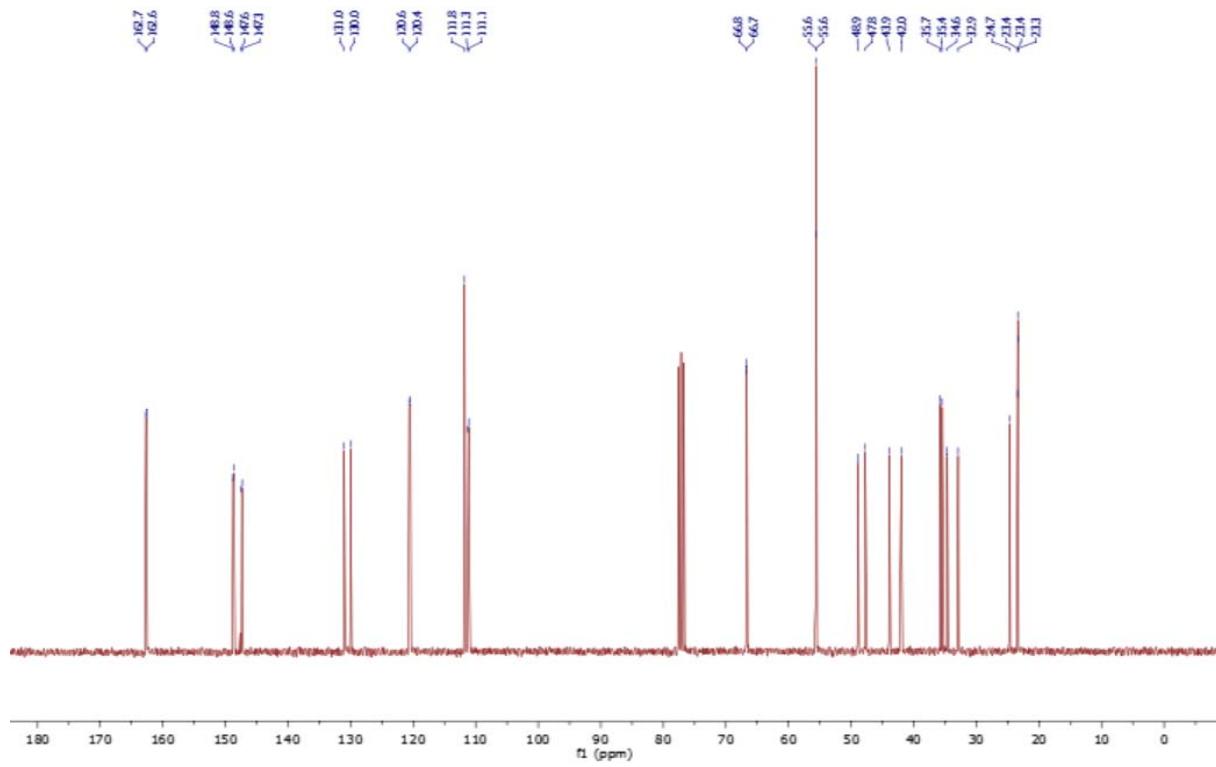


N-(3,4-Dimethoxyphenethyl)-N-(4-hydroxypentyl)formamide (2-26)

¹H RMN spectrum

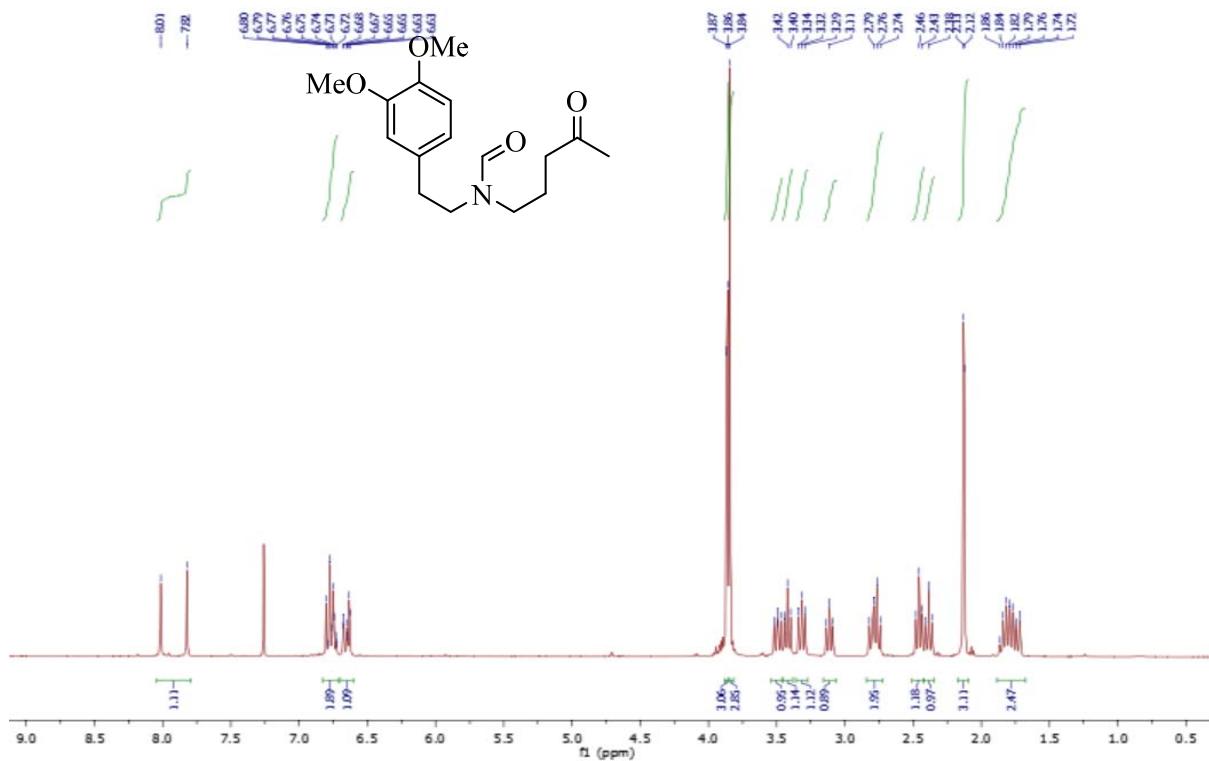


¹³C RMN spectrum

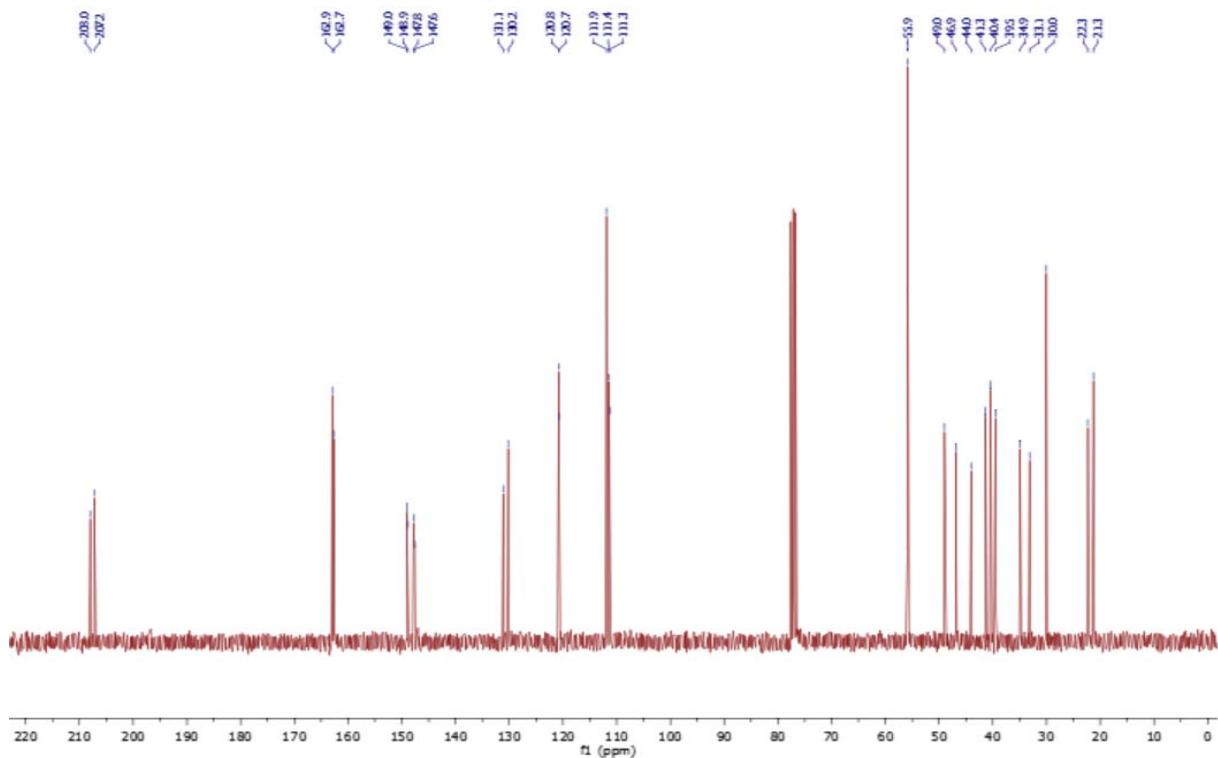


N-(3,4-Dimethoxyphenethyl)-N-(4-oxopentyl)formamide (2-27)

^1H RMN spectrum

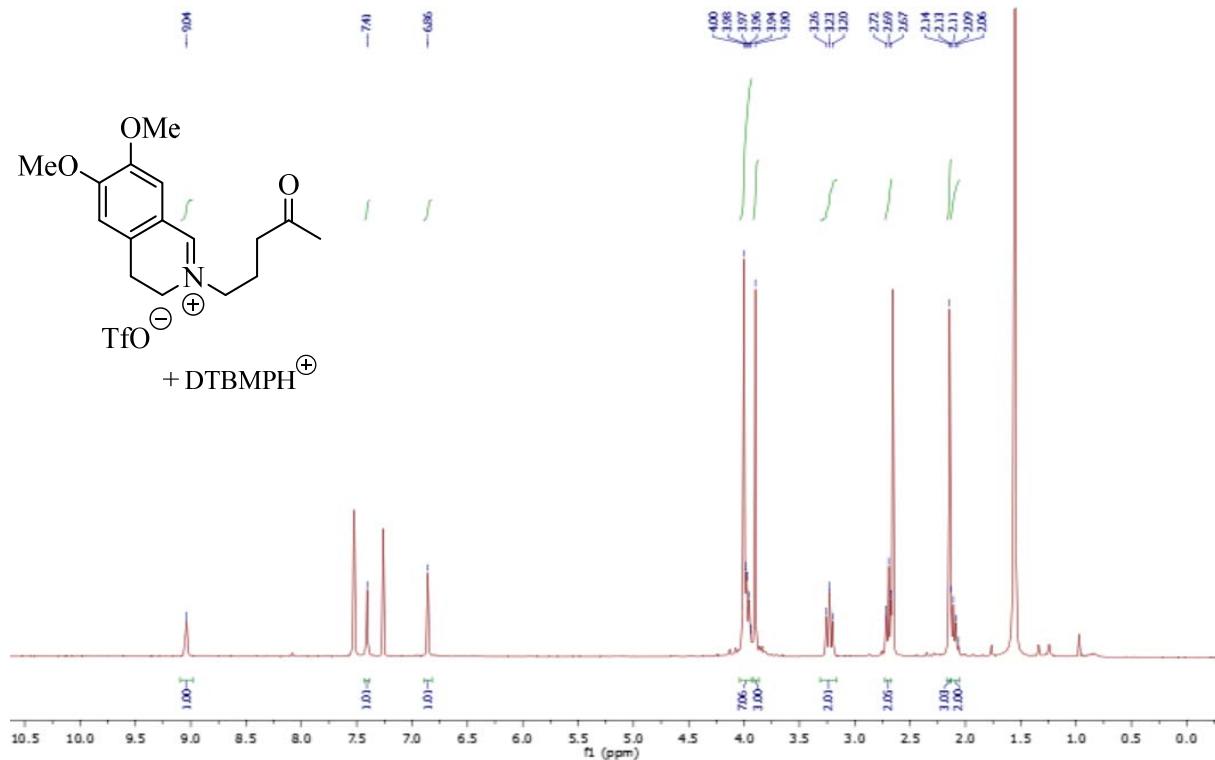


^{13}C RMN spectrum



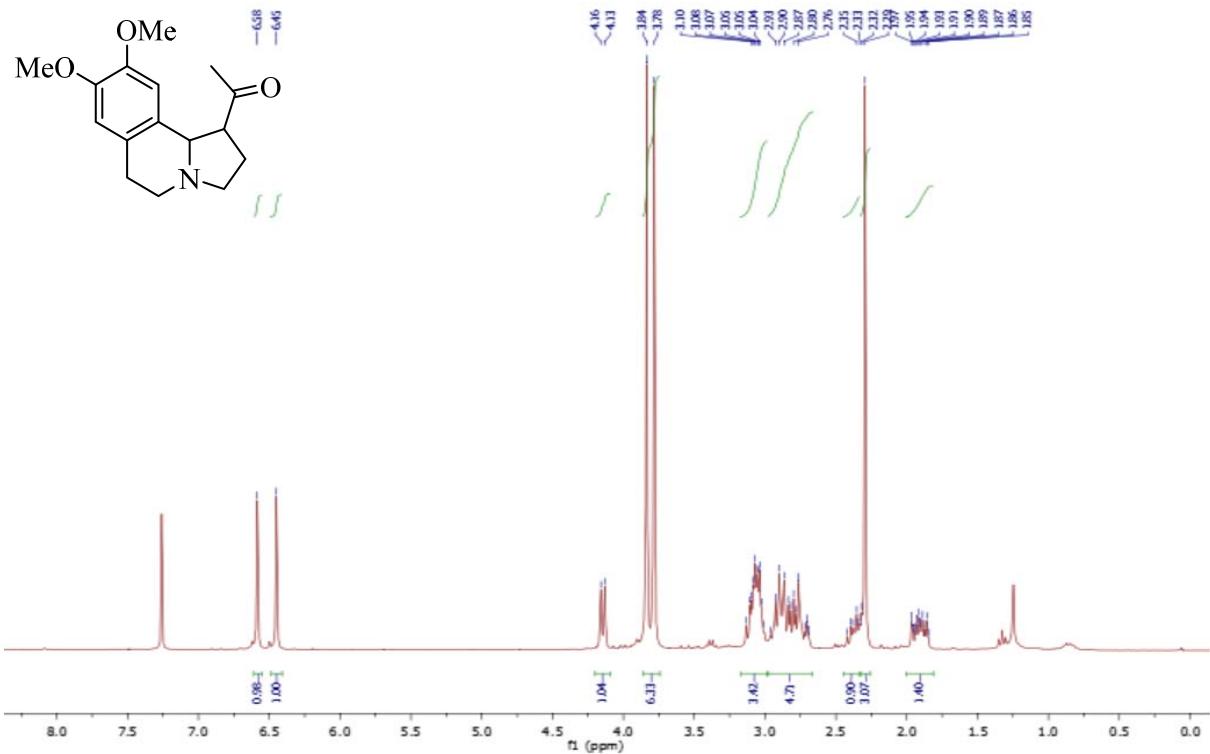
Iminium ion (2-27a)

¹H RMN spectrum

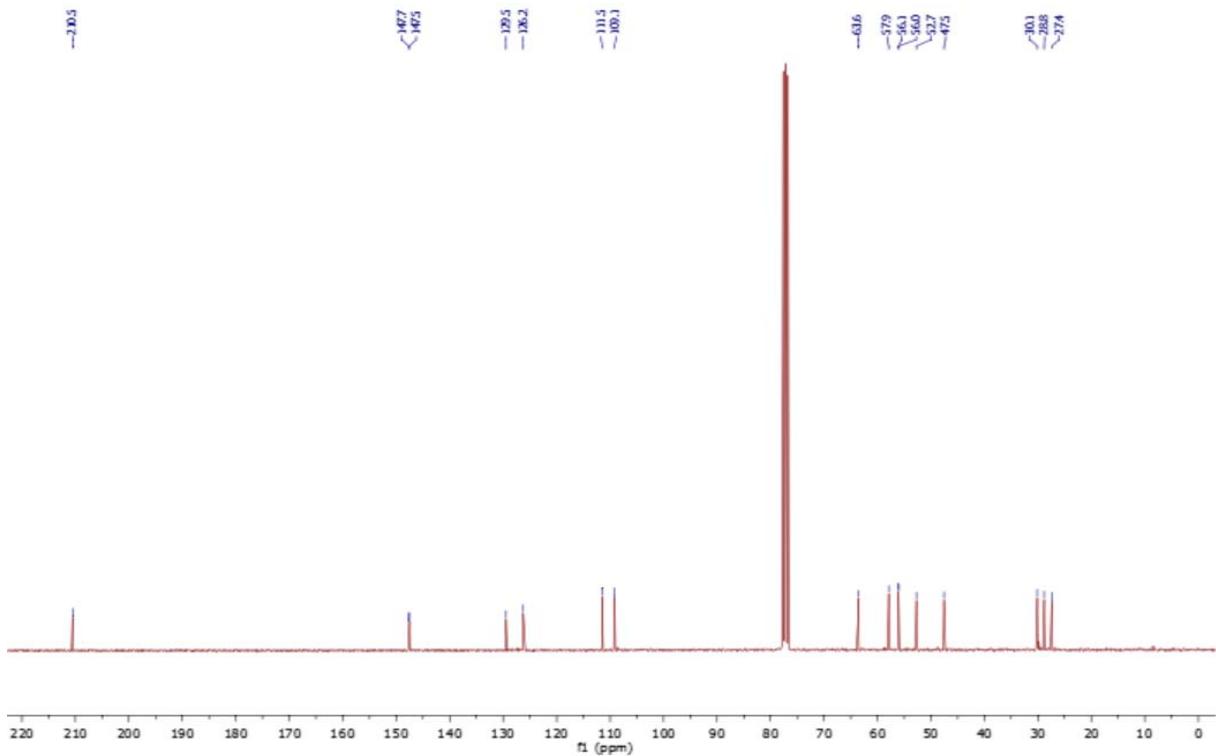


1-(8,9-Dimethoxy-1,2,3,5,6,10b-hexahydropyrrolo[2,1-a]isoquinolin-1-yl)ethan-1-one (2-28)

¹H RMN spectrum

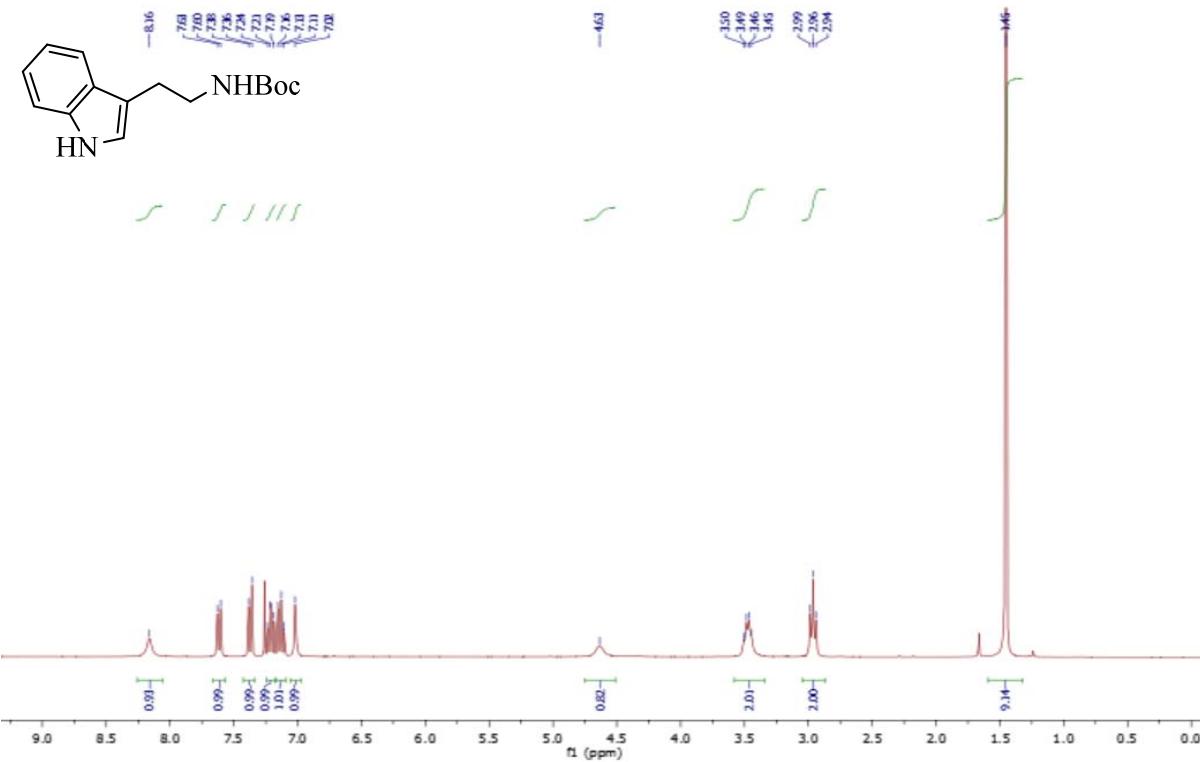


¹³C RMN spectrum

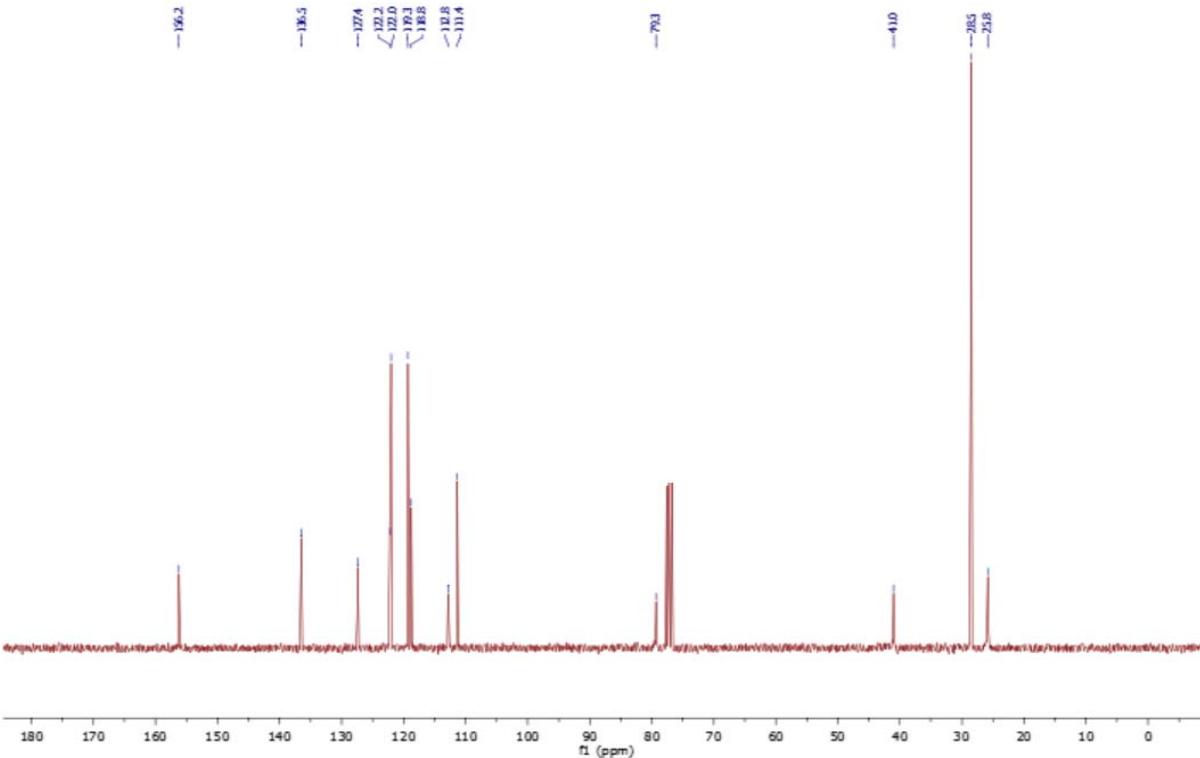


tert-Butyl (2-(1H-indol-3-yl)ethyl)carbamate (3-2)

^1H RMN spectrum

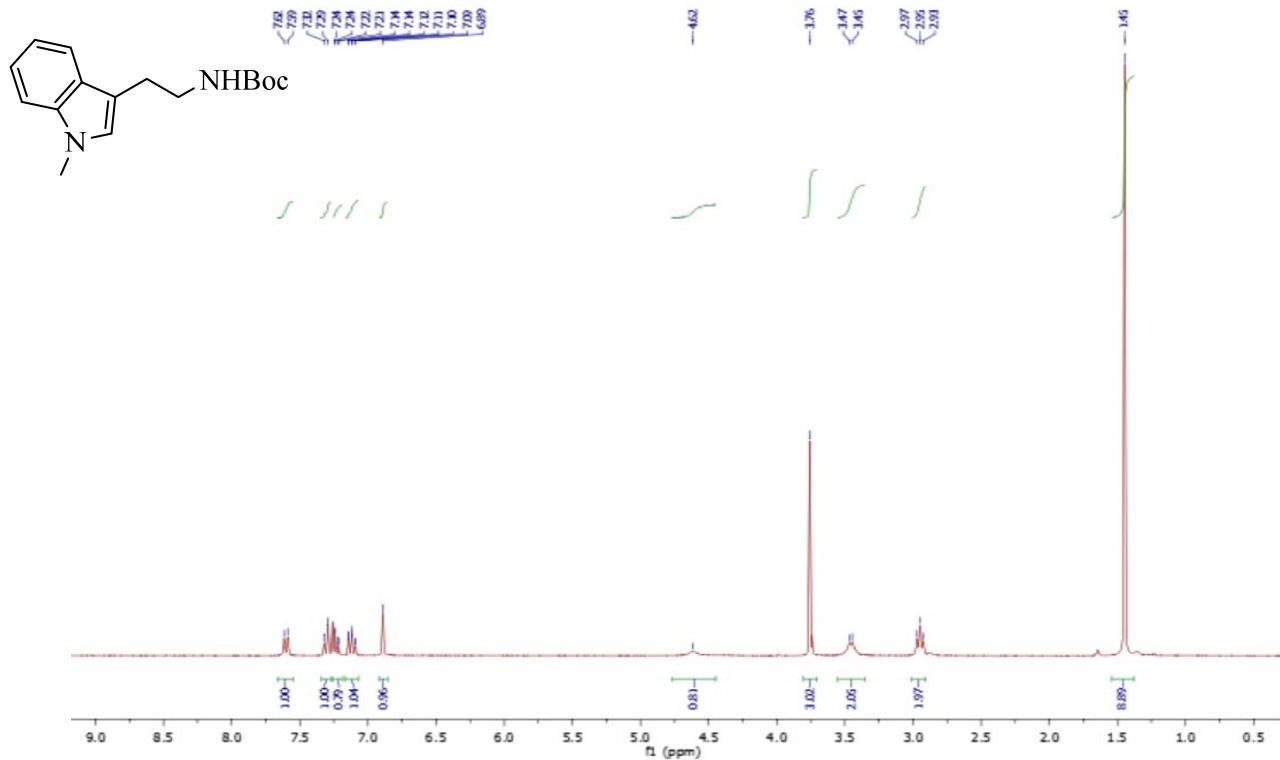


^{13}C RMN spectrum

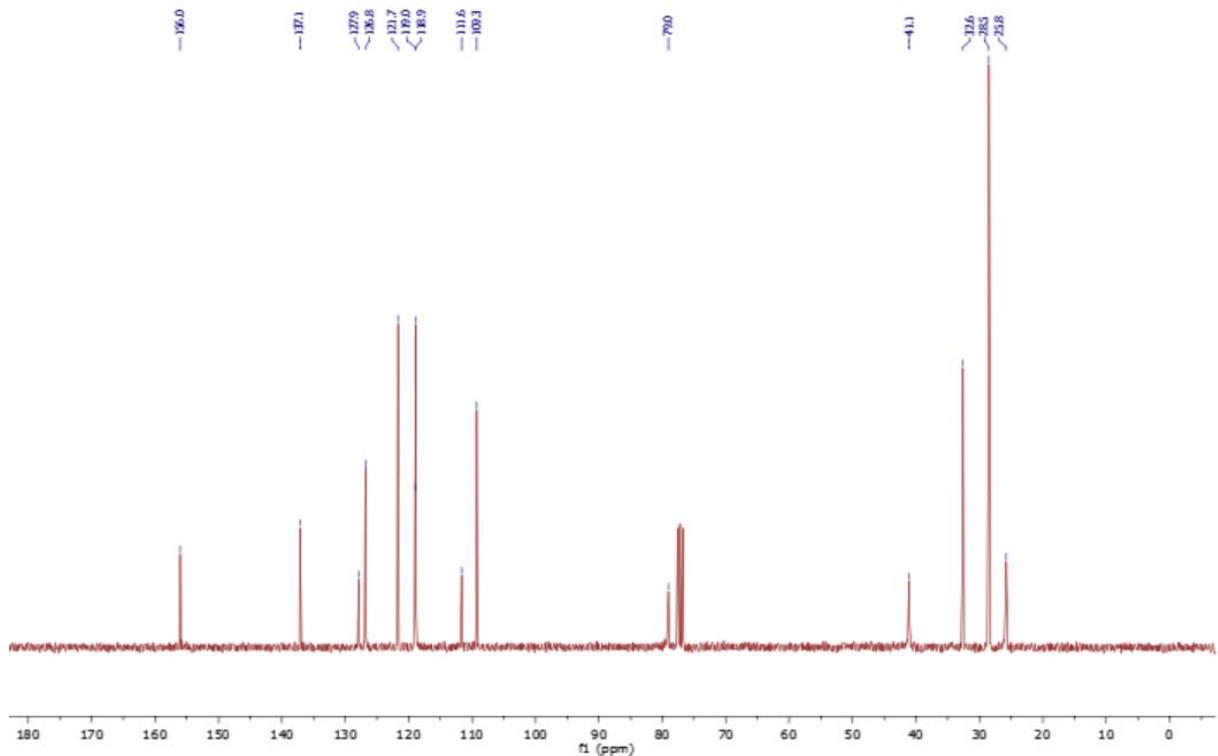


tert-Butyl (2-(1-methyl-1*H*-indol-3-yl)ethyl)carbamate (3-3)

^1H RMN spectrum

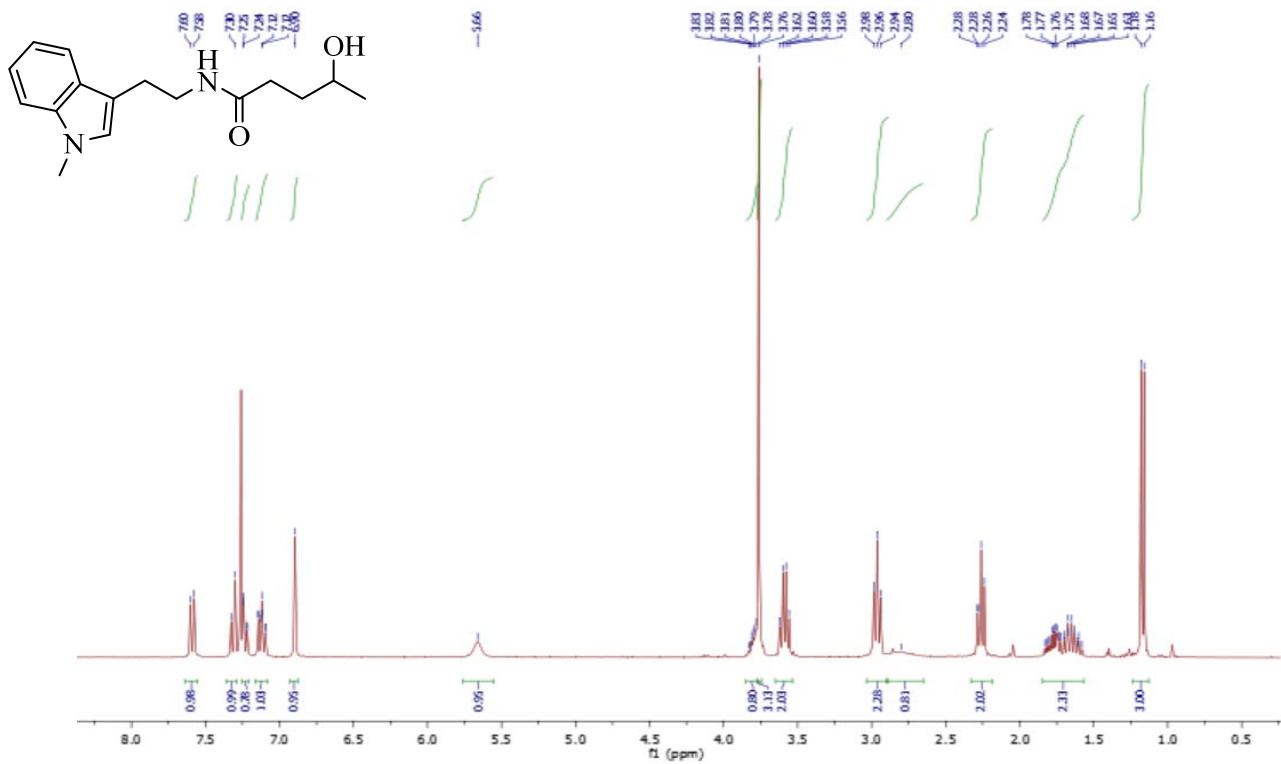


^{13}C RMN spectrum

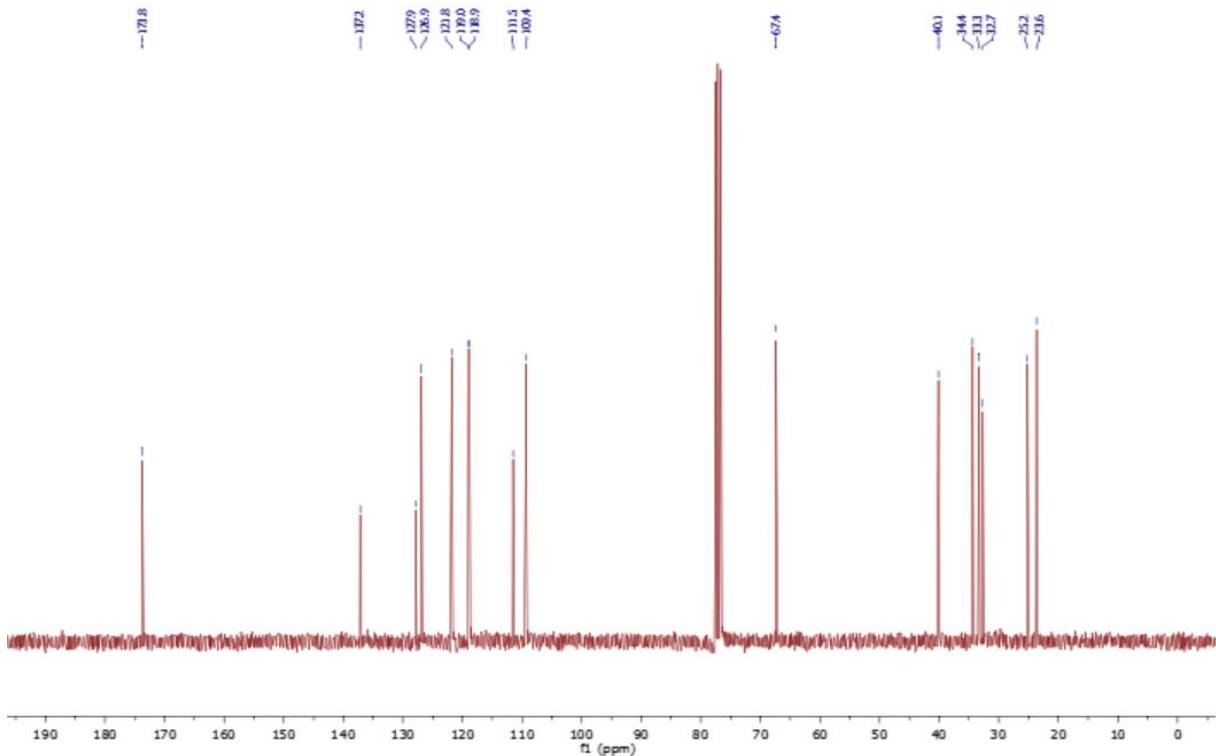


4-Hydroxy-N-(2-(1-methyl-1*H*-indol-3-yl)ethyl)pentanamide (3-4)

¹H RMN spectrum

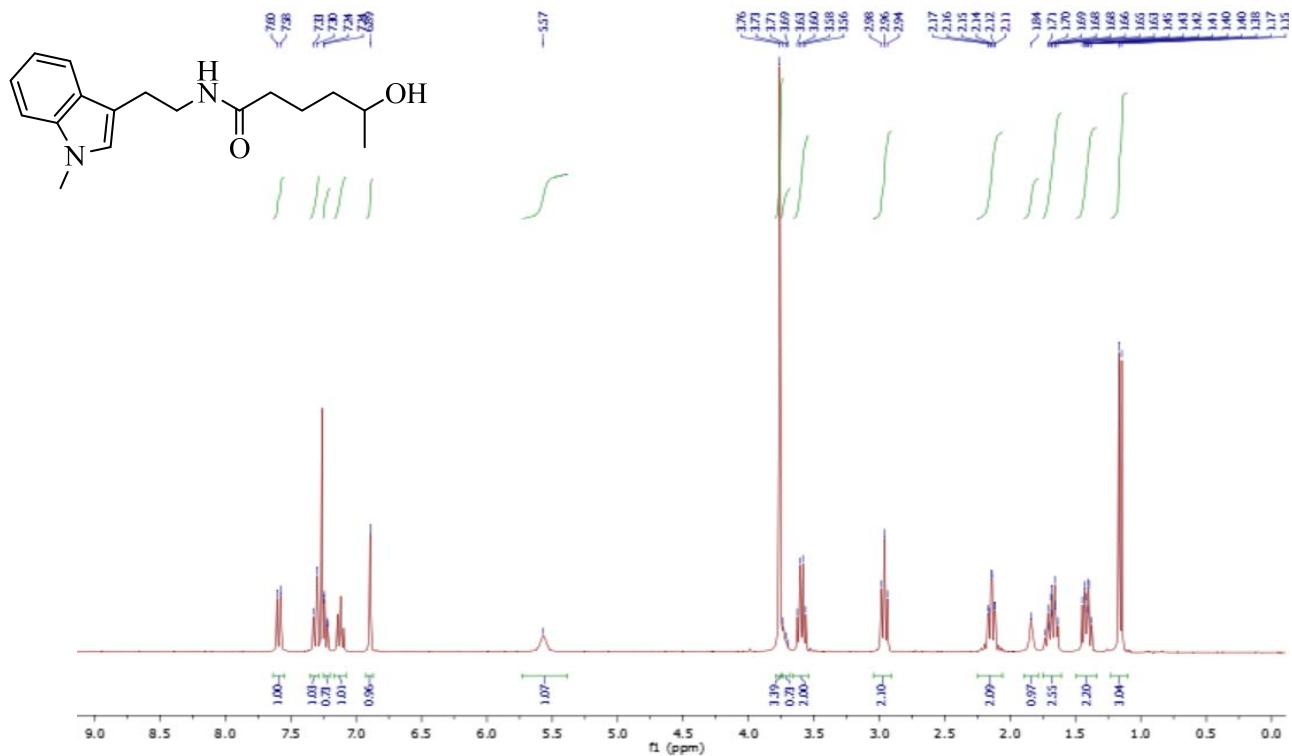


¹³C RMN spectrum

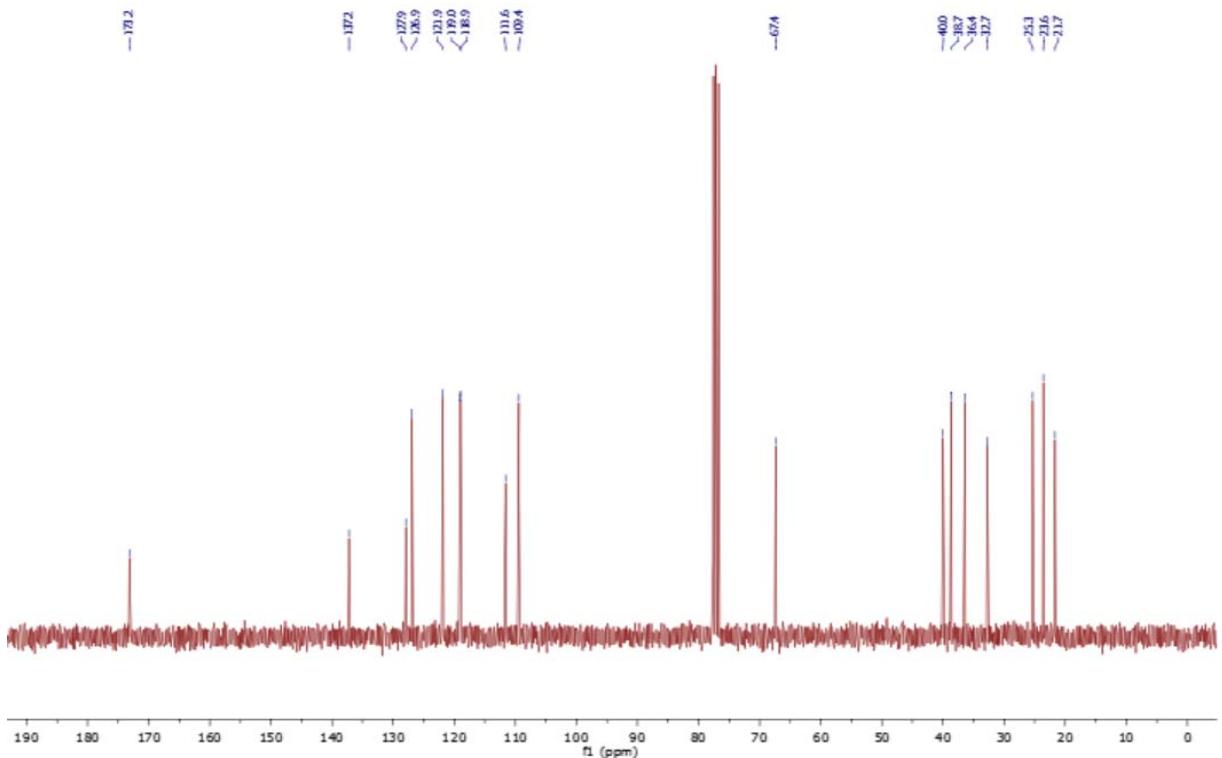


5-Hydroxy-N-(2-(1-methyl-1*H*-indol-3-yl)ethyl)hexanamide (3-5)

¹H RMN spectrum

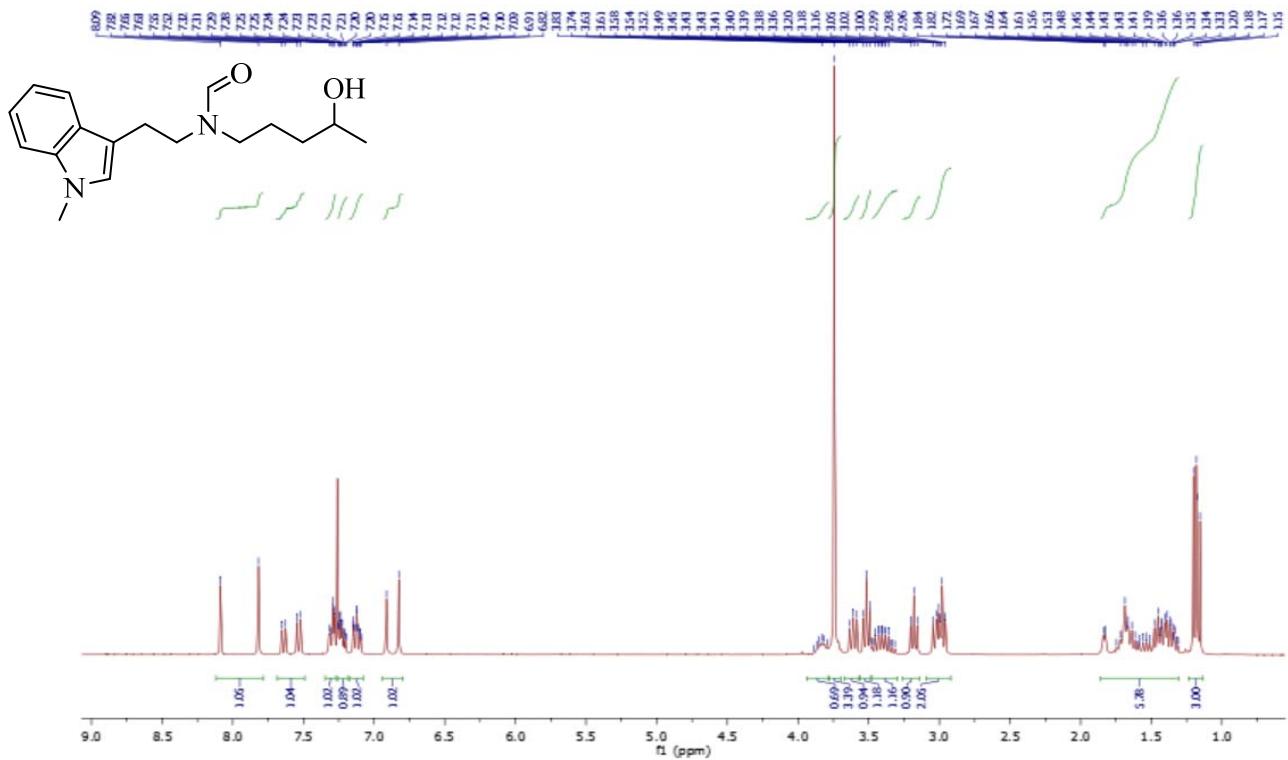


¹³C RMN spectrum

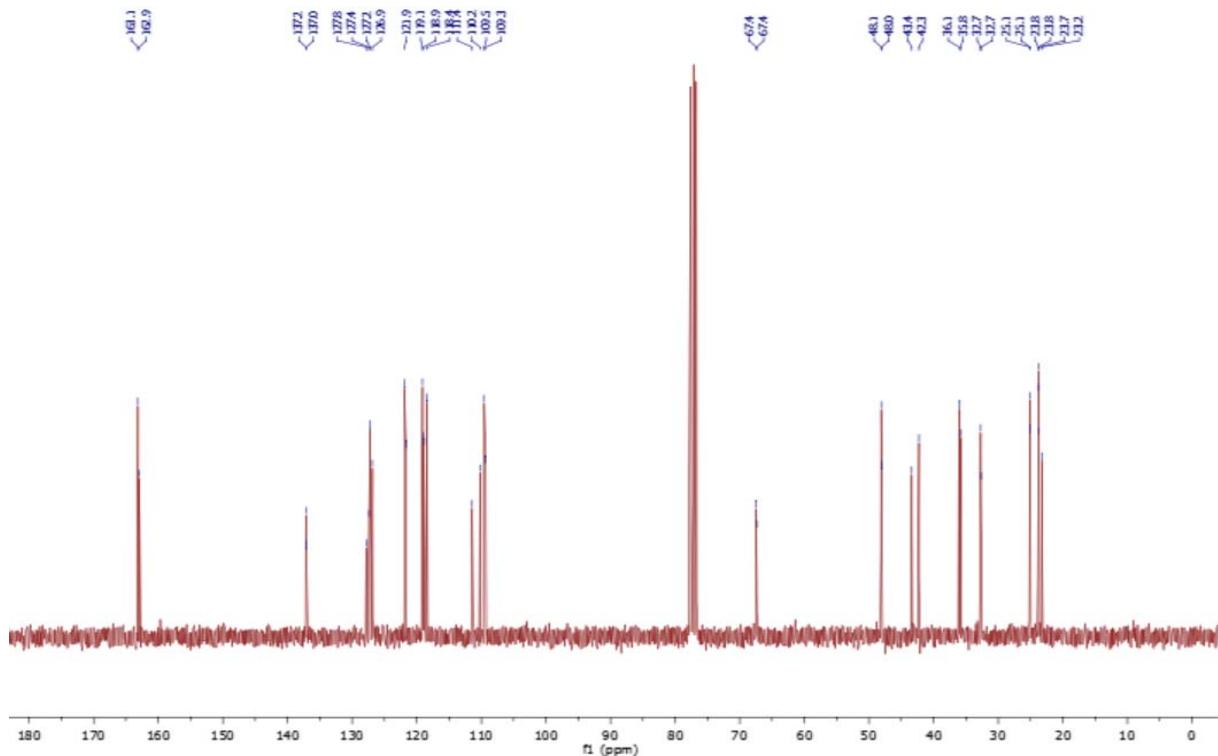


N-(4-Hydroxypentyl)-N-(2-(1-methyl-1*H*-indol-3-yl)ethyl)formamide (3-6)

¹H RMN spectrum

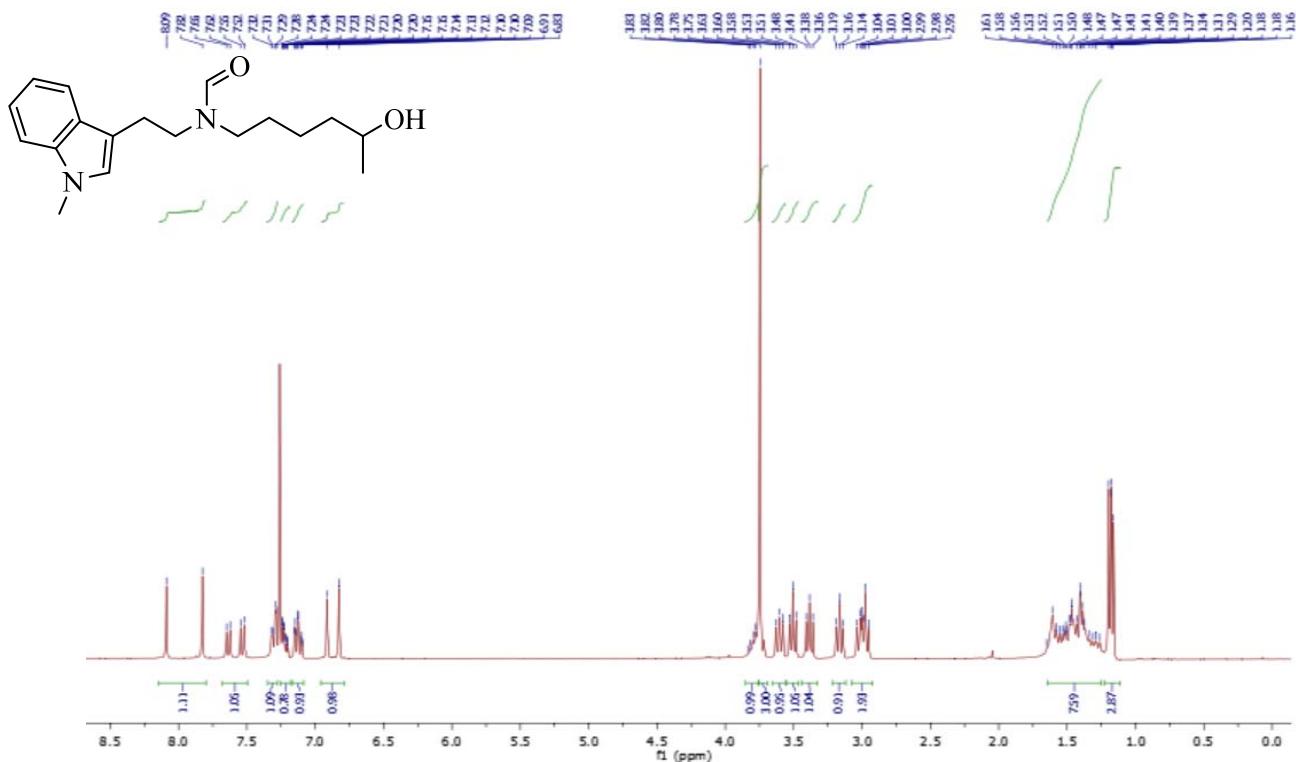


¹³C RMN spectrum

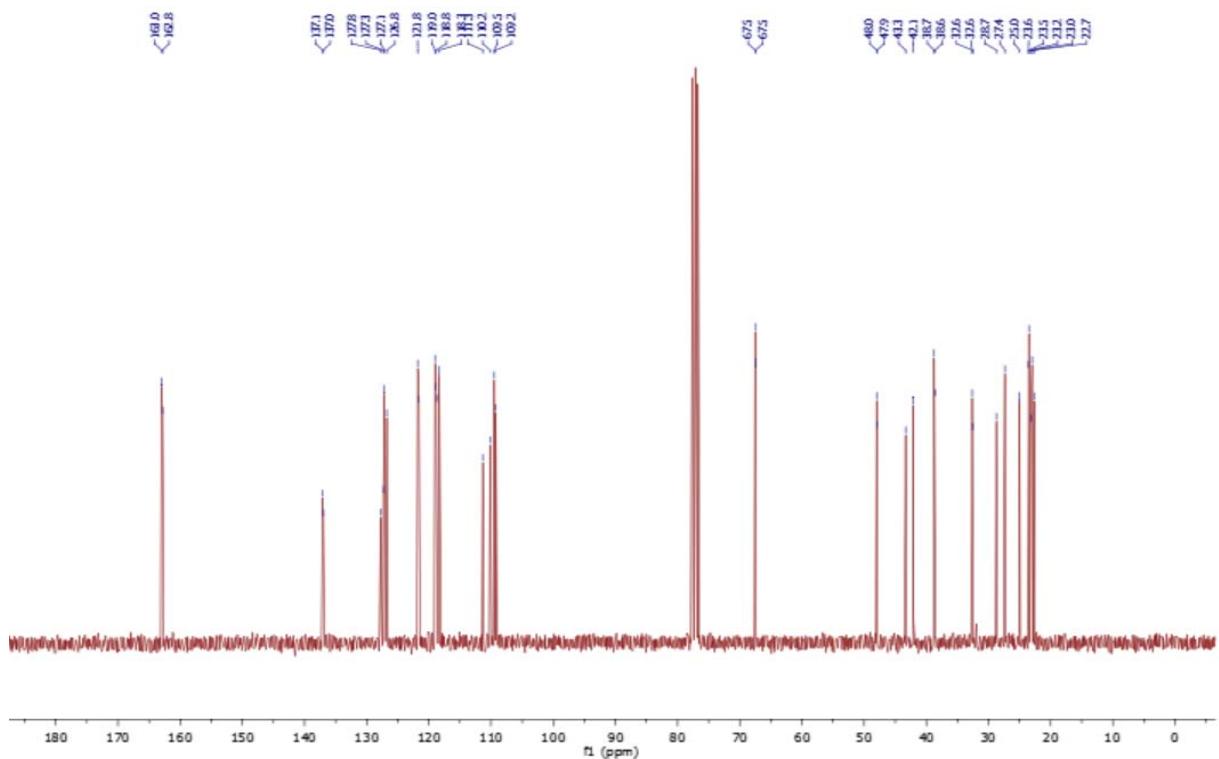


N-(5-Hydroxyhexyl)-N-(2-(1-methyl-1*H*-indol-3-yl)ethyl)formamide (3-7)

¹H RMN spectrum

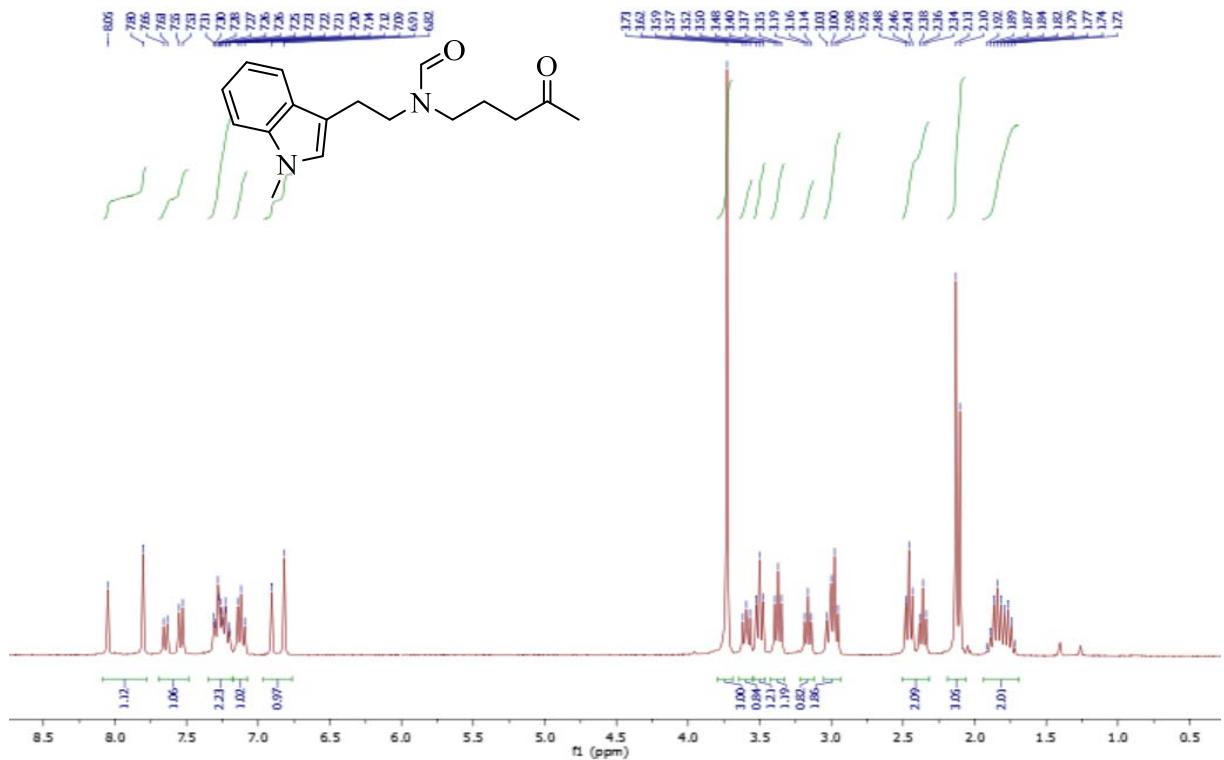


¹³C RMN spectrum

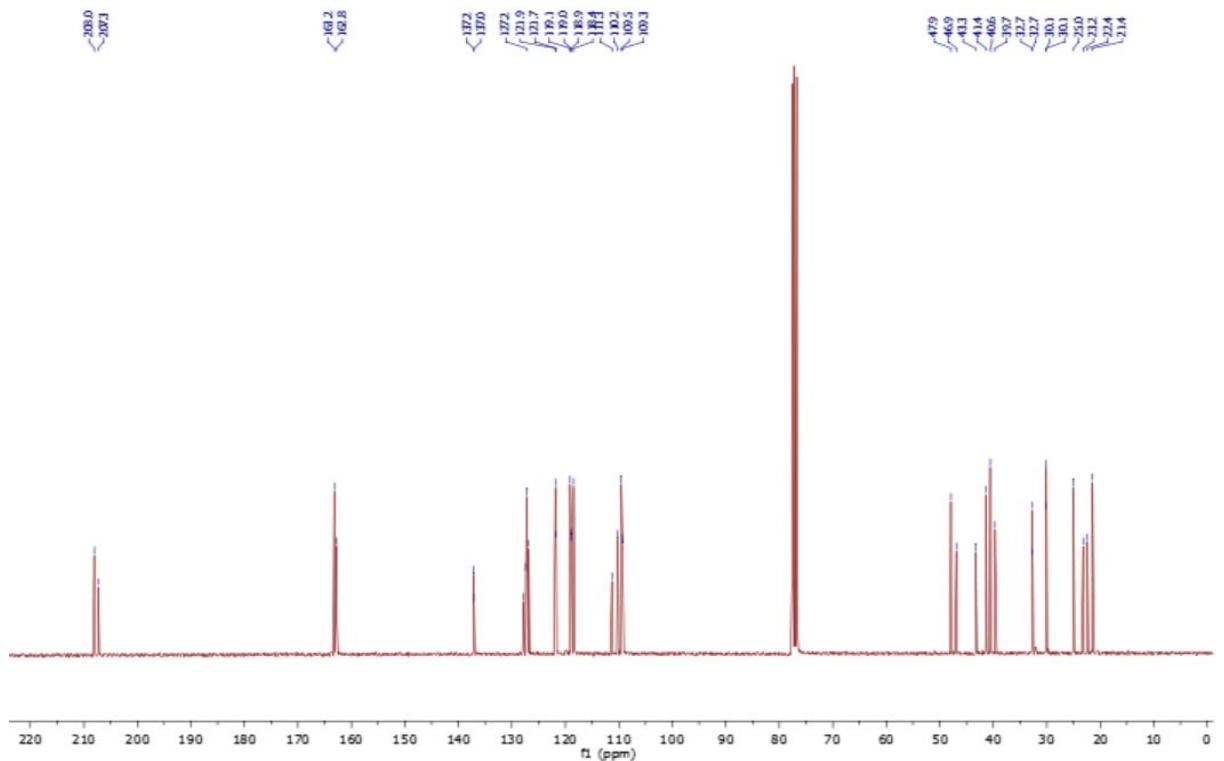


N-(2-(1-Methyl-1*H*-indol-3-yl)ethyl)-*N*-(4-oxopentyl)formamide (3-8)

¹H RMN spectrum

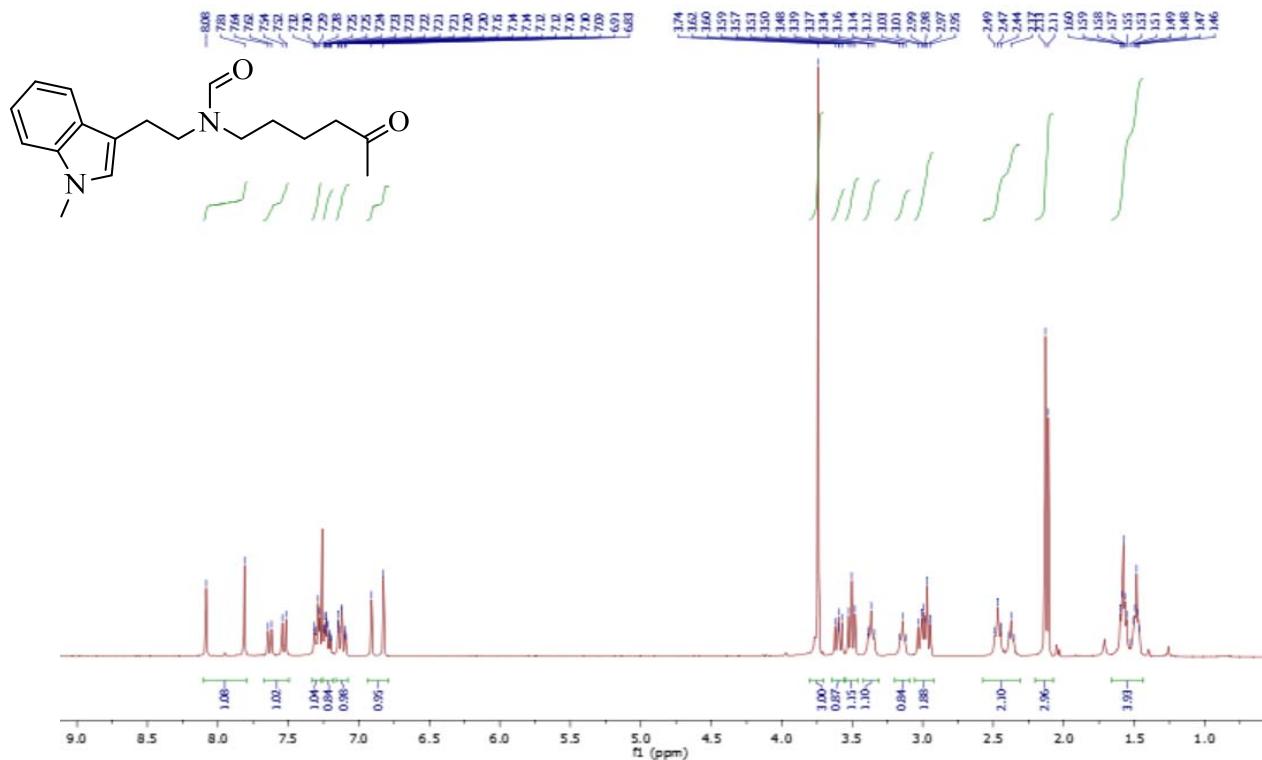


¹³C RMN spectrum

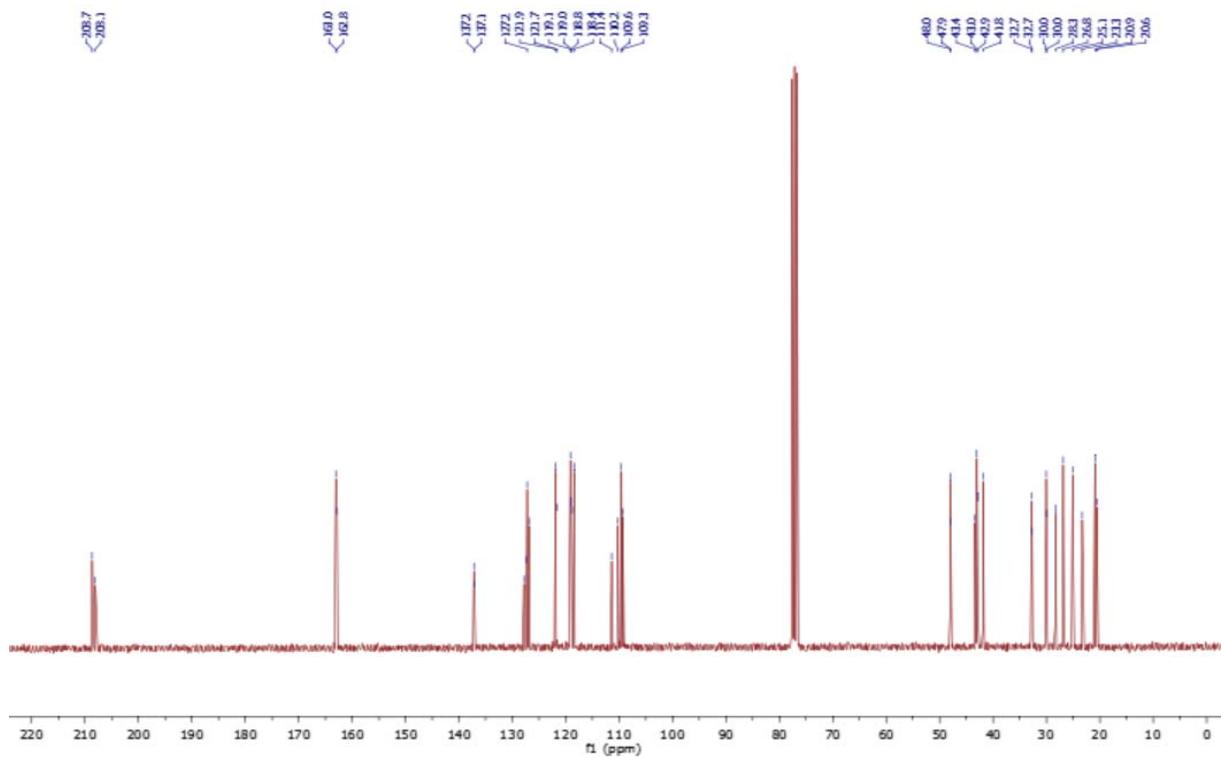


N-(2-(1-Methyl-1*H*-indol-3-yl)ethyl)-*N*-(5-oxohexyl)formamideformamide (3-9)

¹H RMN spectrum

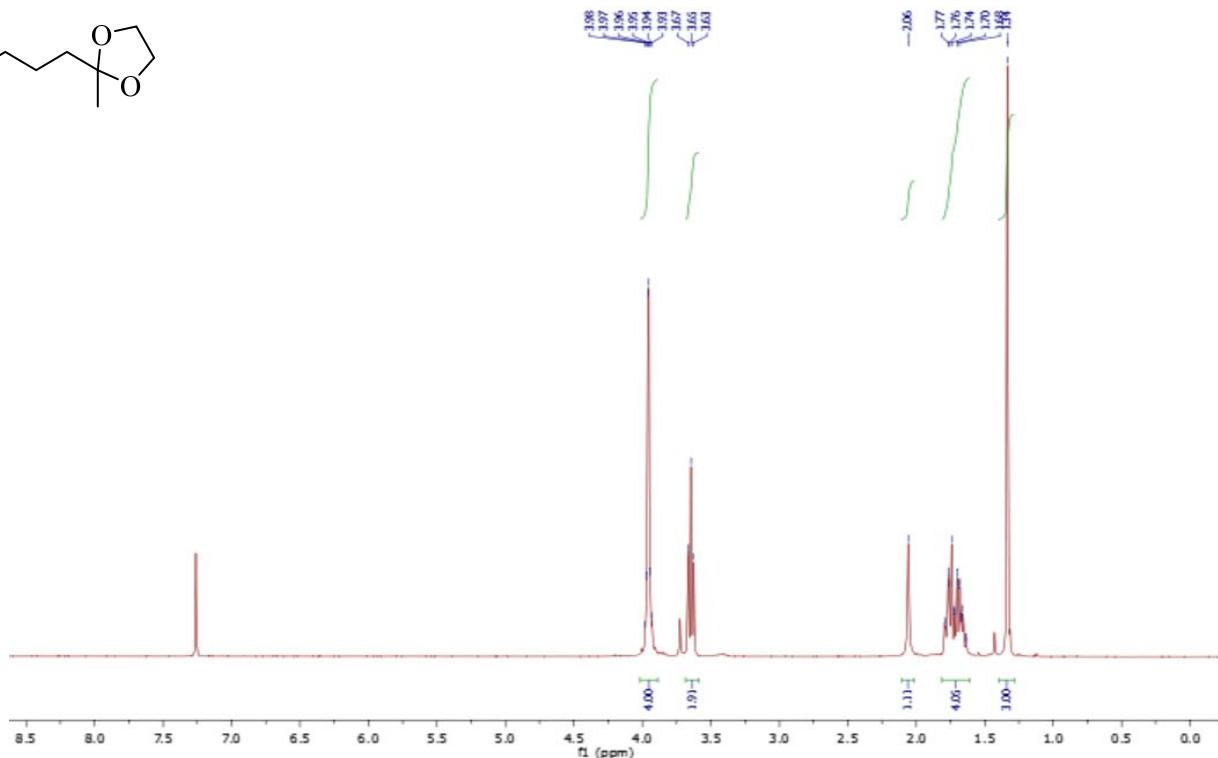
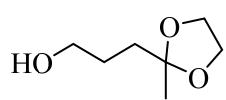


¹³C RMN spectrum

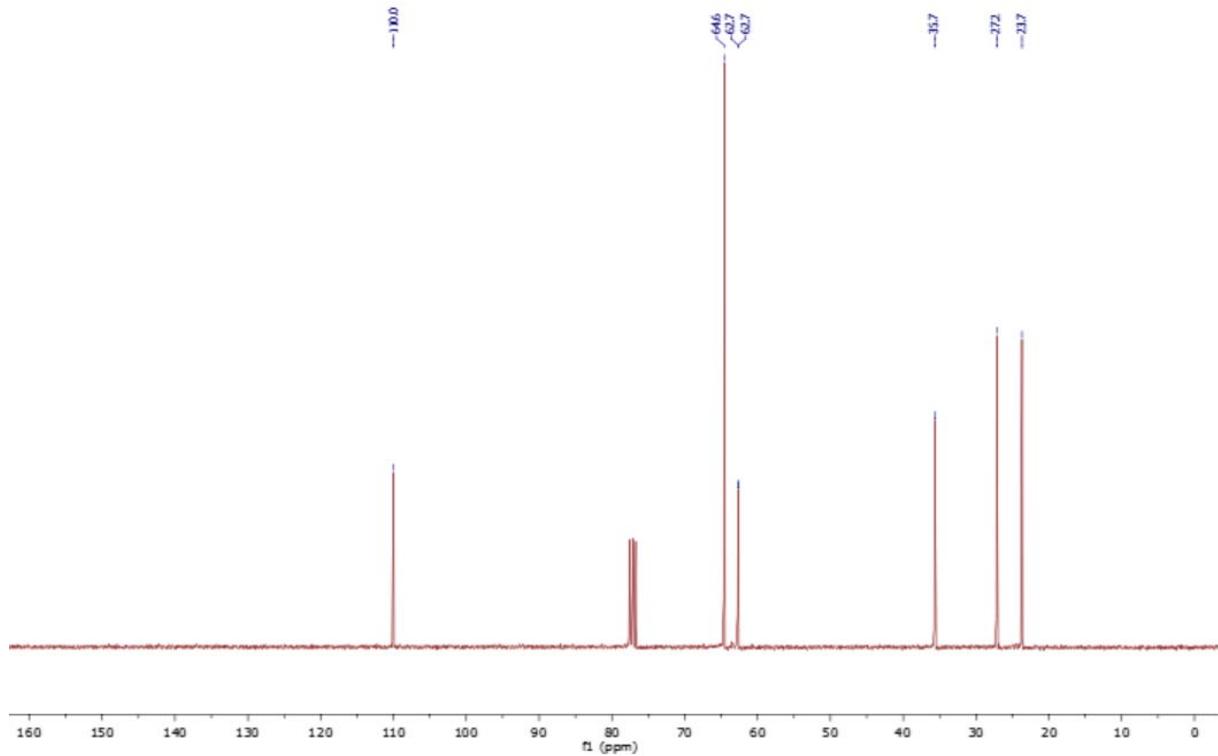


3-(2-Methyl-1,3-dioxolan-2-yl)propan-1-ol (3-11)

^1H RMN spectrum

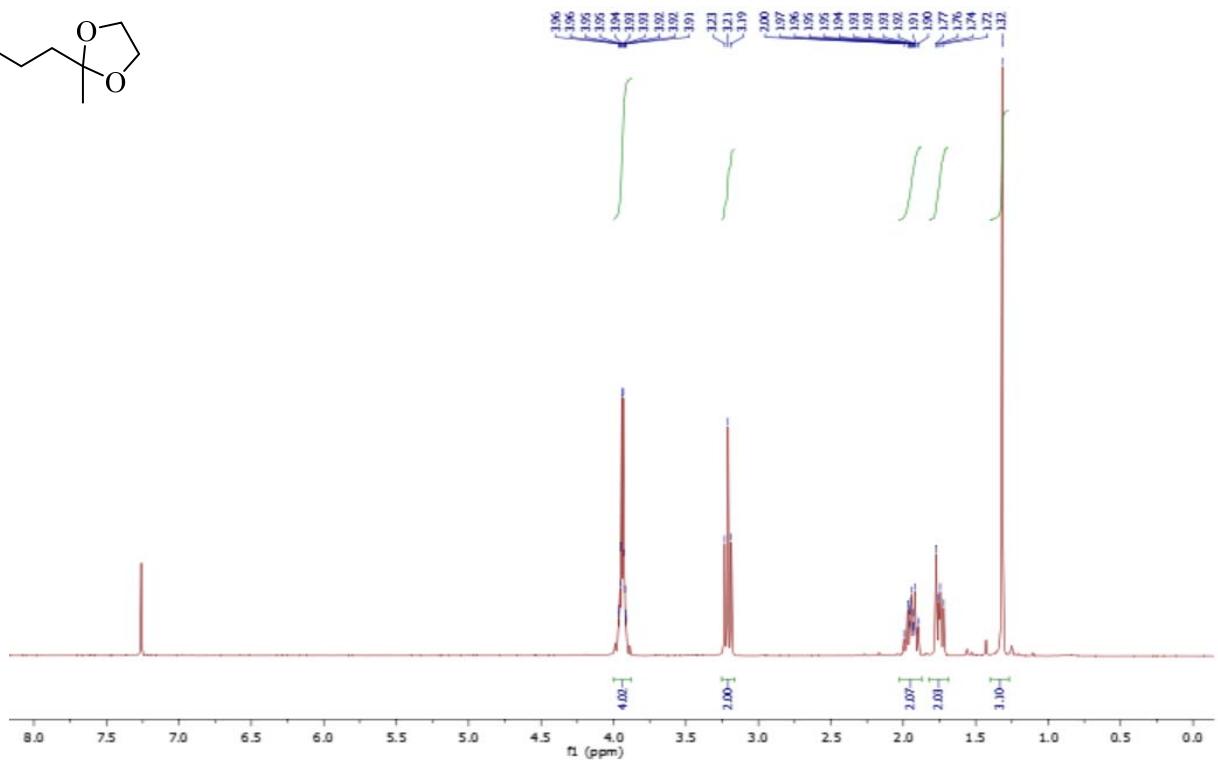
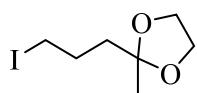


^{13}C RMN spectrum

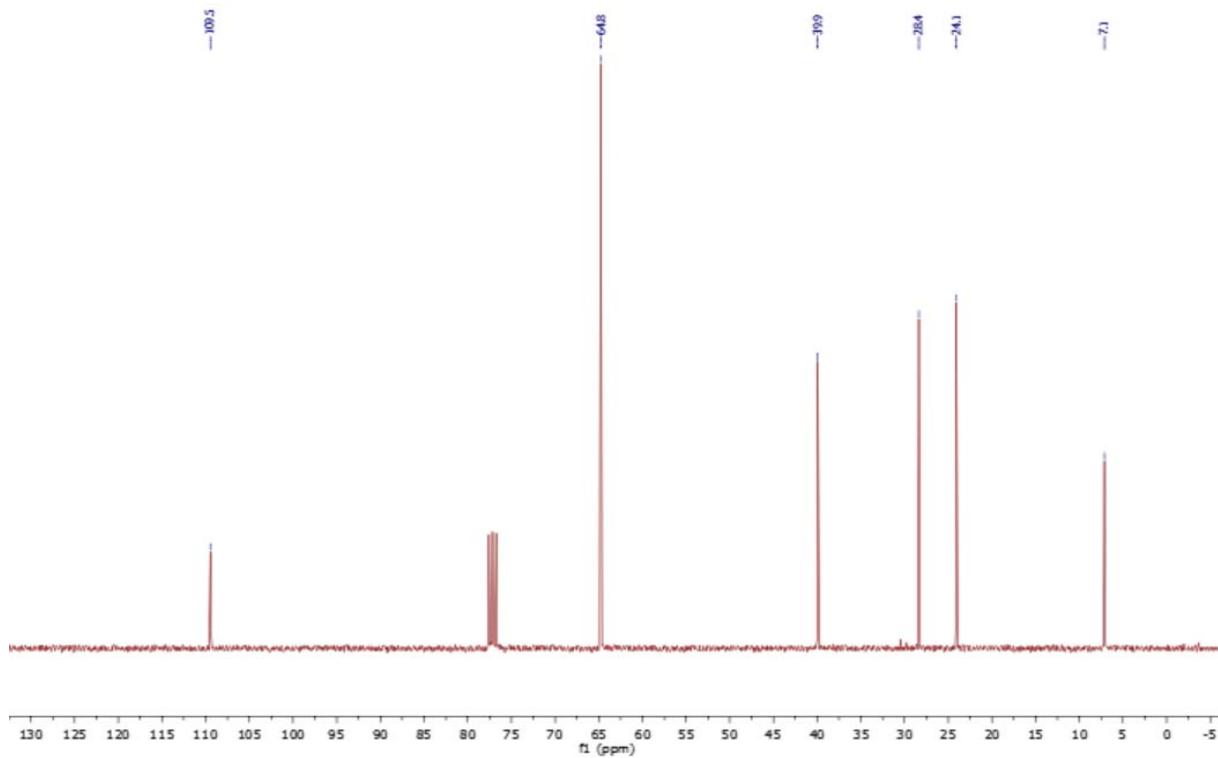


2-(3-Iodopropyl)-2-methyl-1,3-dioxolane (3-12)

^1H RMN spectrum

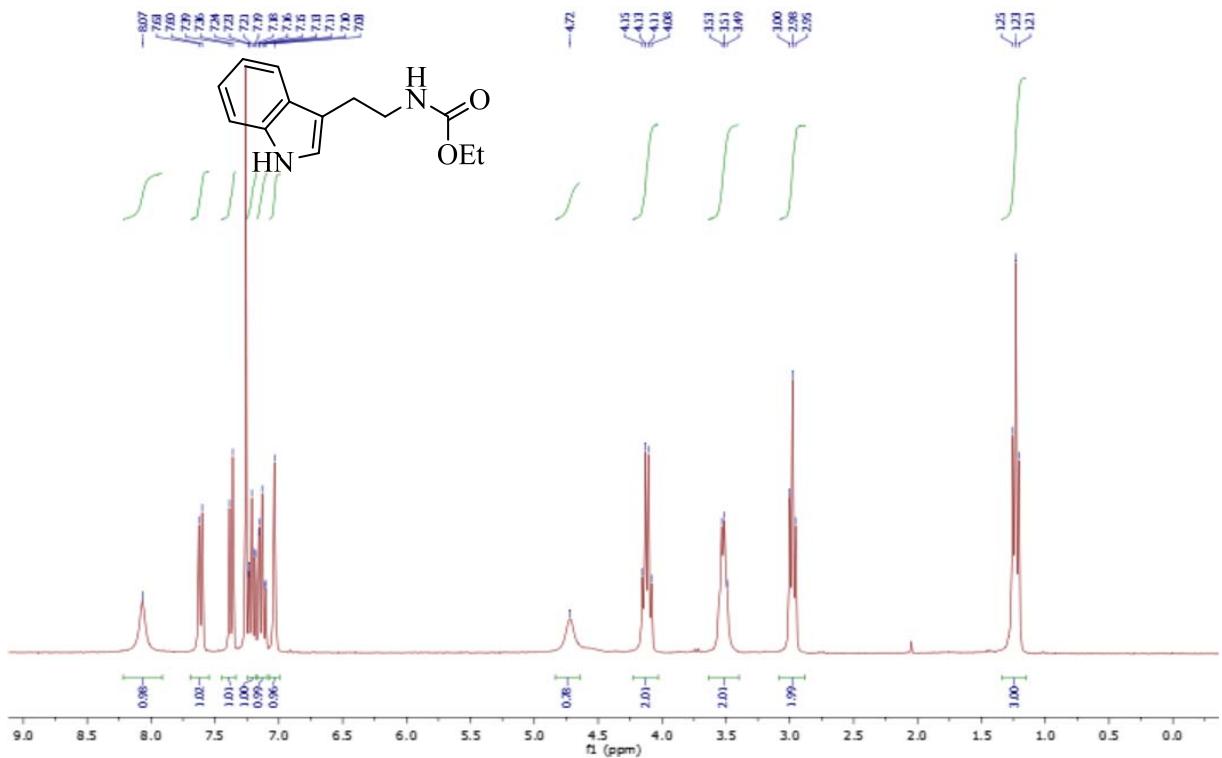


^{13}C RMN spectrum

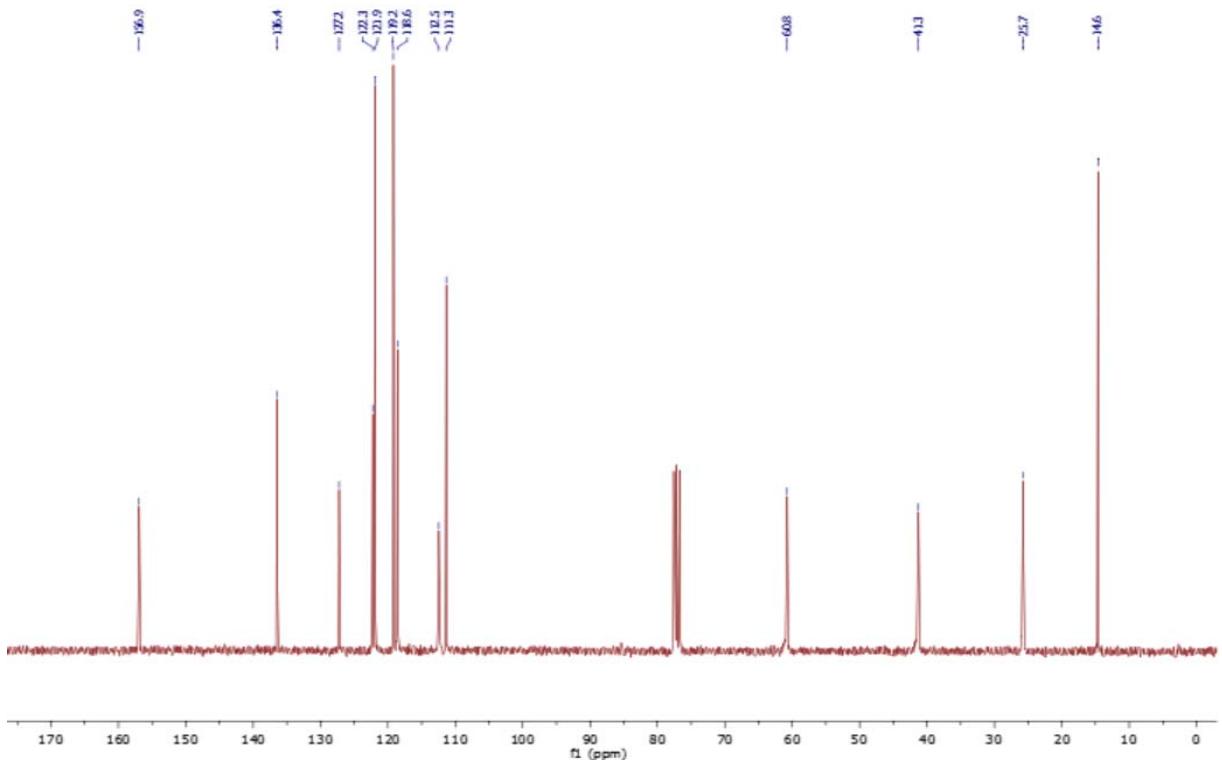


Ethyl (2-(1*H*-indol-3-yl)ethyl)carbamate (3-13)

^1H RMN spectrum

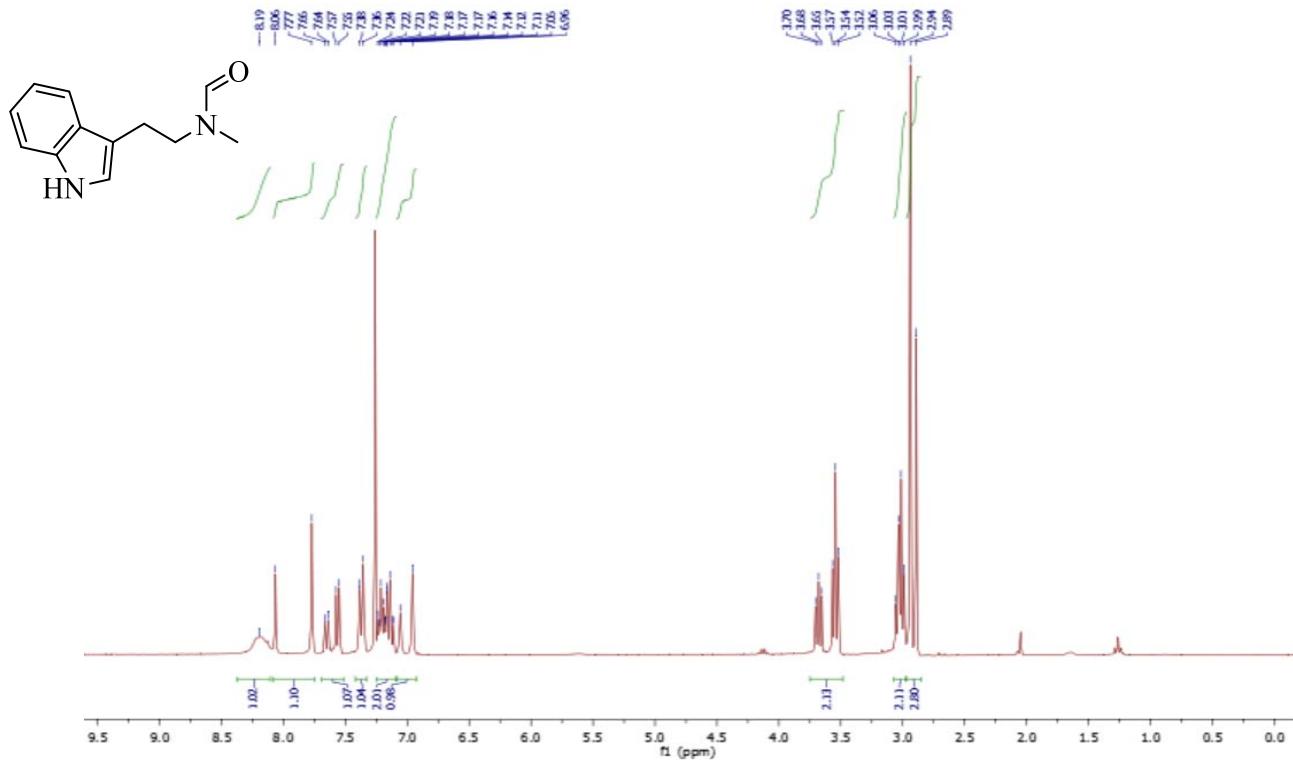


^{13}C RMN spectrum

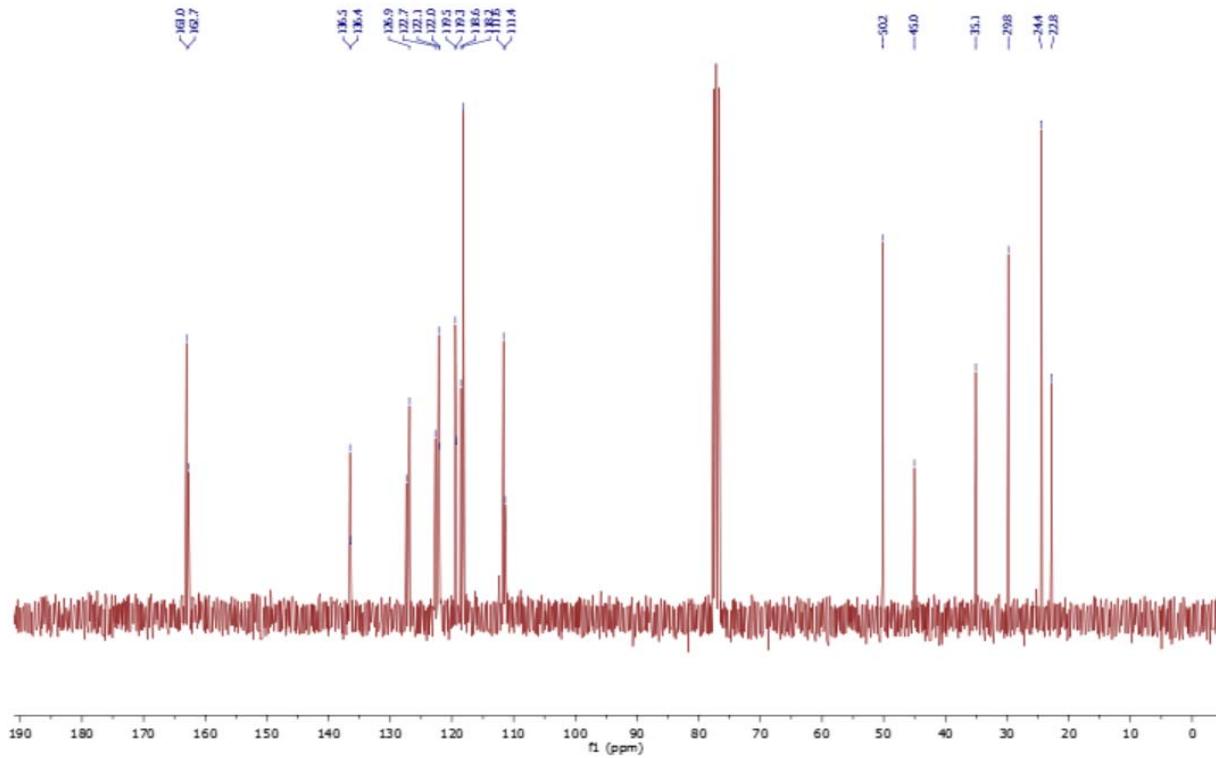


N-(2-(1*H*-Indol-3-yl)ethyl)-*N*-methylformamide (3-14)

¹H RMN spectrum

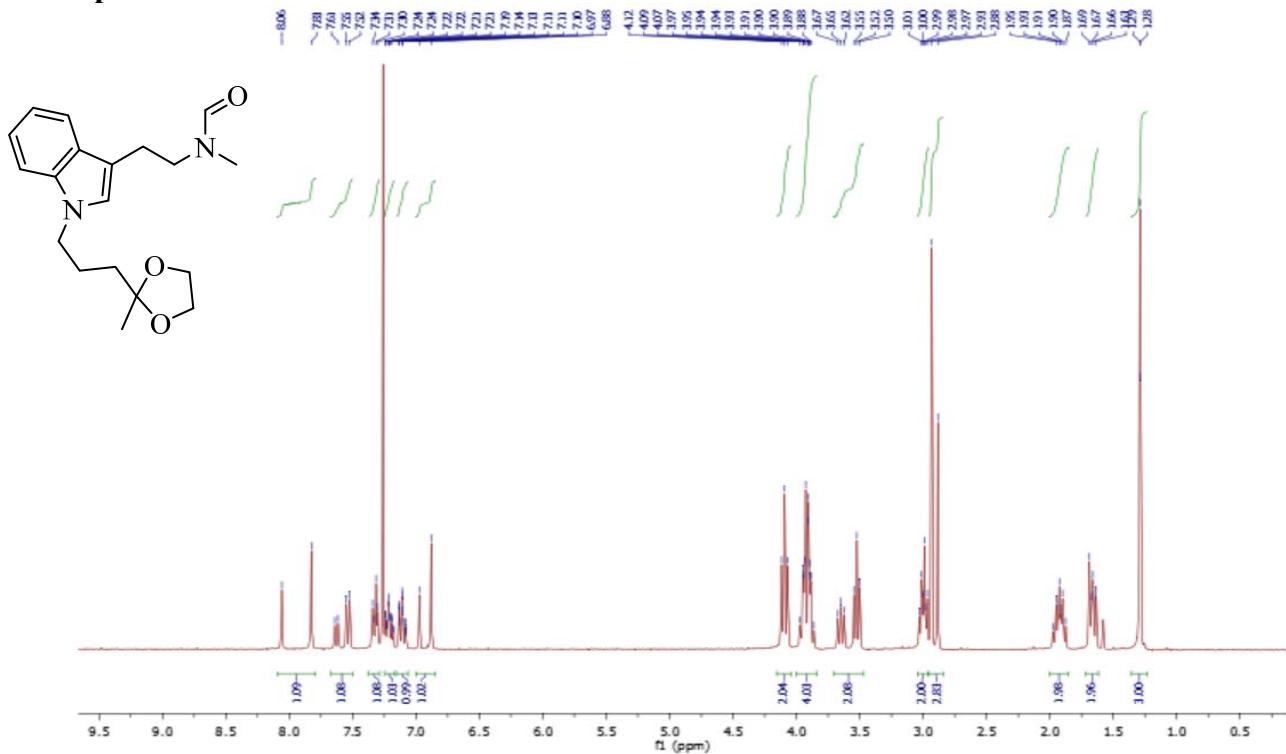


¹³C RMN spectrum

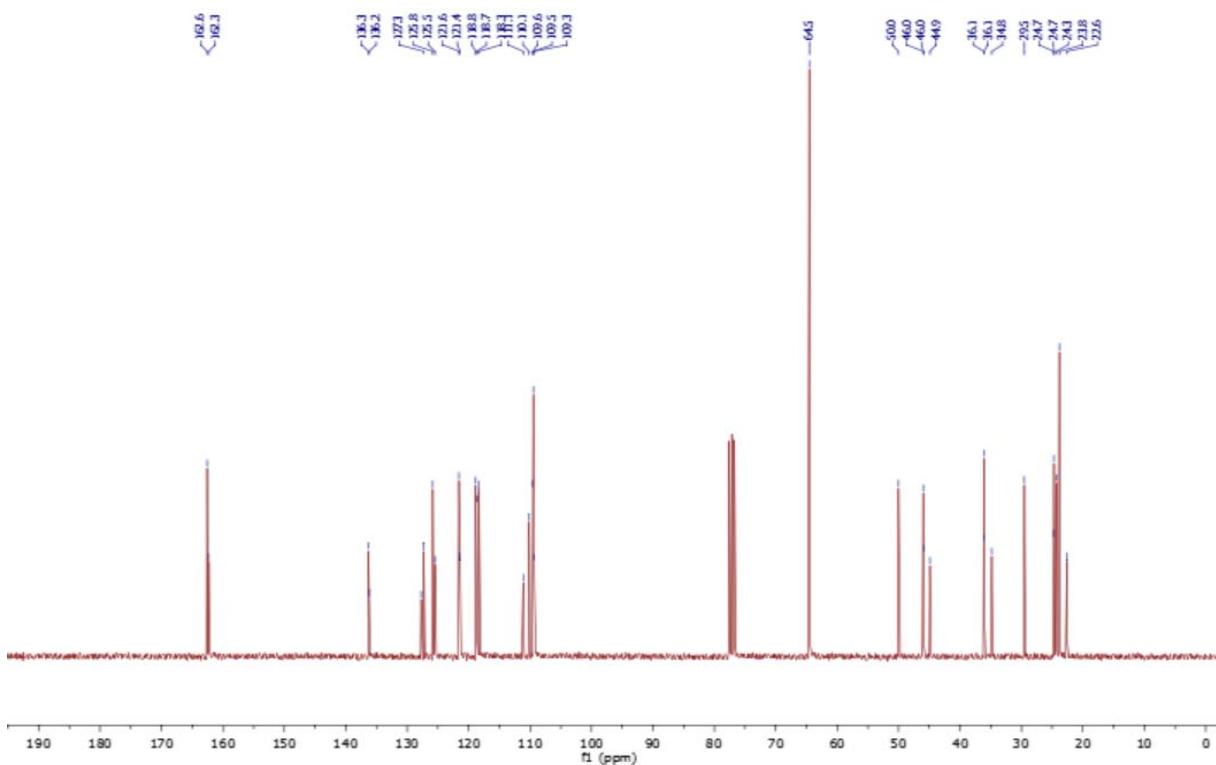


**N-Methyl-N-(2-(1-(3-(2-methyl-1,3-dioxolan-2-yl)propyl)-1*H*-indol-3-yl)ethyl)formamide
(3-15)**

¹H RMN spectrum

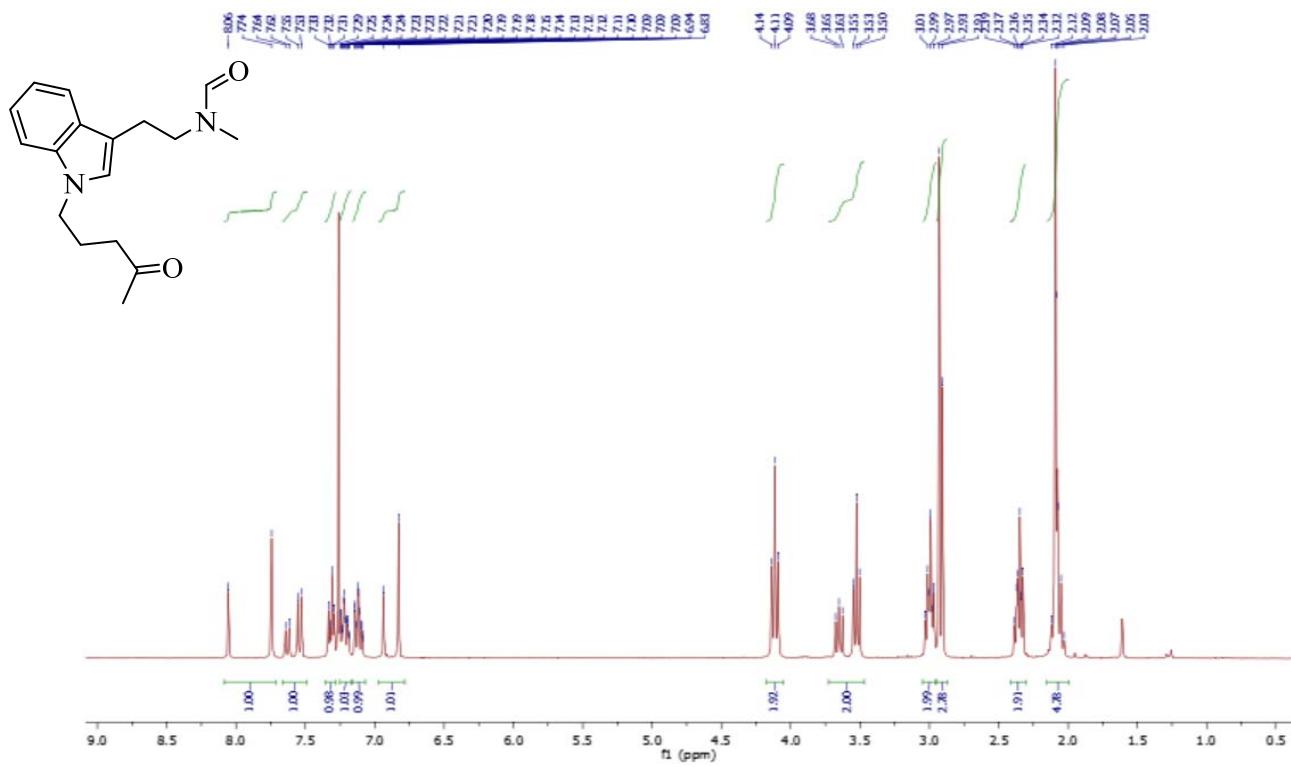


¹³C RMN spectrum

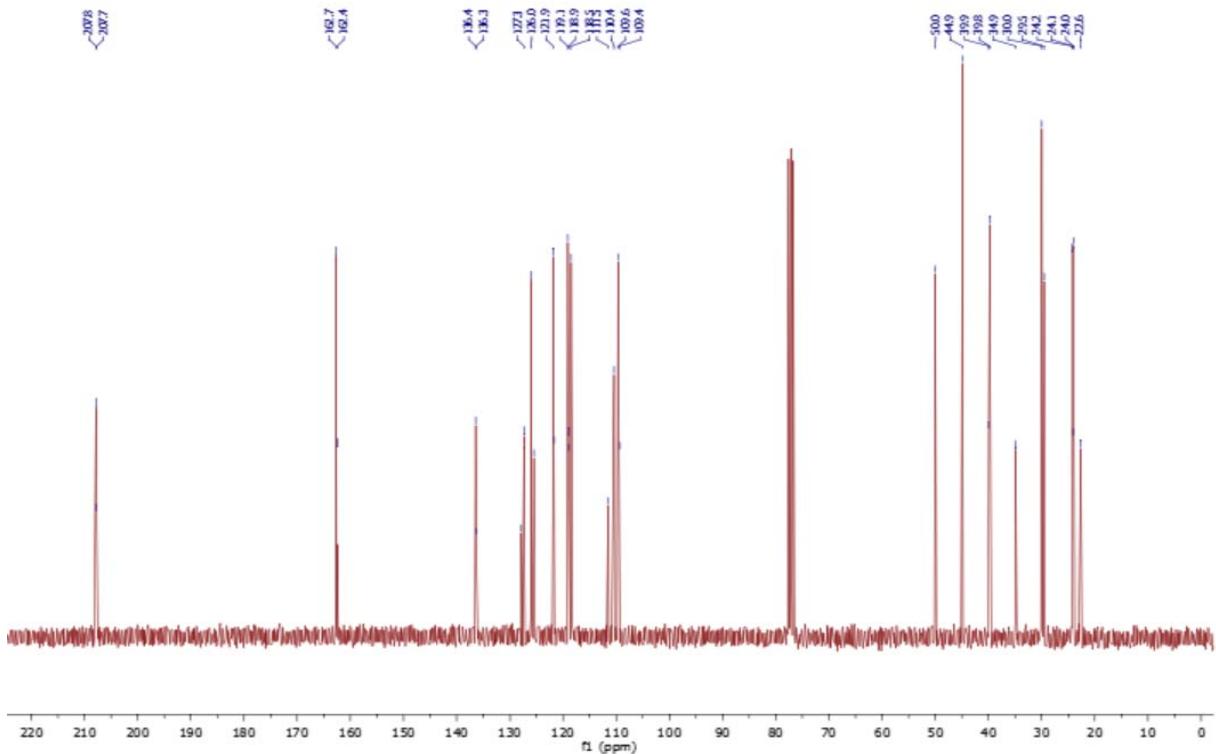


N-Methyl-N-(2-(1-(4-oxopentyl)-1*H*-indol-3-yl)ethyl)formamide (3-16)

^1H RMN spectrum

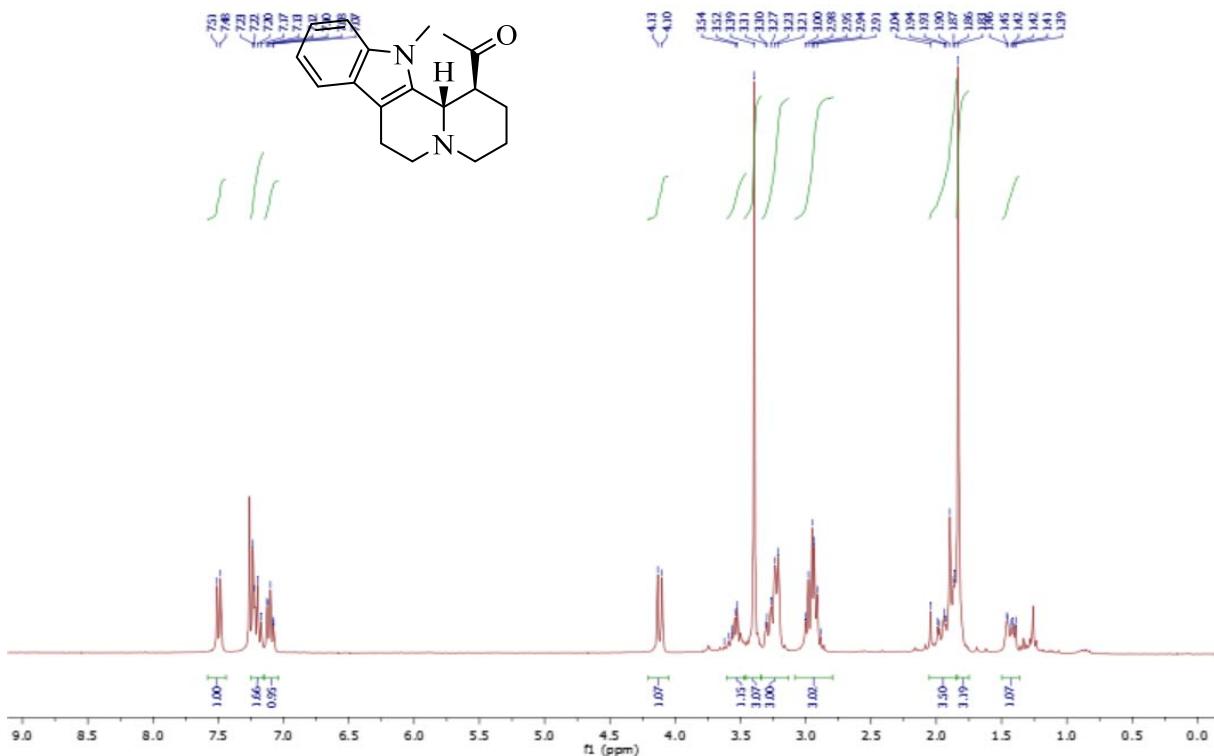


^{13}C RMN spectrum

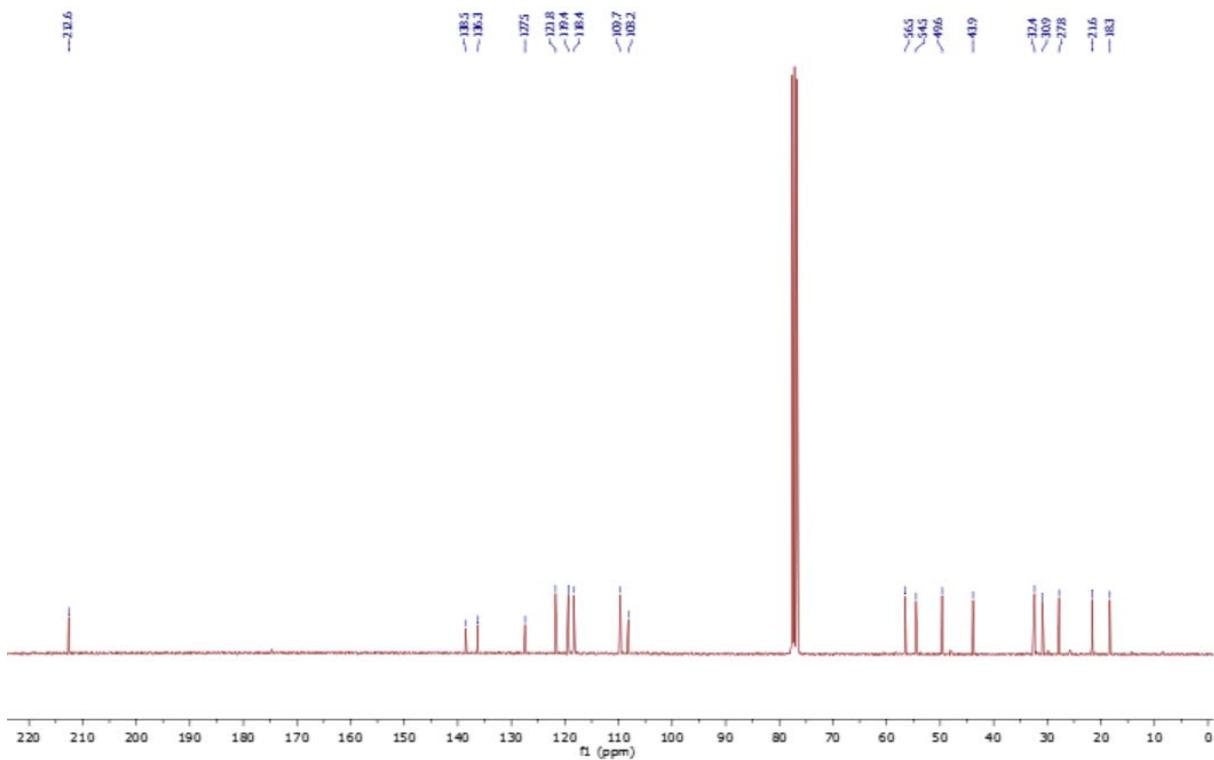


***trans*-1-(12-Methyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizin-1-yl)ethan-1-one (3-24)**

¹H RMN spectrum

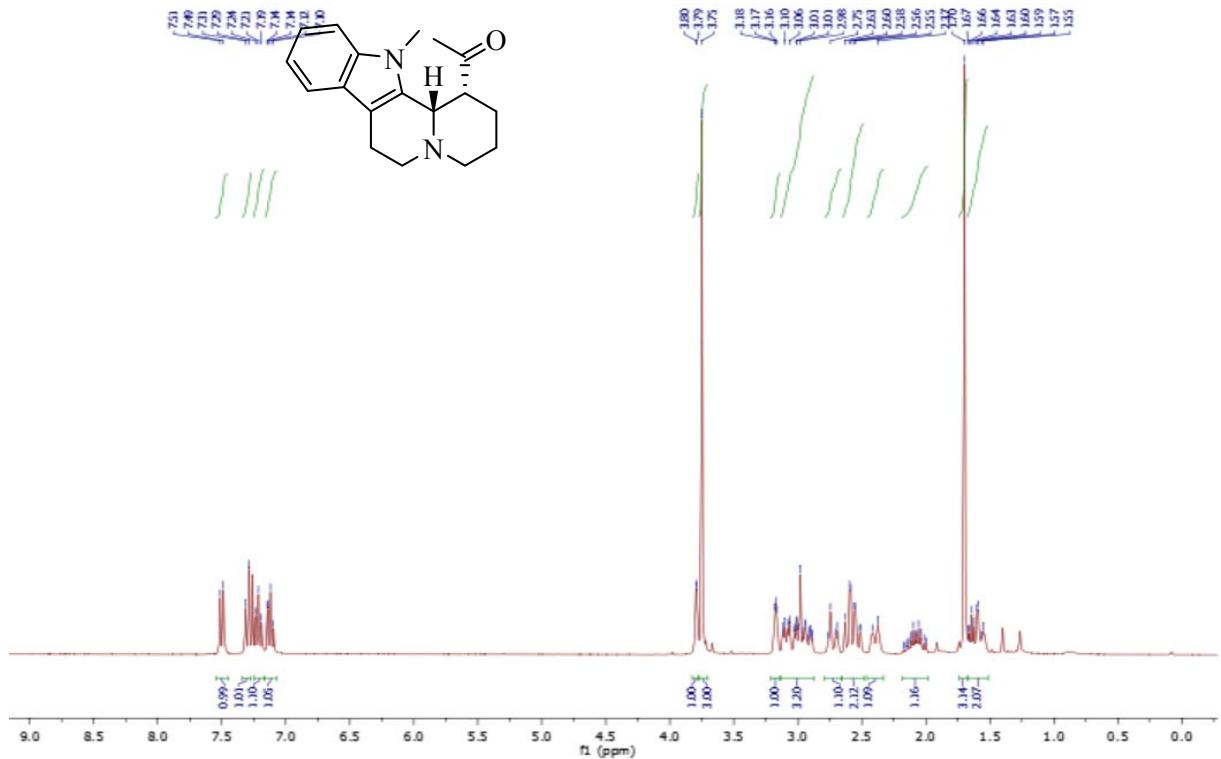


¹³C RMN spectrum

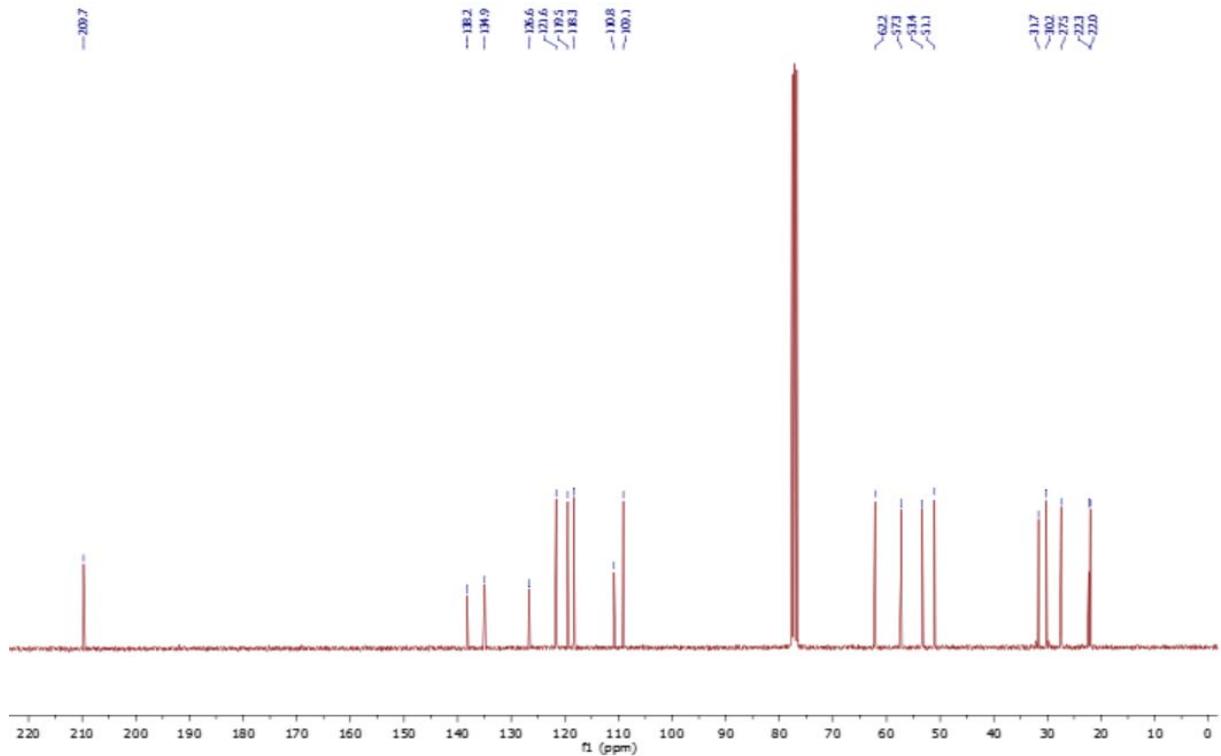


cis-1-(12-methyl-1,2,3,4,6,7,12,12b-octahydroindolo[2,3-a]quinolizin-1-yl)ethan-1-one (3-24)

¹H RMN spectrum

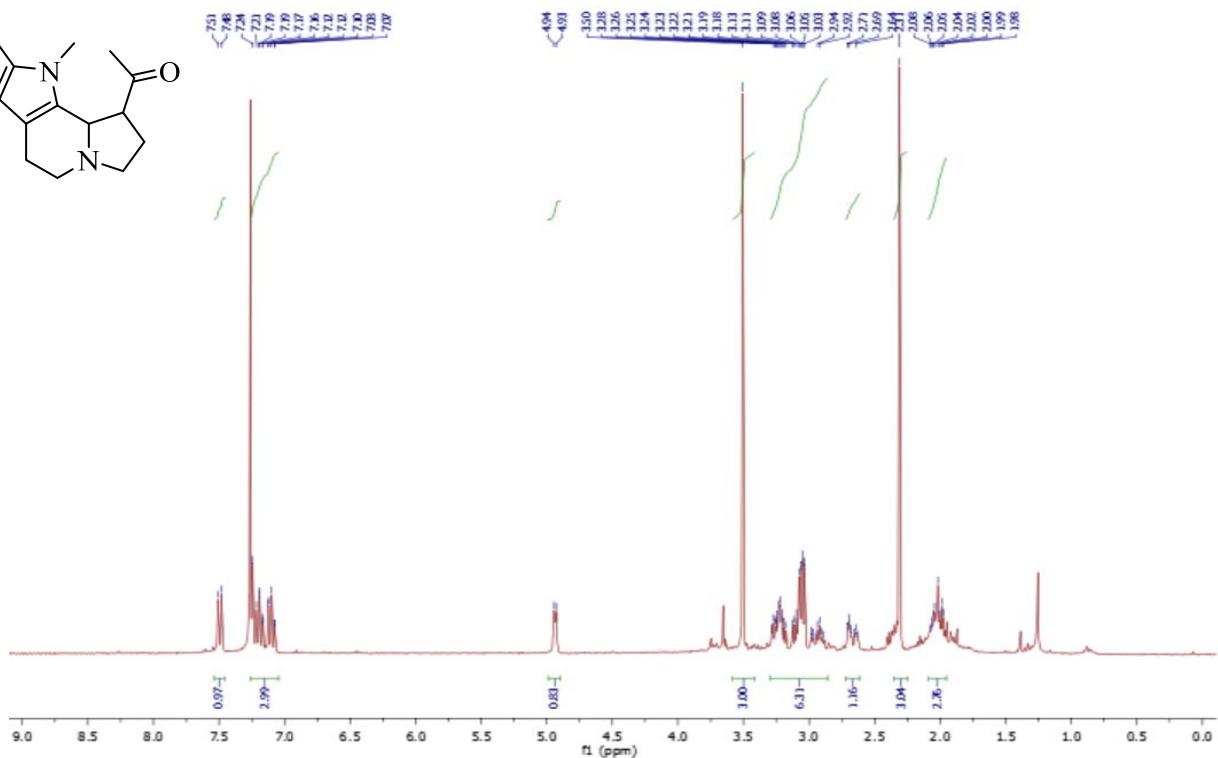
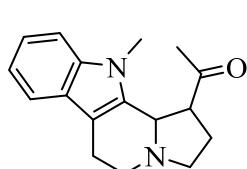


¹³C RMN spectrum

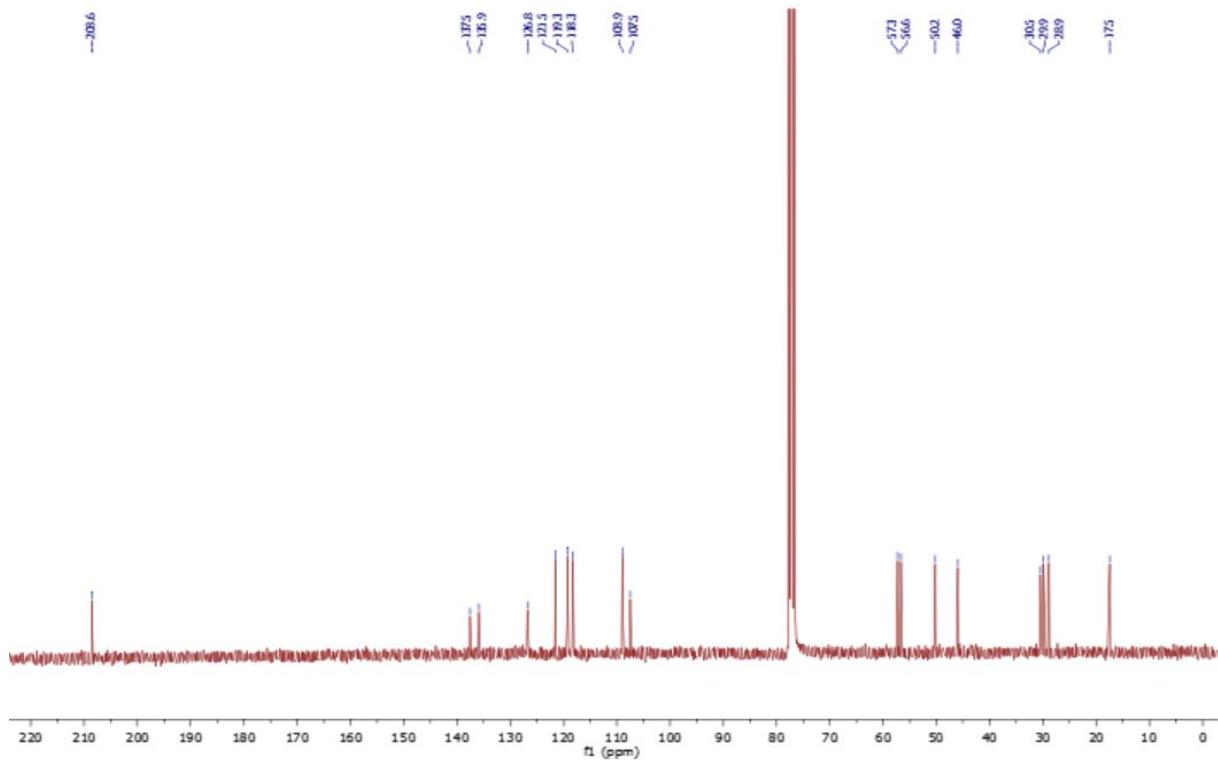


1-(11-Methyl-2,3,5,6,11,11b-hexahydro-1*H*-indolizino[8,7-*b*]indol-1-yl)ethan-1-one (3-32)

¹H RMN spectrum

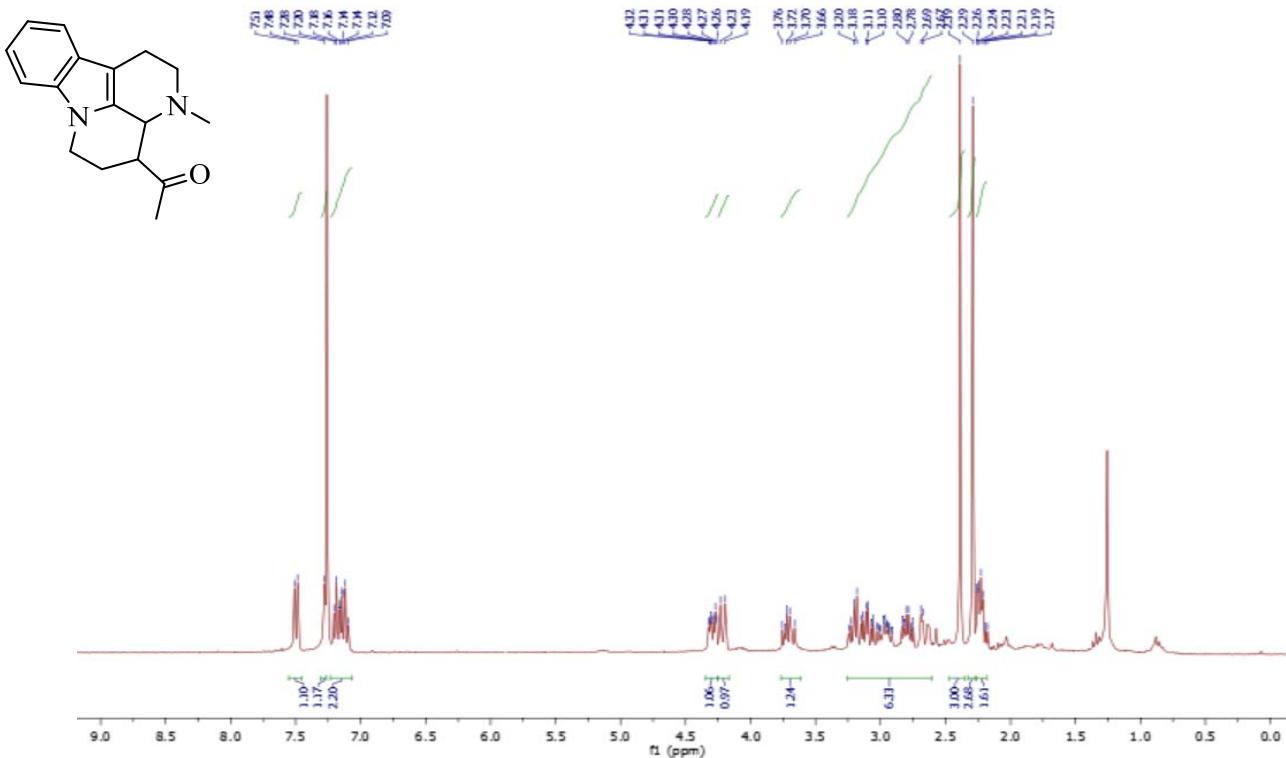


¹³C RMN spectrum

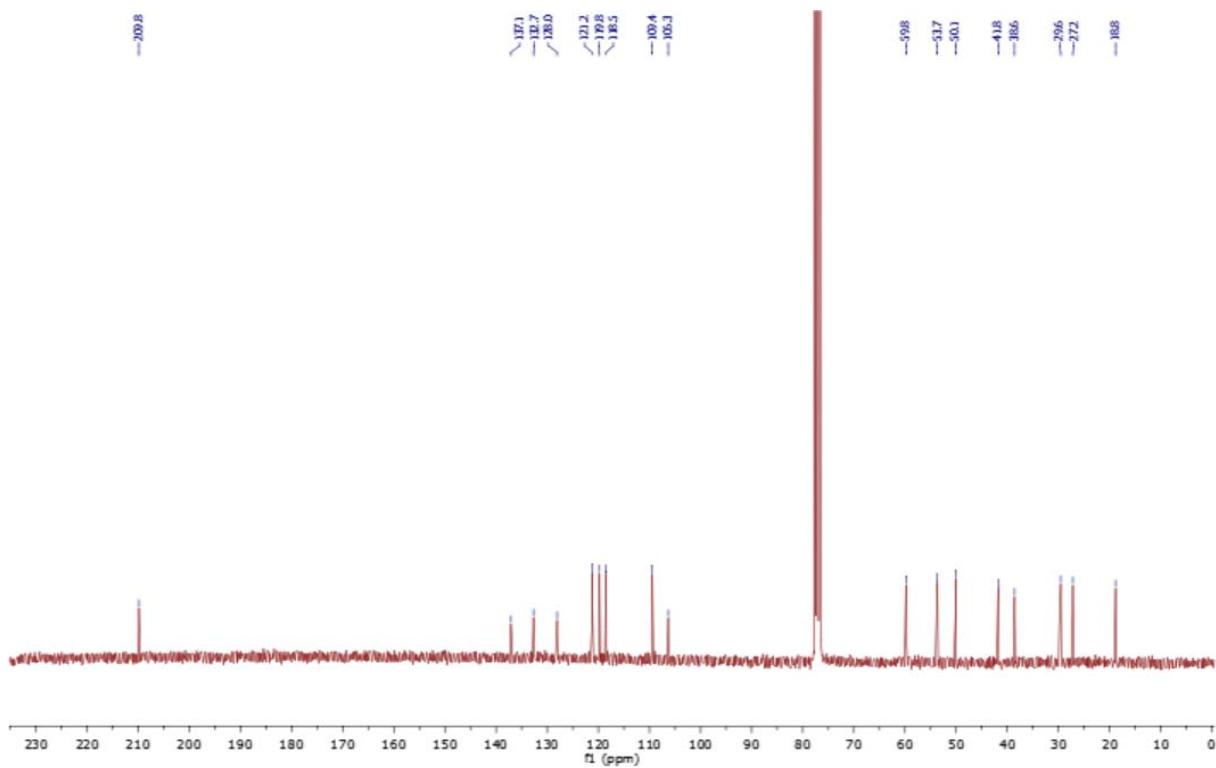


1-(3-Methyl-2,3,3a,4,5,6-hexahydro-1*H*-indolo[3,2,1-*de*][1,5]naphthyridin-4-yl)ethan-1-one (3-34)

¹H RMN spectrum

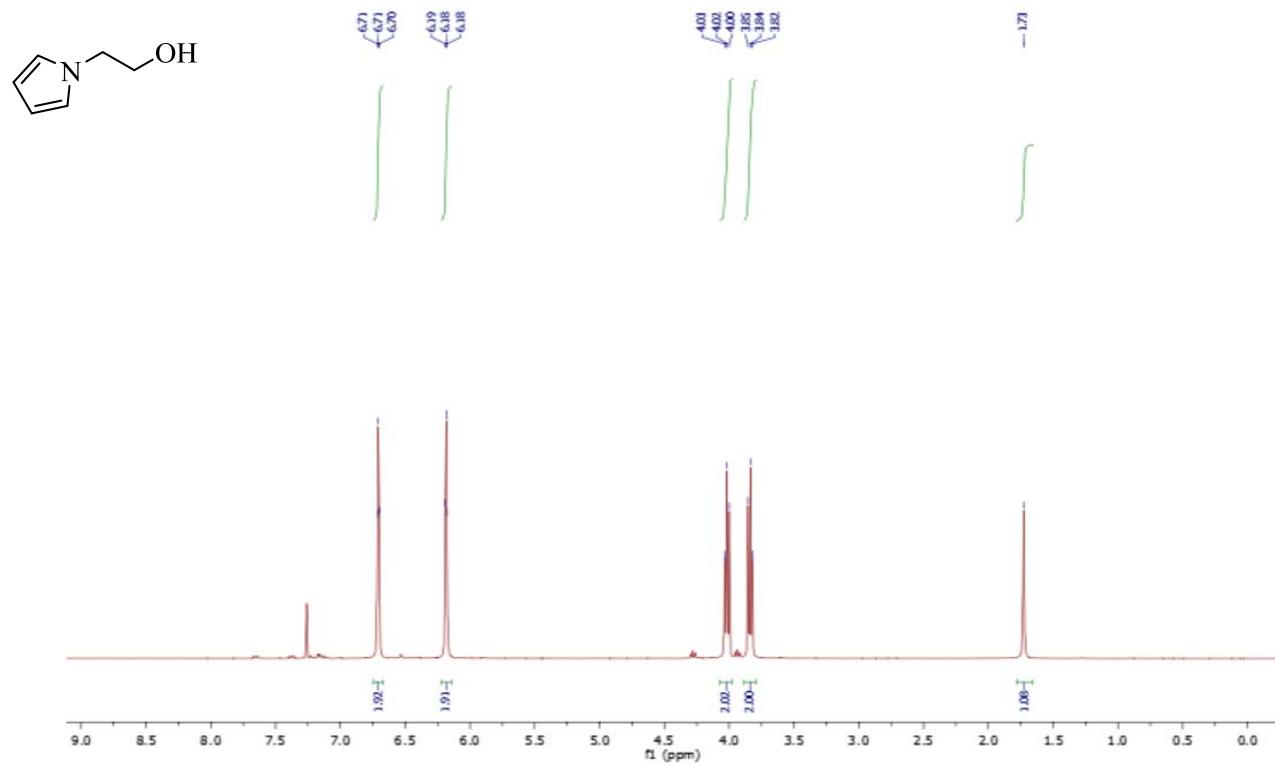


¹³C RMN spectrum

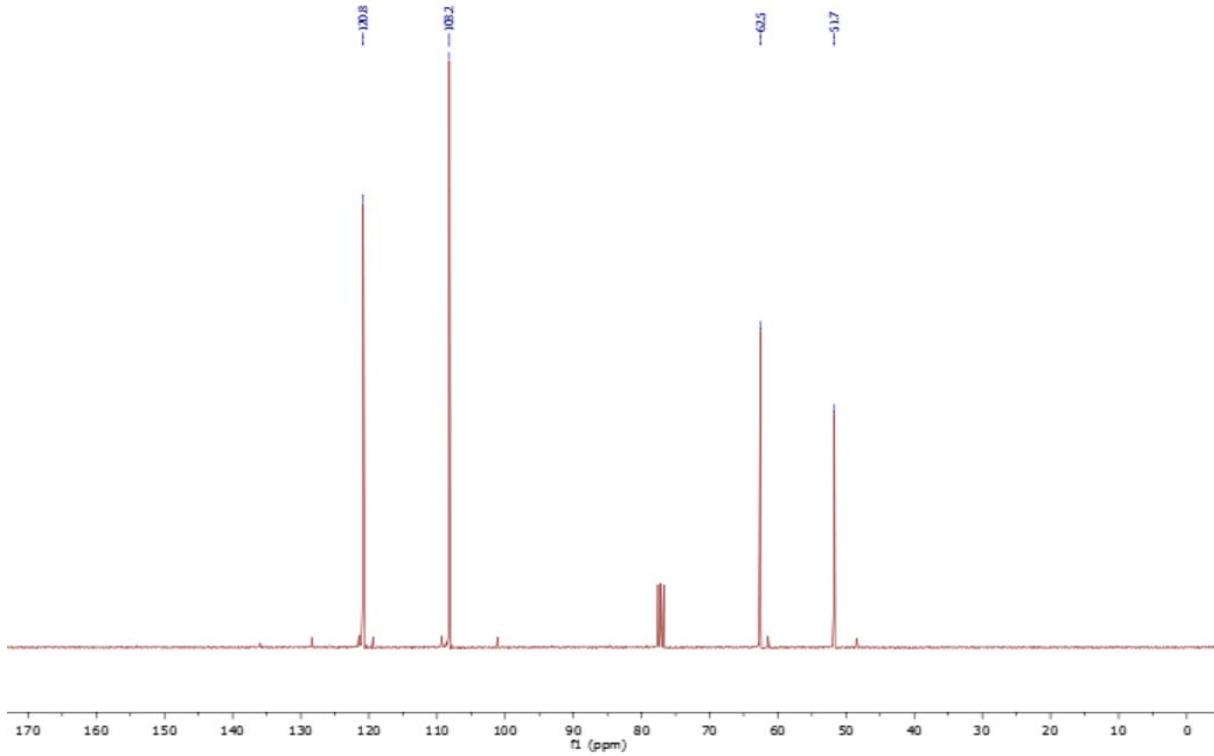


2-(1*H*-Pyrrol-1-yl)ethan-1-ol (3-36)

^1H RMN spectrum

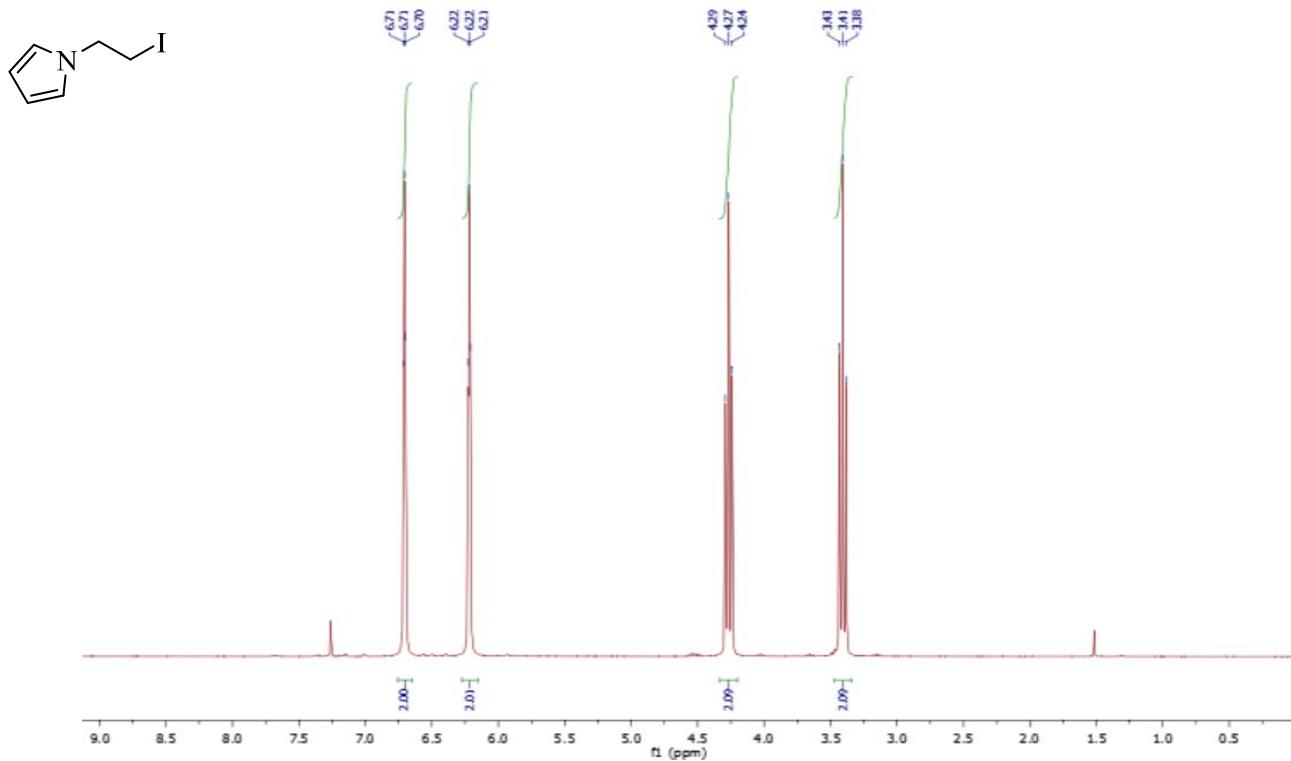


^{13}C RMN spectrum

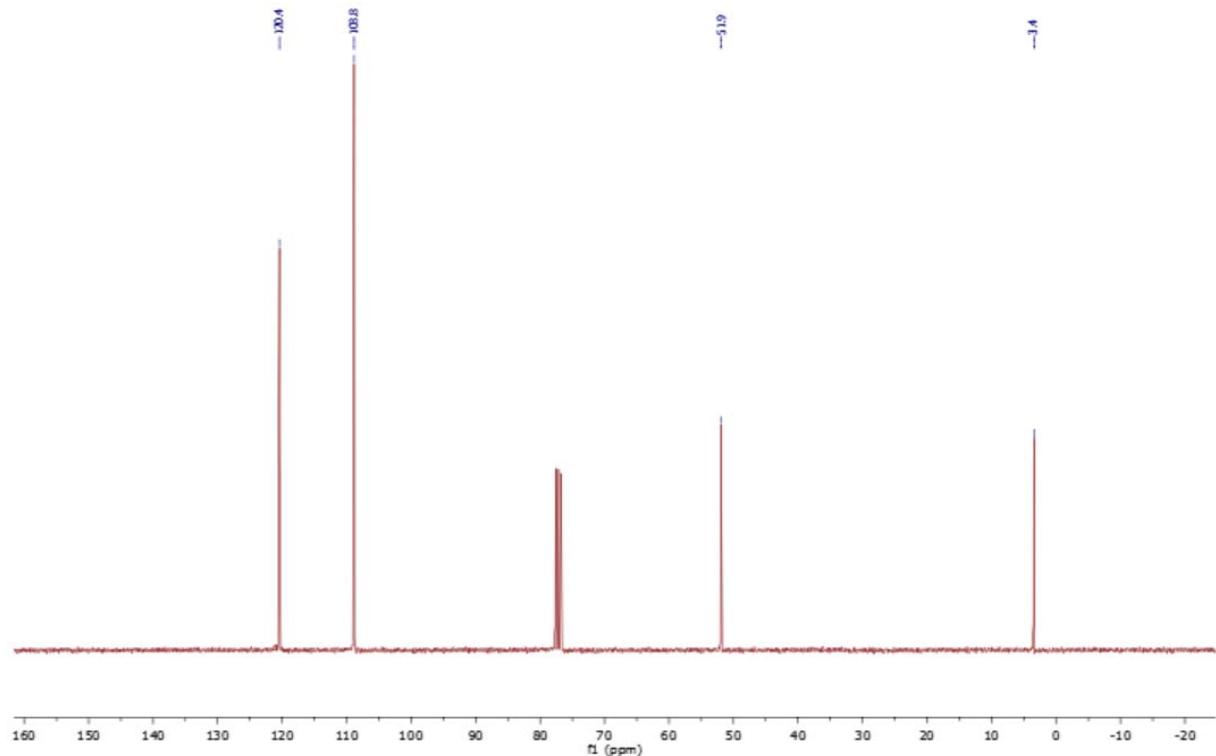


1-(2-Iodoethyl)-1*H*-pyrrole (3-37)

^1H RMN spectrum

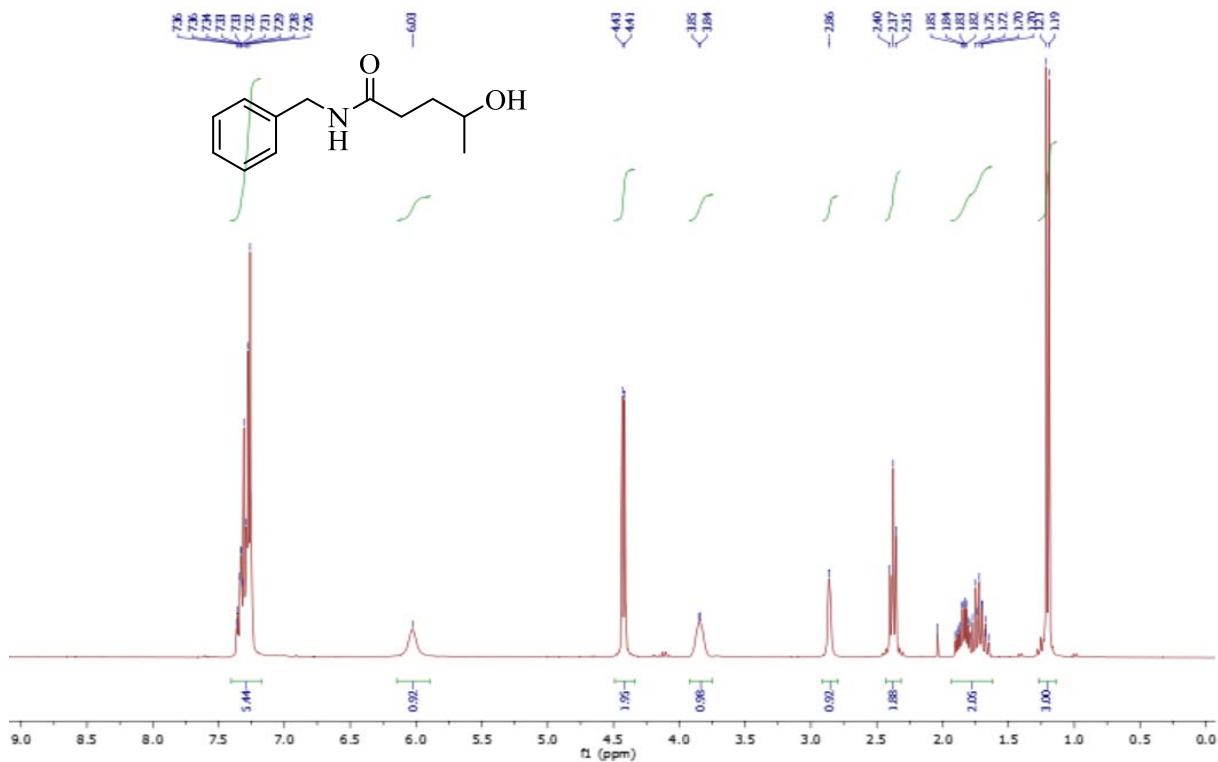


^{13}C RMN spectrum

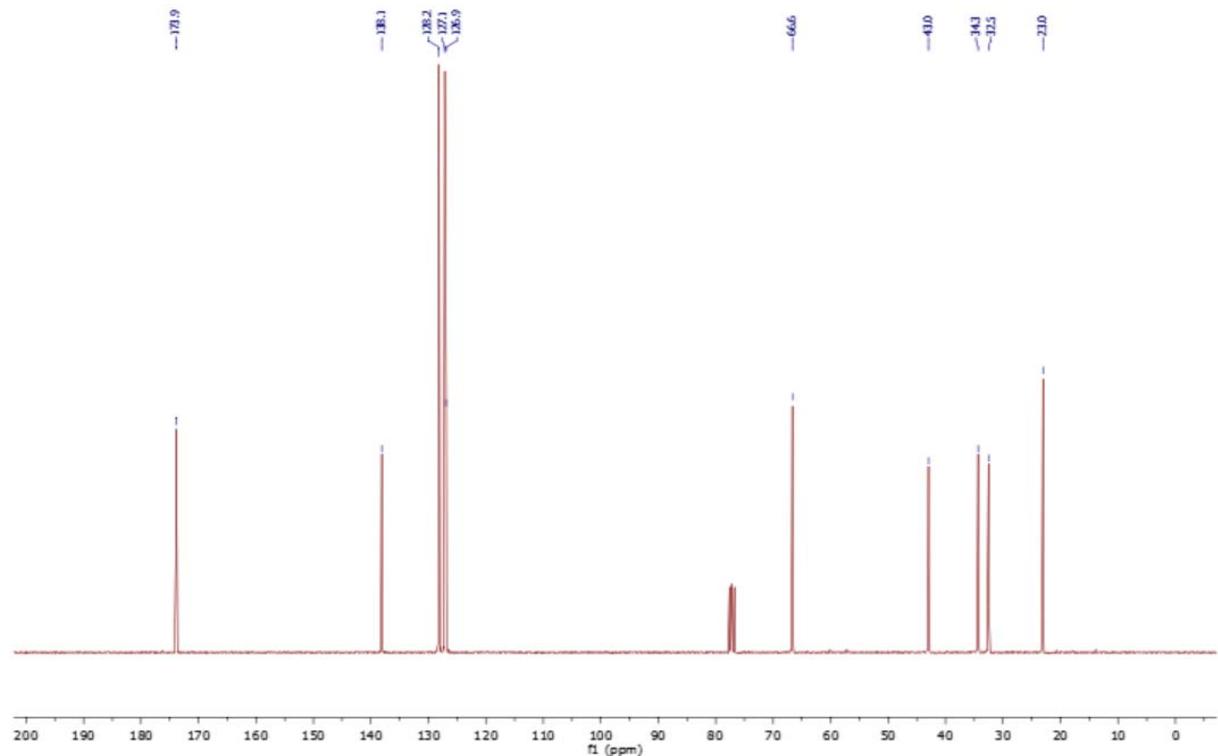


N-Benzyl-4-hydroxypentanamide (3-39)

^1H RMN spectrum

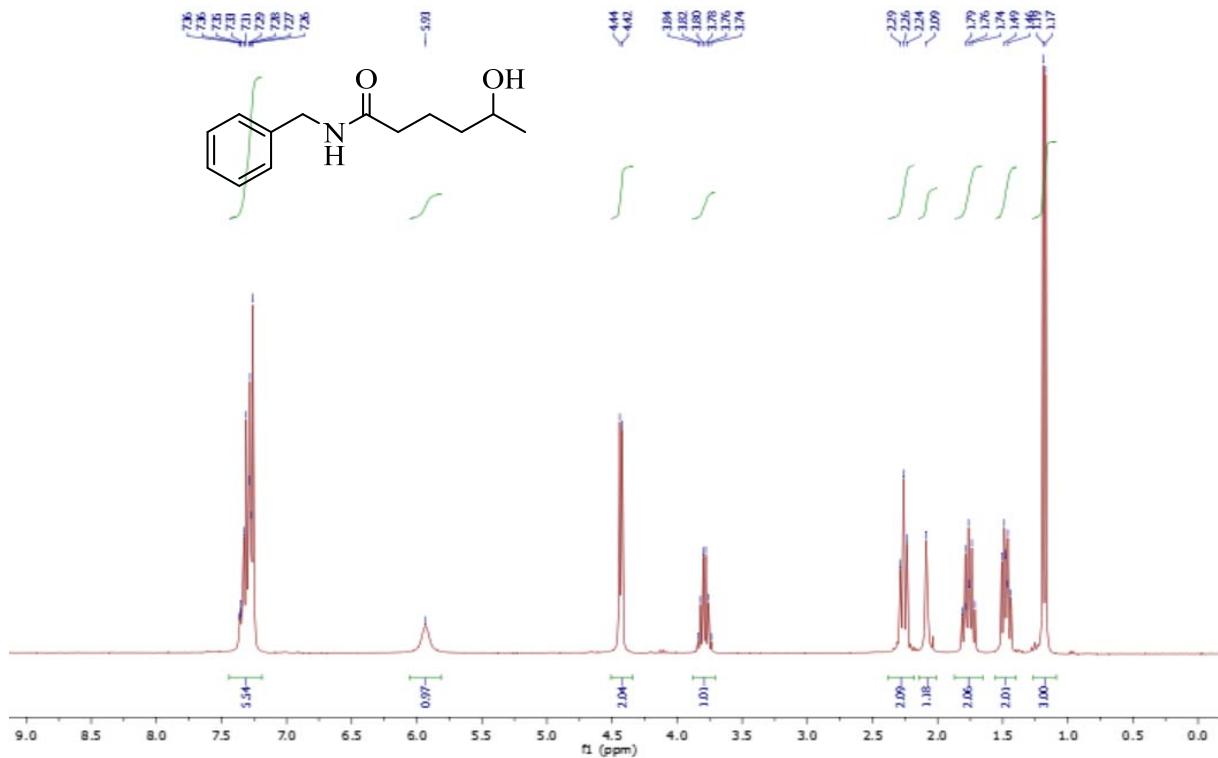


^{13}C RMN spectrum

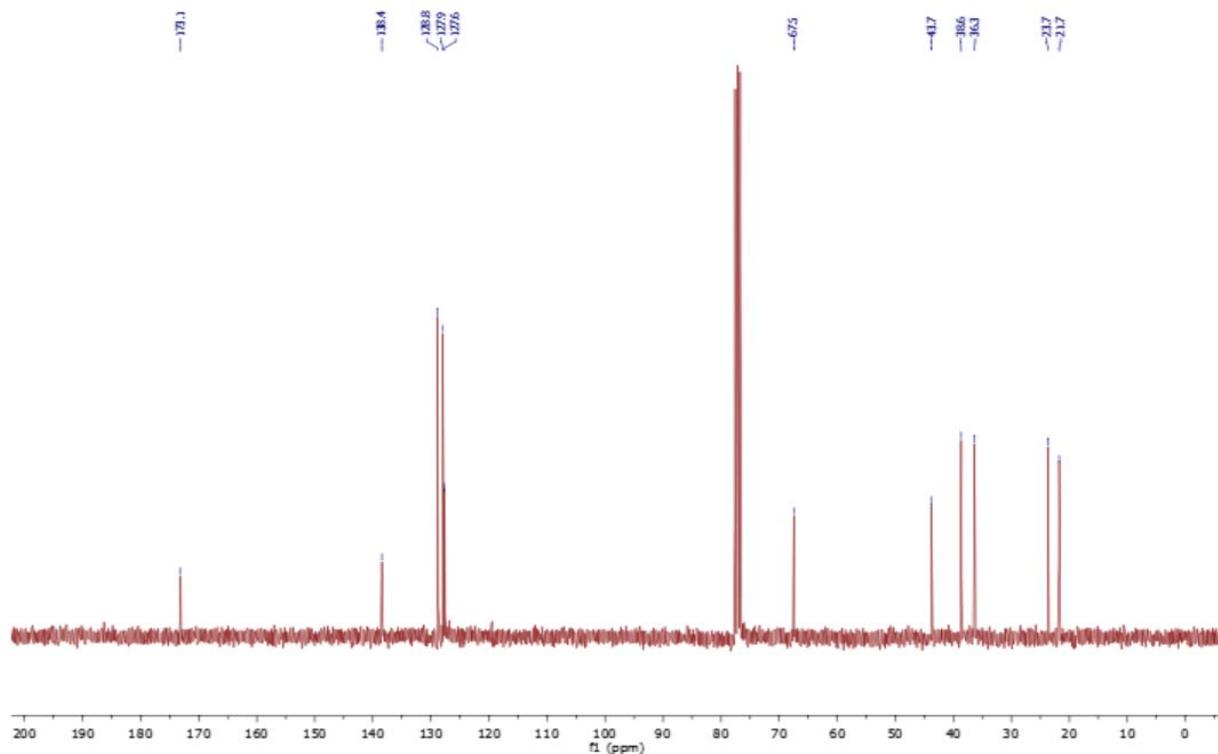


N-Benzyl-5-hydroxyhexanamide (3-40)

^1H RMN spectrum

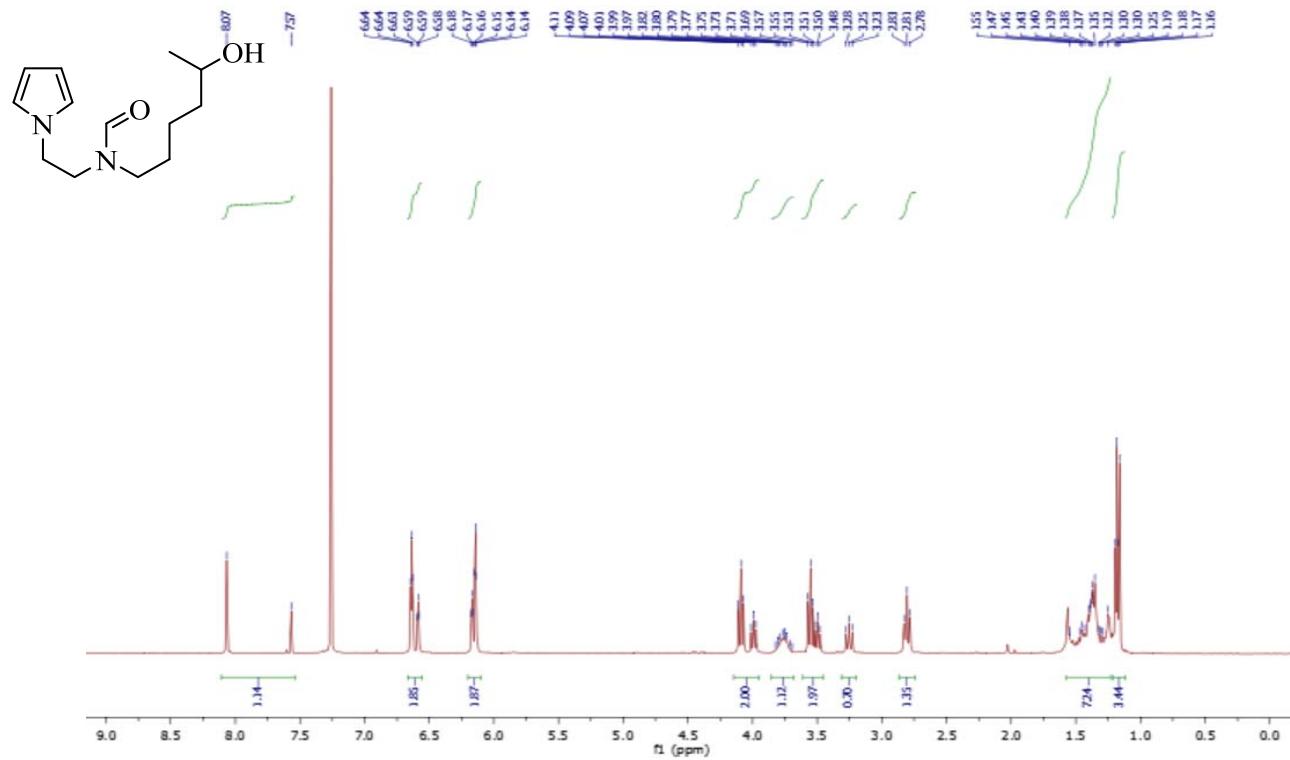


^{13}C RMN spectrum

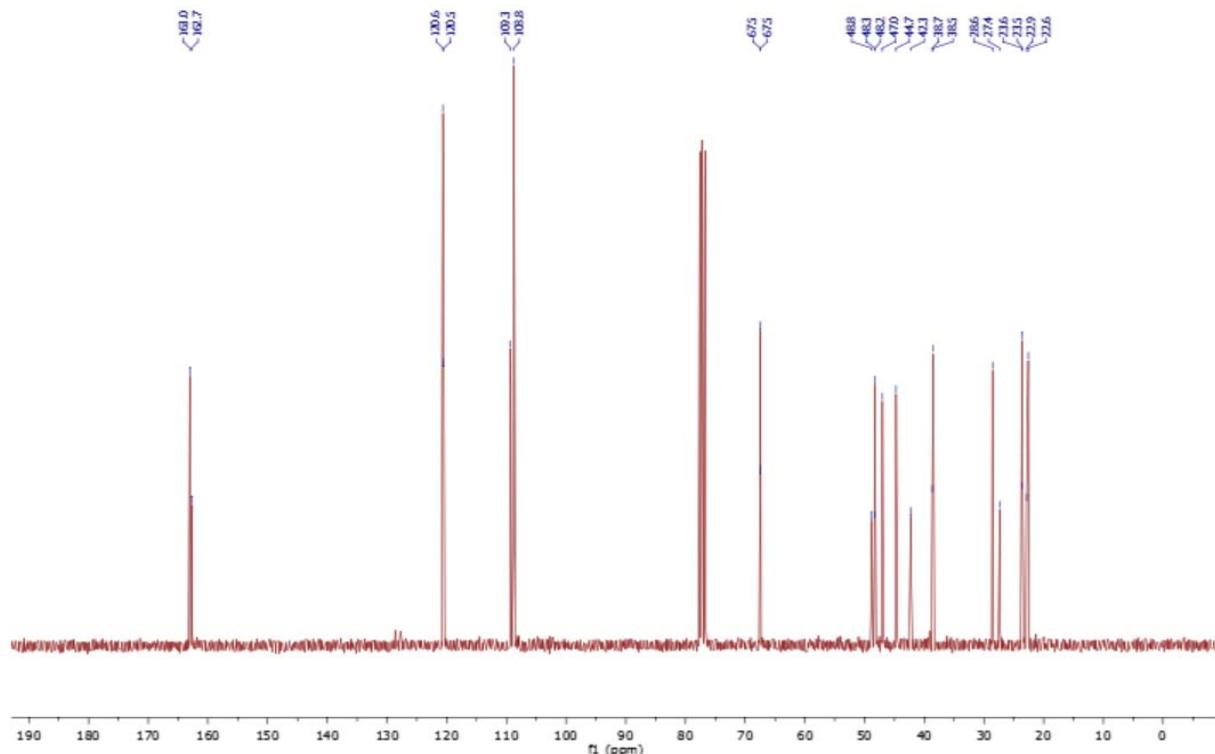


N-(2-(1*H*-Pyrrol-1-yl)ethyl)-N-(5-hydroxyhexyl)formamide (3-44)

¹H RMN spectrum

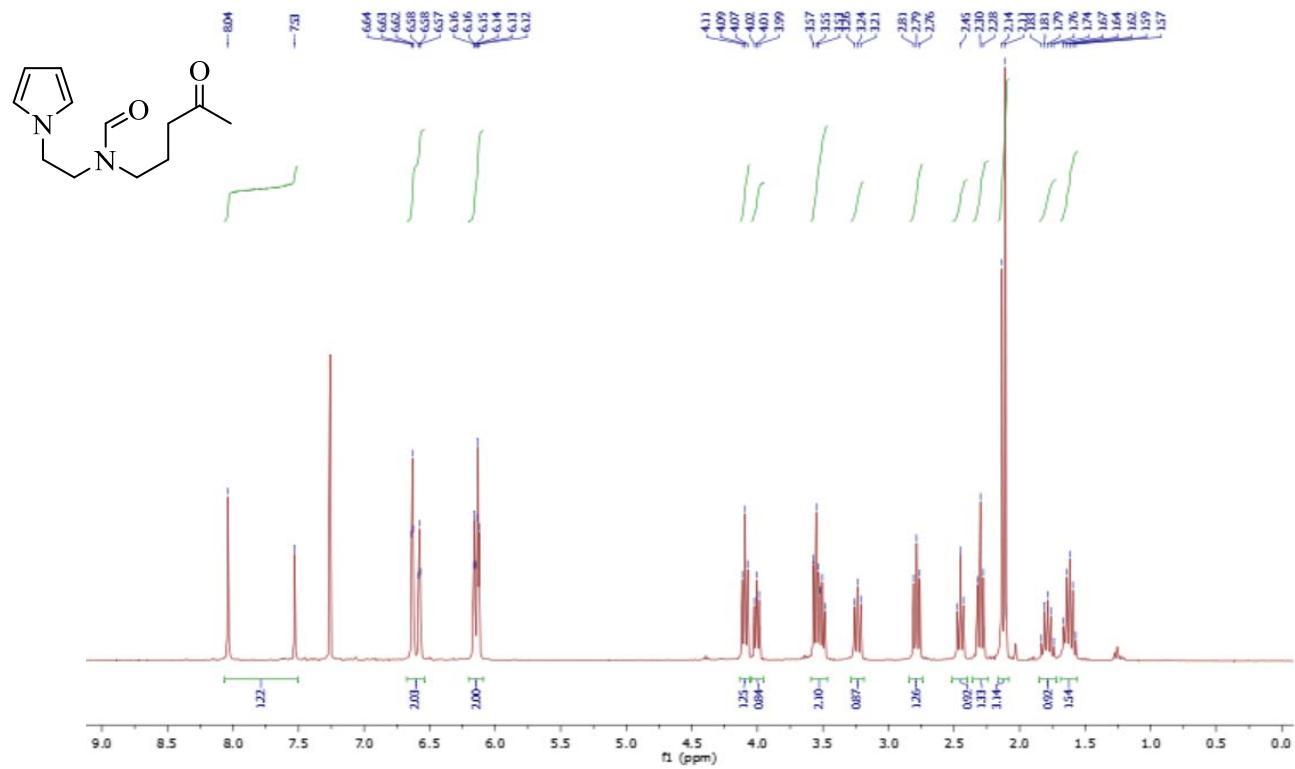


¹³C RMN spectrum

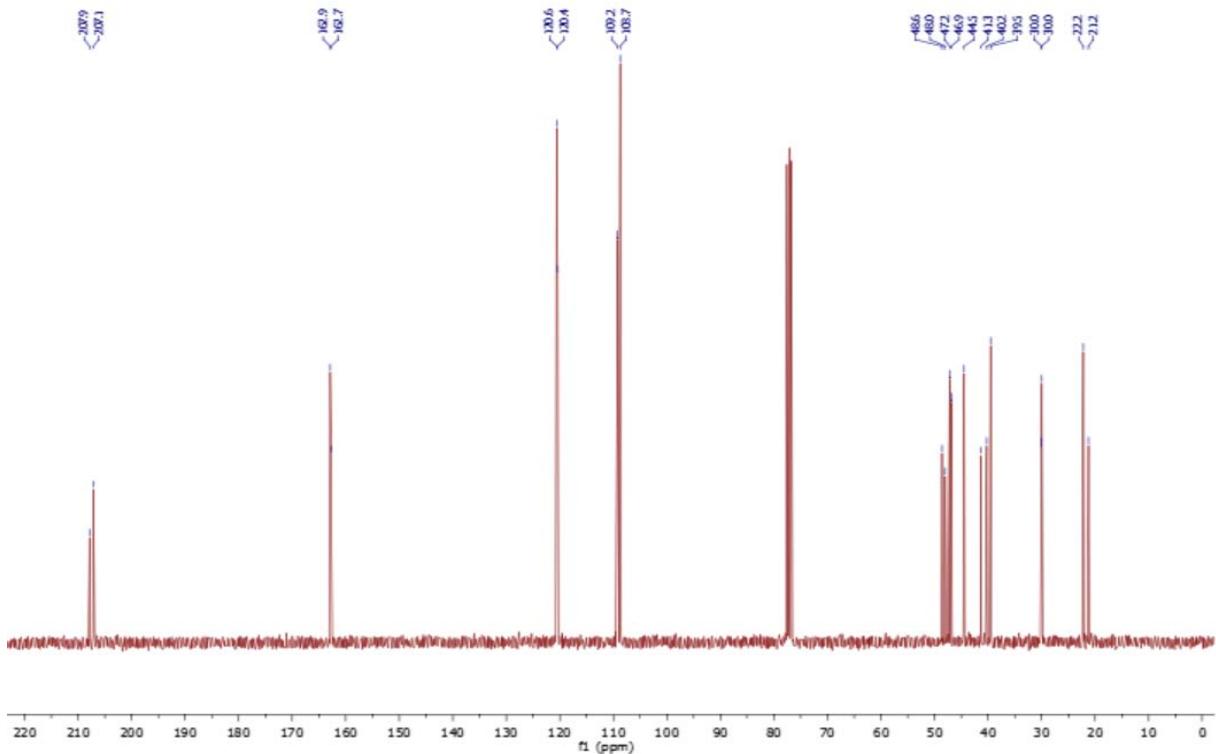


N-(2-(1*H*-Pyrrol-1-yl)ethyl)-N-(4-oxopentyl)formamide (3-45)

¹H RMN spectrum

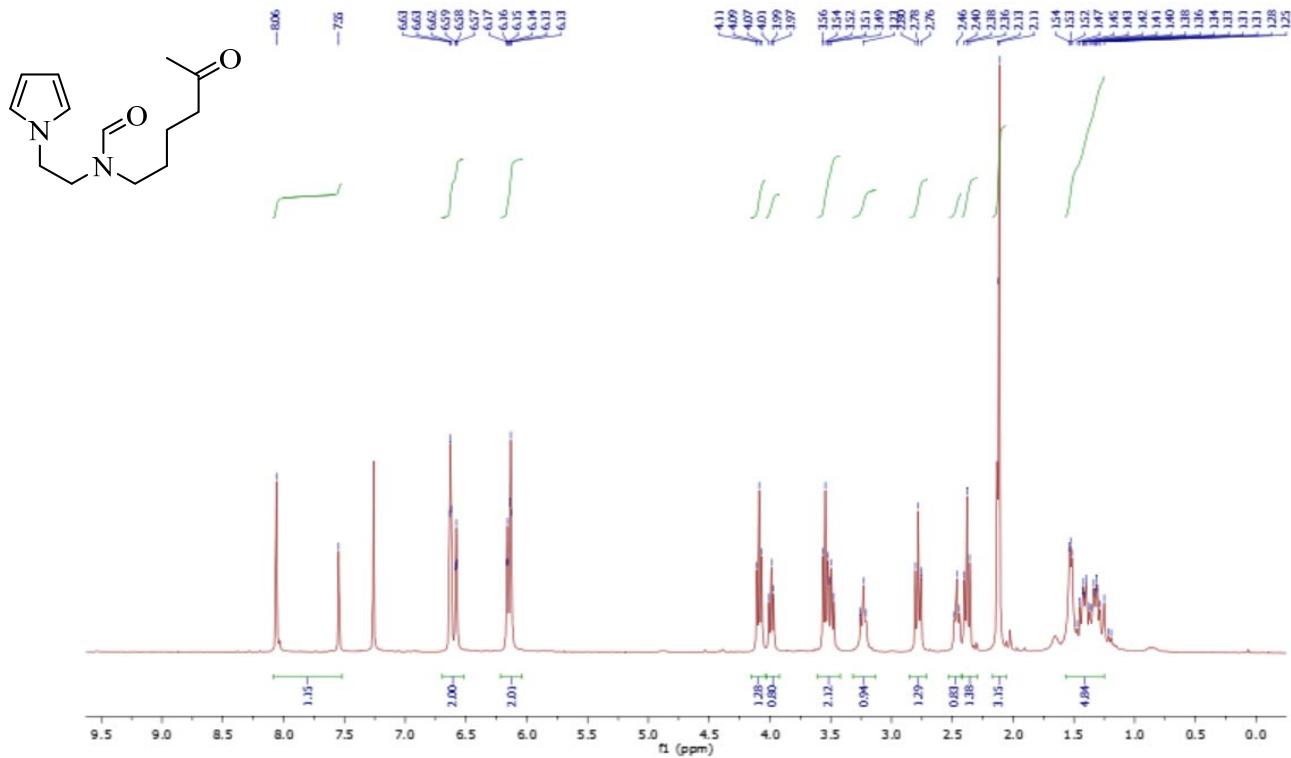


¹³C RMN spectrum

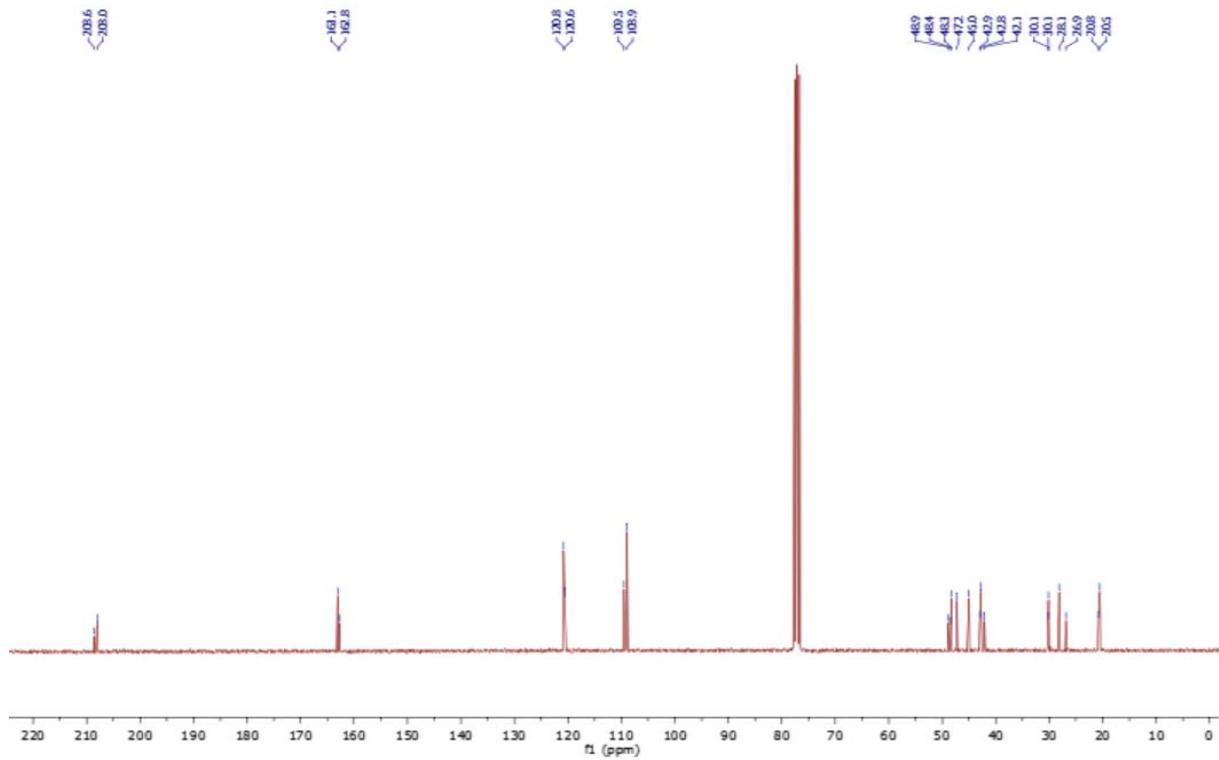


N-(2-(1*H*-Pyrrol-1-yl)ethyl)-N-(5-oxohexyl)formamide (3-46)

¹H RMN spectrum

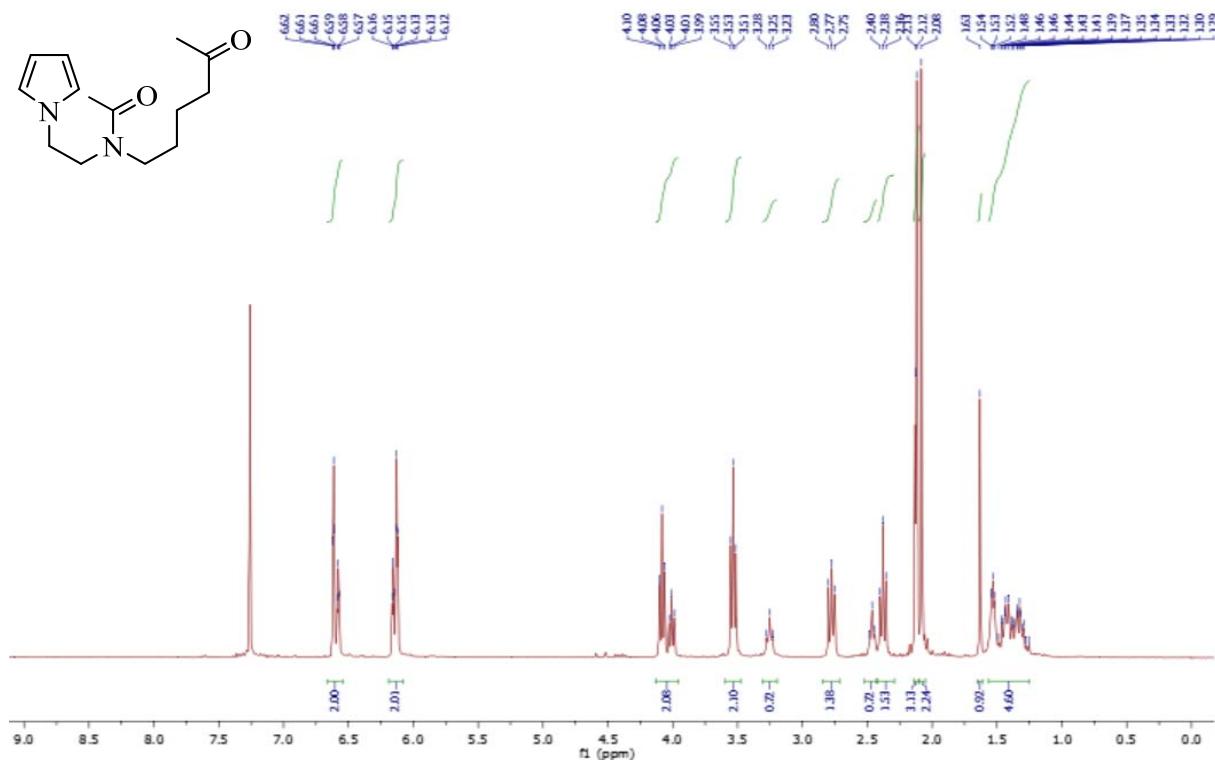


¹³C RMN spectrum

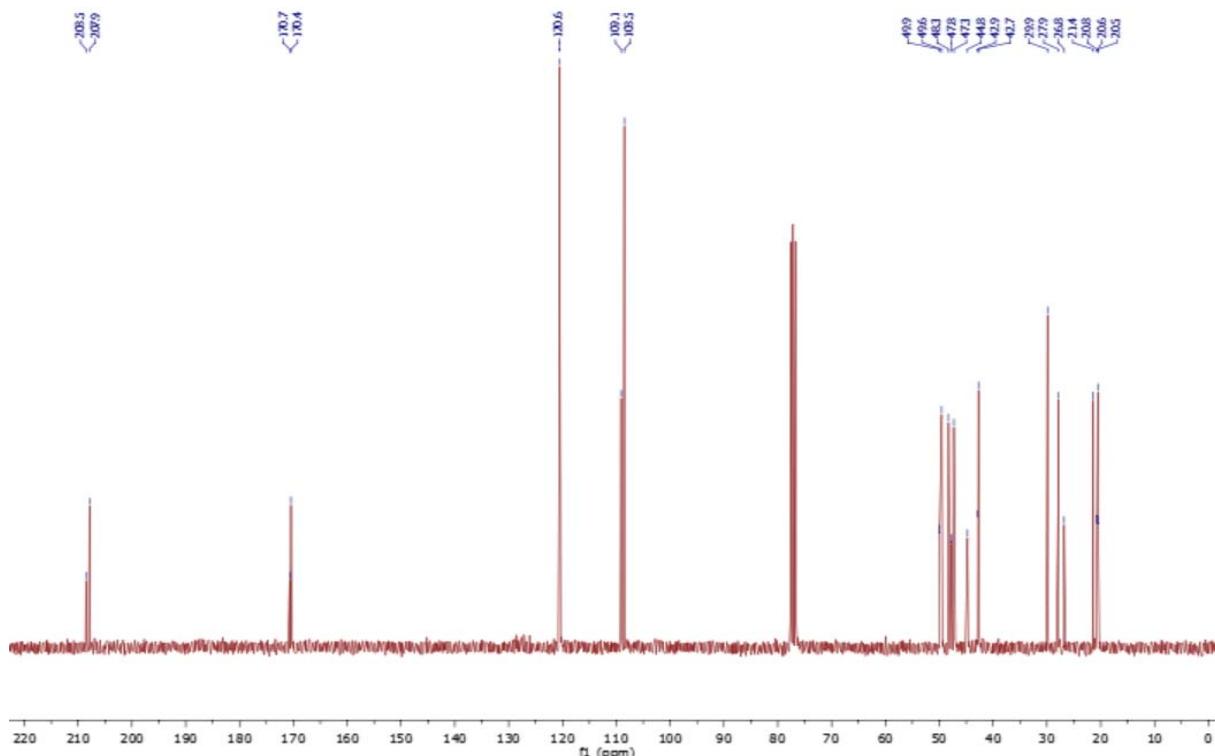


N-(2-(1*H*-Pyrrol-1-yl)ethyl)-N-(5-oxohexyl)acetamide (3-47)

¹H RMN spectrum

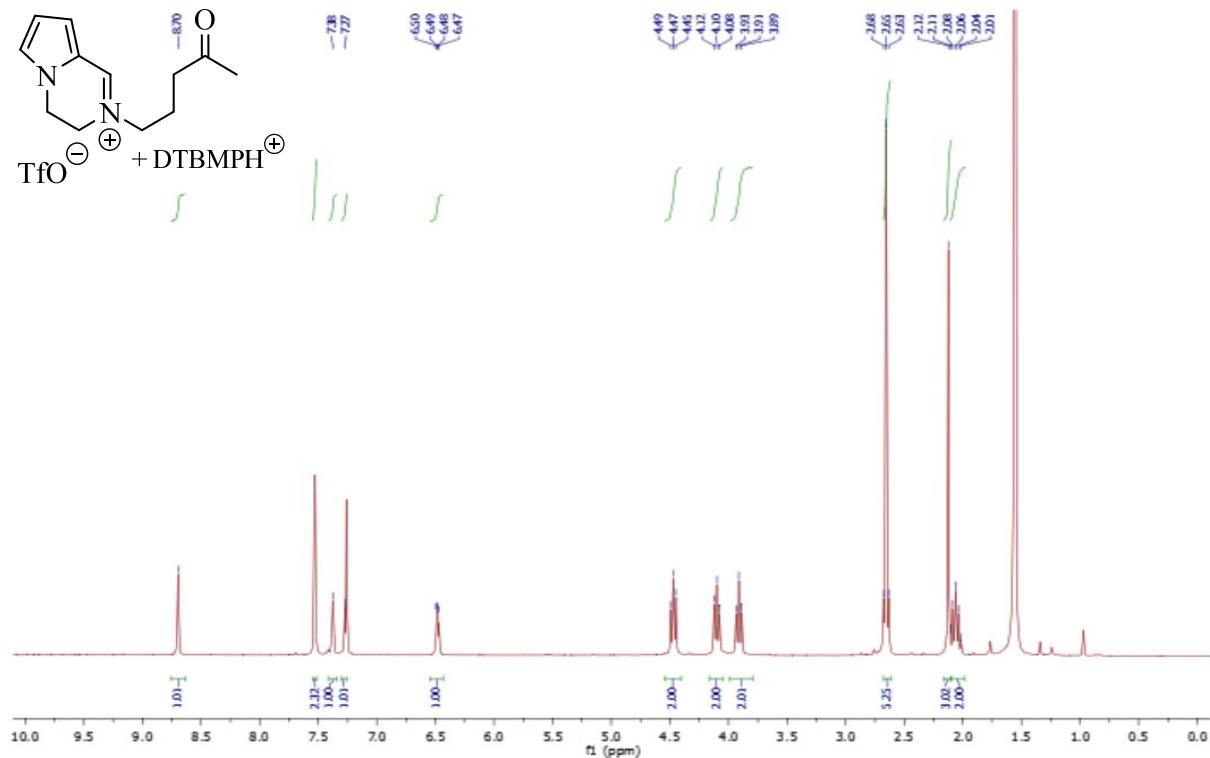


¹³C RMN spectrum



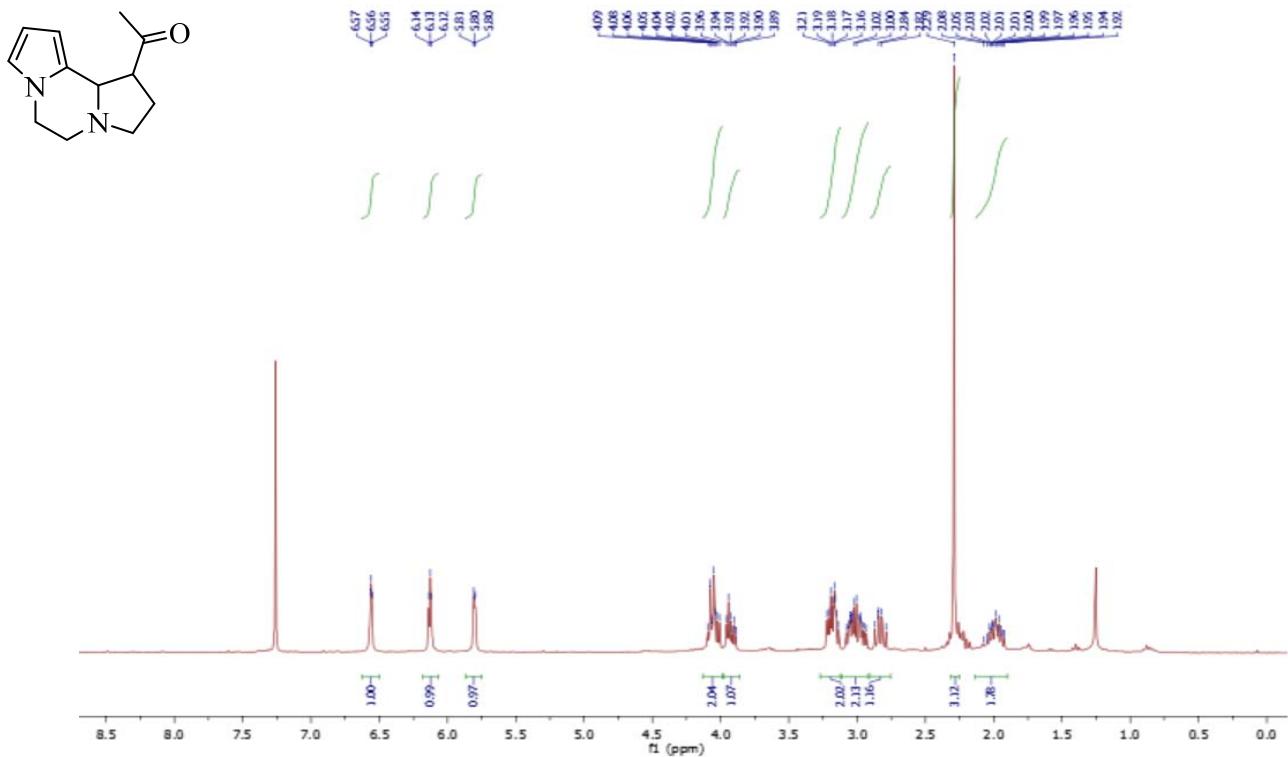
Iminium ion (3-48)

^1H RMN spectrum

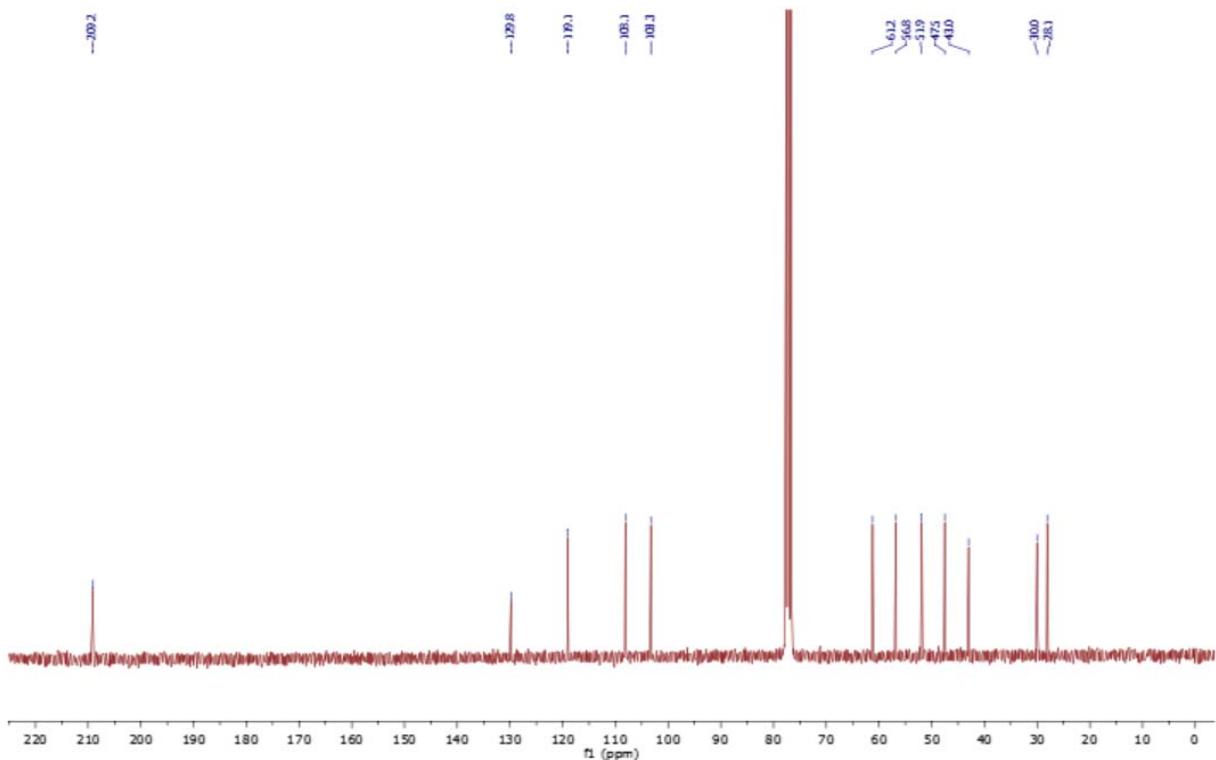


1-(1,2,3,5,6,10b-hexahydrodipyrrolo[1,2-*a*:2',1'-*c*]pyrazin-1-yl)ethan-1-one (3-49)

^1H RMN spectrum

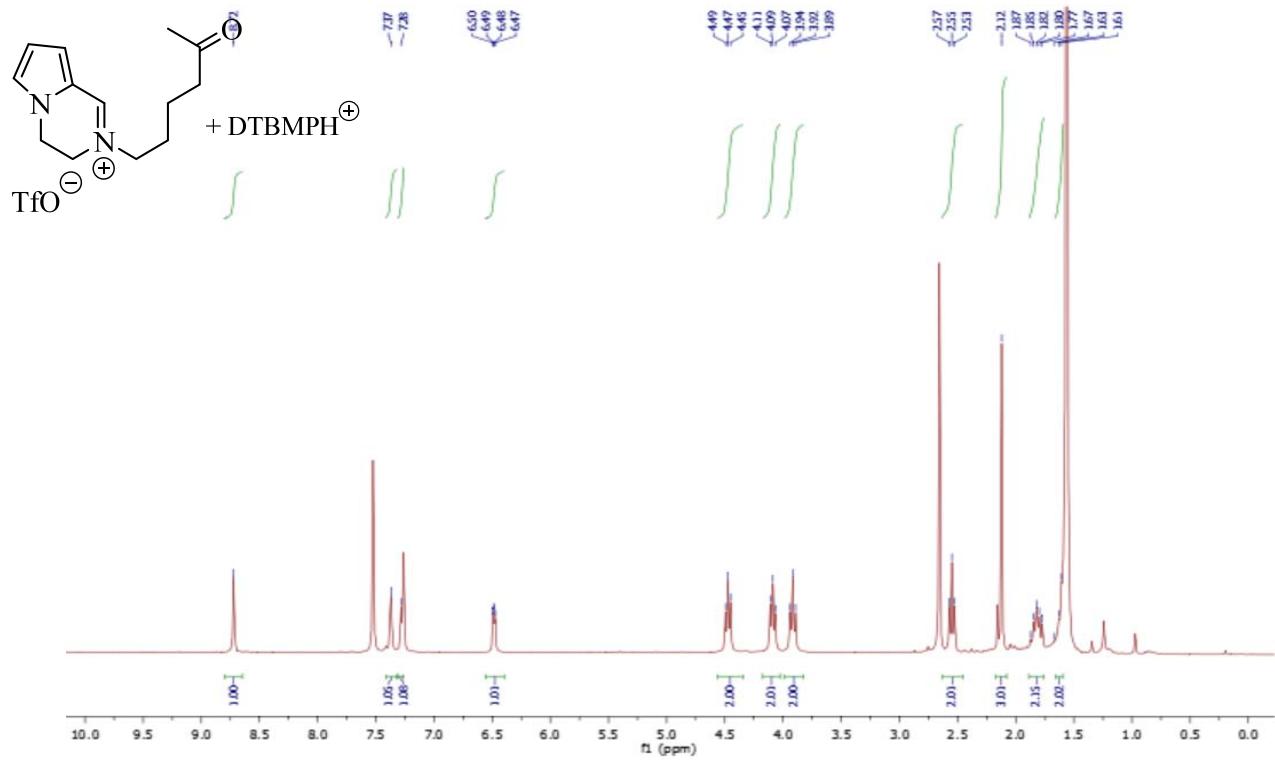


^{13}C RMN spectrum



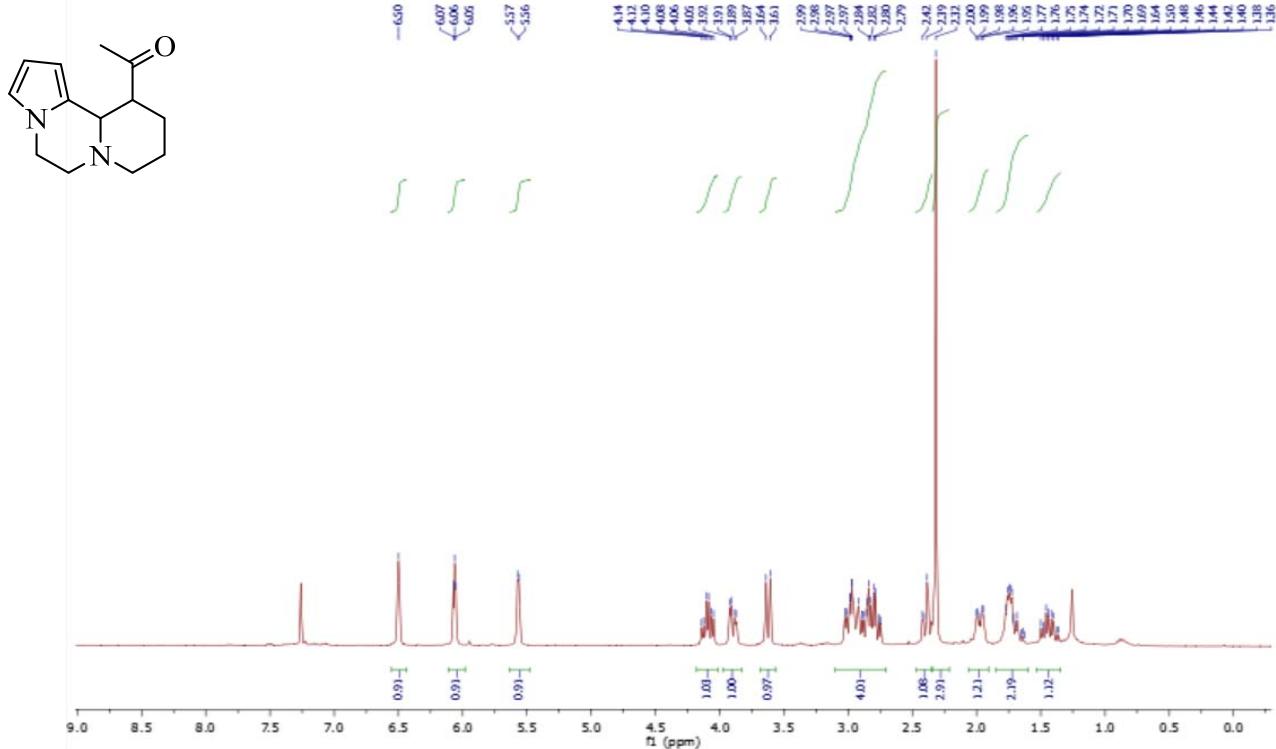
Iminium ion (3-50)

^1H RMN spectrum

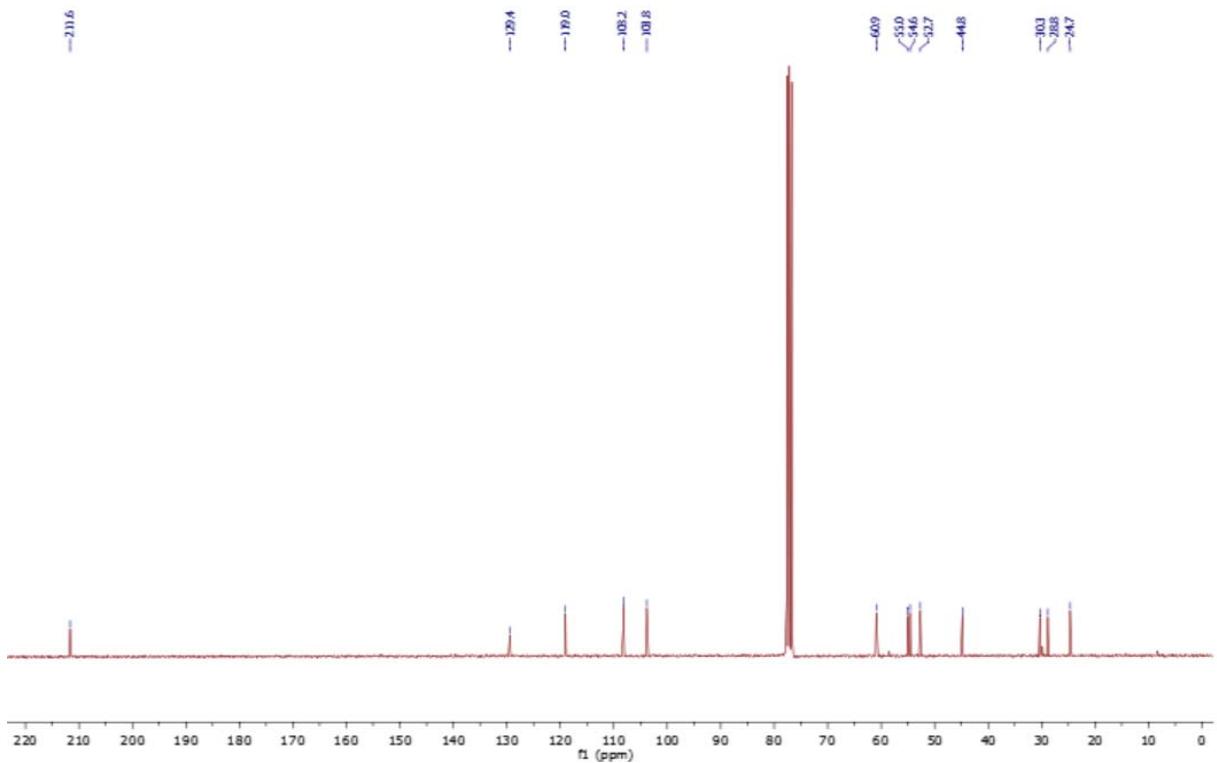


**1-(5,6,9,10,11,11a-Hexahydro-8H-pyrrido[1,2-a]pyrrolo[2,1-c]pyrazin-11-yl)ethan-1-one
(3-51)**

^1H RMN spectrum

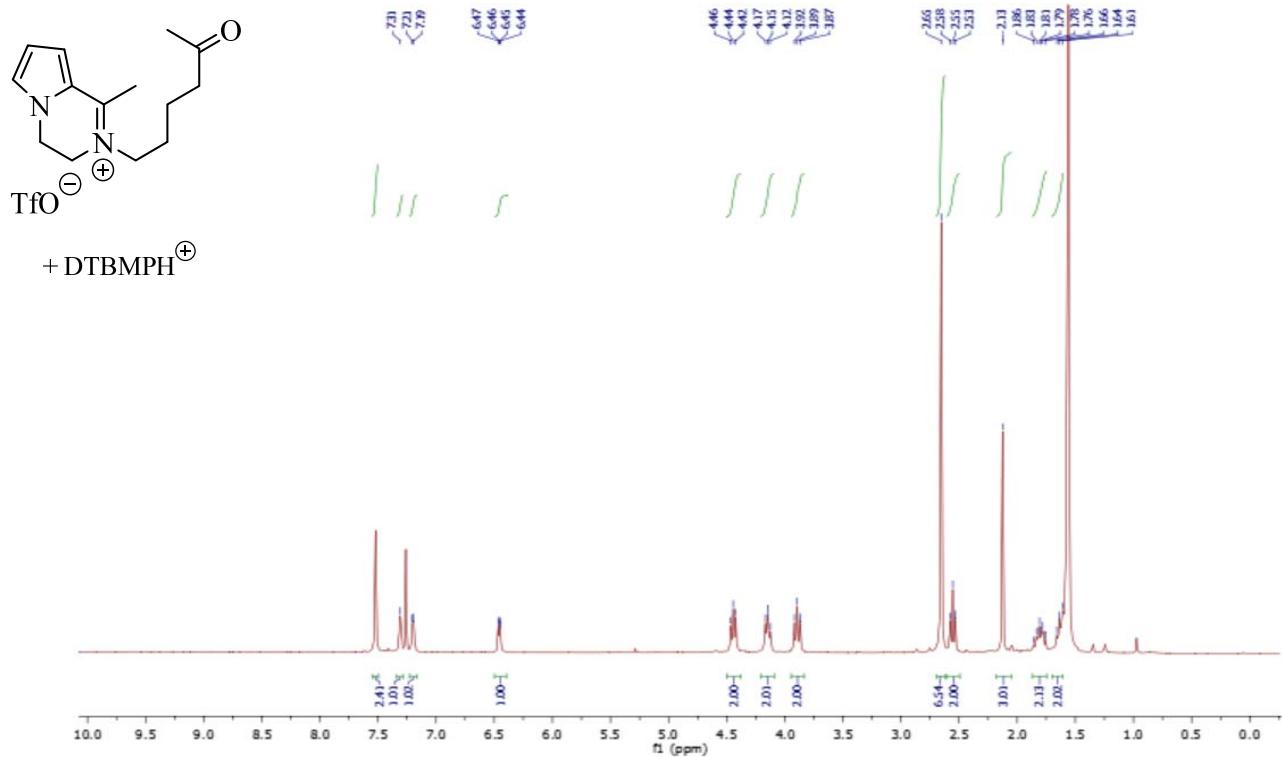


^{13}C RMN spectrum



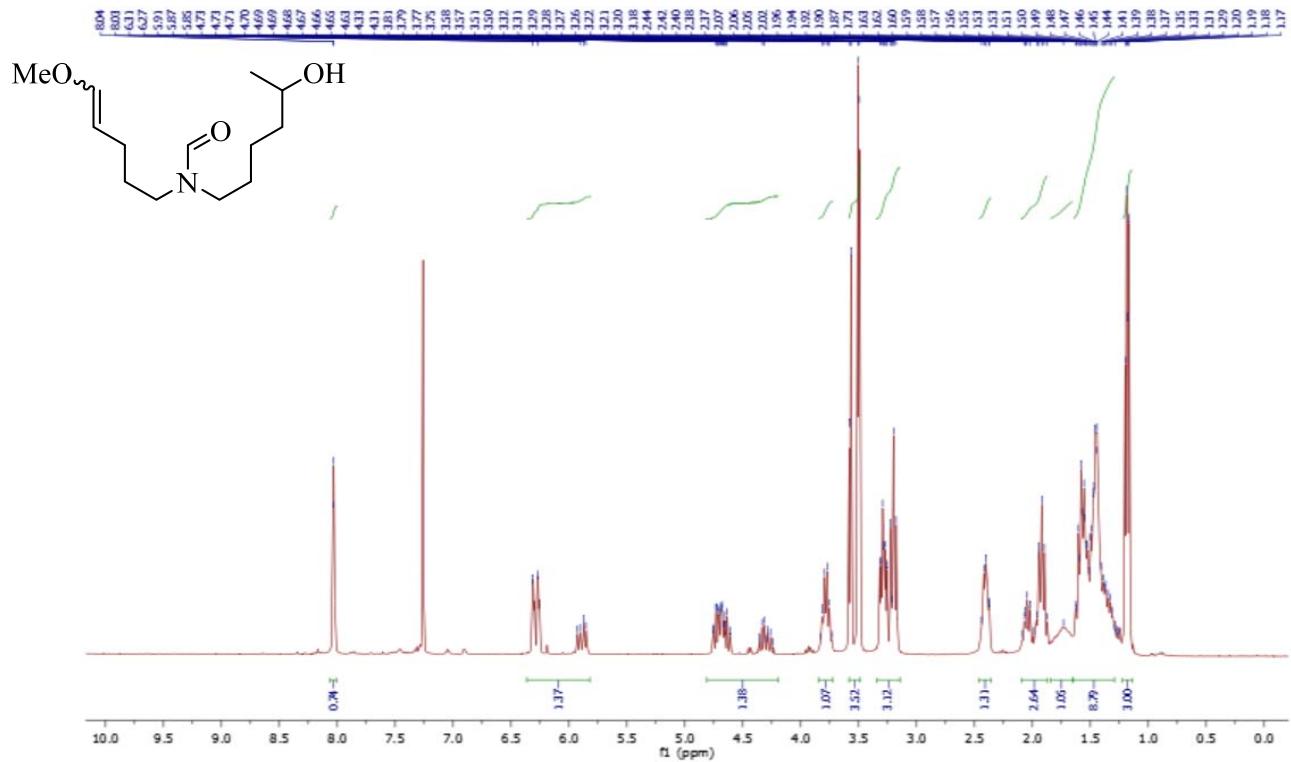
Iminium ion (3-52)

^1H RMN spectrum

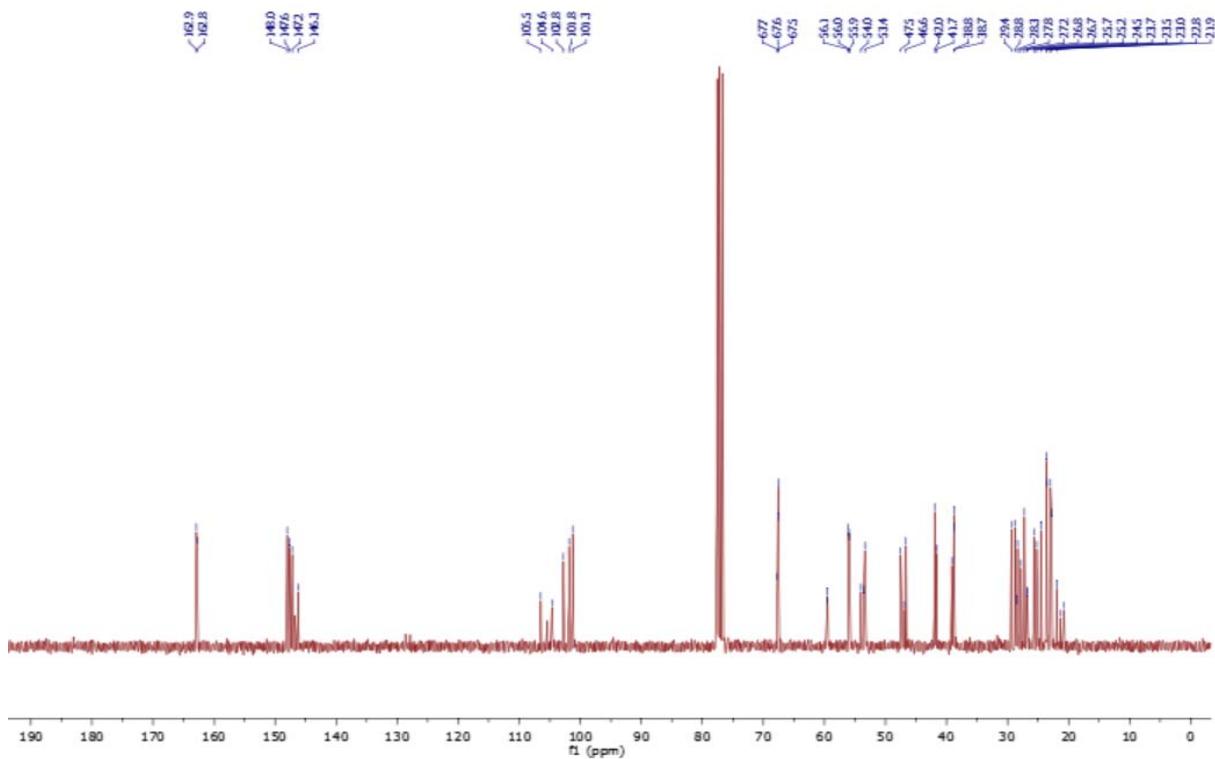


N-(5-Hydroxyhexyl)-N-(5-methoxypent-4-en-1-yl)formamide (3-56)

¹H RMN spectrum

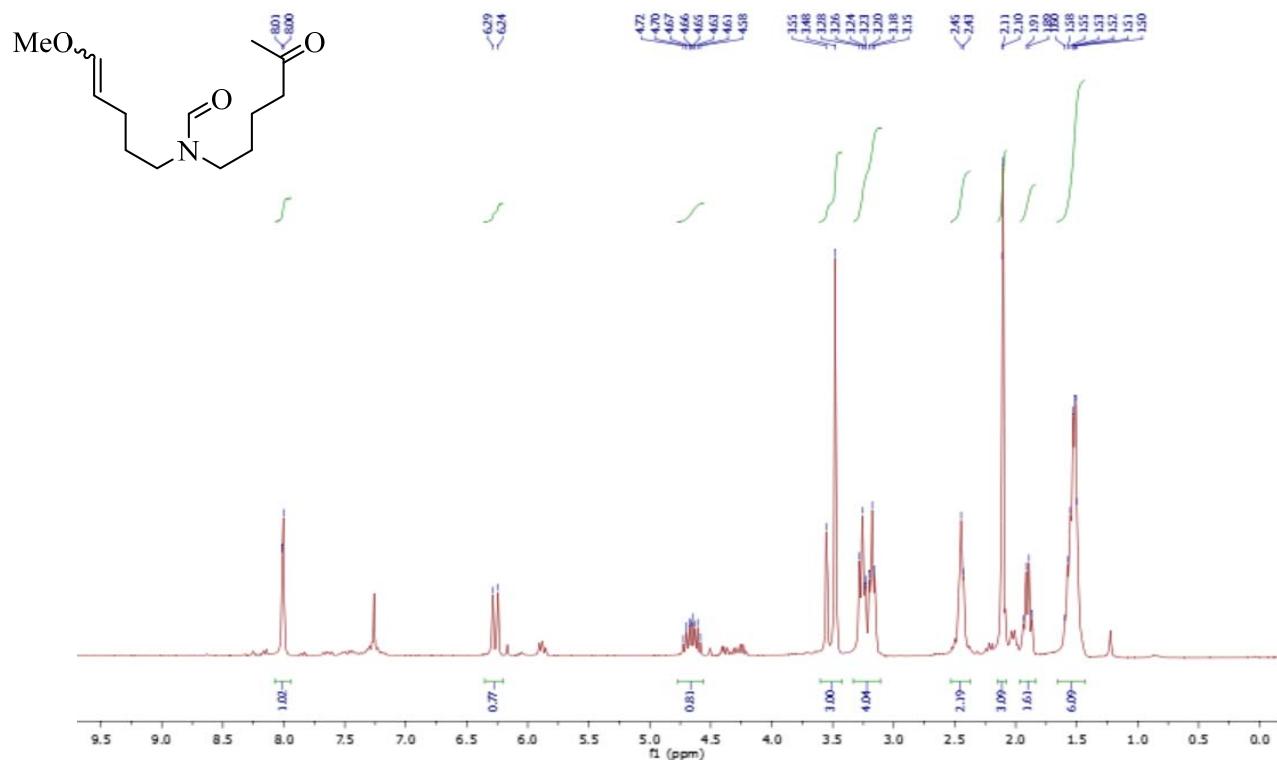


¹³C RMN spectrum

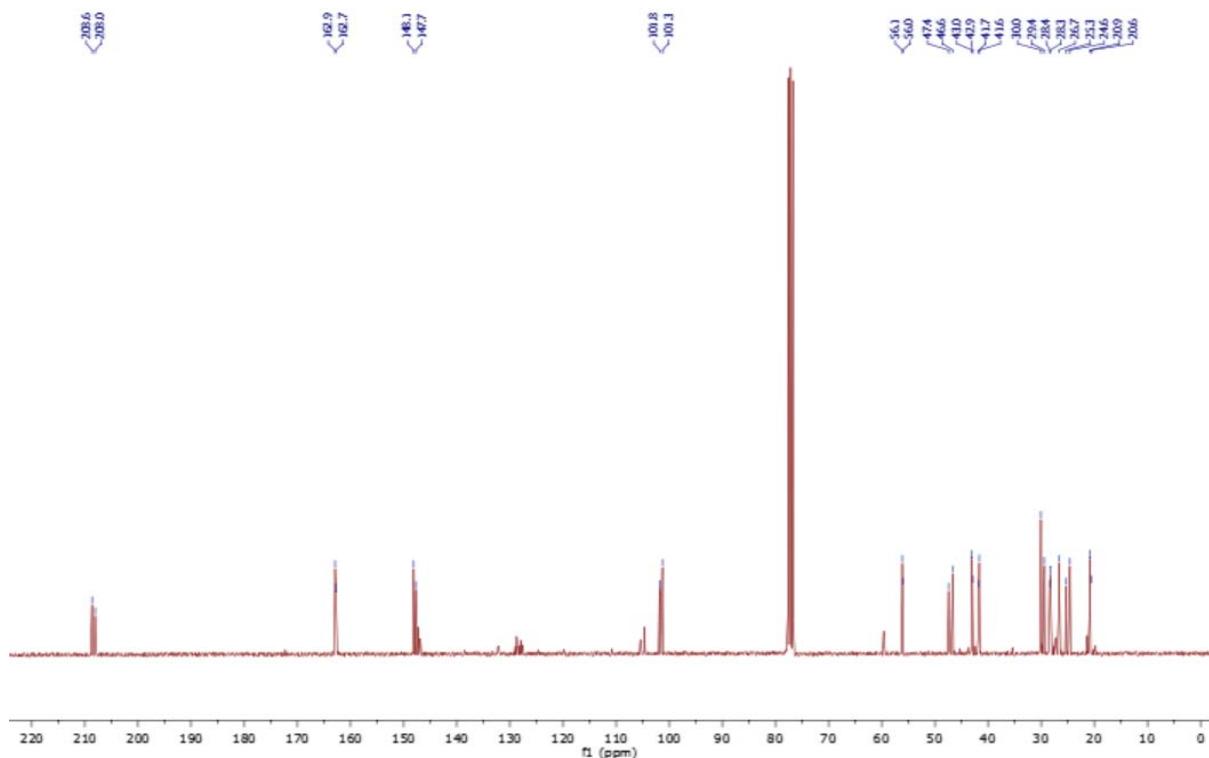


N-(5-Methoxypent-4-en-1-yl)-N-(5-oxohexyl)formamide (3-57)

¹H RMN spectrum

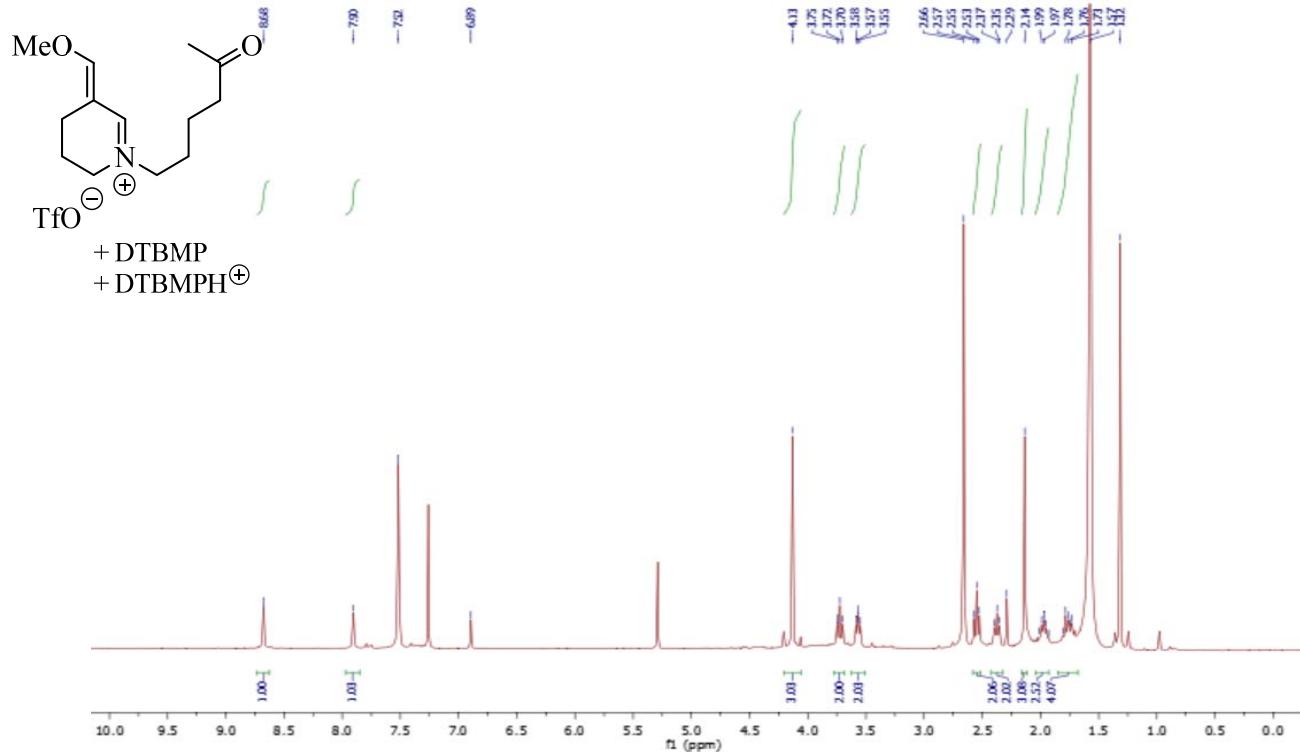


¹³C RMN spectrum



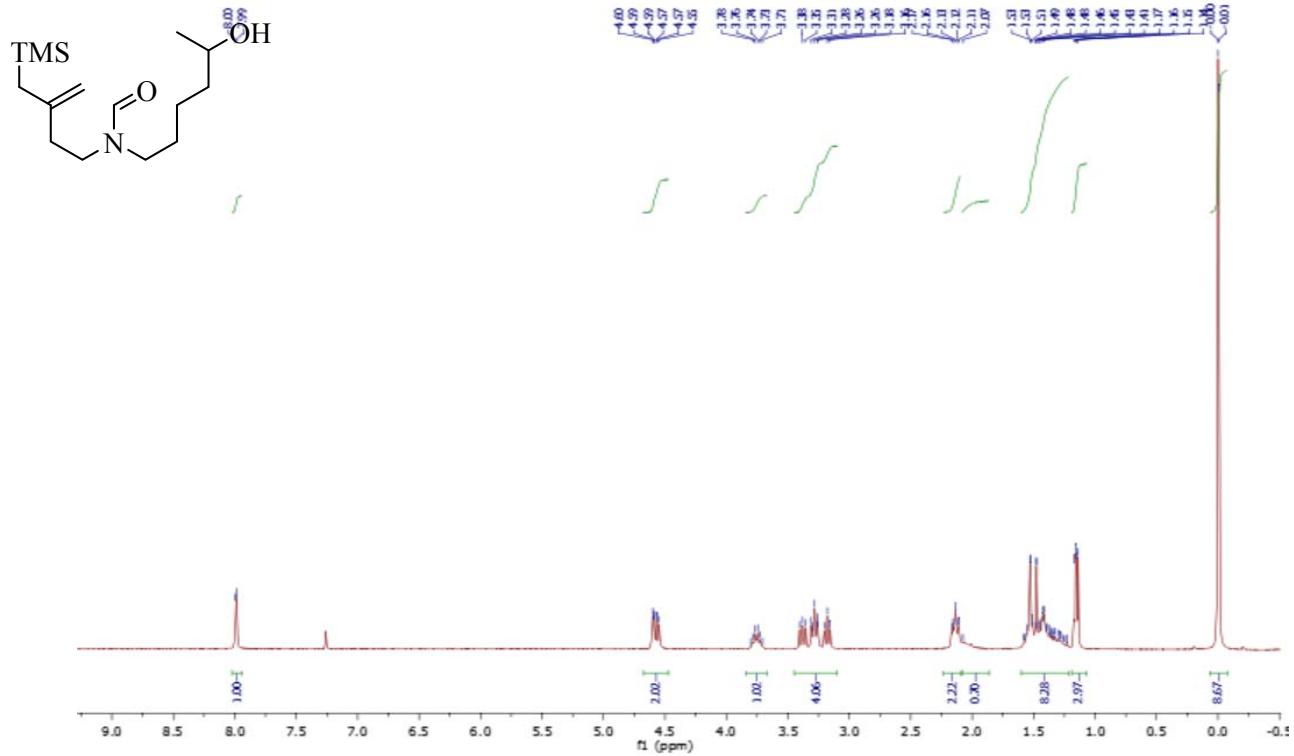
Iminium ion (3-58)

^1H RMN spectrum

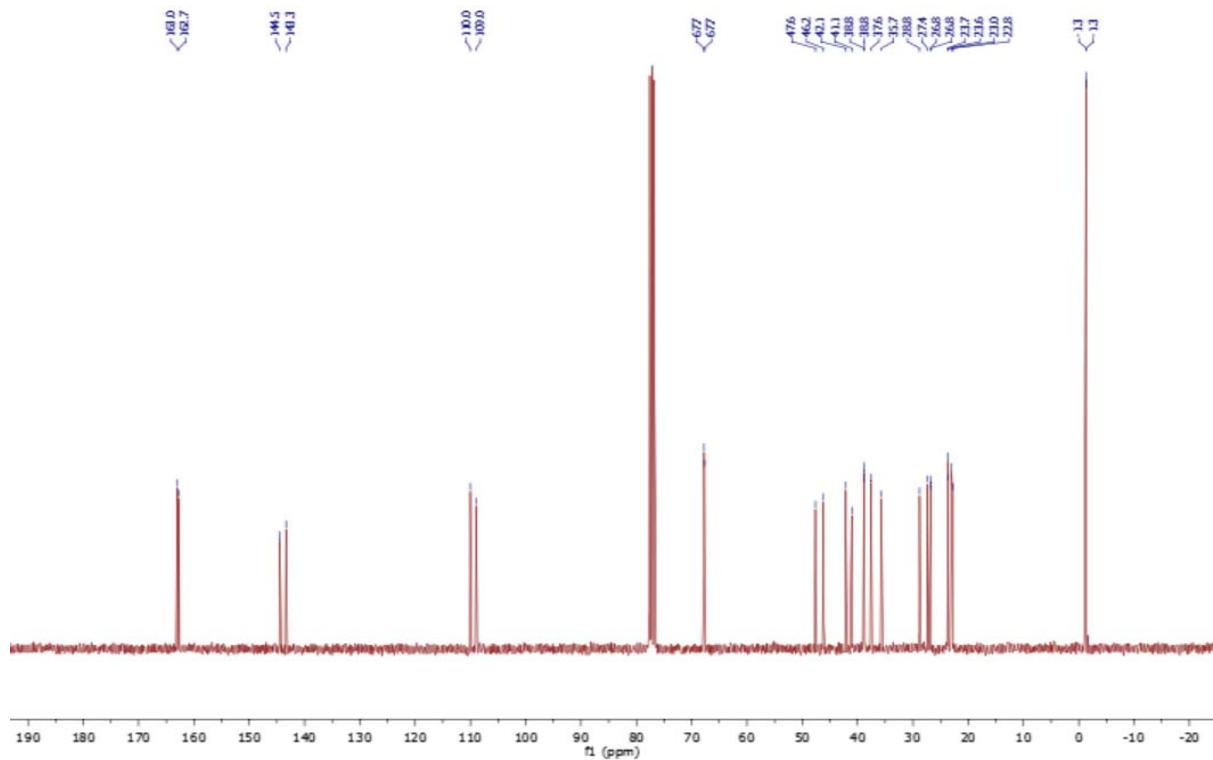


N-(5-Hydroxyhexyl)-N-(3-((trimethylsilyl)methyl)but-3-en-1-yl)formamide (3-60)

^1H RMN spectrum

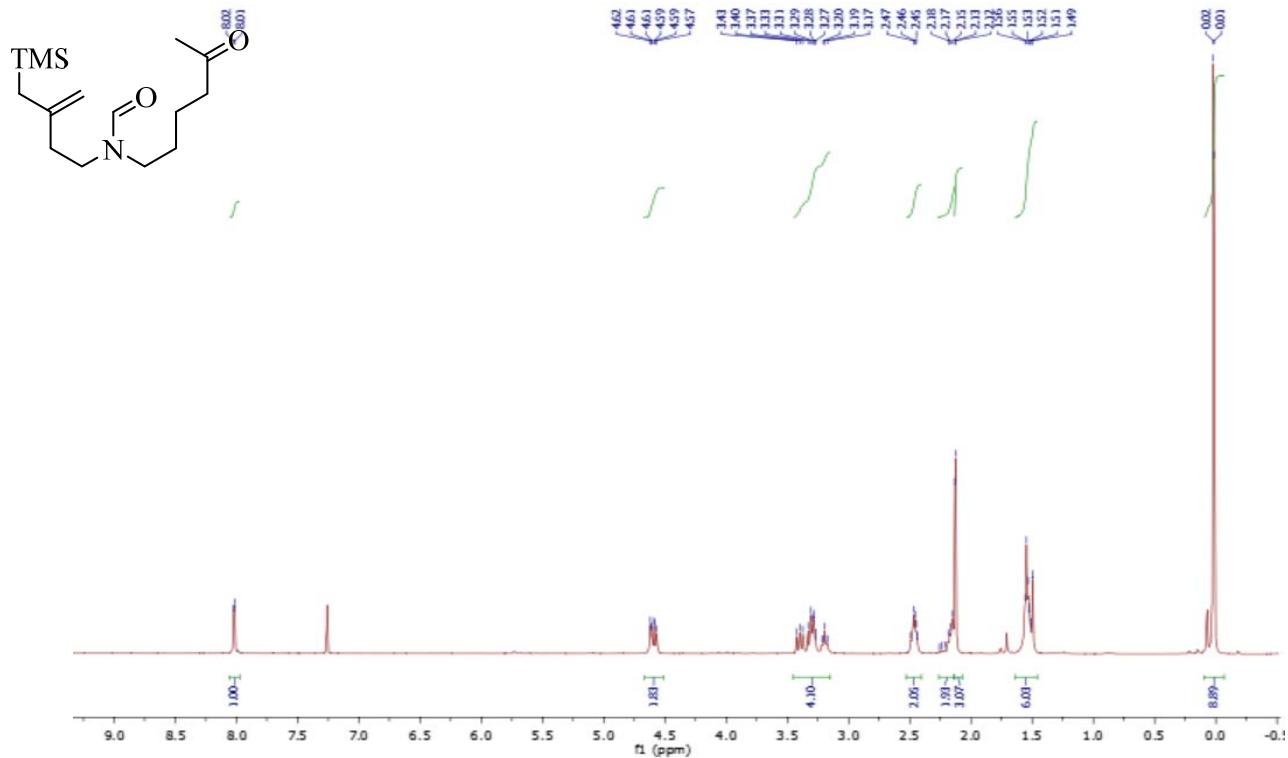


^{13}C RMN spectrum

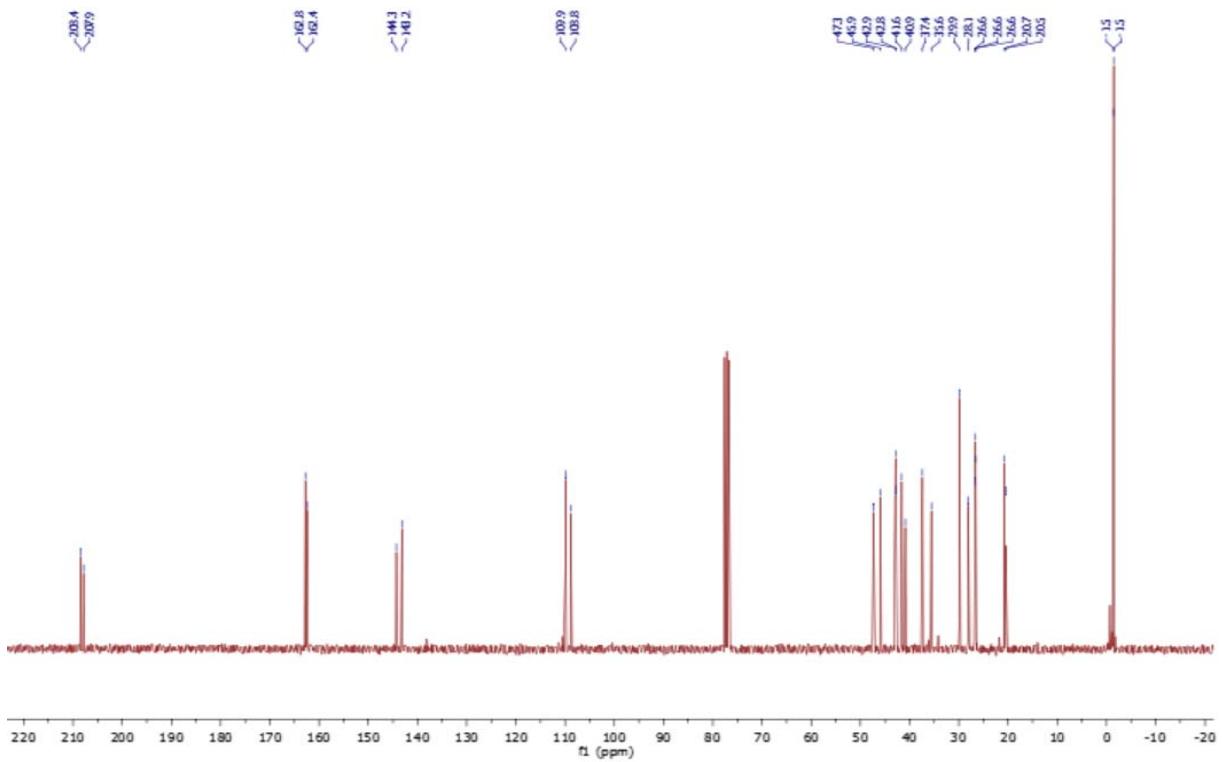


N-(5-Oxohexyl)-N-((trimethylsilyl)methyl)but-3-en-1-ylformamide (3-61)

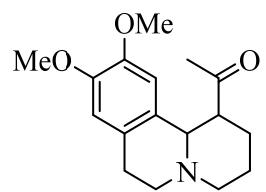
^1H RMN spectrum



^{13}C RMN spectrum



ANNEXE 3: TRACE HPLC DU COMPOSÉ 2-4



Column: Chiralcel OZ-H 250 x 4.6 mm, 5 μ m ID.

Rate: 0.8 mL/min.

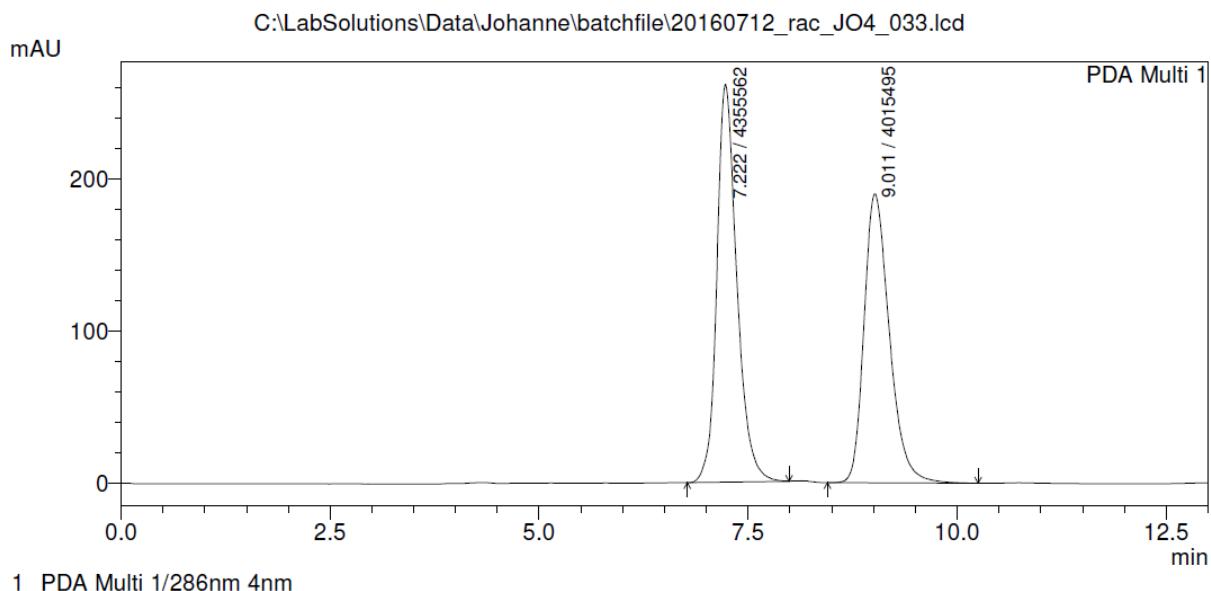
Eluent: 60% *i*-PrOH/Hex. (+ 0.1% DEA).

[C]_{sample}: 1 mg/mL (dissolved in *i*-PrOH).

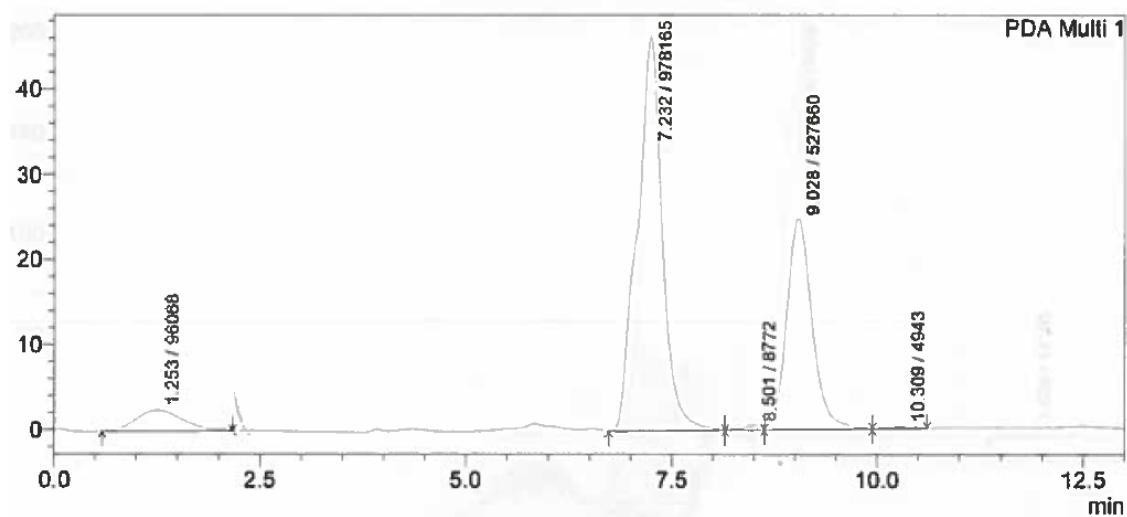
V_{injected}: 10 μ L.

λ : 286 nm.

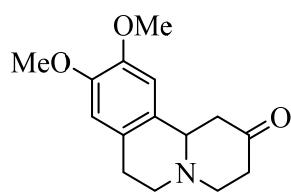
Racemic reaction:



Enantiomeric reaction (using L-proline):



ANNEXE 4: TRACE HPLC DU COMPOSÉ 2-24



Column: Chiralcel OD-H 250 x 4.6 mm, 5 μm ID.

Rate: 1.0 mL/min.

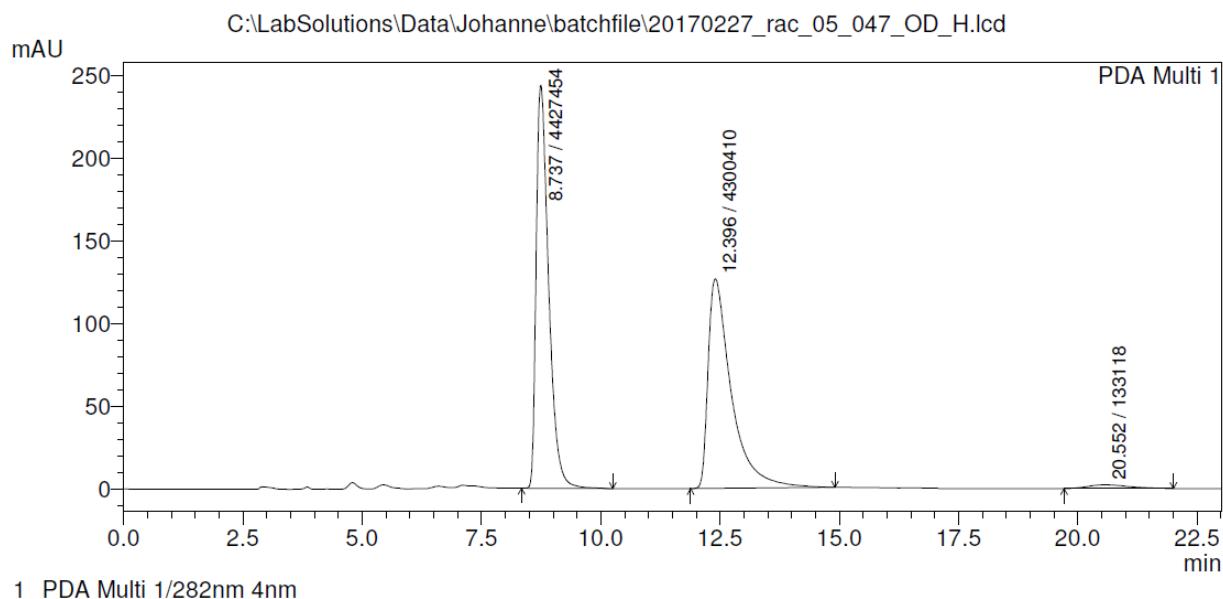
Eluent: 20% i-PrOH/Hex. (+ 0.1% DEA).

[C]_{sample}: 1 mg/mL (dissolved in *i*-PrOH).

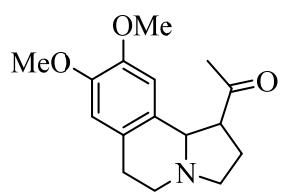
V_{injected}: 10 μL .

λ : 282 nm.

Racemic reaction:



ANNEXE 5: TRACE HPLC DU COMPOSÉ 2-28



Column: Chiralcel AS-H 250 x 4.6 mm, 5 μm ID.

Rate: 1.2 mL/min.

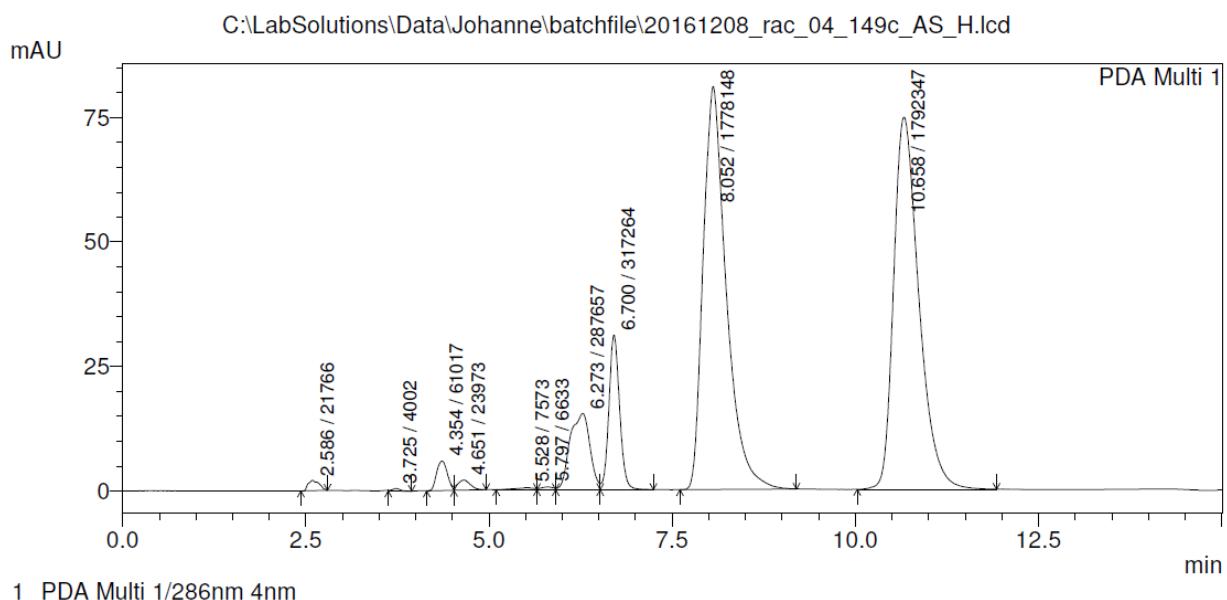
Eluent: 10% i-PrOH/Hex. (+ 0.1% DEA).

[C]_{sample}: 1 mg/mL (dissolved in *i*-PrOH).

V_{injected}: 10 μL .

λ : 286 nm.

Racemic reaction:



ANNEXE 6: COORDONNÉES DE DIFFRACTION DES RAYONS-X DU COMPOSÉ 1-31

A Needle-like specimen of $C_{16}H_{21}NO_3$, approximate dimensions 0.220 mm x 0.230 mm x 0.320 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker Apex DUO system equipped with a Cu K α ImuS micro-focus source with MX optics ($\lambda = 1.54186 \text{ \AA}$).

The total exposure time was 3.84 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 7513 reflections to a maximum θ angle of 71.04° (0.82 \AA resolution), of which 2700 were independent (average redundancy 2.783, completeness = 97.9%, $R_{\text{int}} = 2.05\%$, $R_{\text{sig}} = 2.73\%$) and 2502 (92.67%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 30.3546(18) \text{ \AA}$, $b = 5.0780(3) \text{ \AA}$, $c = 20.1975(12) \text{ \AA}$, $\beta = 113.6040(10)^\circ$, volume = $2852.8(3) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 9968 reflections above $20\sigma(I)$ with $9.113^\circ < 2\theta < 141.1^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.801. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.8043 and 0.8592.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group C 1 2/c 1, with $Z = 8$ for the formula unit, $C_{16}H_{21}NO_3$. The final anisotropic full-matrix least-squares refinement on F^2 with 184 variables converged at $R1 = 3.62\%$, for the observed data and $wR2 = 9.49\%$ for all data. The goodness-of-fit was 1.043. The largest peak in the final difference electron density synthesis was $0.200 \text{ e}^-/\text{\AA}^3$ and the largest hole was $-0.188 \text{ e}^-/\text{\AA}^3$ with an RMS deviation of $0.046 \text{ e}^-/\text{\AA}^3$. On the basis of the final model, the calculated density was 1.282 g/cm^3 and $F(000) = 1184 \text{ e}^-$.

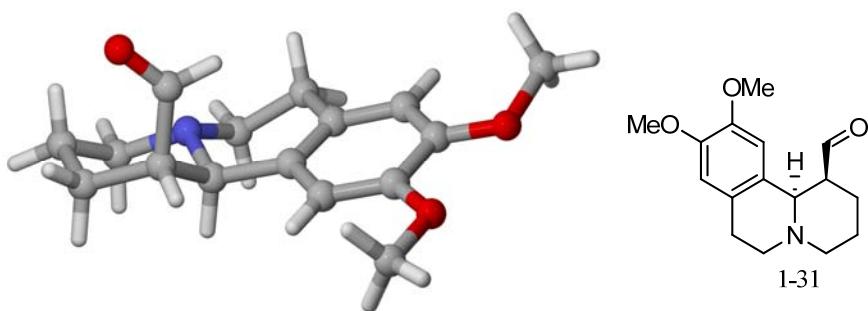


Table 1. Sample and crystal data for Belanger_JO2_016D.

Identification code	Belanger_JO2_016D
Chemical formula	$C_{16}H_{21}NO_3$

Formula weight	275.34
Temperature	173(2) K
Wavelength	1.54186 Å
Crystal size	0.220 x 0.230 x 0.320 mm
Crystal system	monoclinic
Space group	C 1 2/c 1
Unit cell dimensions	a = 30.3546(18) Å α = 90° b = 5.0780(3) Å β = 113.6040(10)° c = 20.1975(12) Å γ = 90°
Volume	2852.8(3) Å ³
Z	8
Density (calculated)	1.282 g/cm ³
Absorption coefficient	0.711 mm ⁻¹
F(000)	1184

Table 2. Data collection and structure refinement for Belanger_JO2_016D.

Diffractometer	Bruker Apex DUO
Radiation source	ImuS micro—focus source with MX optics, Cu K α
Theta range for data collection	4.56 to 71.04°
Index ranges	-34≤h≤36, -6≤k≤5, -22≤l≤24
Reflections collected	7513
Independent reflections	2700 [R(int) = 0.0205]
Coverage of independent reflections	97.9%
Absorption correction	multi-scan
Max. and min. transmission	0.8592 and 0.8043
Structure solution technique	direct methods
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Refinement method	Full-matrix least-squares on F ²
Refinement program	SHELXL-97 (Sheldrick, 2008)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	2700 / 0 / 184
Goodness-of-fit on F²	1.043

Δ/σ_{\max}	0.001	
Final R indices	2502 data; $I > 2\sigma(I)$	$R_1 = 0.0362$, $wR_2 = 0.0930$
	all data	$R_1 = 0.0384$, $wR_2 = 0.0949$
Weighting scheme		$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 1.5156P]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction coefficient	0.0080(3)	
Largest diff. peak and hole	0.200 and -0.188 eÅ ⁻³	
R.M.S. deviation from mean	0.046 eÅ ⁻³	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for Belanger_JO2_016D.

$U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	$U(\text{eq})$
C1	0.11477(5)	0.5432(3)	0.87842(6)	0.0443(3)
C2	0.06045(5)	0.5476(3)	0.84085(6)	0.0386(3)
C3	0.03785(5)	0.3630(2)	0.87797(6)	0.0386(3)
C4	0.05876(4)	0.4126(2)	0.95954(6)	0.0305(3)
C5	0.11409(4)	0.4018(2)	0.99181(6)	0.0306(3)
C6	0.13659(4)	0.4350(2)	0.07370(6)	0.0283(3)
C7	0.11935(4)	0.2824(2)	0.11633(6)	0.0291(3)
C8	0.13830(4)	0.3079(2)	0.19055(6)	0.0285(3)
C9	0.17493(4)	0.4937(2)	0.22422(6)	0.0293(3)
C10	0.19251(4)	0.6397(2)	0.18253(6)	0.0307(3)
C11	0.17419(4)	0.6083(2)	0.10732(6)	0.0294(3)
C12	0.19660(4)	0.7581(3)	0.06403(6)	0.0363(3)
C13	0.18273(4)	0.6372(3)	0.99009(7)	0.0422(3)
C14	0.04209(4)	0.6693(2)	0.97794(6)	0.0330(3)
C15	0.08543(4)	0.9856(3)	0.20475(7)	0.0382(3)
C16	0.21926(5)	0.7352(3)	0.33116(7)	0.0407(3)
N1	0.13050(4)	0.6063(2)	0.95556(5)	0.0348(3)
O1	0.00612(3)	0.7833(2)	0.94091(5)	0.0467(3)
O2	0.12404(3)	0.16724(17)	0.23628(4)	0.0353(2)
O3	0.19033(3)	0.51246(17)	0.29775(4)	0.0366(2)

Table 4. Bond lengths (Å) for Belanger_JO2_016D.

C1-N1	1.4705(15)	C1-C2	1.5142(19)
C2-C3	1.5241(18)	C3-C4	1.5308(15)
C4-C14	1.4982(16)	C4-C5	1.5402(16)
C5-N1	1.4676(15)	C5-C6	1.5250(15)
C6-C11	1.3845(16)	C6-C7	1.4061(16)
C7-C8	1.3800(15)	C8-O2	1.3677(13)
C8-C9	1.4084(16)	C9-O3	1.3701(13)
C9-C10	1.3796(16)	C10-C11	1.4021(15)
C11-C12	1.5107(15)	C12-C13	1.5102(18)
C13-N1	1.4624(16)	C14-O1	1.1969(15)
C15-O2	1.4255(14)	C16-O3	1.4252(15)

Table 5. Bond angles (°) for Belanger_JO2_016D.

N1-C1-C2	110.75(10)	C1-C2-C3	111.02(10)
C2-C3-C4	110.73(10)	C14-C4-C3	111.98(10)
C14-C4-C5	110.21(9)	C3-C4-C5	111.20(10)
N1-C5-C6	111.63(10)	N1-C5-C4	107.11(9)
C6-C5-C4	113.20(9)	C11-C6-C7	119.06(10)
C11-C6-C5	121.70(10)	C7-C6-C5	119.23(10)
C8-C7-C6	121.21(11)	O2-C8-C7	125.29(10)
O2-C8-C9	115.21(9)	C7-C8-C9	119.49(10)
O3-C9-C10	125.22(10)	O3-C9-C8	115.51(10)
C10-C9-C8	119.27(10)	C9-C10-C11	121.17(11)
C6-C11-C10	119.69(10)	C6-C11-C12	120.65(10)
C10-C11-C12	119.64(10)	C13-C12-C11	110.84(11)
N1-C13-C12	109.57(10)	O1-C14-C4	125.45(11)
C13-N1-C5	110.96(9)	C13-N1-C1	111.03(9)
C5-N1-C1	110.08(10)	C8-O2-C15	117.52(9)
C9-O3-C16	116.52(9)		

Table 6. Torsion angles (°) for Belanger_JO2_016D.

N1-C1-C2-C3	-55.45(15)	C1-C2-C3-C4	49.84(14)
C2-C3-C4-C14	71.01(13)	C2-C3-C4-C5	-52.78(13)
C14-C4-C5-N1	-65.10(11)	C3-C4-C5-N1	59.69(12)
C14-C4-C5-C6	58.38(12)	C3-C4-C5-C6	-176.83(9)

N1-C5-C6-C11	-11.01(14)	C4-C5-C6-C11	-131.97(11)
N1-C5-C6-C7	170.17(10)	C4-C5-C6-C7	49.22(14)
C11-C6-C7-C8	1.84(16)	C5-C6-C7-C8	-179.32(10)
C6-C7-C8-O2	-179.61(10)	C6-C7-C8-C9	1.25(17)
O2-C8-C9-O3	-1.58(14)	C7-C8-C9-O3	177.65(10)
O2-C8-C9-C10	178.27(10)	C7-C8-C9-C10	-2.50(16)
O3-C9-C10-C11	-179.47(10)	C8-C9-C10-C11	0.69(17)
C7-C6-C11-C10	-3.64(16)	C5-C6-C11-C10	177.55(10)
C7-C6-C11-C12	174.62(10)	C5-C6-C11-C12	-4.20(16)
C9-C10-C11-C6	2.41(17)	C9-C10-C11-C12	-175.86(10)
C6-C11-C12-C13	-15.85(15)	C10-C11-C12-C13	162.40(10)
C11-C12-C13-N1	51.30(14)	C3-C4-C14-O1	23.71(17)
C5-C4-C14-O1	148.05(12)	C12-C13-N1-C5	-69.40(14)
C12-C13-N1-C1	167.84(11)	C6-C5-N1-C13	47.17(13)
C4-C5-N1-C13	171.60(10)	C6-C5-N1-C1	170.48(9)
C4-C5-N1-C1	-65.09(12)	C2-C1-N1-C13	-172.38(12)
C2-C1-N1-C5	64.35(14)	C7-C8-O2-C15	-2.19(16)
C9-C8-O2-C15	176.99(10)	C10-C9-O3-C16	13.54(16)
C8-C9-O3-C16	-166.61(11)		

Table 7. Anisotropic atomic displacement parameters (\AA^2) for Belanger_JO2_016D.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U₁₁	U₂₂	U₃₃	U₂₃	U₁₃	U₁₂
C1	0.0494(8)	0.0591(9)	0.0285(6)	0.0053(6)	0.0199(6)	0.0077(6)
C2	0.0498(7)	0.0402(7)	0.0244(6)	0.0000(5)	0.0135(5)	0.0038(6)
C3	0.0507(7)	0.0323(6)	0.0277(6)	-0.0033(5)	0.0102(5)	-0.0031(5)
C4	0.0366(6)	0.0278(6)	0.0254(5)	0.0003(4)	0.0107(5)	-0.0025(5)
C5	0.0372(6)	0.0295(6)	0.0267(6)	0.0017(4)	0.0144(5)	0.0062(5)
C6	0.0305(6)	0.0283(6)	0.0271(6)	0.0029(4)	0.0127(4)	0.0062(4)
C7	0.0324(6)	0.0275(6)	0.0265(5)	-0.0003(4)	0.0109(4)	0.0004(4)
C8	0.0308(6)	0.0288(6)	0.0278(5)	0.0029(4)	0.0138(4)	0.0019(4)
C9	0.0272(6)	0.0348(6)	0.0248(5)	0.0010(4)	0.0093(4)	0.0027(4)
C10	0.0257(5)	0.0337(6)	0.0318(6)	0.0017(5)	0.0105(4)	0.0004(4)
C11	0.0267(5)	0.0326(6)	0.0302(6)	0.0055(5)	0.0128(4)	0.0059(4)
C12	0.0287(6)	0.0450(7)	0.0361(6)	0.0097(5)	0.0139(5)	0.0016(5)
C13	0.0340(6)	0.0625(9)	0.0355(6)	0.0098(6)	0.0196(5)	0.0049(6)
C14	0.0323(6)	0.0359(6)	0.0302(6)	-0.0007(5)	0.0118(5)	-0.0002(5)

C15	0.0393(7)	0.0387(7)	0.0341(6)	0.0044(5)	0.0122(5)	-0.0090(5)
C16	0.0413(7)	0.0444(7)	0.0301(6)	-0.0042(5)	0.0076(5)	-0.0081(6)
N1	0.0346(5)	0.0463(6)	0.0263(5)	0.0067(4)	0.0151(4)	0.0031(4)
O1	0.0397(5)	0.0521(6)	0.0441(5)	0.0041(4)	0.0125(4)	0.0136(4)
O2	0.0409(5)	0.0394(5)	0.0258(4)	0.0016(3)	0.0135(3)	-0.0092(4)
O3	0.0376(5)	0.0456(5)	0.0246(4)	-0.0028(3)	0.0105(3)	-0.0099(4)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (\AA^2) for Belanger_JO2_016D.

	x/a	y/b	z/c	U(eq)
H1A	0.1268	0.3665	-0.1268	0.053
H1B	0.1285	0.6730	-0.1446	0.053
H2A	0.0506	0.4934	-0.2102	0.046
H2B	0.0487	0.7292	-0.1586	0.046
H3A	0.0439	0.1781	-0.1314	0.046
H3B	0.0026	0.3910	-0.1422	0.046
H4	0.0471	0.2688	-0.0177	0.037
H5	0.1240	0.2266	-0.0202	0.037
H7	0.0942	0.1595	0.0936	0.035
H10	0.2175	0.7638	0.2052	0.037
H12A	0.1858	0.9437	0.0589	0.044
H12B	0.2320	0.7560	0.0900	0.044
H13A	0.1937	0.7517	-0.0399	0.051
H13B	0.1984	0.4633	-0.0054	0.051
H14	0.0617	0.7483	0.0227	0.04
H15A	0.0567	0.0801	0.1724	0.057
H15B	0.0786	-0.1007	0.2430	0.057
H15C	0.0944	-0.1473	0.1772	0.057
H16A	0.2504	0.7203	0.3275	0.061
H16B	0.2243	0.7423	0.3822	0.061
H16C	0.2030	0.8962	0.3067	0.061

ANNEXE 7: COORDONNÉES DE DIFFRACTION DES RAYONS-X DU COMPOSÉ 2-4

A Needle-like specimen of C₁₇H₂₃NO₃, approximate dimensions 0.040 mm x 0.110 mm x 0.450 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker Apex DUO system equipped with a Cu K α ImuS micro-focus source with MX optics ($\lambda = 1.54186 \text{ \AA}$).

Table 1: Data collection details for Belanger_JO3_130.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Widt h/°	Fram es	Time /s	Wavelen gth/ \AA	Vo lta ge/ k V	Cur rent /m A	Temper ature/K
Phi	79.723	-46.00	305.43	-13.34	-30.75	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	86.00	103.39	-351.72	19.47	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	116.00	77.76	-339.07	46.12	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	118.00	116.80	-350.39	22.49	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	86.00	241.66	-36.72	-70.95	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	122.00	93.61	-344.21	35.94	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	114.00	230.75	-30.28	-62.00	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	72.00	24.26	-268.44	48.96	7.00	39	10.00	1.54184	45	0.7	173.20
Phi	79.723	54.00	26.90	-44.98	-80.22	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	122.00	236.31	-40.72	-75.73	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	120.00	149.15	-350.39	22.49	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	104.00	69.58	-339.07	46.12	7.00	47	10.00	1.54184	45	0.7	173.20
Phi	79.723	66.00	98.78	-9.35	-27.76	7.00	44	10.00	1.54184	45	0.7	173.20
Phi	79.723	50.00	201.72	-34.69	-68.46	7.00	49	10.00	1.54184	45	0.7	173.20
Phi	79.723	-76.00	302.84	-288.75	32.98	7.00	36	10.00	1.54184	45	0.7	173.20
Omega	79.723	-28.00	-93.52	76.66	-30.75	7.00	28	10.00	1.54184	45	0.7	173.20
Phi	79.723	120.00	36.25	-281.19	98.91	7.00	40	10.00	1.54184	45	0.7	173.20
Omega	79.723	82.00	-17.79	-182.66	96.94	7.00	10	10.00	1.54184	45	0.7	173.20
Phi	79.723	108.00	57.47	-313.35	81.82	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	118.00	43.74	-321.81	72.77	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	80.00	80.63	36.38	48.96	7.00	18	10.00	1.54184	45	0.7	173.20
Omega	79.723	-22.00	-36.98	-55.88	-67.19	7.00	7	10.00	1.54184	45	0.7	173.20
Omega	79.723	70.00	-11.20	18.98	88.60	7.00	9	10.00	1.54184	45	0.7	173.20
Omega	79.723	102.00	61.87	63.45	-45.40	7.00	10	10.00	1.54184	45	0.7	173.20
Omega	79.723	-28.00	-189.96	2.44	95.88	7.00	7	10.00	1.54184	45	0.7	173.20
Omega	79.723	120.00	-194.14	-13.70	-74.56	7.00	10	10.00	1.54184	45	0.7	173.20
Phi	79.723	-28.00	233.44	81.94	48.96	7.00	9	10.00	1.54184	45	0.7	173.20
Omega	79.723	-72.00	-203.21	-45.49	86.79	7.00	10	10.00	1.54184	45	0.7	173.20

Omega	79.723	-20.00	-200.06	57.53	90.31	7.00	9	10.00	1.54184	45	0.7	173.20
Omega	79.723	-66.00	-36.98	-199.88	-67.19	7.00	10	10.00	1.54184	45	0.7	173.20
Omega	79.723	-38.00	-146.52	-190.61	-24.76	7.00	34	10.00	1.54184	45	0.7	173.20
Phi	79.723	104.00	17.74	-210.70	90.31	7.00	16	10.00	1.54184	45	0.7	173.20
Omega	79.723	-40.00	-178.06	-192.21	35.94	7.00	13	10.00	1.54184	45	0.7	173.20
Omega	79.723	82.00	-10.87	-5.72	19.47	7.00	24	10.00	1.54184	45	0.7	173.20
Phi	79.723	90.00	212.69	-42.82	-78.01	7.00	26	10.00	1.54184	45	0.7	173.20
Omega	79.723	96.00	45.06	-14.15	-36.68	7.00	26	10.00	1.54184	45	0.7	173.20
Phi	79.723	112.00	236.46	-43.89	-79.13	7.00	18	10.00	1.54184	45	0.7	173.20
Phi	79.723	-76.00	180.66	-229.12	-21.74	7.00	28	10.00	1.54184	45	0.7	173.20
Phi	79.723	120.00	214.34	-39.70	-74.56	7.00	21	10.00	1.54184	45	0.7	173.20
Phi	79.723	110.00	20.51	-185.59	99.62	7.00	17	10.00	1.54184	45	0.7	173.20
Phi	79.723	94.00	62.69	-179.25	-56.62	7.00	22	10.00	1.54184	45	0.7	173.20
Omega	79.723	-40.00	-65.67	-71.31	-46.83	7.00	23	10.00	1.54184	45	0.7	173.20
Omega	79.723	-78.00	-208.76	-189.00	30.00	7.00	26	10.00	1.54184	45	0.7	173.20
Phi	79.723	-22.00	314.65	-29.41	-60.67	7.00	34	10.00	1.54184	45	0.7	173.20
Phi	79.723	110.00	98.34	-203.54	54.56	7.00	33	10.00	1.54184	45	0.7	173.20
Phi	79.723	110.00	119.38	-243.88	43.24	7.00	38	10.00	1.54184	45	0.7	173.20
Phi	79.723	120.00	175.91	-84.18	-99.71	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	-56.00	7.76	-49.52	-84.38	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	-72.00	157.14	-314.47	80.76	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	60.00	-9.36	-303.76	89.47	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	88.00	157.07	-347.69	28.51	7.00	52	10.00	1.54184	45	0.7	173.20
Phi	79.723	120.00	201.98	-21.31	-46.83	7.00	52	10.00	1.54184	45	0.7	173.20

A total of 1727 frames were collected. The total exposure time was 4.80 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a triclinic unit cell yielded a total of 4292 reflections to a maximum θ angle of 70.77° (0.82 \AA resolution), of which 2853 were independent (average redundancy 1.504, completeness = 97.1%, $R_{\text{int}} = 2.44\%$, $R_{\text{sig}} = 4.38\%$) and 2276 (79.78%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 5.45370(10) \text{ \AA}$, $b = 10.2807(3) \text{ \AA}$, $c = 14.4580(4) \text{ \AA}$, $\alpha = 71.7500(10)^\circ$, $\beta = 81.1190(10)^\circ$, $\gamma = 85.2000(10)^\circ$, volume = $760.08(3) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 6796 reflections above $20 \sigma(I)$ with $6.497^\circ < 2\theta < 140.7^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.767. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7460 and 0.9729.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P -1, with Z = 2 for the formula unit, C₁₇H₂₃NO₃. The final anisotropic full-matrix least-squares refinement on F² with 202 variables converged at R1 = 3.96%, for the observed data and wR2 = 10.97% for all data. The goodness-of-fit was 1.066. The largest peak in the final difference electron density synthesis was 0.232 e⁻/Å³ and the largest hole was -0.154 e⁻/Å³ with an RMS deviation of 0.043 e⁻/Å³. On the basis of the final model, the calculated density was 1.264 g/cm³ and F(000), 312 e⁻.

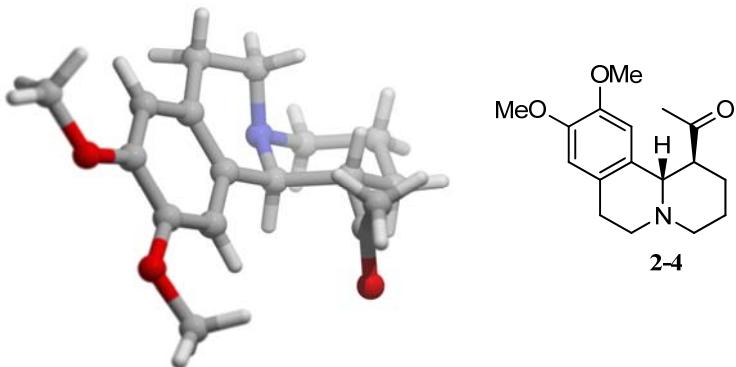


Table 2. Sample and crystal data for Belanger_JO3_130.

Identification code	Belanger_JO3_130		
Chemical formula	C ₁₇ H ₂₃ NO ₃		
Formula weight	289.36		
Temperature	173(2) K		
Wavelength	1.54186 Å		
Crystal size	0.040 x 0.110 x 0.450 mm		
Crystal system	triclinic		
Space group	P -1		
Unit cell dimensions	a = 5.45370(10) Å	α = 71.7500(10)°	
	b = 10.2807(3) Å	β = 81.1190(10)°	
	c = 14.4580(4) Å	γ = 85.2000(10)°	
Volume	760.08(3) Å ³		
Z	2		
Density (calculated)	1.264 g/cm ³		
Absorption coefficient	0.692 mm ⁻¹		
F(000)	312		

Table 3. Data collection and structure refinement for Belanger_JO3_130.

Diffractometer	Bruker Apex DUO
Radiation source	ImuS micro—focus source with MX optics, Cu K α
Theta range for data collection	3.25 to 70.77°
Index ranges	-5≤h≤6, -12≤k≤12, -16≤l≤17
Reflections collected	4292
Independent reflections	2853 [R(int) = 0.0244]
Coverage of independent reflections	97.1%
Absorption correction	multi-scan
Max. and min. transmission	0.9729 and 0.7460
Structure solution technique	direct methods
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Refinement method	Full-matrix least-squares on F ²
Refinement program	SHELXL-97 (Sheldrick, 2008)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	2853 / 0 / 202
Goodness-of-fit on F²	1.066
Δ/σ_{\max}	0.004
Final R indices	2276 data; I>2σ(I) R1 = 0.0396, wR2 = 0.1026
	all data R1 = 0.0528, wR2 = 0.1097
Weighting scheme	w=1/[$\sigma^2(F_o^2)+(0.0489P)^2+0.1022P$] where P=(F _o ² +2F _c ²)/3
Extinction coefficient	0.0096(12)
Largest diff. peak and hole	0.232 and -0.154 eÅ ⁻³
R.M.S. deviation from mean	0.043 eÅ ⁻³

Table 4. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for Belanger_JO3_130.U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C17	0.8619(3)	0.39452(18)	0.95588(13)	0.0452(4)
C1	0.9975(3)	0.69275(14)	0.61988(11)	0.0313(3)
C2	0.1466(3)	0.58816(15)	0.59832(11)	0.0312(3)

C3	0.0828(2)	0.45067(14)	0.64037(10)	0.0287(3)
C4	0.2424(3)	0.34055(15)	0.60904(11)	0.0324(3)
C5	0.1794(3)	0.19766(15)	0.67543(11)	0.0336(3)
C6	0.8370(3)	0.04170(15)	0.74202(12)	0.0369(4)
C7	0.8853(3)	0.97902(15)	0.84810(12)	0.0397(4)
C8	0.7612(3)	0.06842(16)	0.90919(12)	0.0388(4)
C9	0.8449(3)	0.21643(15)	0.86598(11)	0.0316(3)
C10	0.7937(2)	0.27204(14)	0.75639(11)	0.0287(3)
C11	0.8670(2)	0.41901(14)	0.70648(10)	0.0280(3)
C12	0.7129(2)	0.52577(14)	0.72709(11)	0.0298(3)
C13	0.7769(3)	0.66053(15)	0.68593(11)	0.0311(3)
C14	0.2644(3)	0.86538(16)	0.51245(12)	0.0403(4)
C15	0.4331(3)	0.74116(17)	0.77852(14)	0.0460(4)
C16	0.7093(3)	0.30416(16)	0.92603(11)	0.0359(4)
N1	0.9099(2)	0.18471(12)	0.69763(9)	0.0306(3)
O1	0.0452(2)	0.82913(10)	0.58225(8)	0.0404(3)
O2	0.63838(19)	0.77052(10)	0.70270(8)	0.0402(3)
O3	0.4857(2)	0.29976(13)	0.94908(9)	0.0493(3)

Tale 5. Bond lengths (\AA) for Belanger_JO3_130.

C17-C16	1.494(2)	C1-O1	1.3671(17)
C1-C2	1.378(2)	C1-C13	1.408(2)
C2-C3	1.403(2)	C3-C11	1.3880(19)
C3-C4	1.5097(19)	C4-C5	1.516(2)
C5-N1	1.4632(17)	C6-N1	1.4696(18)
C6-C7	1.521(2)	C7-C8	1.518(2)
C8-C9	1.532(2)	C9-C16	1.515(2)
C9-C10	1.568(2)	C10-N1	1.4647(18)
C10-C11	1.5143(19)	C11-C12	1.4048(19)
C12-C13	1.377(2)	C13-O2	1.3712(17)
C14-O1	1.4330(18)	C15-O2	1.4200(19)
C16-O3	1.2145(18)		

Table 6. Bond angles ($^\circ$) for Belanger_JO3_130.

O1-C1-C2	125.31(13)	O1-C1-C13	115.54(12)
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C2-C1-C13	119.15(13)	C1-C2-C3	121.46(13)
C11-C3-C2	119.29(13)	C11-C3-C4	121.12(12)
C2-C3-C4	119.52(12)	C3-C4-C5	112.50(12)
N1-C5-C4	110.34(11)	N1-C6-C7	114.08(12)
C8-C7-C6	109.74(12)	C7-C8-C9	111.58(13)
C16-C9-C8	110.23(12)	C16-C9-C10	111.20(11)
C8-C9-C10	108.62(12)	N1-C10-C11	110.09(11)
N1-C10-C9	112.58(11)	C11-C10-C9	113.15(11)
C3-C11-C12	119.21(13)	C3-C11-C10	121.44(12)
C12-C11-C10	119.34(12)	C13-C12-C11	121.21(13)
O2-C13-C12	124.92(13)	O2-C13-C1	115.45(12)
C12-C13-C1	119.62(13)	O3-C16-C17	121.88(15)
O3-C16-C9	120.77(14)	C17-C16-C9	117.34(13)
C5-N1-C10	112.27(11)	C5-N1-C6	112.74(11)
C10-N1-C6	111.83(11)	C1-O1-C14	116.79(12)
C13-O2-C15	116.83(12)		

Table 7. Torsion angles ($^{\circ}$) for Belanger_JO3_130.

O1-C1-C2-C3	-179.91(13)	C13-C1-C2-C3	0.3(2)
C1-C2-C3-C11	-1.1(2)	C1-C2-C3-C4	175.67(13)
C11-C3-C4-C5	-14.91(19)	C2-C3-C4-C5	168.34(12)
C3-C4-C5-N1	43.53(16)	N1-C6-C7-C8	54.49(17)
C6-C7-C8-C9	-55.36(16)	C7-C8-C9-C16	177.53(12)
C7-C8-C9-C10	55.47(15)	C16-C9-C10-N1	-176.52(11)
C8-C9-C10-N1	-55.05(15)	C16-C9-C10-C11	57.85(15)
C8-C9-C10-C11	179.32(11)	C2-C3-C11-C12	2.2(2)
C4-C3-C11-C12	-174.51(12)	C2-C3-C11-C10	-178.09(12)
C4-C3-C11-C10	5.2(2)	N1-C10-C11-C3	-23.58(17)
C9-C10-C11-C3	103.37(15)	N1-C10-C11-C12	156.10(12)
C9-C10-C11-C12	-76.95(15)	C3-C11-C12-C13	-2.6(2)
C10-C11-C12-C13	177.75(12)	C11-C12-C13-O2	-179.48(13)
C11-C12-C13-C1	1.7(2)	O1-C1-C13-O2	0.69(19)
C2-C1-C13-O2	-179.46(12)	O1-C1-C13-C12	179.62(12)
C2-C1-C13-C12	-0.5(2)	C8-C9-C16-O3	-47.60(19)
C10-C9-C16-O3	72.92(18)	C8-C9-C16-C17	131.96(14)
C10-C9-C16-C17	-107.51(15)	C4-C5-N1-C10	-65.93(15)
C4-C5-N1-C6	166.68(12)	C11-C10-N1-C5	53.92(14)
C9-C10-N1-C5	-73.35(15)	C11-C10-N1-C6	-178.20(10)

C9-C10-N1-C6	54.53(15)	C7-C6-N1-C5	73.02(16)
C7-C6-N1-C10	-54.61(16)	C2-C1-O1-C14	2.1(2)
C13-C1-O1-C14	-178.10(13)	C12-C13-O2-C15	9.4(2)
C1-C13-O2-C15	-171.77(13)		

Table 8. Anisotropic atomic displacement parameters (\AA^2) for Belanger_JO3_130.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U₁₁	U₂₂	U₃₃	U₂₃	U₁₃	U₁₂
C17	0.0466(9)	0.0510(10)	0.0454(10)	-0.0248(8)	-0.0090(7)	0.0018(7)
C1	0.0366(7)	0.0261(7)	0.0318(8)	-0.0088(6)	-0.0065(6)	-0.0021(6)
C2	0.0310(7)	0.0316(8)	0.0305(8)	-0.0103(6)	-0.0008(6)	-0.0017(6)
C3	0.0281(6)	0.0309(8)	0.0285(7)	-0.0109(6)	-0.0055(5)	0.0014(6)
C4	0.0302(7)	0.0350(8)	0.0332(8)	-0.0144(6)	-0.0016(6)	0.0023(6)
C5	0.0328(7)	0.0322(8)	0.0378(8)	-0.0153(6)	-0.0037(6)	0.0048(6)
C6	0.0380(8)	0.0271(8)	0.0486(9)	-0.0137(7)	-0.0112(7)	0.0001(6)
C7	0.0406(8)	0.0274(8)	0.0480(10)	-0.0068(7)	-0.0076(7)	0.0002(6)
C8	0.0355(8)	0.0372(9)	0.0383(9)	-0.0045(7)	-0.0043(6)	0.0000(6)
C9	0.0258(7)	0.0362(8)	0.0329(8)	-0.0111(6)	-0.0043(6)	0.0013(6)
C10	0.0249(6)	0.0280(7)	0.0347(8)	-0.0113(6)	-0.0058(6)	0.0004(5)
C11	0.0277(6)	0.0288(7)	0.0300(7)	-0.0118(6)	-0.0063(6)	0.0006(5)
C12	0.0263(6)	0.0312(8)	0.0334(8)	-0.0130(6)	-0.0027(5)	0.0002(6)
C13	0.0327(7)	0.0291(7)	0.0342(8)	-0.0144(6)	-0.0053(6)	0.0040(6)
C14	0.0439(8)	0.0332(8)	0.0389(9)	-0.0042(7)	-0.0022(7)	-0.0066(7)
C15	0.0417(9)	0.0420(9)	0.0551(11)	-0.0240(8)	0.0064(7)	0.0056(7)
C16	0.0337(7)	0.0402(9)	0.0316(8)	-0.0095(7)	-0.0027(6)	0.0023(6)
N1	0.0323(6)	0.0253(6)	0.0373(7)	-0.0138(5)	-0.0061(5)	0.0005(5)
O1	0.0476(6)	0.0259(5)	0.0438(6)	-0.0086(5)	0.0020(5)	-0.0033(4)
O2	0.0415(6)	0.0294(6)	0.0494(7)	-0.0166(5)	0.0009(5)	0.0055(4)
O3	0.0335(6)	0.0587(8)	0.0562(8)	-0.0247(6)	0.0060(5)	0.0000(5)

Table 9. Hydrogen atomic coordinates and isotropic atomic displacement parameters (\AA^2) for Belanger_JO3_130.

	x/a	y/b	z/c	U(eq)
H17A	-0.2481	0.4558	0.9851	0.068
H17B	-0.0322	0.4491	0.8980	0.068
H17C	-0.0340	0.3379	1.0041	0.068

H10	-0.394(3)	0.2706(15)	0.7594(11)	0.027(4)
H9	0.020(3)	0.2165(16)	0.8699(12)	0.035(4)
H2	0.2958	0.6097	0.5540	0.037
H4A	0.4191	0.3557	0.6094	0.039
H4B	0.2203	0.3478	0.5409	0.039
H5A	0.2524	0.1293	0.6427	0.04
H5B	0.2514	0.1791	0.7373	0.04
H6A	-0.0711	-0.0141	0.7023	0.044
H6B	-0.3423	0.0372	0.7393	0.044
H7A	-0.1812	-0.1141	0.8750	0.048
H7B	0.0664	-0.0289	0.8509	0.048
H8A	-0.1977	0.0303	0.9771	0.047
H8B	-0.4213	0.0675	0.9122	0.047
H12	-0.4383	0.5046	0.7702	0.036
H14A	0.4113	0.8259	0.5443	0.06
H14B	0.2738	0.9654	0.4878	0.06
H14C	0.2582	0.8296	0.4575	0.06
H15A	-0.6862	0.6887	0.7619	0.069
H15B	-0.6474	0.8272	0.7853	0.069
H15C	-0.5087	0.6874	0.8406	0.069

ANNEXE 8: COORDONNÉES DE DIFFRACTION DES RAYONS-X DU COMPOSÉ 2-9

A Prism-like specimen of C₁₉H₂₇NO₃, approximate dimensions 0.110 mm x 0.330 mm x 0.350 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker Apex DUO system equipped with a Cu K α ImuS micro-focus source with MX optics ($\lambda = 1.54186 \text{ \AA}$).

The total exposure time was 4.80 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a triclinic unit cell yielded a total of 22493 reflections to a maximum θ angle of 70.89° (0.82 Å resolution), of which 6351 were independent (average redundancy 3.542, completeness = 96.9%, R_{int} = 5.24%, R_{sig} = 4.69%) and 5319 (83.75%) were greater than 2 σ (F²). The final cell constants of $a = 9.1272(2) \text{ \AA}$, $b = 9.8101(2) \text{ \AA}$, $c = 19.4090(5) \text{ \AA}$, $\alpha = 81.5820(10)^\circ$, $\beta = 82.6750(10)^\circ$, $\gamma = 85.3380(10)^\circ$, volume = 1701.63(7) Å³, are based upon the refinement of the XYZ-centroids of 9938 reflections above 20 σ(I) with 9.131° < 2θ < 141.5°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.789. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.8017 and 0.9309.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P -1, with Z = 4 for the formula unit, C₁₉H₂₇NO₃. The final anisotropic full-matrix least-squares refinement on F² with 437 variables converged at R1 = 4.73%, for the observed data and wR2 = 14.31% for all data. The goodness-of-fit was 1.003. The largest peak in the final difference electron density synthesis was 0.184 e⁻/Å³ and the largest hole was -0.257 e⁻/Å³ with an RMS deviation of 0.050 e⁻/Å³. On the basis of the final model, the calculated density was 1.239 g/cm³ and F(000), 688 e⁻.

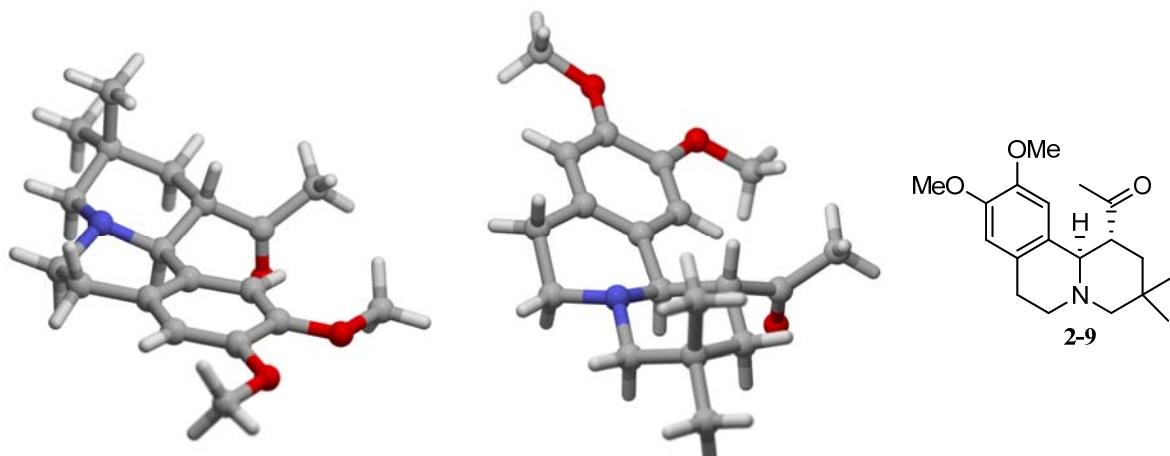


Table 1. Sample and crystal data for Belanger_JO4_116.

Identification code	Belanger_JO4_116		
Chemical formula	C ₁₉ H ₂₇ NO ₃		
Formula weight	317.42		
Temperature	173(2) K		
Wavelength	1.54186 Å		
Crystal size	0.110 x 0.330 x 0.350 mm		
Crystal system	triclinic		
Space group	P -1		
Unit cell dimensions	a = 9.1272(2) Å	α = 81.5820(10)°	
	b = 9.8101(2) Å	β = 82.6750(10)°	
	c = 19.4090(5) Å	γ = 85.3380(10)°	
Volume	1701.63(7) Å ³		
Z	4		
Density (calculated)	1.239 g/cm ³		
Absorption coefficient	0.661 mm ⁻¹		
F(000)	688		

Table 2. Data collection and structure refinement for Belanger_JO4_116.

Diffractometer	Bruker Apex DUO
Radiation source	ImuS micro—focus source with MX optics, Cu Kα
Theta range for data collection	2.32 to 70.89°
Index ranges	-9<=h<=11, -10<=k<=11, -23<=l<=23
Reflections collected	22493
Independent reflections	6351 [R(int) = 0.0524]
Coverage of independent reflections	96.9%
Absorption correction	multi-scan
Max. and min. transmission	0.9309 and 0.8017
Structure solution technique	direct methods
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Refinement method	Full-matrix least-squares on F ²
Refinement program	SHELXL-97 (Sheldrick, 2008)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	6351 / 0 / 437
Goodness-of-fit on F²	1.003

Δ/σ_{\max}	0.007	
Final R indices	5319 data; $I > 2\sigma(I)$	$R_1 = 0.0473, wR_2 = 0.1343$
	all data	$R_1 = 0.0562, wR_2 = 0.1431$
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0878P)^2 + 0.3397P]$ where $P = (F_o^2 + 2F_c^2)/3$	
Largest diff. peak and hole	0.184 and -0.257 e \AA^{-3}	
R.M.S. deviation from mean	0.050 e \AA^{-3}	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (\AA^2) for Belanger_JO4_116.

$U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	$U(\text{eq})$
C20	0.64209(18)	0.04774(16)	0.29159(9)	0.0311(3)
C21	0.57647(17)	0.98118(17)	0.36298(8)	0.0320(4)
C22	0.42205(17)	0.93980(15)	0.36100(8)	0.0267(3)
C23	0.32943(17)	0.91377(15)	0.42438(8)	0.0286(3)
C24	0.18848(18)	0.87156(15)	0.42652(8)	0.0285(3)
C25	0.13898(17)	0.85116(15)	0.36348(8)	0.0279(3)
C26	0.22937(17)	0.87785(15)	0.30106(8)	0.0273(3)
C27	0.37184(17)	0.92469(14)	0.29800(8)	0.0255(3)
C28	0.46494(16)	0.96661(15)	0.22762(8)	0.0255(3)
C29	0.44517(17)	0.87997(15)	0.16975(8)	0.0269(3)
C30	0.55260(18)	0.92031(16)	0.10252(8)	0.0317(4)
C31	0.71337(18)	0.91878(16)	0.11654(8)	0.0311(4)
C32	0.71536(18)	0.01032(15)	0.17309(8)	0.0308(4)
O5	0.09022(13)	0.84626(12)	0.48561(6)	0.0359(3)
O6	0.99895(12)	0.80602(12)	0.36940(6)	0.0368(3)
C35	0.29413(18)	0.90111(16)	0.14287(8)	0.0299(3)
C36	0.2382(2)	0.77798(19)	0.11951(9)	0.0388(4)
C37	0.8054(2)	0.9815(2)	0.04951(9)	0.0423(4)
C38	0.77606(19)	0.77246(17)	0.13988(9)	0.0348(4)
N2	0.62320(14)	0.96141(12)	0.23835(7)	0.0267(3)
O4	0.22708(14)	0.01395(12)	0.13670(6)	0.0393(3)
C33	0.1376(2)	0.8701(2)	0.54967(9)	0.0466(5)
C34	0.94855(19)	0.77906(19)	0.30671(10)	0.0393(4)

C1	0.63116(18)	0.55446(17)	0.32390(8)	0.0323(4)
C2	0.74482(17)	0.47256(17)	0.28045(9)	0.0330(4)
C3	0.67748(17)	0.43460(15)	0.21975(8)	0.0284(3)
C4	0.77156(17)	0.40401(15)	0.16038(8)	0.0303(3)
C5	0.71733(18)	0.37389(15)	0.10181(8)	0.0303(3)
C6	0.56314(18)	0.37567(15)	0.10150(8)	0.0303(3)
C7	0.47009(17)	0.40725(15)	0.15970(8)	0.0293(3)
C8	0.52455(16)	0.43491(14)	0.22050(8)	0.0262(3)
C9	0.41821(16)	0.47143(15)	0.28409(8)	0.0260(3)
C10	0.29419(17)	0.36952(15)	0.30818(8)	0.0279(3)
C11	0.20039(17)	0.40582(17)	0.37609(8)	0.0329(4)
C12	0.29183(18)	0.42092(16)	0.43503(8)	0.0313(4)
C13	0.40889(18)	0.52156(16)	0.40349(8)	0.0307(3)
C14	0.9580(2)	0.3211(2)	0.04554(11)	0.0492(5)
C15	0.3608(2)	0.3519(2)	0.03949(10)	0.0409(4)
C16	0.17914(17)	0.37073(16)	0.25825(8)	0.0294(3)
C17	0.11779(19)	0.23498(17)	0.25484(10)	0.0388(4)
C18	0.3632(2)	0.28220(18)	0.46455(9)	0.0406(4)
C19	0.1938(2)	0.4836(2)	0.49394(9)	0.0419(4)
N1	0.50158(14)	0.47272(13)	0.34402(7)	0.0283(3)
O1	0.13165(12)	0.47761(12)	0.22564(6)	0.0367(3)
O2	0.80242(13)	0.34293(13)	0.04203(6)	0.0393(3)
O3	0.51657(13)	0.34438(13)	0.04172(6)	0.0376(3)

Table 4. Bond lengths (Å) for Belanger_JO4_116.

C20-N2	1.463(2)	C20-C21	1.514(2)
C21-C22	1.505(2)	C22-C27	1.391(2)
C22-C23	1.404(2)	C23-C24	1.377(2)
C24-O5	1.3682(18)	C24-C25	1.403(2)
C25-O6	1.3720(18)	C25-C26	1.380(2)
C26-C27	1.405(2)	C27-C28	1.5341(19)
C28-N2	1.4813(19)	C28-C29	1.540(2)
C29-C35	1.523(2)	C29-C30	1.551(2)
C30-C31	1.525(2)	C31-C32	1.519(2)
C31-C38	1.528(2)	C31-C37	1.532(2)

C32-N2	1.4691(18)	O5-C33	1.424(2)
O6-C34	1.421(2)	C35-O4	1.218(2)
C35-C36	1.498(2)	C1-N1	1.4626(18)
C1-C2	1.508(2)	C2-C3	1.502(2)
C3-C8	1.394(2)	C3-C4	1.401(2)
C4-C5	1.375(2)	C5-O2	1.3699(19)
C5-C6	1.407(2)	C6-O3	1.368(2)
C6-C7	1.381(2)	C7-C8	1.407(2)
C8-C9	1.537(2)	C9-N1	1.4710(19)
C9-C10	1.5529(19)	C10-C16	1.515(2)
C10-C11	1.548(2)	C11-C12	1.530(2)
C12-C13	1.523(2)	C12-C18	1.526(2)
C12-C19	1.530(2)	C13-N1	1.458(2)
C14-O2	1.428(2)	C15-O3	1.424(2)
C16-O1	1.2205(19)	C16-C17	1.499(2)

Table 5. Bond angles ($^{\circ}$) for Belanger_JO4_116.

N2-C20-C21	109.78(12)	C22-C21-C20	111.26(13)
C27-C22-C23	120.14(14)	C27-C22-C21	121.09(14)
C23-C22-C21	118.76(14)	C24-C23-C22	121.74(14)
O5-C24-C23	125.59(14)	O5-C24-C25	115.95(14)
C23-C24-C25	118.46(14)	O6-C25-C26	124.57(14)
O6-C25-C24	115.58(13)	C26-C25-C24	119.85(14)
C25-C26-C27	122.18(14)	C22-C27-C26	117.56(14)
C22-C27-C28	120.97(13)	C26-C27-C28	121.36(13)
N2-C28-C27	109.55(12)	N2-C28-C29	109.97(12)
C27-C28-C29	114.47(12)	C35-C29-C28	114.97(13)
C35-C29-C30	102.78(12)	C28-C29-C30	111.24(12)
C31-C30-C29	113.22(13)	C32-C31-C30	106.51(13)
C32-C31-C38	111.00(13)	C30-C31-C38	111.64(13)
C32-C31-C37	108.94(13)	C30-C31-C37	108.85(13)
C38-C31-C37	109.81(14)	N2-C32-C31	112.57(12)
C24-O5-C33	116.51(13)	C25-O6-C34	116.67(12)
O4-C35-C36	121.94(15)	O4-C35-C29	121.70(14)
C36-C35-C29	116.21(14)	C20-N2-C32	108.85(11)
C20-N2-C28	109.76(12)	C32-N2-C28	111.36(12)
N1-C1-C2	107.71(13)	C3-C2-C1	109.58(13)
C8-C3-C4	120.01(15)	C8-C3-C2	121.35(14)

C4-C3-C2	118.59(14)	C5-C4-C3	121.74(15)
O2-C5-C4	124.96(15)	O2-C5-C6	116.19(15)
C4-C5-C6	118.85(15)	O3-C6-C7	124.60(15)
O3-C6-C5	115.88(14)	C7-C6-C5	119.52(15)
C6-C7-C8	122.04(15)	C3-C8-C7	117.80(14)
C3-C8-C9	121.31(14)	C7-C8-C9	120.82(13)
N1-C9-C8	109.70(12)	N1-C9-C10	107.45(11)
C8-C9-C10	114.02(12)	C16-C10-C11	102.91(12)
C16-C10-C9	115.90(12)	C11-C10-C9	110.87(13)
C12-C11-C10	114.00(13)	C13-C12-C18	111.00(14)
C13-C12-C19	108.28(14)	C18-C12-C19	109.52(14)
C13-C12-C11	106.47(12)	C18-C12-C11	111.45(14)
C19-C12-C11	110.05(14)	N1-C13-C12	111.59(13)
O1-C16-C17	121.29(15)	O1-C16-C10	121.98(14)
C17-C16-C10	116.58(13)	C13-N1-C1	110.66(12)
C13-N1-C9	112.02(12)	C1-N1-C9	111.26(11)
C5-O2-C14	116.43(14)	C6-O3-C15	116.43(13)

Table 6. Torsion angles ($^{\circ}$) for Belanger_JO4_116.

N2-C20-C21-C22	-49.57(18)	C20-C21-C22-C27	19.3(2)
C20-C21-C22-C23	-161.99(14)	C27-C22-C23-C24	0.7(2)
C21-C22-C23-C24	-178.04(14)	C22-C23-C24-O5	-178.82(14)
C22-C23-C24-C25	1.6(2)	O5-C24-C25-O6	-1.1(2)
C23-C24-C25-O6	178.52(13)	O5-C24-C25-C26	178.25(13)
C23-C24-C25-C26	-2.1(2)	O6-C25-C26-C27	179.66(14)
C24-C25-C26-C27	0.4(2)	C23-C22-C27-C26	-2.4(2)
C21-C22-C27-C26	176.30(13)	C23-C22-C27-C28	173.72(13)
C21-C22-C27-C28	-7.5(2)	C25-C26-C27-C22	1.9(2)
C25-C26-C27-C28	-174.23(14)	C22-C27-C28-N2	24.72(18)
C26-C27-C28-N2	-159.26(13)	C22-C27-C28-C29	148.77(14)
C26-C27-C28-C29	-35.21(19)	N2-C28-C29-C35	-167.57(11)
C27-C28-C29-C35	68.60(16)	N2-C28-C29-C30	-51.29(16)
C27-C28-C29-C30	-175.11(12)	C35-C29-C30-C31	175.42(13)
C28-C29-C30-C31	51.87(17)	C29-C30-C31-C32	-53.86(16)
C29-C30-C31-C38	67.45(17)	C29-C30-C31-C37	-171.17(14)
C30-C31-C32-N2	59.82(16)	C38-C31-C32-N2	-61.90(17)
C37-C31-C32-N2	177.07(13)	C23-C24-O5-C33	2.0(2)
C25-C24-O5-C33	-178.38(15)	C26-C25-O6-C34	3.2(2)

C24-C25-O6-C34	-177.46(14)	C28-C29-C35-O4	36.5(2)
C30-C29-C35-O4	-84.48(17)	C28-C29-C35-C36	-147.86(13)
C30-C29-C35-C36	91.11(15)	C21-C20-N2-C32	-167.45(13)
C21-C20-N2-C28	70.43(16)	C31-C32-N2-C20	174.94(13)
C31-C32-N2-C28	-63.92(16)	C27-C28-N2-C20	-55.35(15)
C29-C28-N2-C20	178.01(11)	C27-C28-N2-C32	-175.96(12)
C29-C28-N2-C32	57.40(15)	N1-C1-C2-C3	55.05(16)
C1-C2-C3-C8	-20.09(19)	C1-C2-C3-C4	157.28(13)
C8-C3-C4-C5	0.1(2)	C2-C3-C4-C5	-177.34(14)
C3-C4-C5-O2	-179.93(14)	C3-C4-C5-C6	0.8(2)
O2-C5-C6-O3	0.8(2)	C4-C5-C6-O3	-179.82(13)
O2-C5-C6-C7	-179.43(13)	C4-C5-C6-C7	-0.1(2)
O3-C6-C7-C8	178.21(13)	C5-C6-C7-C8	-1.5(2)
C4-C3-C8-C7	-1.6(2)	C2-C3-C8-C7	175.76(13)
C4-C3-C8-C9	-178.52(13)	C2-C3-C8-C9	-1.2(2)
C6-C7-C8-C3	2.3(2)	C6-C7-C8-C9	179.27(13)
C3-C8-C9-N1	-12.45(18)	C7-C8-C9-N1	170.70(12)
C3-C8-C9-C10	-133.00(14)	C7-C8-C9-C10	50.15(18)
N1-C9-C10-C16	170.05(13)	C8-C9-C10-C16	-68.15(17)
N1-C9-C10-C11	53.25(16)	C8-C9-C10-C11	175.05(12)
C16-C10-C11-C12	-176.26(12)	C9-C10-C11-C12	-51.72(17)
C10-C11-C12-C13	51.89(17)	C10-C11-C12-C18	-69.28(17)
C10-C11-C12-C19	169.02(13)	C18-C12-C13-N1	63.16(18)
C19-C12-C13-N1	-176.61(13)	C11-C12-C13-N1	-58.30(17)
C11-C10-C16-O1	82.14(17)	C9-C10-C16-O1	-39.0(2)
C11-C10-C16-C17	-93.43(16)	C9-C10-C16-C17	145.41(14)
C12-C13-N1-C1	-168.10(13)	C12-C13-N1-C9	67.12(16)
C2-C1-N1-C13	161.35(13)	C2-C1-N1-C9	-73.44(16)
C8-C9-N1-C13	173.69(11)	C10-C9-N1-C13	-61.85(15)
C8-C9-N1-C1	49.24(16)	C10-C9-N1-C1	173.70(13)
C4-C5-O2-C14	9.6(2)	C6-C5-O2-C14	-171.13(15)
C7-C6-O3-C15	3.0(2)	C5-C6-O3-C15	-177.26(14)

Table 7. Anisotropic atomic displacement parameters (\AA^2) for Belanger_JO4_116.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h \cdot k \cdot a^* b^* U_{12}]$

	U₁₁	U₂₂	U₃₃	U₂₃	U₁₃	U₁₂
C20	0.0272(8)	0.0288(8)	0.0380(9)	-0.0069(6)	-0.0012(6)	-0.0054(6)
C21	0.0275(8)	0.0387(9)	0.0312(8)	-0.0093(7)	-0.0028(6)	-0.0039(7)
C22	0.0274(8)	0.0246(7)	0.0280(8)	-0.0045(6)	-0.0023(6)	-0.0016(6)
C23	0.0316(8)	0.0292(7)	0.0255(8)	-0.0054(6)	-0.0038(6)	-0.0023(6)
C24	0.0319(8)	0.0251(7)	0.0270(8)	-0.0028(6)	0.0017(6)	-0.0026(6)
C25	0.0255(8)	0.0250(7)	0.0326(8)	-0.0025(6)	-0.0011(6)	-0.0042(6)
C26	0.0296(8)	0.0262(7)	0.0255(7)	-0.0007(6)	-0.0045(6)	-0.0019(6)
C27	0.0273(8)	0.0216(7)	0.0266(8)	-0.0018(5)	-0.0024(6)	0.0001(6)
C28	0.0254(8)	0.0236(7)	0.0262(7)	0.0000(6)	-0.0022(6)	-0.0002(6)
C29	0.0287(8)	0.0247(7)	0.0254(7)	0.0002(6)	-0.0008(6)	-0.0008(6)
C30	0.0348(9)	0.0324(8)	0.0253(8)	0.0007(6)	-0.0010(6)	0.0004(6)
C31	0.0313(8)	0.0316(8)	0.0266(8)	0.0034(6)	0.0025(6)	-0.0014(6)
C32	0.0299(8)	0.0262(7)	0.0328(8)	0.0039(6)	0.0024(6)	-0.0049(6)
O5	0.0354(6)	0.0449(7)	0.0270(6)	-0.0072(5)	0.0056(5)	-0.0120(5)
O6	0.0284(6)	0.0482(7)	0.0351(6)	-0.0073(5)	0.0000(5)	-0.0131(5)
C35	0.0319(8)	0.0359(8)	0.0204(7)	-0.0013(6)	-0.0006(6)	-0.0023(7)
C36	0.0385(9)	0.0455(10)	0.0340(9)	-0.0055(7)	-0.0067(7)	-0.0086(8)
C37	0.0428(10)	0.0450(10)	0.0330(9)	0.0052(7)	0.0084(7)	-0.0052(8)
C38	0.0336(9)	0.0339(8)	0.0336(8)	-0.0023(7)	0.0027(7)	0.0023(7)
N2	0.0250(6)	0.0266(6)	0.0271(6)	-0.0010(5)	0.0003(5)	-0.0038(5)
O4	0.0404(7)	0.0417(7)	0.0351(6)	-0.0042(5)	-0.0097(5)	0.0086(5)
C33	0.0505(11)	0.0642(12)	0.0260(9)	-0.0096(8)	0.0054(8)	-0.0196(9)
C34	0.0286(9)	0.0482(10)	0.0446(10)	-0.0136(8)	-0.0055(7)	-0.0083(7)
C1	0.0314(8)	0.0350(8)	0.0309(8)	-0.0023(6)	-0.0026(6)	-0.0106(7)
C2	0.0253(8)	0.0377(8)	0.0355(9)	-0.0004(7)	-0.0042(6)	-0.0057(6)
C3	0.0269(8)	0.0233(7)	0.0333(8)	0.0014(6)	-0.0027(6)	-0.0024(6)
C4	0.0243(7)	0.0276(8)	0.0364(8)	0.0010(6)	-0.0005(6)	-0.0013(6)
C5	0.0307(8)	0.0250(7)	0.0324(8)	-0.0015(6)	0.0029(6)	0.0011(6)
C6	0.0335(8)	0.0262(7)	0.0306(8)	-0.0007(6)	-0.0044(6)	-0.0029(6)
C7	0.0256(8)	0.0285(7)	0.0326(8)	-0.0004(6)	-0.0025(6)	-0.0025(6)
C8	0.0262(8)	0.0213(7)	0.0298(8)	0.0014(6)	-0.0037(6)	-0.0028(6)
C9	0.0252(7)	0.0241(7)	0.0275(8)	0.0003(6)	-0.0027(6)	-0.0014(6)
C10	0.0254(7)	0.0253(7)	0.0314(8)	0.0000(6)	-0.0017(6)	-0.0011(6)

C11	0.0271(8)	0.0363(8)	0.0329(8)	0.0002(7)	-0.0004(6)	-0.0028(6)
C12	0.0286(8)	0.0338(8)	0.0287(8)	0.0016(6)	-0.0001(6)	-0.0017(6)
C13	0.0314(8)	0.0321(8)	0.0277(8)	-0.0038(6)	-0.0014(6)	-0.0012(6)
C14	0.0322(10)	0.0599(12)	0.0540(12)	-0.0183(9)	0.0092(8)	0.0026(8)
C15	0.0383(10)	0.0484(10)	0.0391(9)	-0.0107(8)	-0.0109(8)	-0.0025(8)
C16	0.0222(7)	0.0324(8)	0.0313(8)	-0.0027(6)	0.0030(6)	-0.0007(6)
C17	0.0328(9)	0.0361(9)	0.0492(10)	-0.0093(7)	-0.0075(8)	-0.0023(7)
C18	0.0452(10)	0.0361(9)	0.0374(9)	0.0065(7)	-0.0056(8)	-0.0031(8)
C19	0.0355(9)	0.0547(11)	0.0324(9)	-0.0029(8)	0.0022(7)	0.0002(8)
N1	0.0264(7)	0.0311(7)	0.0270(6)	-0.0020(5)	-0.0018(5)	-0.0057(5)
O1	0.0291(6)	0.0362(6)	0.0422(7)	0.0043(5)	-0.0065(5)	0.0001(5)
O2	0.0344(6)	0.0449(7)	0.0362(6)	-0.0092(5)	0.0048(5)	0.0035(5)
O3	0.0358(6)	0.0452(7)	0.0333(6)	-0.0105(5)	-0.0043(5)	-0.0019(5)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (\AA^2) for Belanger_JO4_116.

	x/a	y/b	z/c	U(eq)
H9	0.3663(18)	0.5638(17)	0.2723(8)	0.023(4)
H29	1.4648(19)	0.7770(18)	0.1864(9)	0.030(4)
H28	1.4323(19)	1.0657(18)	0.2099(9)	0.028(4)
H20A	1.5923	1.1403	0.2802	0.037
H20B	1.7487	1.0591	0.2921	0.037
H21A	1.6398	0.8985	0.3786	0.038
H21B	1.5742	1.0469	0.3973	0.038
H23	1.3649	0.9256	0.4669	0.034
H26	1.1941	0.8641	0.2588	0.033
H30A	1.5454	0.8553	0.0688	0.038
H30B	1.5214	1.0139	0.0806	0.038
H32A	1.8186	1.0135	0.1832	0.037
H32B	1.6794	1.1054	0.1555	0.037
H36A	1.1480	0.8059	0.0972	0.058
H36B	1.2163	0.7079	0.1602	0.058
H36C	1.3139	0.7397	0.0858	0.058
H37A	1.7670	1.0765	0.0358	0.063
H37B	1.7991	0.9266	0.0118	0.063
H37C	1.9089	0.9816	0.0581	0.063

H38A	1.8803	0.7749	0.1467	0.052
H38B	1.7680	0.7140	0.1038	0.052
H38C	1.7201	0.7346	0.1841	0.052
H33A	1.2254	0.8092	0.5592	0.07
H33B	1.0581	0.8509	0.5881	0.07
H33C	1.1620	0.9666	0.5459	0.07
H34A	0.9508	0.8631	0.2725	0.059
H34B	0.8470	0.7499	0.3170	0.059
H34C	1.0131	0.7056	0.2874	0.059
H1A	0.6719	0.5732	0.3662	0.039
H1B	0.6034	0.6438	0.2963	0.039
H2A	0.8311	0.5281	0.2629	0.04
H2B	0.7796	0.3878	0.3097	0.04
H4	0.8756	0.4041	0.1606	0.036
H7	0.3661	0.4104	0.1586	0.035
H10	0.3406	0.2737	0.3174	0.033
H11A	0.1410	0.4935	0.3648	0.039
H11B	0.1306	0.3327	0.3931	0.039
H13A	0.3597	0.6126	0.3880	0.037
H13B	0.4718	0.5337	0.4398	0.037
H14A	0.9970	0.4060	0.0547	0.074
H14B	1.0072	0.2957	0.0009	0.074
H14C	0.9767	0.2464	0.0835	0.074
H15A	0.3159	0.2823	0.0758	0.061
H15B	0.3403	0.3345	-0.0067	0.061
H15C	0.3189	0.4440	0.0479	0.061
H17A	0.0156	0.2507	0.2436	0.058
H17B	0.1197	0.1766	0.3003	0.058
H17C	0.1780	0.1889	0.2184	0.058
H18A	0.4296	0.2441	0.4273	0.061
H18B	0.2860	0.2182	0.4829	0.061
H18C	0.4199	0.2951	0.5025	0.061
H19A	0.1241	0.4165	0.5180	0.063
H19B	0.1388	0.5670	0.4741	0.063
H19C	0.2559	0.5076	0.5275	0.063

ANNEXE 9: COORDONNÉES DE DIFFRACTION DES RAYONS-X DU COMPOSÉ 2-24

A Plate-like specimen of C₁₅H₁₉NO₃, approximate dimensions 0.040 mm x 0.350 mm x 0.430 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker Apex DUO system equipped with a Cu K α ImuS micro-focus source with MX optics ($\lambda = 1.54186 \text{ \AA}$).

The total exposure time was 4.80 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 7185 reflections to a maximum θ angle of 70.88° (0.82 Å resolution), of which 2520 were independent (average redundancy 2.851, completeness = 98.2%, R_{int} = 5.81%, R_{sig} = 6.09%) and 2044 (81.11%) were greater than 2 $\sigma(F^2)$. The final cell constants of $a = 12.9004(2) \text{ \AA}$, $b = 7.78420(10) \text{ \AA}$, $c = 13.6850(3) \text{ \AA}$, $\beta = 104.8200(10)^\circ$, volume = 1328.52(4) Å³, are based upon the refinement of the XYZ-centroids of 9878 reflections above 20 $\sigma(I)$ with $8.408^\circ < 2\theta < 141.7^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.659. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7424 and 0.9711.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1 21/n 1, with Z = 4 for the formula unit, C₁₅H₁₉NO₃. The final anisotropic full-matrix least-squares refinement on F² with 187 variables converged at R1 = 4.80%, for the observed data and wR2 = 13.67% for all data. The goodness-of-fit was 1.040. The largest peak in the final difference electron density synthesis was 0.228 e⁻/Å³ and the largest hole was -0.236 e⁻/Å³ with an RMS deviation of 0.053 e⁻/Å³. On the basis of the final model, the calculated density was 1.306 g/cm³ and F(000), 560 e⁻.

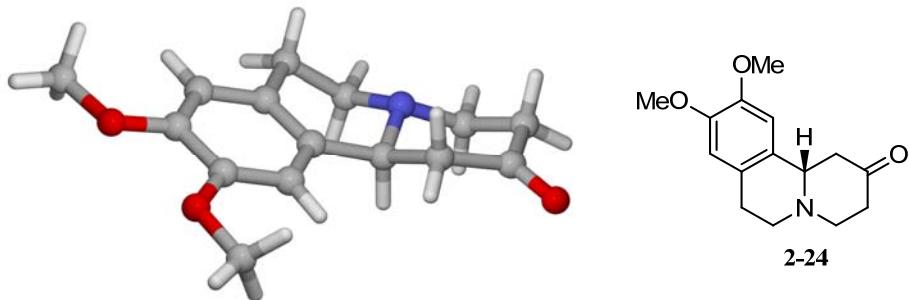


Table 1. Sample and crystal data for Belanger_JO5_047.

Identification code	Blanger_JO5_047
Chemical formula	C ₁₅ H ₁₉ NO ₃
Formula weight	261.31

Temperature	173(2) K	
Wavelength	1.54186 Å	
Crystal size	0.040 x 0.350 x 0.430 mm	
Crystal system	monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 12.9004(2) Å b = 7.78420(10) Å c = 13.6850(3) Å	α = 90° β = 104.8200(10)° γ = 90°
Volume	1328.52(4) Å ³	
Z	4	
Density (calculated)	1.306 g/cm ³	
Absorption coefficient	0.736 mm ⁻¹	
F(000)	560	

Table 2. Data collection and structure refinement for Blanger_JO5_047.

Diffractometer	Bruker Apex DUO
Radiation source	ImuS micro—focus source with MX optics, Cu K α
Theta range for data collection	4.20 to 70.88°
Index ranges	-14<=h<=15, -9<=k<=9, -13<=l<=16
Reflections collected	7185
Independent reflections	2520 [R(int) = 0.0581]
Coverage of independent reflections	98.2%
Absorption correction	multi-scan
Max. and min. transmission	0.9711 and 0.7424
Structure solution technique	direct methods
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Refinement method	Full-matrix least-squares on F ²
Refinement program	SHELXL-97 (Sheldrick, 2008)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	2520 / 0 / 187
Goodness-of-fit on F²	1.040
Δ/σ_{max}	0.001
Final R indices	2044 data; I>2σ(I) R1 = 0.0480, wR2 = 0.1295 all data R1 = 0.0580, wR2 = 0.1367
Weighting scheme	w=1/[σ ² (F _o ²)+(0.0805P) ² +0.0000P] where P=(F _o ² +2F _c ²)/3

Extinction coefficient	0.0044(8)
Largest diff. peak and hole	0.228 and -0.236 eÅ ⁻³
R.M.S. deviation from mean	0.053 eÅ ⁻³

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for Blanger_JO5_047.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
C1	0.40882(12)	0.0986(2)	0.56845(11)	0.0312(4)
C2	0.50763(13)	0.1812(2)	0.63751(12)	0.0347(4)
C3	0.51247(12)	0.1579(2)	0.74746(12)	0.0307(4)
C4	0.40740(12)	0.1756(2)	0.77546(10)	0.0271(4)
C5	0.31377(11)	0.08871(19)	0.69843(10)	0.0237(3)
C6	0.20752(11)	0.11983(18)	0.72480(10)	0.0221(3)
C7	0.20147(11)	0.09619(19)	0.82469(10)	0.0229(3)
C8	0.10662(11)	0.11907(19)	0.85208(10)	0.0236(3)
C9	0.01331(12)	0.16576(19)	0.77791(11)	0.0271(4)
C10	0.01885(12)	0.1869(2)	0.67938(10)	0.0284(4)
C11	0.11510(12)	0.16329(18)	0.65130(10)	0.0250(3)
C12	0.11745(12)	0.1826(2)	0.54221(10)	0.0297(4)
C13	0.21494(12)	0.0935(2)	0.52283(10)	0.0308(4)
C14	0.18447(12)	0.0452(2)	0.02343(10)	0.0306(4)
C15	0.82603(12)	0.2227(3)	0.74025(12)	0.0431(5)
N1	0.31142(10)	0.15261(16)	0.59704(8)	0.0252(3)
O1	0.59581(9)	0.12930(19)	0.81101(8)	0.0476(4)
O2	0.09318(8)	0.09844(15)	0.94703(6)	0.0297(3)
O3	0.92362(8)	0.18390(16)	0.81305(8)	0.0376(3)

Table 4. Bond lengths (Å) for Blanger_JO5_047.

C1-N1	1.4699(18)	C1-C2	1.522(2)
C2-C3	1.501(2)	C3-O1	1.2179(19)
C3-C4	1.506(2)	C4-C5	1.5413(19)
C5-N1	1.4670(17)	C5-C6	1.5234(19)
C6-C11	1.3908(19)	C6-C7	1.4011(19)
C7-C8	1.3799(19)	C8-O2	1.3636(16)
C8-C9	1.409(2)	C9-O3	1.3691(18)
C9-C10	1.378(2)	C10-C11	1.403(2)
C11-C12	1.5085(18)	C12-C13	1.518(2)

C13-N1	1.4648(18)	C14-O2	1.4220(16)
C15-O3	1.4238(17)		

Table 5. Bond angles ($^{\circ}$) for Blanger_JO5_047.

N1-C1-C2	110.55(12)	C3-C2-C1	112.65(13)
O1-C3-C2	122.74(14)	O1-C3-C4	121.49(14)
C2-C3-C4	115.76(13)	C3-C4-C5	112.62(12)
N1-C5-C6	111.59(11)	N1-C5-C4	108.73(11)
C6-C5-C4	111.31(11)	C11-C6-C7	119.09(13)
C11-C6-C5	121.61(12)	C7-C6-C5	119.23(12)
C8-C7-C6	121.51(12)	O2-C8-C7	125.54(12)
O2-C8-C9	115.04(12)	C7-C8-C9	119.41(12)
O3-C9-C10	126.24(13)	O3-C9-C8	114.61(12)
C10-C9-C8	119.14(13)	C9-C10-C11	121.56(13)
C6-C11-C10	119.27(12)	C6-C11-C12	120.63(13)
C10-C11-C12	120.09(13)	C11-C12-C13	110.77(12)
N1-C13-C12	109.30(11)	C13-N1-C5	110.96(11)
C13-N1-C1	110.99(11)	C5-N1-C1	110.03(11)
C8-O2-C14	116.86(10)	C9-O3-C15	116.84(11)

Table 6. Torsion angles ($^{\circ}$) for Blanger_JO5_047.

N1-C1-C2-C3	50.62(18)	C1-C2-C3-O1	141.12(16)
C1-C2-C3-C4	-40.07(19)	O1-C3-C4-C5	-139.84(16)
C2-C3-C4-C5	41.34(19)	C3-C4-C5-N1	-52.60(16)
C3-C4-C5-C6	-175.92(12)	N1-C5-C6-C11	14.70(19)
C4-C5-C6-C11	136.37(14)	N1-C5-C6-C7	-168.38(12)
C4-C5-C6-C7	-46.70(18)	C11-C6-C7-C8	-1.6(2)
C5-C6-C7-C8	-178.58(13)	C6-C7-C8-O2	179.78(12)
C6-C7-C8-C9	0.6(2)	O2-C8-C9-O3	0.3(2)
C7-C8-C9-O3	179.56(13)	O2-C8-C9-C10	-179.03(13)
C7-C8-C9-C10	0.2(2)	O3-C9-C10-C11	-179.30(14)
C8-C9-C10-C11	-0.1(2)	C7-C6-C11-C10	1.7(2)
C5-C6-C11-C10	178.64(13)	C7-C6-C11-C12	-177.45(13)
C5-C6-C11-C12	-0.5(2)	C9-C10-C11-C6	-0.9(2)
C9-C10-C11-C12	178.25(13)	C6-C11-C12-C13	19.0(2)
C10-C11-C12-C13	-160.20(14)	C11-C12-C13-N1	-51.98(16)
C12-C13-N1-C5	69.34(15)	C12-C13-N1-C1	-168.00(12)
C6-C5-N1-C13	-48.63(16)	C4-C5-N1-C13	-171.78(12)
C6-C5-N1-C1	-171.84(11)	C4-C5-N1-C1	65.00(15)

C2-C1-N1-C13	172.08(12)	C2-C1-N1-C5	-64.71(16)
C7-C8-O2-C14	-1.4(2)	C9-C8-O2-C14	177.77(13)
C10-C9-O3-C15	2.2(2)	C8-C9-O3-C15	-177.01(14)

Table 7. Anisotropic atomic displacement parameters (\AA^2) for Blanger_JO5_047.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + \dots + 2 h k a^{*} b^{*} U_{12}]$

	U₁₁	U₂₂	U₃₃	U₂₃	U₁₃	U₁₂
C1	0.0328(9)	0.0385(9)	0.0269(7)	-0.0014(6)	0.0159(6)	0.0031(7)
C2	0.0283(8)	0.0406(9)	0.0402(9)	0.0001(7)	0.0178(7)	0.0004(7)
C3	0.0238(8)	0.0344(9)	0.0356(8)	-0.0045(6)	0.0110(6)	-0.0023(6)
C4	0.0216(8)	0.0355(9)	0.0251(7)	-0.0013(6)	0.0074(6)	-0.0004(6)
C5	0.0242(8)	0.0273(8)	0.0209(7)	-0.0003(6)	0.0080(6)	0.0002(6)
C6	0.0217(7)	0.0222(7)	0.0225(7)	-0.0006(5)	0.0060(6)	-0.0024(5)
C7	0.0207(7)	0.0266(8)	0.0213(7)	0.0011(6)	0.0051(6)	0.0005(6)
C8	0.0233(7)	0.0275(8)	0.0204(6)	0.0030(6)	0.0064(5)	-0.0011(6)
C9	0.0193(7)	0.0357(9)	0.0276(7)	0.0032(6)	0.0082(6)	-0.0014(6)
C10	0.0202(8)	0.0398(9)	0.0229(7)	0.0052(6)	0.0012(6)	-0.0034(6)
C11	0.0250(8)	0.0291(8)	0.0204(7)	0.0004(5)	0.0049(6)	-0.0054(6)
C12	0.0267(8)	0.0407(9)	0.0196(7)	0.0006(6)	0.0024(6)	-0.0067(7)
C13	0.0354(9)	0.0363(9)	0.0211(7)	-0.0035(6)	0.0078(6)	-0.0052(7)
C14	0.0269(8)	0.0436(9)	0.0200(7)	0.0030(6)	0.0037(6)	0.0030(6)
C15	0.0176(8)	0.0709(13)	0.0384(8)	0.0095(8)	0.0028(7)	0.0024(8)
N1	0.0245(7)	0.0332(7)	0.0195(6)	-0.0006(5)	0.0086(5)	-0.0004(5)
O1	0.0208(6)	0.0796(10)	0.0414(7)	-0.0057(6)	0.0064(5)	0.0004(6)
O2	0.0250(6)	0.0452(7)	0.0206(5)	0.0060(4)	0.0087(4)	0.0055(4)
O3	0.0173(6)	0.0663(8)	0.0305(6)	0.0111(5)	0.0083(4)	0.0048(5)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (\AA^2) for Blanger_JO5_047.

	x/a	y/b	z/c	U(eq)
H5	0.3289(12)	-0.040(2)	0.7031(11)	0.026(4)
H7	0.2628(14)	0.057(2)	0.8769(11)	0.031(4)
H10	-0.0448(15)	0.223(2)	0.6271(12)	0.038(5)
H1A	0.4034	0.1322	0.4976	0.037

H1B	0.4156	-0.0280	0.5732	0.037
H2A	0.5726	0.1303	0.6234	0.042
H2B	0.5075	0.3055	0.6223	0.042
H4A	0.3910	0.2991	0.7801	0.033
H4B	0.4144	0.1236	0.8429	0.033
H12A	0.1193	0.3061	0.5255	0.036
H12B	0.0515	0.1323	0.4980	0.036
H13A	0.2076	-0.0324	0.5284	0.037
H13B	0.2207	0.1202	0.4537	0.037
H14A	0.2106	-0.0647	1.0041	0.046
H14B	0.1647	0.0316	1.0875	0.046
H14C	0.2411	0.1320	1.0313	0.046
H15A	-0.1679	0.3351	0.7099	0.065
H15B	-0.2330	0.2253	0.7733	0.065
H15C	-0.1883	0.1345	0.6874	0.065

ANNEXE 10: COORDONNÉES DE DIFFRACTION DES RAYONS-X DU COMPOSÉ 3-51

A Newedle-like specimen of C₁₃H₁₈N₂O, approximate dimensions 0.020 mm x 0.120 mm x 0.360 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker Apex DUO system equipped with a Cu K α ImuS micro-focus source with MX optics ($\lambda = 1.54186 \text{ \AA}$).

The total exposure time was 7.20 hours. The frames were integrated with the Bruker SAINT software package using a wide-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 6120 reflections to a maximum θ angle of 70.65° (0.82 Å resolution), of which 2185 were independent (average redundancy 2.801, completeness = 98.9%, R_{int} = 7.83%, R_{sig} = 6.91%) and 1628 (74.51%) were greater than 2 $\sigma(F^2)$. The final cell constants of $a = 9.6553(2) \text{ \AA}$, $b = 11.4000(3) \text{ \AA}$, $c = 11.0500(3) \text{ \AA}$, $\beta = 109.0350(10)^\circ$, volume = 1149.77(5) Å³, are based upon the refinement of the XYZ-centroids of 7339 reflections above 20 $\sigma(I)$ with 9.690° < 2θ < 141.2°. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.645. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.8032 and 0.9874.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P 1 21/c 1, with Z = 4 for the formula unit, C₁₃H₁₈N₂O. The final anisotropic full-matrix least-squares refinement on F² with 154 variables converged at R1 = 5.03%, for the observed data and wR2 = 14.91% for all data. The goodness-of-fit was 1.012. The largest peak in the final difference electron density synthesis was 0.185 e⁻/Å³ and the largest hole was -0.246 e⁻/Å³ with an RMS deviation of 0.053 e⁻/Å³. On the basis of the final model, the calculated density was 1.261 g/cm³ and F(000), 472 e⁻.

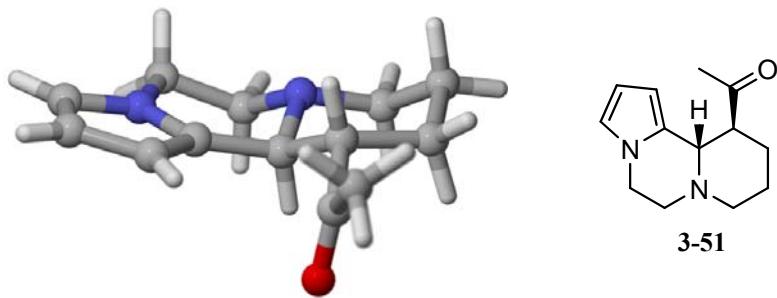


Table 1. Sample and crystal data for Belanger_JO5_129.

Identification code	Belanger_JO5_129
Chemical formula	C ₁₃ H ₁₈ N ₂ O
Formula weight	218.29 g/mol

Temperature	173(2) K	
Wavelength	1.54186 Å	
Crystal size	0.020 x 0.120 x 0.360 mm	
Crystal system	monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 9.6553(2) Å b = 11.4000(3) Å c = 11.0500(3) Å	α = 90° β = 109.0350(10)° γ = 90°
Volume	1149.77(5) Å ³	
Z	4	
Density (calculated)	1.261 g/cm ³	
Absorption coefficient	0.637 mm ⁻¹	
F(000)	472	

Table 2. Data collection and structure refinement for Belanger_JO5_129.

Diffractometer	Bruker Apex DUO
Radiation source	ImuS micro—focus source with MX optics, Cu K α
Theta range for data collection	4.85 to 70.65°
Index ranges	-8<=h<=11, -13<=k<=13, -13<=l<=12
Reflections collected	6120
Independent reflections	2185 [R(int) = 0.0783]
Coverage of independent reflections	98.9%
Absorption correction	multi-scan
Max. and min. transmission	0.9874 and 0.8032
Structure solution technique	direct methods
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Refinement method	Full-matrix least-squares on F ²
Refinement program	SHELXL-97 (Sheldrick, 2008)
Function minimized	Σ w(F _o ² - F _c ²) ²
Data / restraints / parameters	2185 / 0 / 154
Goodness-of-fit on F²	1.012
Δ/σ_{max}	0.016

Final R indices	1628 data; I>2σ(I)	R1 = 0.0503, wR2 = 0.1331
	all data	R1 = 0.0685, wR2 = 0.1491
Weighting scheme	w=1/[$\sigma^2(F_o^2)+(0.0854P)^2+0.0000P$] where P=($F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	0.185 and -0.246 eÅ ⁻³	
R.M.S. deviation from mean	0.053 eÅ ⁻³	

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for Belanger_JO5_129.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

x/a	y/b	z/c	U(eq)
C1 0.67776(17)	0.40763(16)	0.13620(17)	0.0263(4)
C2 0.80587(16)	0.47128(15)	0.11043(17)	0.0251(4)
C3 0.76638(16)	0.50970(16)	0.97399(18)	0.0285(4)
C4 0.67559(18)	0.46311(17)	0.85999(18)	0.0319(4)
C5 0.67867(19)	0.53857(18)	0.75995(19)	0.0356(5)
C6 0.77054(19)	0.62942(18)	0.81373(19)	0.0366(5)
C7 0.91698(18)	0.69220(17)	0.03756(19)	0.0344(5)
C8 0.97315(17)	0.63206(17)	0.16558(18)	0.0327(4)
C9 0.89565(19)	0.53995(17)	0.32896(18)	0.0327(4)
C10 0.76500(19)	0.49134(17)	0.35964(19)	0.0337(5)
C11 0.70868(18)	0.38324(16)	0.27968(17)	0.0307(4)
C12 0.64613(18)	0.28903(16)	0.06999(18)	0.0302(4)
C13 0.4902(2)	0.2480(2)	0.0302(2)	0.0446(5)
N1 0.85159(14)	0.57475(13)	0.19456(14)	0.0277(4)
N2 0.82472(14)	0.61131(14)	0.94281(15)	0.0312(4)
O1 0.74309(13)	0.22897(12)	0.05591(14)	0.0395(4)

Table 4. Bond lengths (Å) for Belanger_JO5_129.

C1-C12	1.520(2)	C1-C2	1.539(2)
C1-C11	1.540(2)	C2-N1	1.477(2)
C2-C3	1.495(3)	C3-N2	1.380(2)
C3-C4	1.385(3)	C4-C5	1.409(3)
C5-C6	1.367(3)	C6-N2	1.365(2)

C7-N2	1.460(2)	C7-C8	1.505(3)
C8-N1	1.467(2)	C9-N1	1.460(2)
C9-C10	1.514(2)	C10-C11	1.510(3)
C12-O1	1.210(2)	C12-C13	1.499(2)

Table 5. Bond angles ($^{\circ}$) for Belanger_JO5_129.

C12-C1-C2	112.20(14)	C12-C1-C11	106.02(15)
C2-C1-C11	112.22(14)	N1-C2-C3	109.04(15)
N1-C2-C1	110.69(14)	C3-C2-C1	111.64(13)
N2-C3-C4	106.48(16)	N2-C3-C2	120.47(16)
C4-C3-C2	133.05(17)	C3-C4-C5	108.09(17)
C6-C5-C4	107.36(18)	N2-C6-C5	108.23(17)
N2-C7-C8	109.57(15)	N1-C8-C7	109.84(13)
N1-C9-C10	109.85(14)	C11-C10-C9	109.57(15)
C10-C11-C1	111.52(15)	O1-C12-C13	122.07(17)
O1-C12-C1	121.51(15)	C13-C12-C1	116.32(15)
C9-N1-C8	110.77(13)	C9-N1-C2	110.56(14)
C8-N1-C2	108.32(14)	C6-N2-C3	109.83(15)
C6-N2-C7	126.15(16)	C3-N2-C7	123.67(15)

Table 6. Torsion angles ($^{\circ}$) for Belanger_JO5_129.

C12-C1-C2-N1	-169.39(14)	C11-C1-C2-N1	-50.14(18)
C12-C1-C2-C3	68.94(19)	C11-C1-C2-C3	-171.81(14)
N1-C2-C3-N2	24.57(19)	C1-C2-C3-N2	147.18(15)
N1-C2-C3-C4	-155.11(18)	C1-C2-C3-C4	-32.5(3)
N2-C3-C4-C5	-0.65(19)	C2-C3-C4-C5	179.06(16)
C3-C4-C5-C6	0.0(2)	C4-C5-C6-N2	0.6(2)
N2-C7-C8-N1	-48.9(2)	N1-C9-C10-C11	61.94(19)
C9-C10-C11-C1	-53.32(19)	C12-C1-C11-C10	171.10(14)
C2-C1-C11-C10	48.30(19)	C2-C1-C12-O1	33.7(2)
C11-C1-C12-O1	-89.1(2)	C2-C1-C12-C13	-149.68(17)
C11-C1-C12-C13	87.50(19)	C10-C9-N1-C8	174.78(14)
C10-C9-N1-C2	-65.13(18)	C7-C8-N1-C9	-166.62(15)
C7-C8-N1-C2	71.96(18)	C3-C2-N1-C9	-178.14(12)
C1-C2-N1-C9	58.67(17)	C3-C2-N1-C8	-56.59(16)
C1-C2-N1-C8	-179.78(13)	C5-C6-N2-C3	-1.07(19)

C5-C6-N2-C7	-174.45(15)	C4-C3-N2-C6	1.06(19)
C2-C3-N2-C6	-178.69(14)	C4-C3-N2-C7	174.64(14)
C2-C3-N2-C7	-5.1(2)	C8-C7-N2-C6	-170.86(15)
C8-C7-N2-C3	16.6(2)		

Table 7. Anisotropic atomic displacement parameters (\AA^2) for Belanger_JO5_129.

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h \cdot k \cdot a^* b^* U_{12}]$

	U₁₁	U₂₂	U₃₃	U₂₃	U₁₃	U₁₂
C1	0.0234(7)	0.0270(10)	0.0268(10)	0.0004(7)	0.0056(7)	0.0018(7)
C2	0.0220(7)	0.0259(9)	0.0251(10)	-0.0005(7)	0.0046(7)	0.0031(7)
C3	0.0242(7)	0.0305(10)	0.0303(11)	0.0015(8)	0.0081(7)	0.0016(7)
C4	0.0289(8)	0.0354(11)	0.0291(11)	0.0018(8)	0.0065(7)	-0.0010(7)
C5	0.0326(8)	0.0447(12)	0.0259(11)	0.0036(9)	0.0048(8)	0.0026(8)
C6	0.0365(9)	0.0407(12)	0.0329(11)	0.0092(9)	0.0115(8)	0.0031(8)
C7	0.0327(8)	0.0322(10)	0.0385(12)	0.0001(9)	0.0118(8)	-0.0050(8)
C8	0.0260(8)	0.0336(10)	0.0367(12)	-0.0012(8)	0.0078(8)	-0.0032(7)
C9	0.0346(9)	0.0317(11)	0.0259(11)	-0.0035(8)	0.0018(8)	-0.0002(8)
C10	0.0408(9)	0.0328(11)	0.0263(11)	-0.0003(8)	0.0094(8)	0.0015(8)
C11	0.0323(8)	0.0298(10)	0.0289(11)	0.0023(8)	0.0084(7)	0.0017(7)
C12	0.0349(8)	0.0279(10)	0.0258(10)	-0.0012(7)	0.0072(7)	-0.0001(7)
C13	0.0405(10)	0.0405(12)	0.0491(13)	-0.0083(10)	0.0094(9)	-0.0114(9)
N1	0.0269(7)	0.0266(8)	0.0275(9)	-0.0004(6)	0.0058(6)	-0.0015(6)
N2	0.0279(7)	0.0324(9)	0.0321(9)	0.0032(7)	0.0082(6)	-0.0008(6)
O1	0.0448(7)	0.0317(8)	0.0410(9)	-0.0052(6)	0.0128(6)	0.0057(6)

Table 8. Hydrogen atomic coordinates and isotropic atomic displacement parameters (\AA^2) for Belanger_JO_129.

	x/a	y/b	z/c	U(eq)
H4	0.6208	0.3925	-0.1492	0.038
H5	0.6265	0.5282	-0.3285	0.043
H6	0.7928	0.6940	-0.2310	0.044
H7A	0.8595	0.7624	0.0441	0.041
H7B	1.0005	0.7179	0.0107	0.041
H8A	1.0477	0.5729	0.1642	0.039
H8B	1.0200	0.6903	0.2329	0.039

H9A	0.9355	0.6087	0.3841	0.039
H9B	0.9734	0.4796	0.3463	0.039
H10A	0.7940	0.4711	0.4517	0.04
H10B	0.6867	0.5513	0.3413	0.04
H11A	0.6175	0.3564	0.2935	0.037
H11B	0.7821	0.3197	0.3075	0.037
H13A	0.4861	0.1641	0.0102	0.067
H13B	0.4320	0.2917	-0.0458	0.067
H13C	0.4505	0.2616	0.1001	0.067
H1	0.5860(18)	0.4566(16)	0.1048(18)	0.025(5)
H2	0.8909(18)	0.4181(16)	0.1322(17)	0.020(4)

RÉFÉRENCES ET NOTES POUR LA PARTIE EXPÉRIMENTALE

- 93 Anderson, A. G.; Stang, P. J. *Org. Synth.* **1981**, *60*, 34.
- 94 Selander, N.; Paasch, J. R.; Szabo, K. J. *J. Am. Chem. Soc.* **2011**, *133*, 409.
- 95 Saito, T.; Nishimoto, Y.; Yasuda, M.; Baba, A. *J. Org. Chem.* **2006**, *71*, 8516-8522.
- 96 Niggemann, M.; Jelonek, A.; Biber, N.; Wuchrer, M.; Plietker, B. *J. Org. Chem.* **2008**, *73*, 7028-7036.
- 97 Paquette, L. A.; Mendez-Andino, J. L. *J. Org. Chem.* **1998**, *63*, 9061-9068.
- 98 Tietze, L. F.; Kahle, K.; Raschke, T. *Chem. Eur. J.* **2002**, *8*, 401-407.
- 99 Vohra, R.; Maclean, D. B. *Can. J. Chem.* **1994**, *72*, 1660-1667.
- 100 Evans, D. M.; Horton, P. N.; Hursthouse, M. B.; Murphy, P. J. *RSC Adv.* **2014**, *4*, 20744-20751.
- 101 Dutta, A. K.; Xu, C.; Reith, M. E. A. *J. Med. Chem.* **1996**, *39*, 749-756.
- 102 Crestey, F.; Jensen, A. A.; Borch, M.; Andreasen, J. T.; Andersen, J.; Balle, T.; Kristensen, J. L. *J. Med. Chem.* **2013**, *56*, 9673-9682.
- 103 Holzapfel, C.; Dasilva, E.; Den Drijver, L.; Bredenkamp, T. *ChemCatChem* **2016**, *8*, 2912-2915.
- 104 Wang, H.; Liu, D.; Chen, H.; Li, J.; Wang, D. Z. *Tetrahedron* **2015**, *71*, 7073-7076.
- 105 Ameer, F.; Giles, R. G. F.; Green, I. R.; Nagabhushana, K. S. *Synth. Comm.* **2002**, *32*, 369-380.
- 106 Wu, Y.; Ahlberg, P. *Synthesis* **1994**, 463-464.
- 107 Mahale, S.; Bharate, S. B.; Manda, S.; Joshi, P.; Bharate, S. S.; Jenkins, P. R.; Vishwakarma, R. A.; Chaudhuri, B. *J. Med. Chem.* **2014**, *57*, 9658-9672.
- 108 Lindel, T.; Bra1uchle, L.; Golz, G.; Bolhrer, P. *Org. Lett.* **2007**, *9*, 283-286.
- 109 Mecerreyes, D.; Pomposo, J. A.; Bengoetxea, M.; Grande, H. *Macromolecules* **2000**, *33*, 5846-5849.
- 110 Galeazzi, E.; Guzman, A.; Pinedo, A.; Saldana, A.; Torre, D.; Muchowski, J. M. *Can. J. Chem.* **1983**, *61*, 454-456.
- 111 Li, L.; Herzon, S. B. *J. Am. Chem. Soc.* **2012**, *134*, 17376–17379.
- 112 Passacantilli, P. *Tetrahedron Lett.* **1989**, *30*, 5349-5352.