

Supplementary information

A novel one-pot and one-step microwave-assisted cyclization-methylation reaction of amino alcohols and acetylated derivatives with dimethyl carbonate and TBAC

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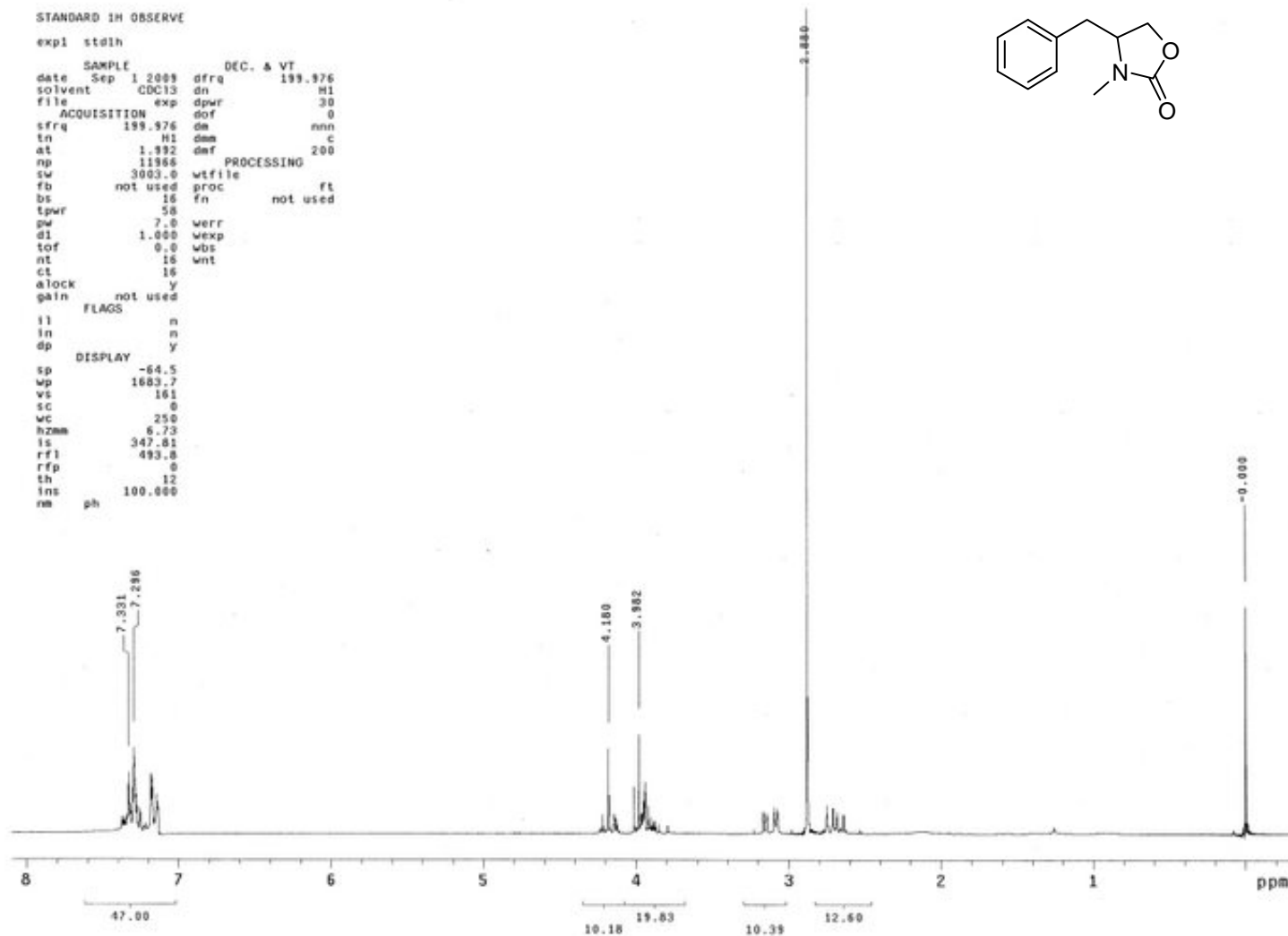


Figure 1: ^1H NMR spectrum of 4-benzyl-3-methyloxazolidin-2-one (**5**)

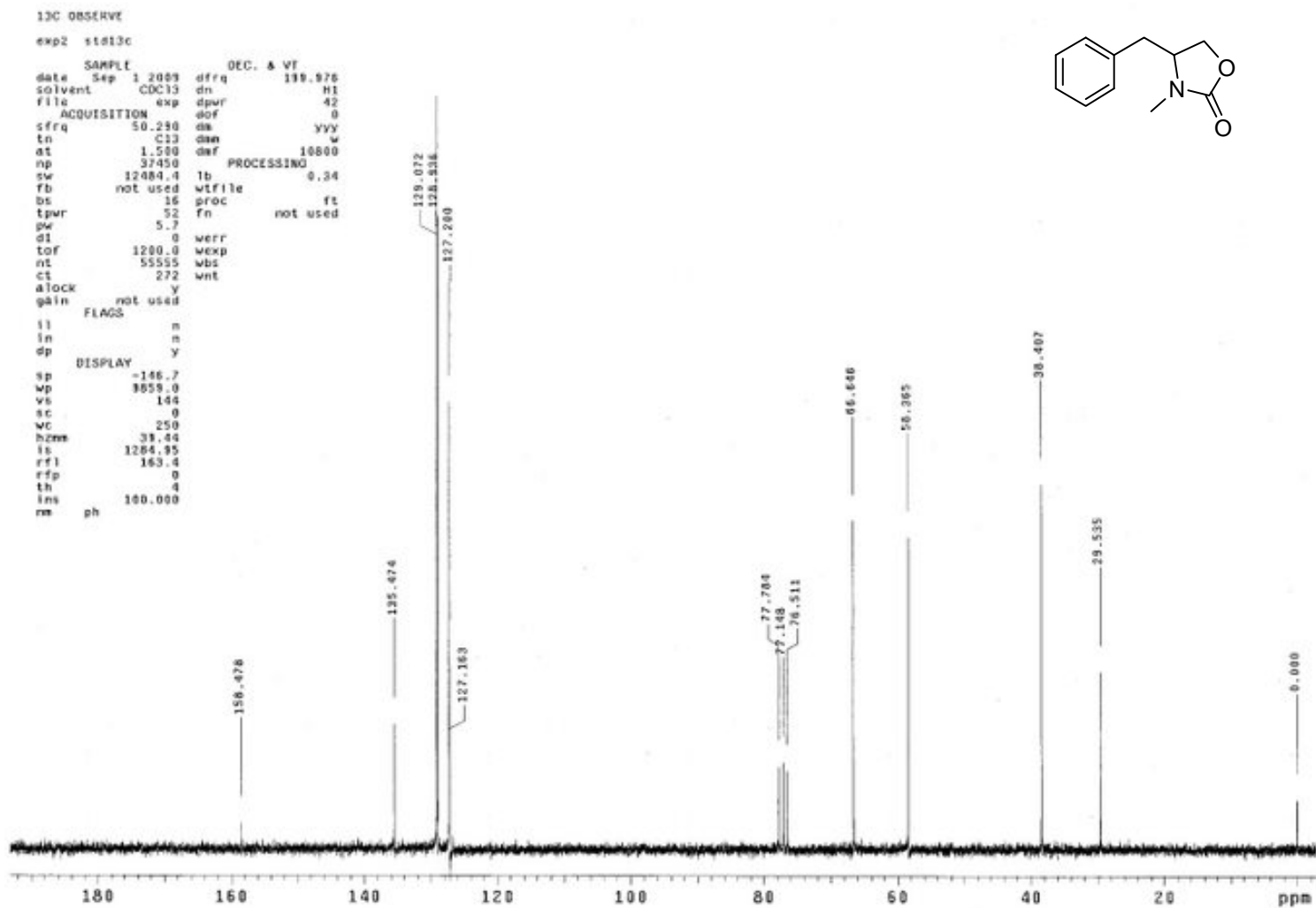


Figure 2: ^{13}C NMR spectrum of 4-benzyl-3-methyloxazolidin-2-one (**5**)

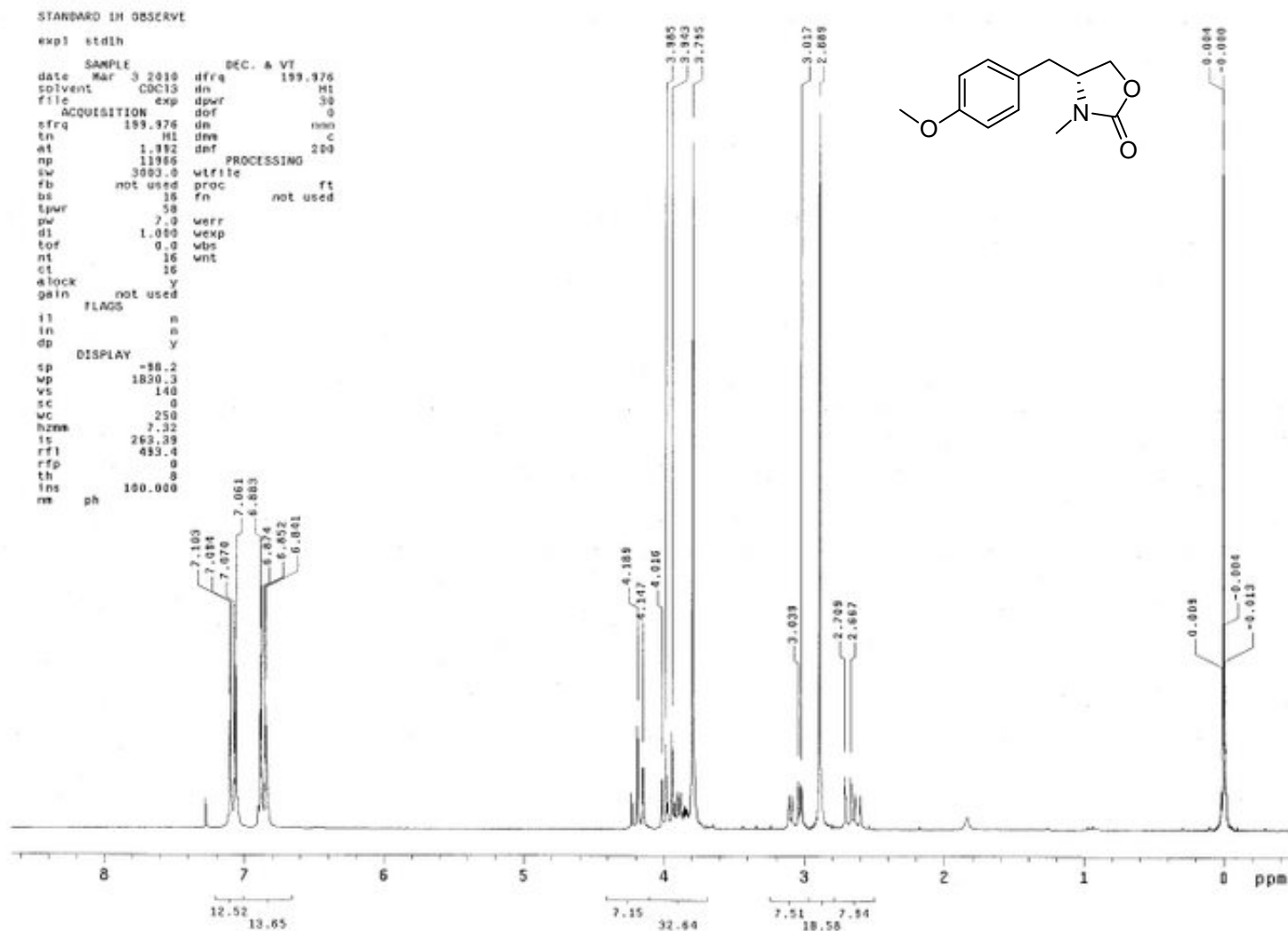


Figure 3: ^1H NMR spectrum of (R)-4-(4-methoxybenzyl)-3-methyloxazolidin-2-one (**10**)

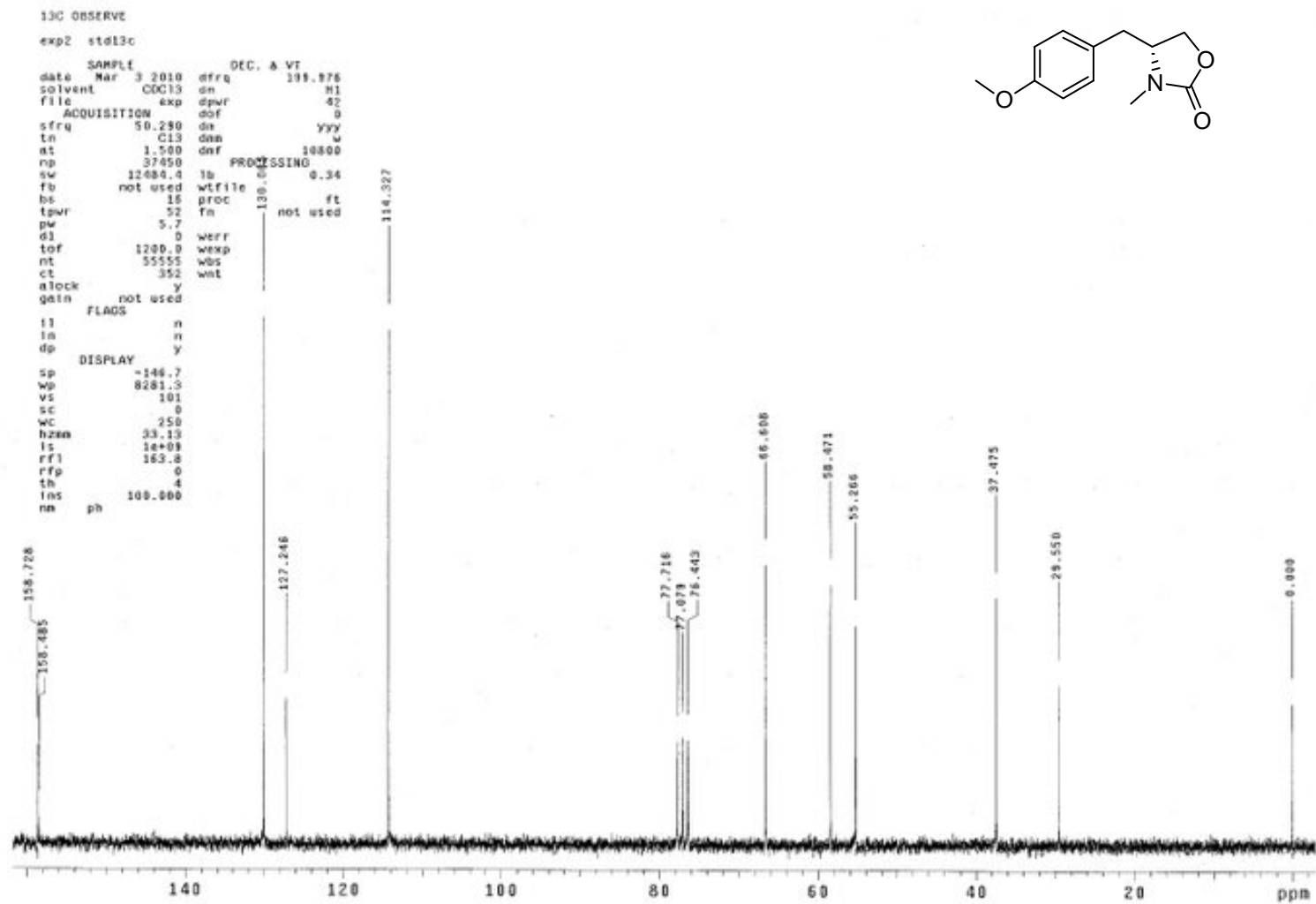


Figure 4: ^{13}C NMR spectrum of (*R*)-4-(4-methoxybenzyl)-3-methyloxazolidin-2-one (**10**)

exp1 stdh

SAMPLE		DEC. & VT	
date	Apr 27 2010	dfrq	193.976
solvent	CDC13	dn	M1
file	exp	dpr	30
ACQUISITION		dof	193.2
sfrq	193.976	dm	nnn
in	M1	dnn	C
at	1.992	daf	200
sw	11966	PROCESSING	
fb	3063.0	wtfile	
bs	not used	proc	ft
tp	16	fn	not used
pr	56		
d1	7.0	verr	
tof	1.000	wexp	
nt	0.0	wbs	
ct	16	wnt	
clock	m		
gain	not used		
FLAGS			
fl	n		
in	n		
dp	y		
DISPLAY			
sp	-114.0		
wp	1641.2		
ws	49		
sc	0		
wc	250		
hzmm	6.56		
ls	239.26		
rff	489.0		
rffp	0		
th	0		
lms	100.000		
al	ph		

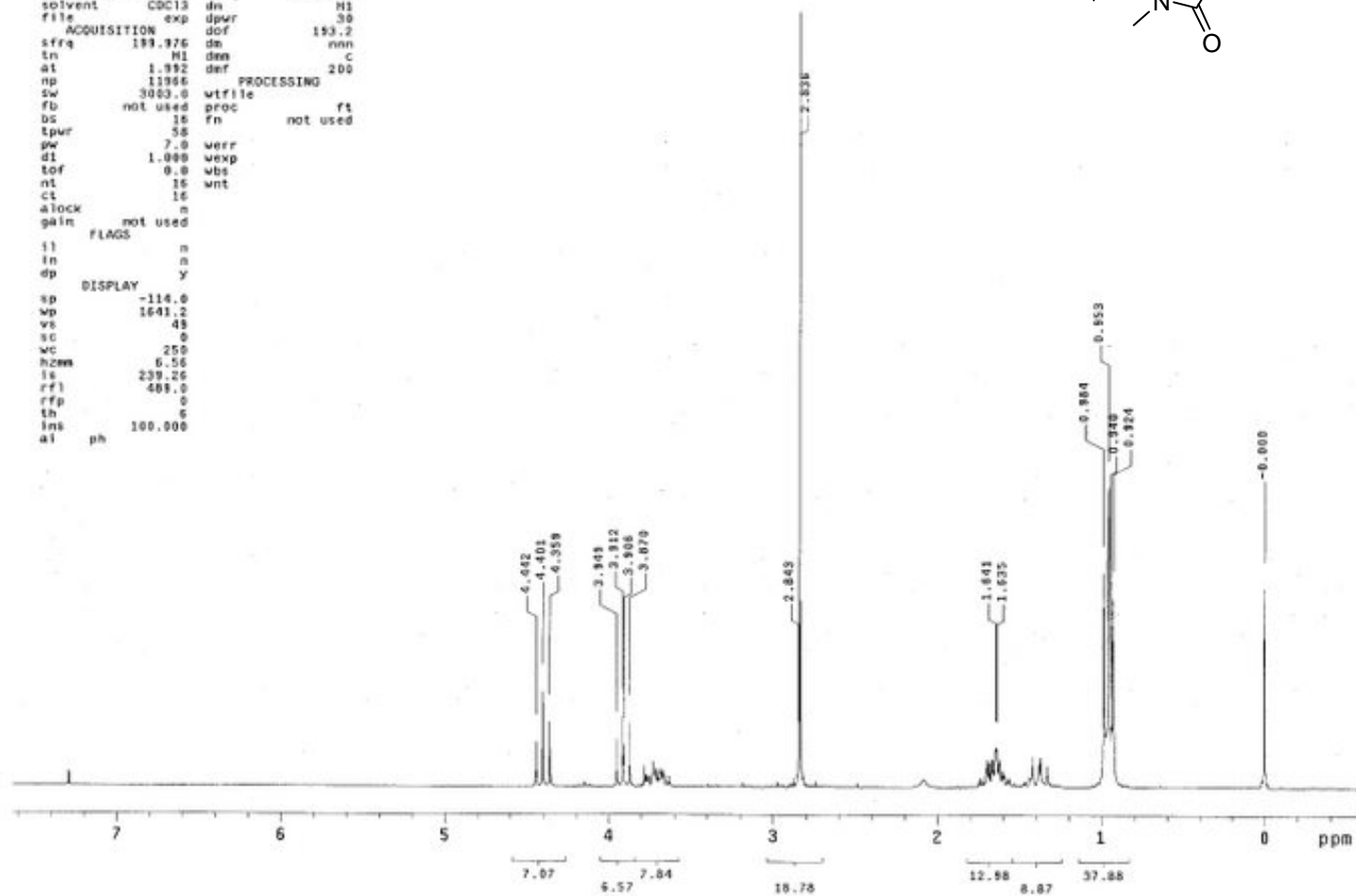
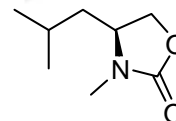


Figure 5: ^1H NMR spectrum of (*S*)-4-isobutyl-3-methyloxazolidin-2-one (**11**)

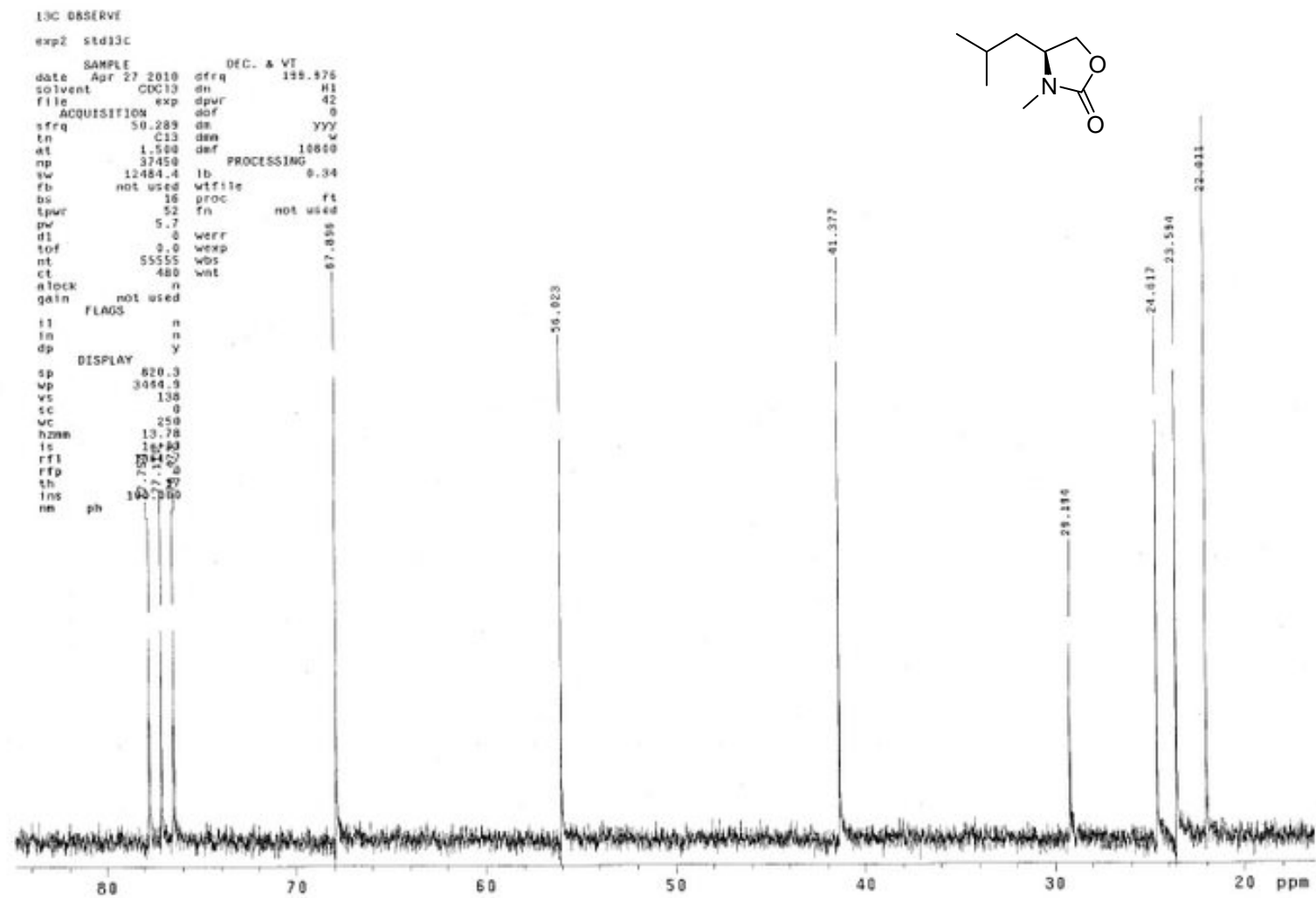


Figure 6: ^{13}C NMR spectrum of (S)-4-isobutyl-3-methyloxazolidin-2-one (**11**)

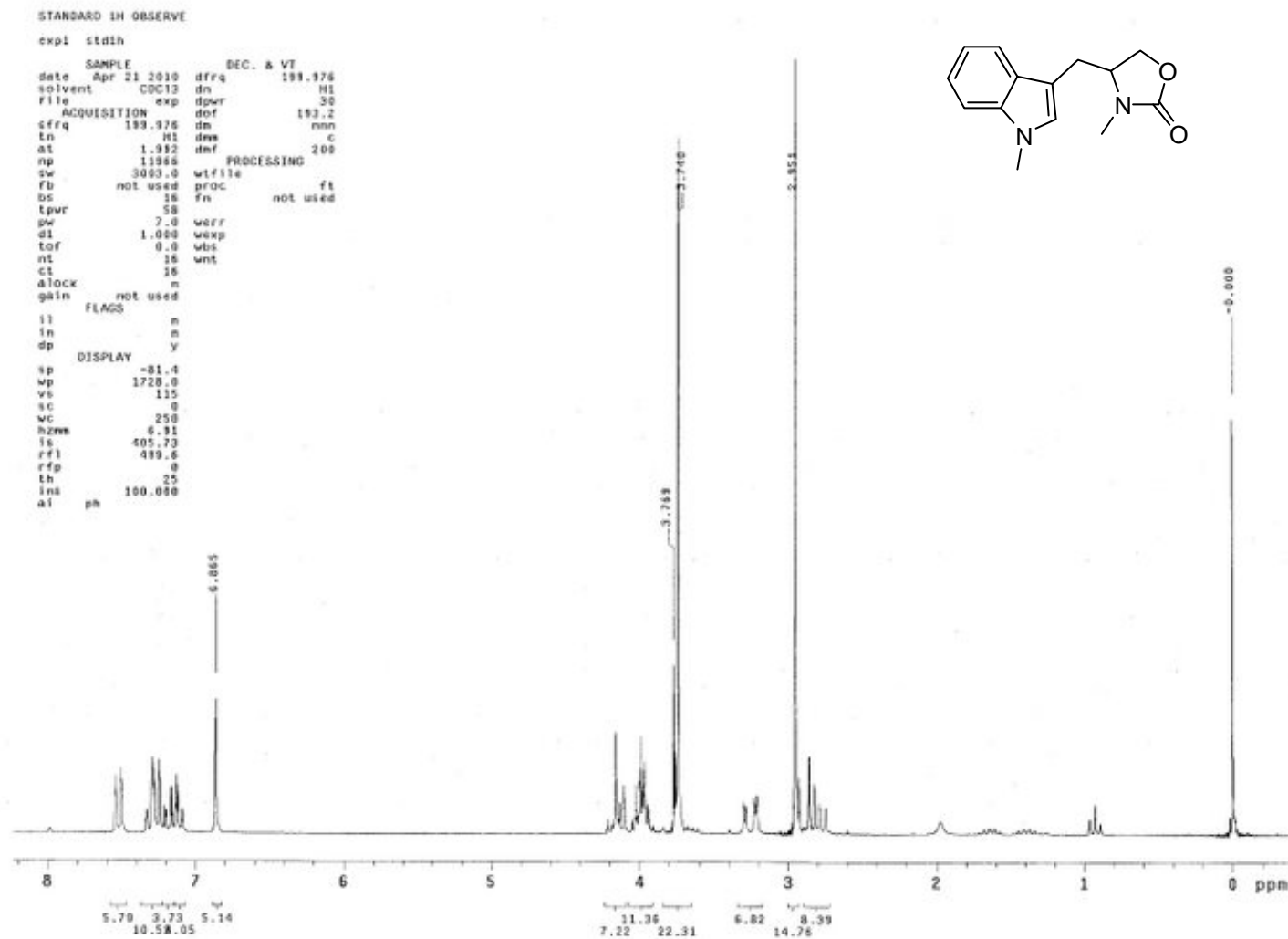


Figure 7: ^1H NMR spectrum of 3-methyl-4-((1-methyl-1*H*-indol-3-yl)methyl)oxazolidin-2-one (**12**)

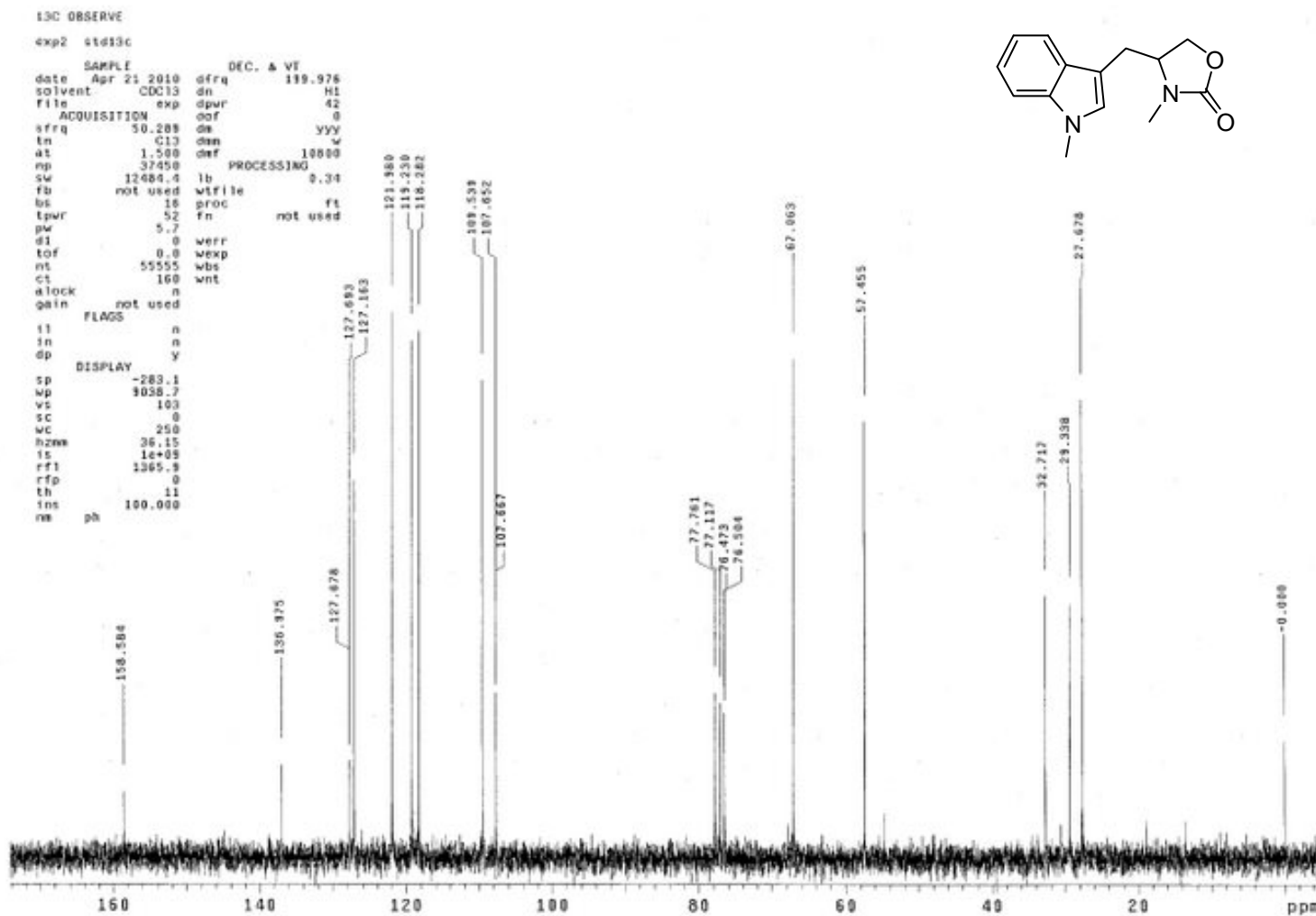


Figure 8: ^{13}C NMR spectrum of 3-methyl-4-((1-methyl-1*H*-indol-3-yl)methyl)oxazolidin-2-one (**12**)

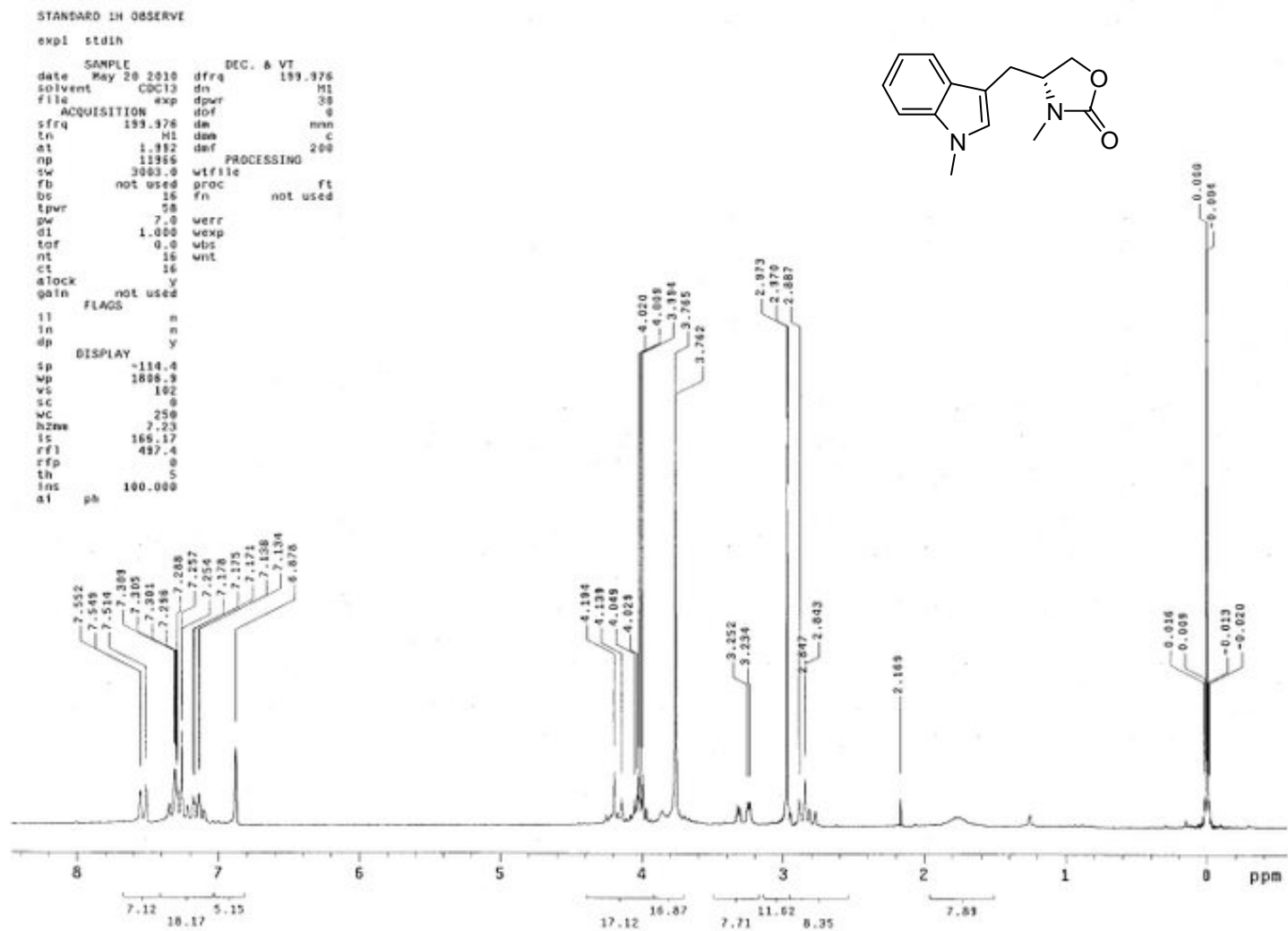


Figure 9: ^1H NMR spectrum of (*R*)-3-methyl-4-((1-methyl-1*H*-indol-3-yl)methyl)oxazolidin-2-one (**13**)

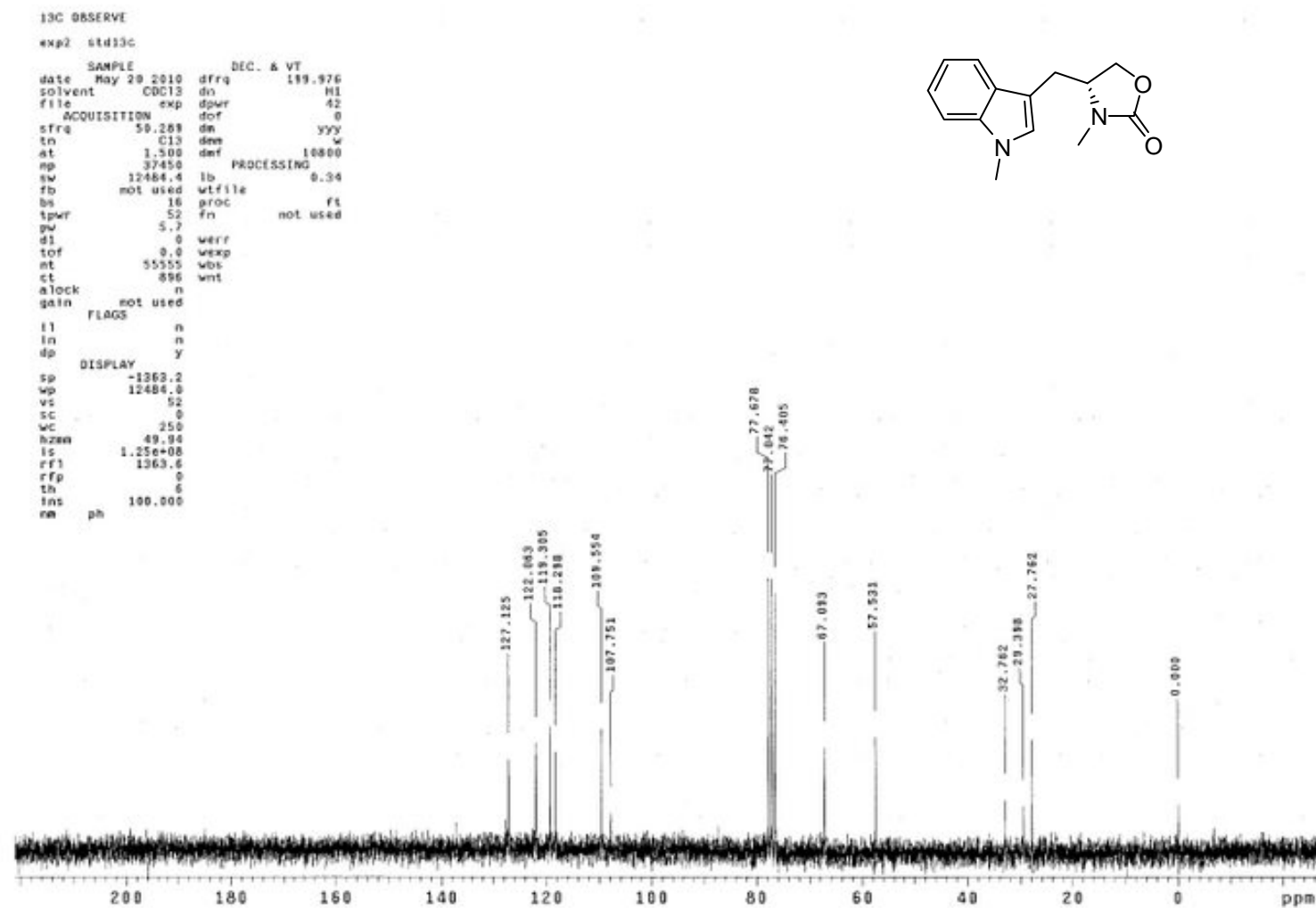


Figure 10: ^{13}C NMR spectrum of (R)-3-methyl-4-((1-methyl-1H-indol-3-yl)methyl)oxazolidin-2-one (**13**)

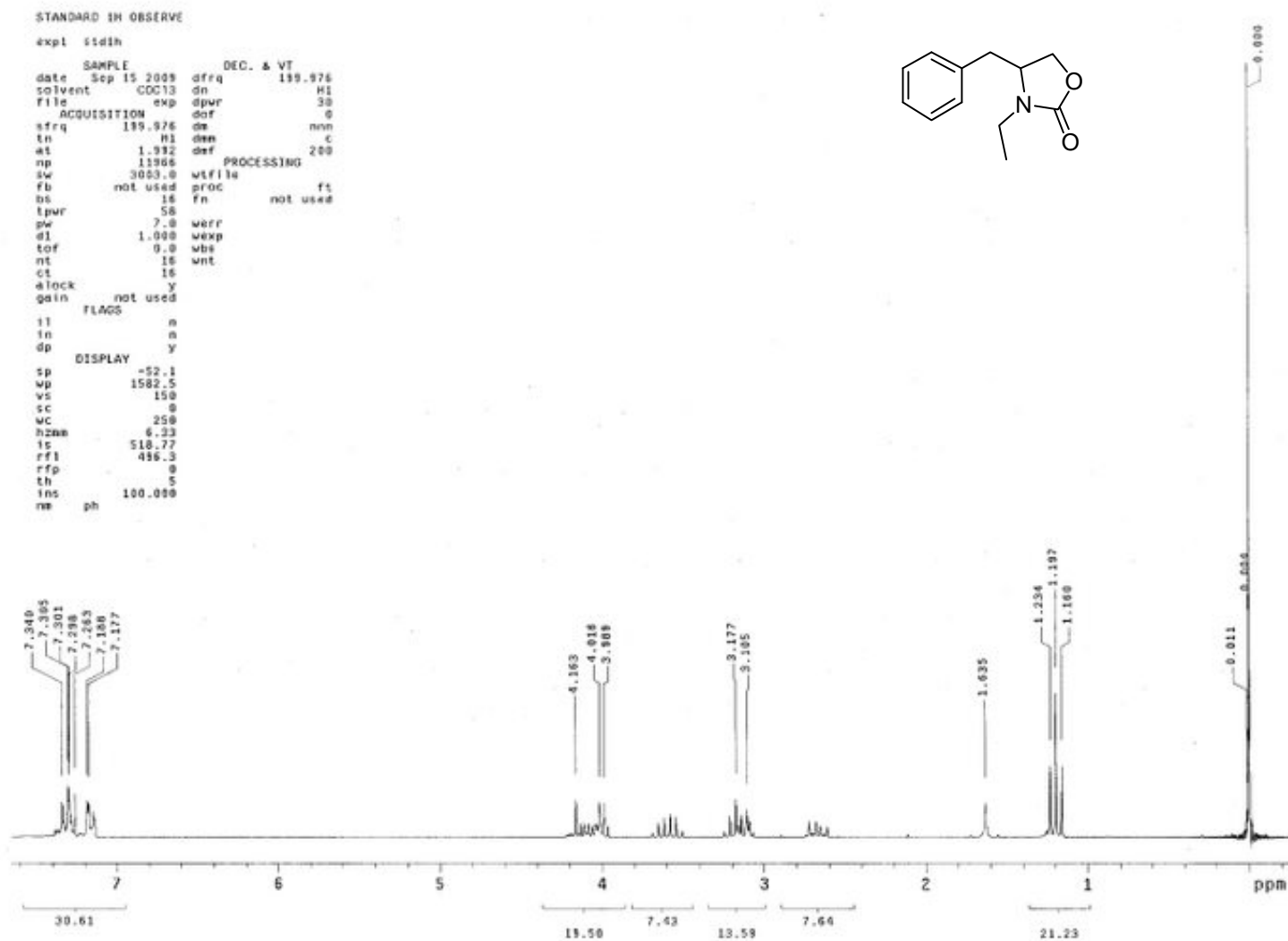


Figure 11: ^1H NMR spectrum of 4-benzyl-3-ethyloxazolidin-2-one (**17**)

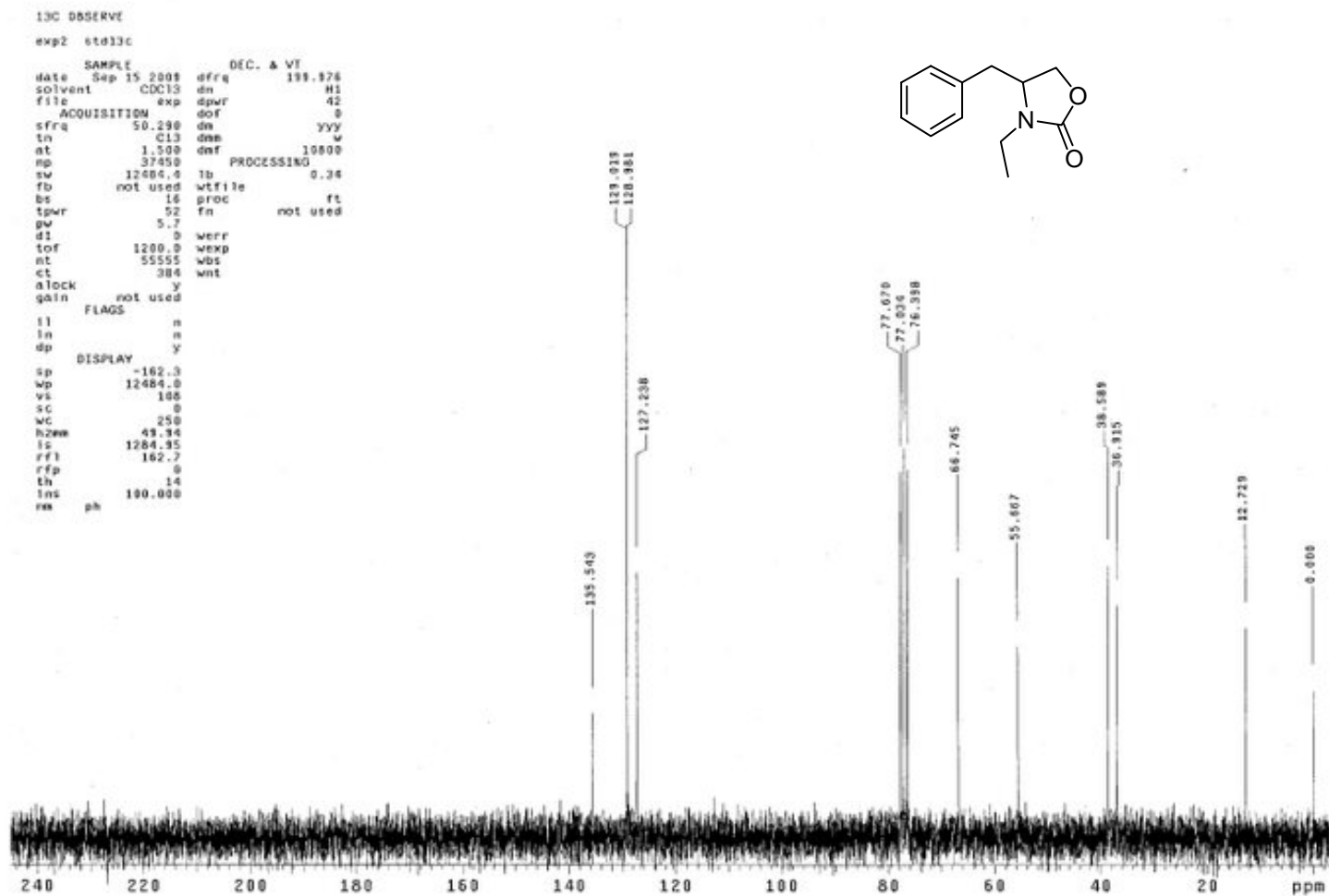


Figure 12: ^{13}C NMR spectrum of 4-benzyl-3-ethyloxazolidin-2-one (17)

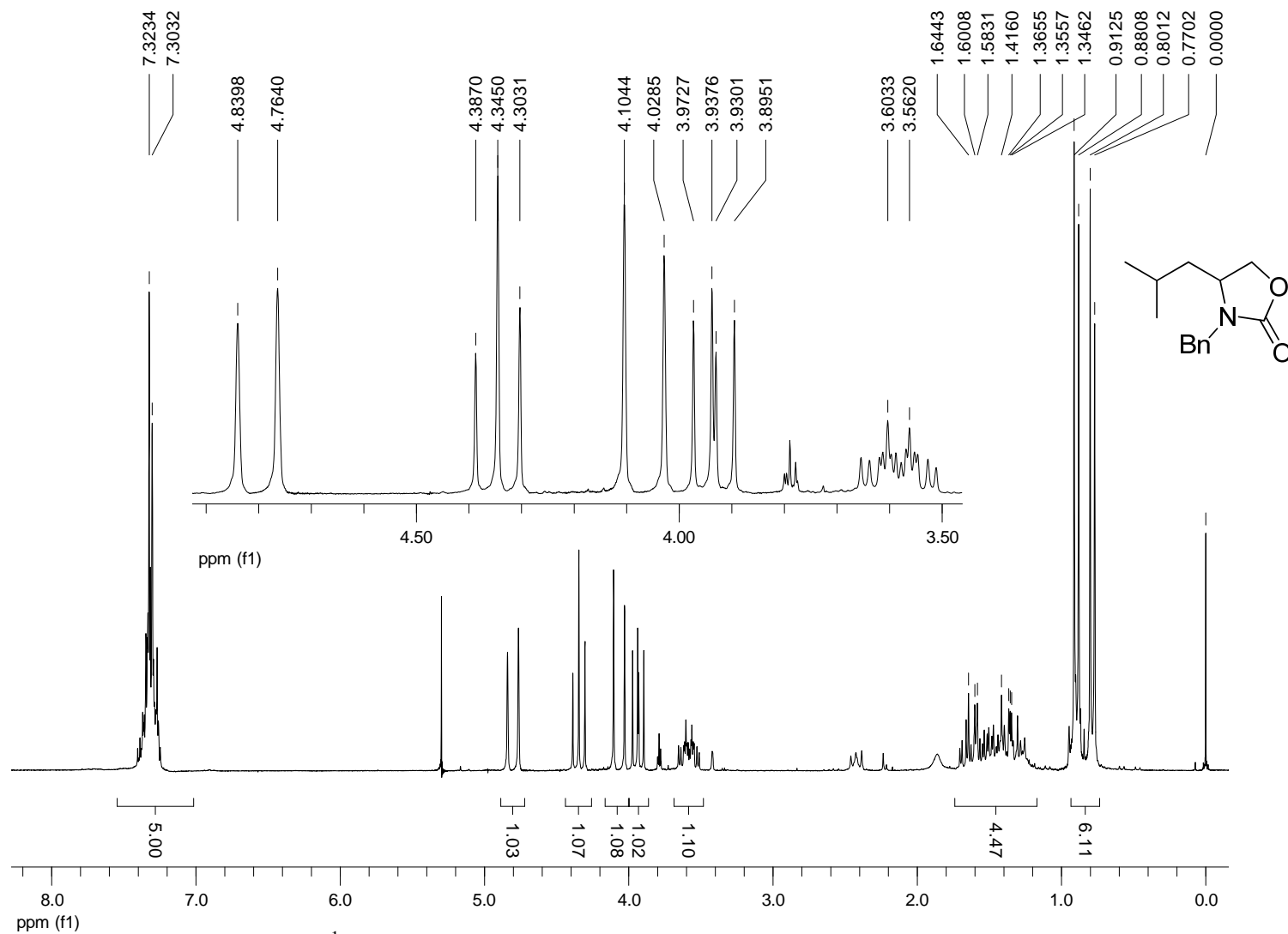


Figure 13: ¹H NMR spectrum of 3-benzyl-4-isobutyloxazolidin-2-one (**19**)

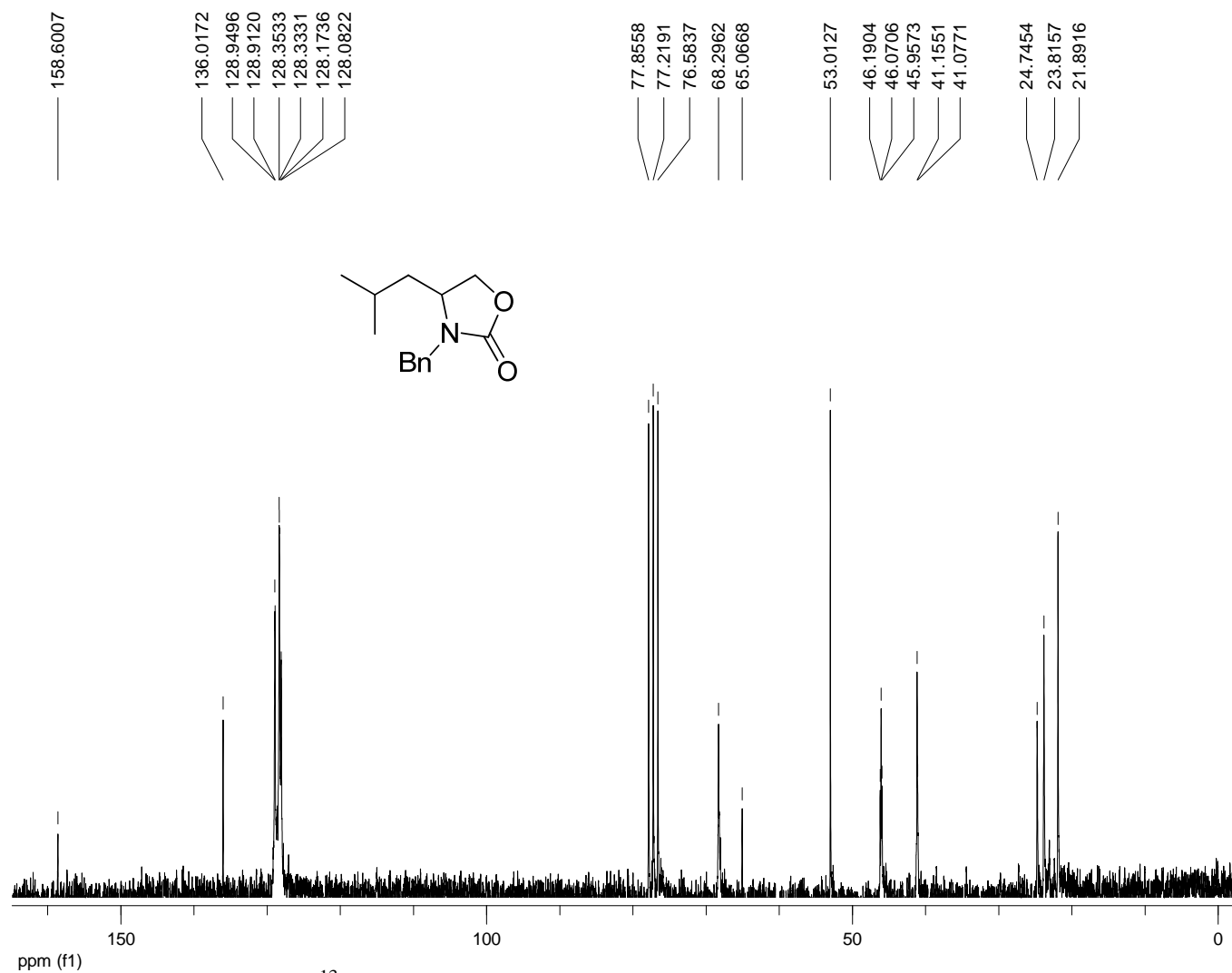


Figure 14: ¹³C NMR spectrum of 3-benzyl-4-isobutyloxazolidin-2-one (**19**)

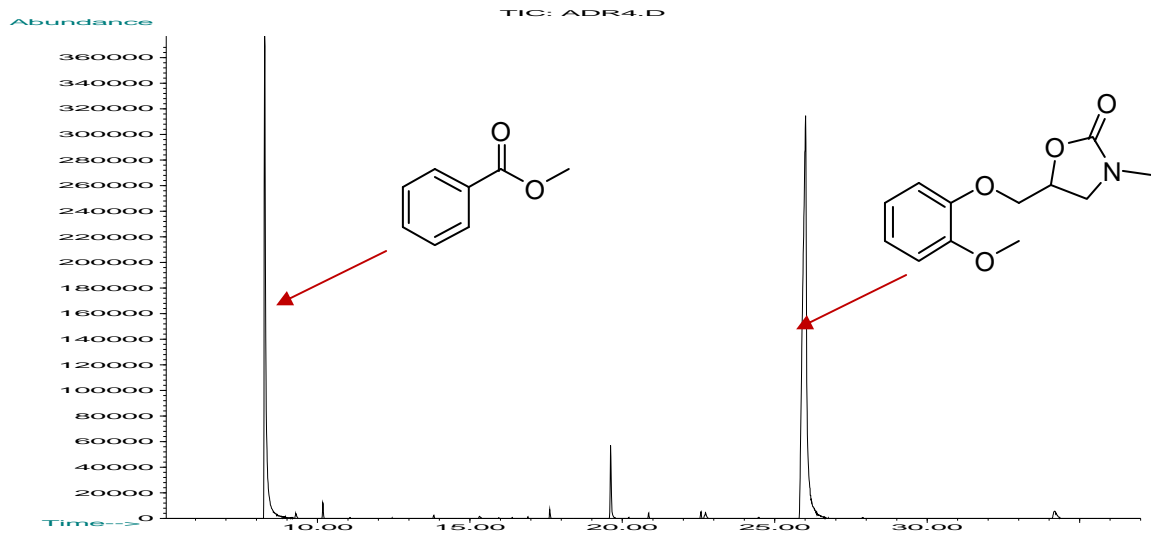


Figure 15: GC of reaction products 5-((2-methoxyphenoxy)-3-methyloxazolidin-2-one (**16**) and methyl benzoate.

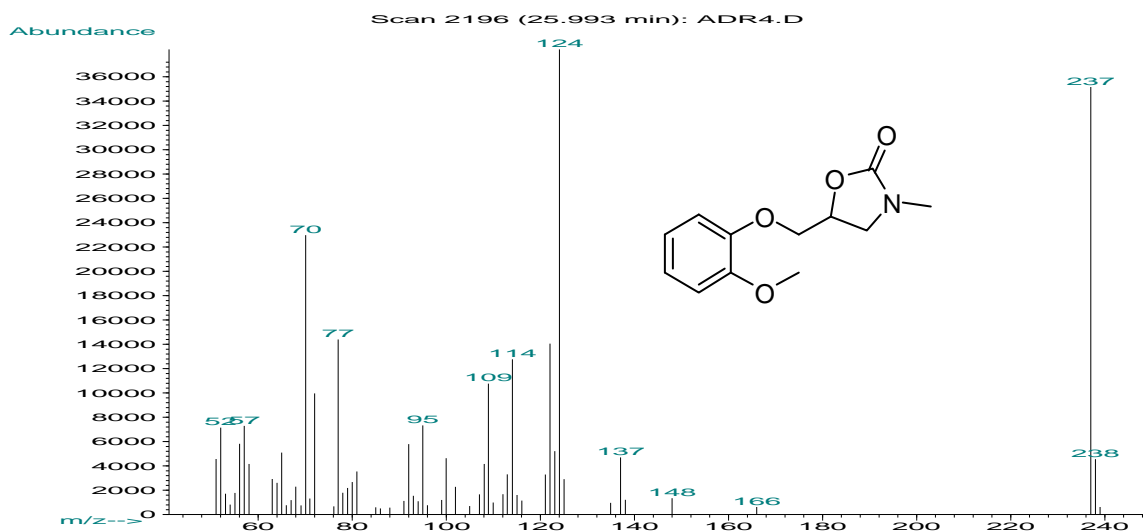


Figure 16: MS of 5-((2-methoxyphenoxy)-3-methyloxazolidin-2-one (**16**)

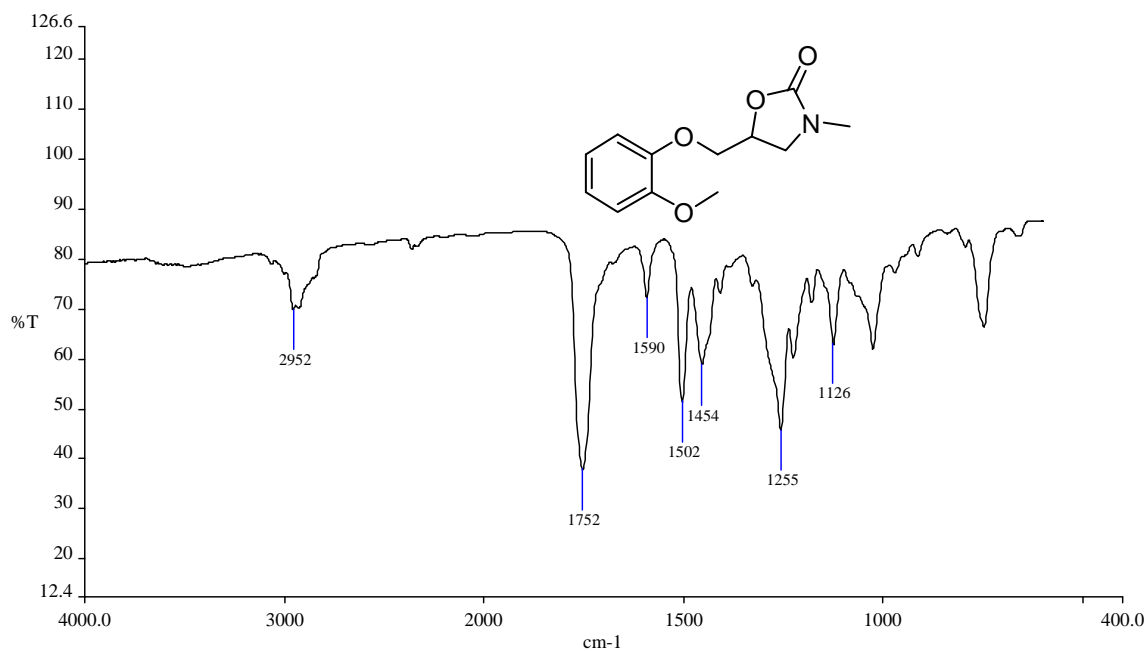


Figure 17: IR spectrum of 5-((2-methoxyphenoxy)-3-methyloxazolidin-2-one (**16**)

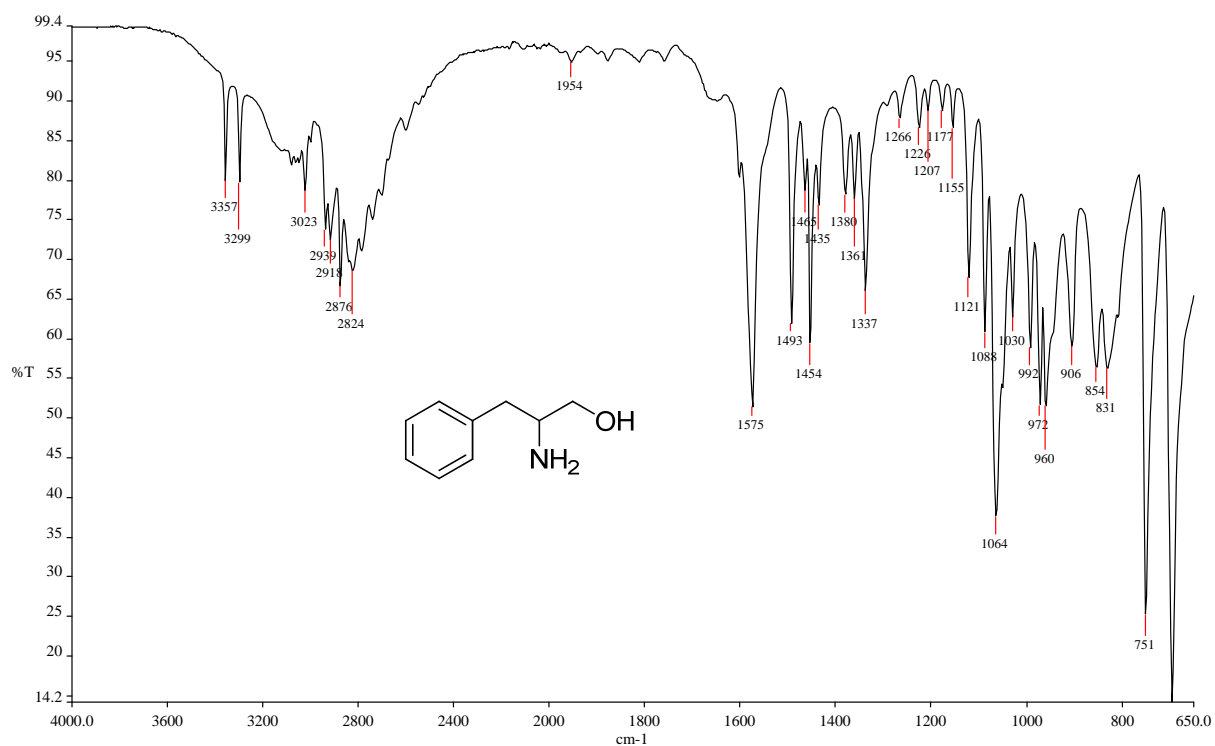


Figure 18: IR spectrum of 2-amino-3-phenylpropan-1-ol (**1**)

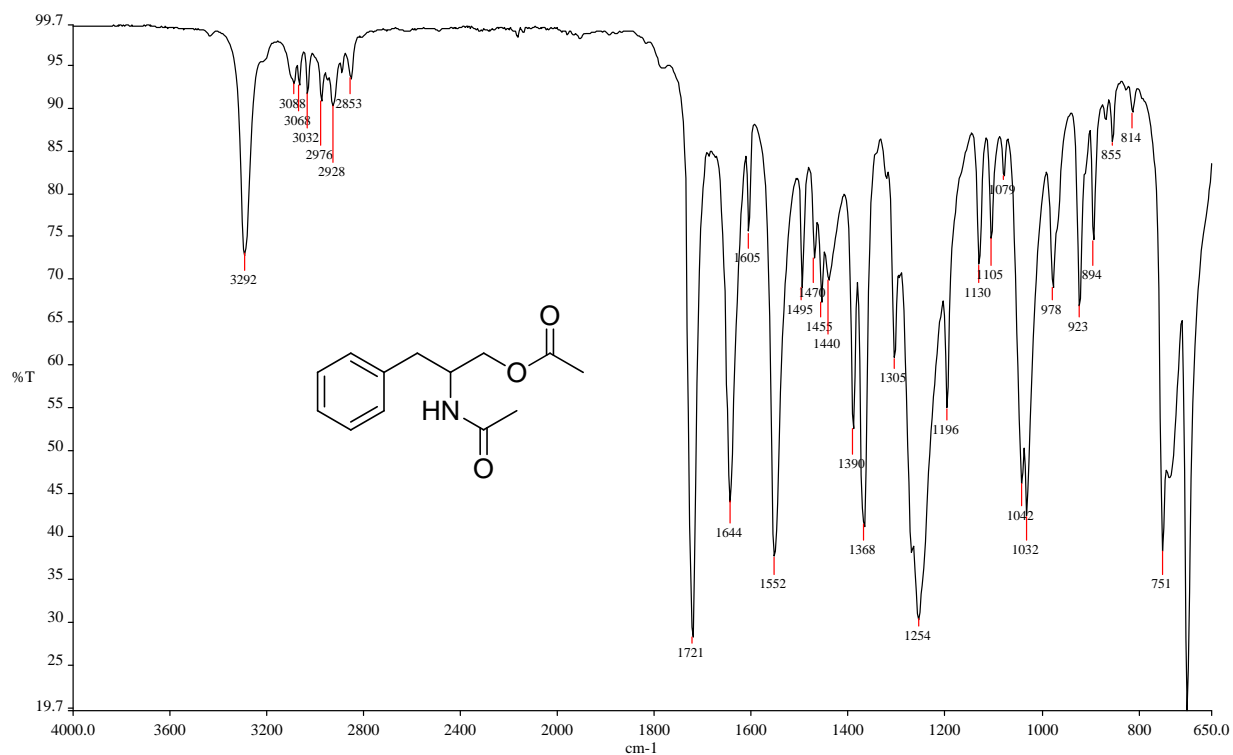


Figure 19: IR spectrum of 2-acetamido-3-phenylpropyl acetate (3)

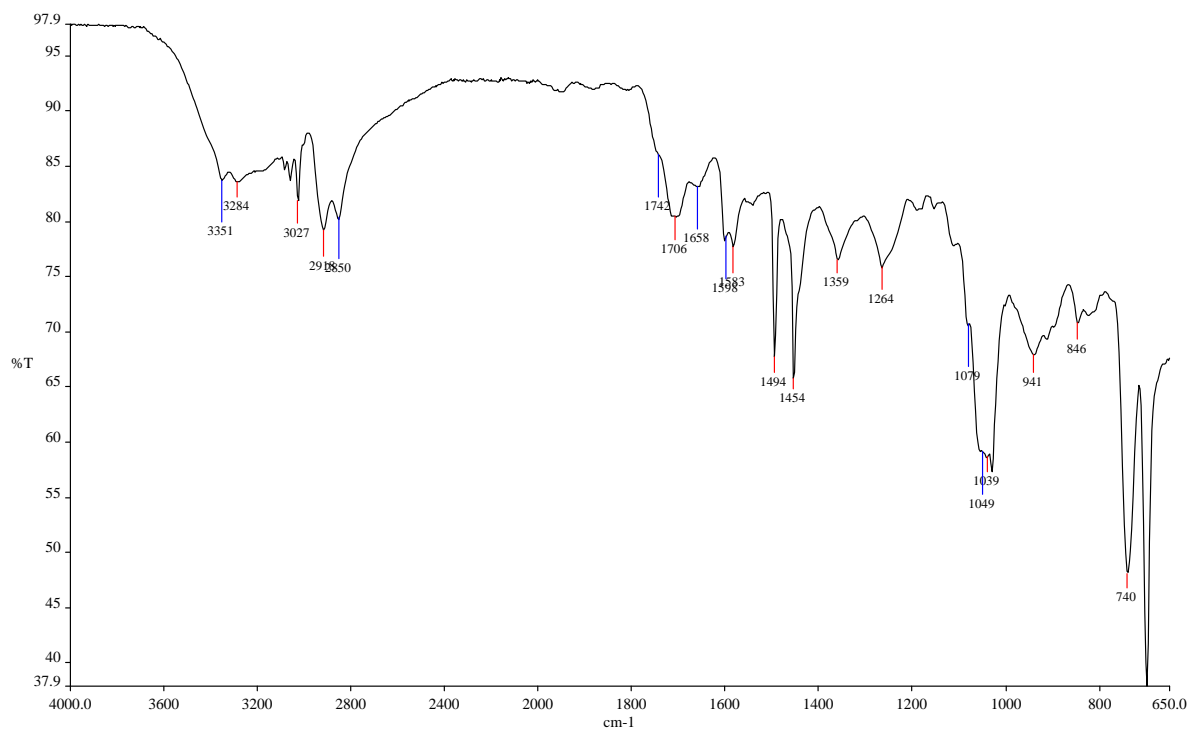


Figure 20: IR spectrum of crude obtained in conditions described in Table 2, entry 1

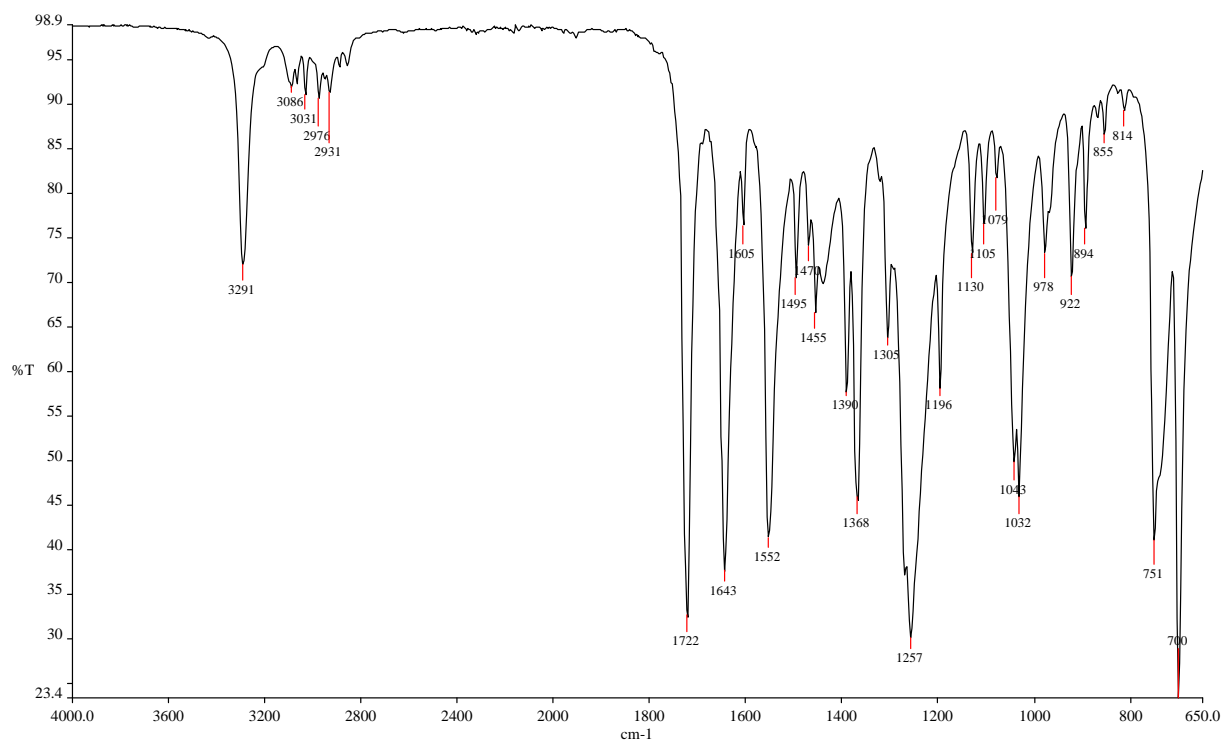


Figure 21: IR spectrum of crude product described in Table 2, entry 2.

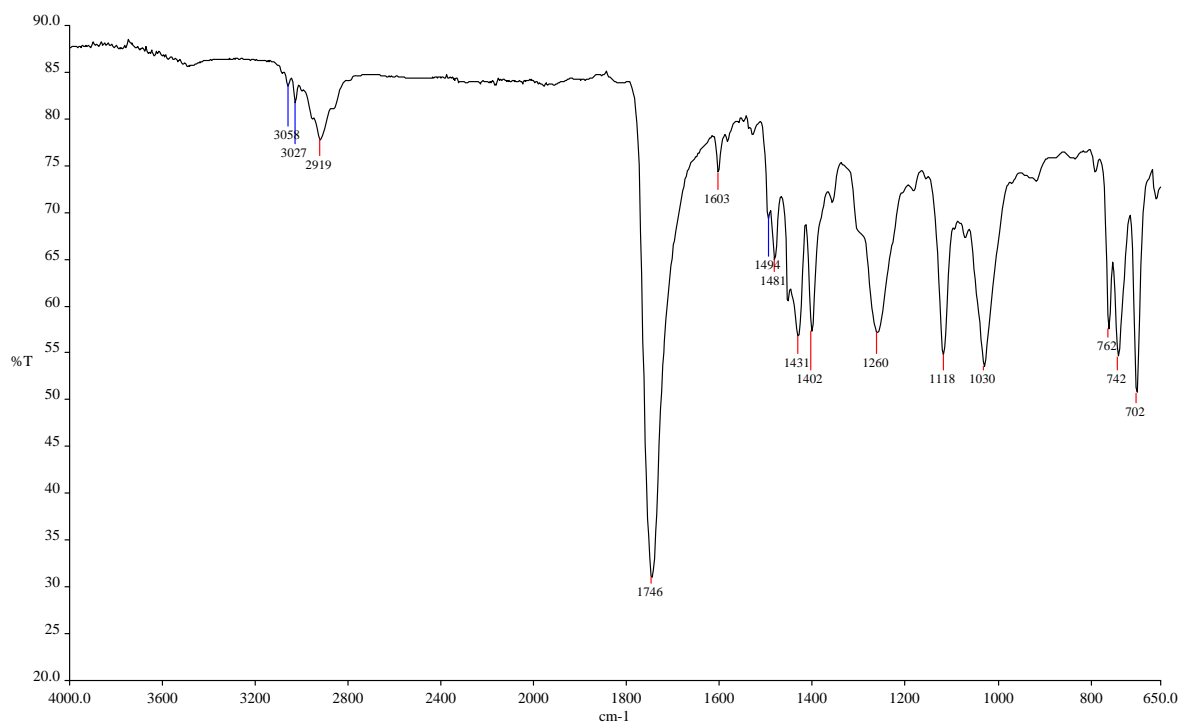


Figure 22: IR spectrum of crude obtained in conditions described in Table 2, entry 3

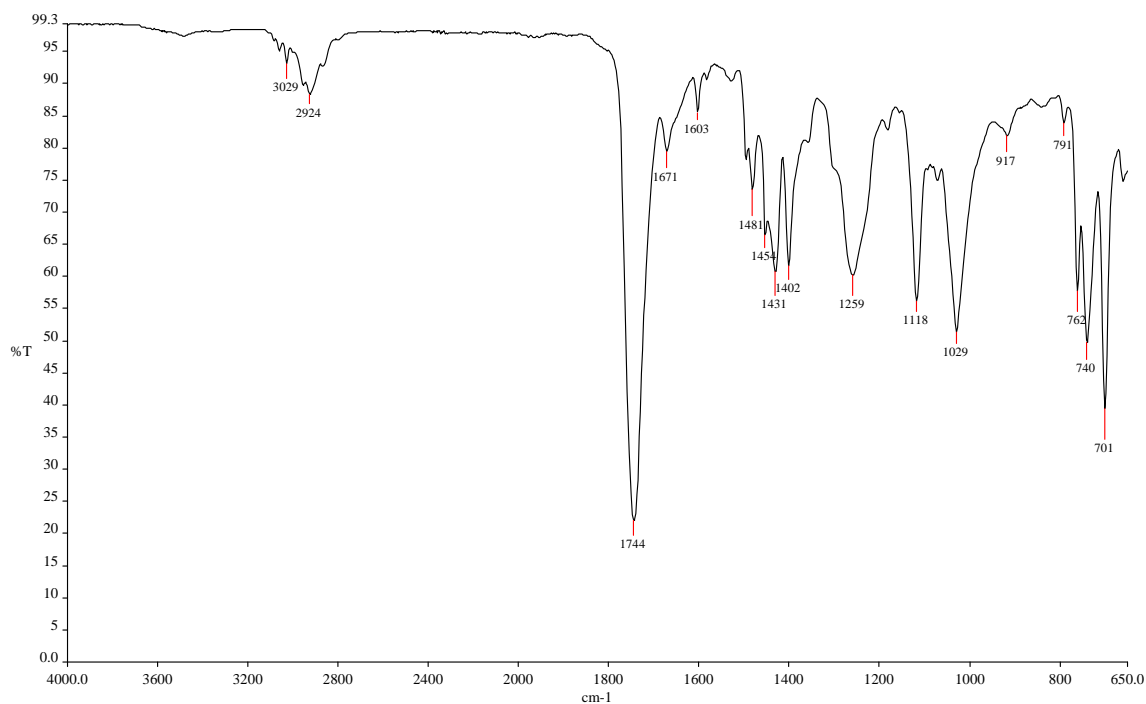


Figure 23: IR spectrum of crude obtained in conditions described in Table 2, entry 4

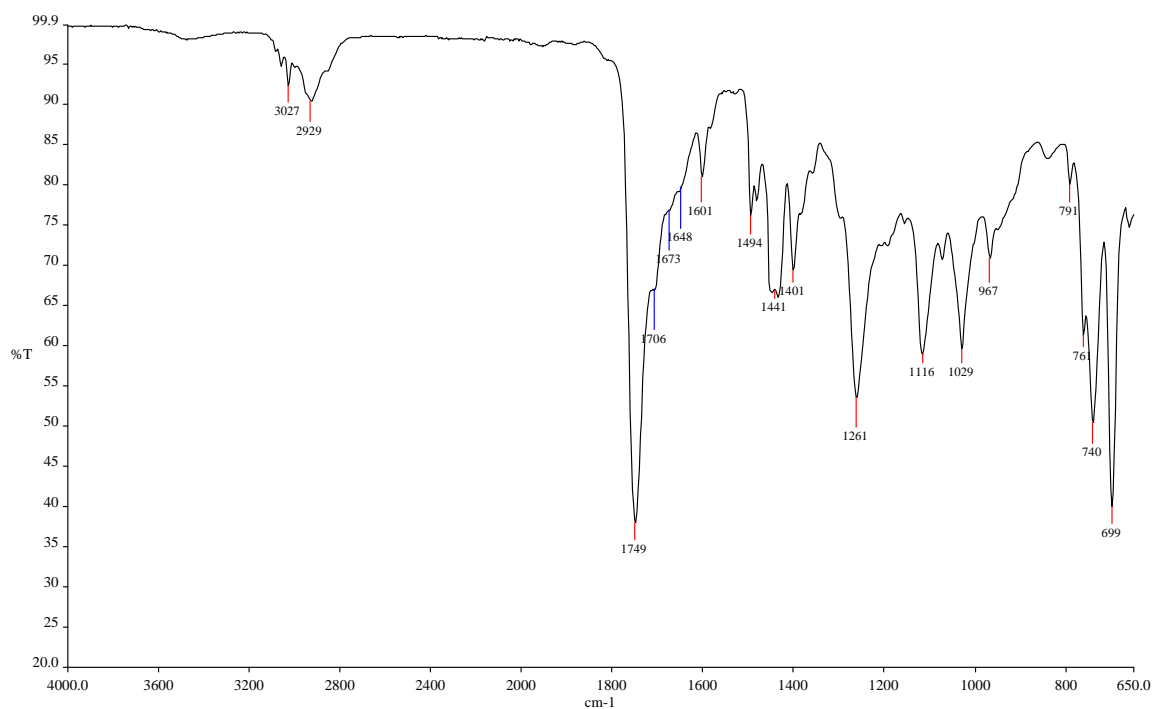


Figure 24.IR spectrum of crude obtained in conditions described in Table 2, entry 5

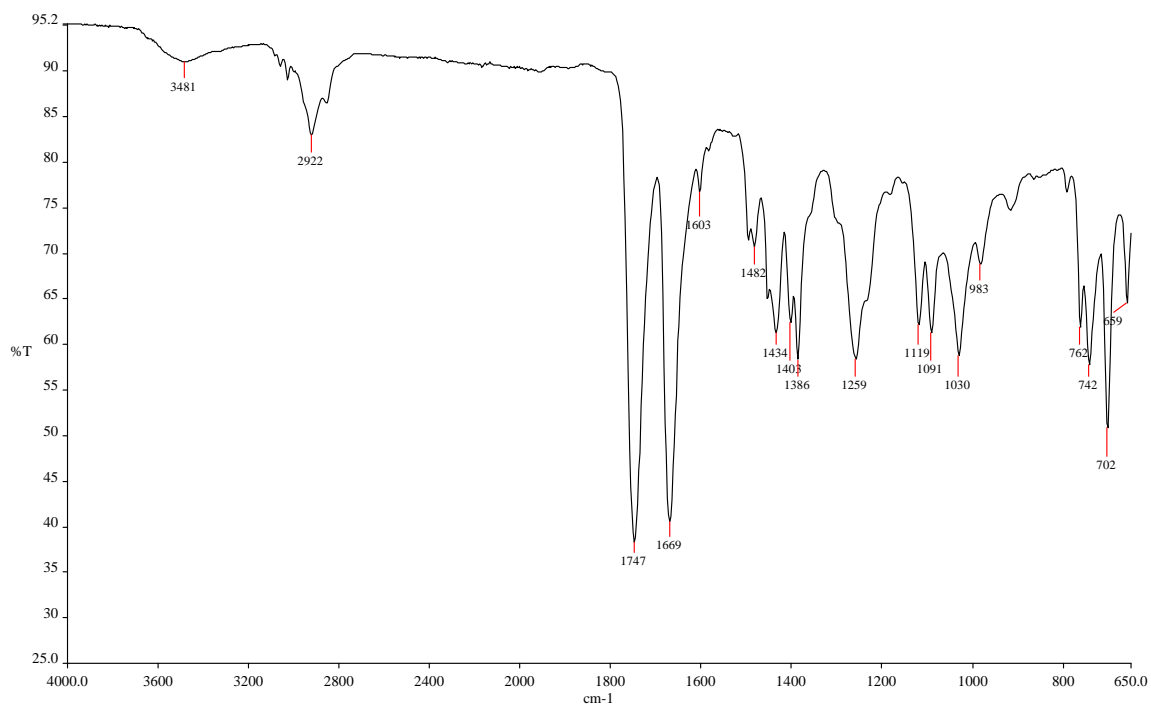


Figure 25: IR spectrum of crude obtained in conditions described in Table 2, entry 6

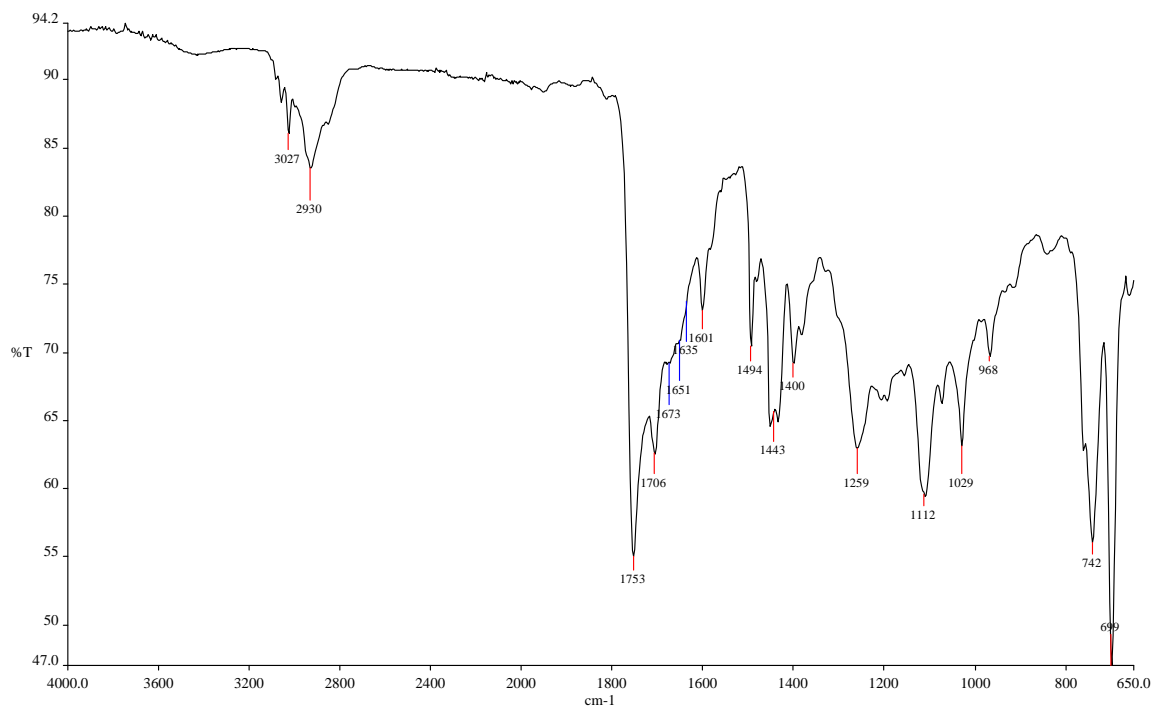


Figure 26: IR spectrum of crude obtained in conditions described in Table 2, entry 7

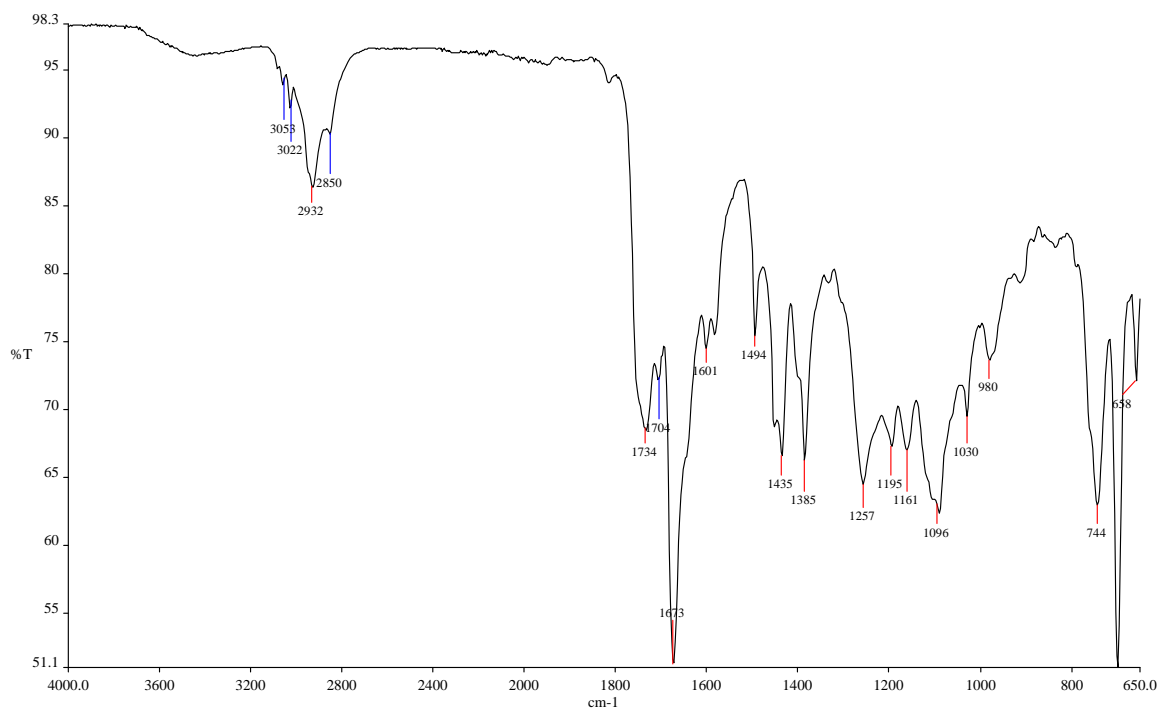


Figure 27: IR spectrum of crude obtained in conditions described in Table 2, entry 8

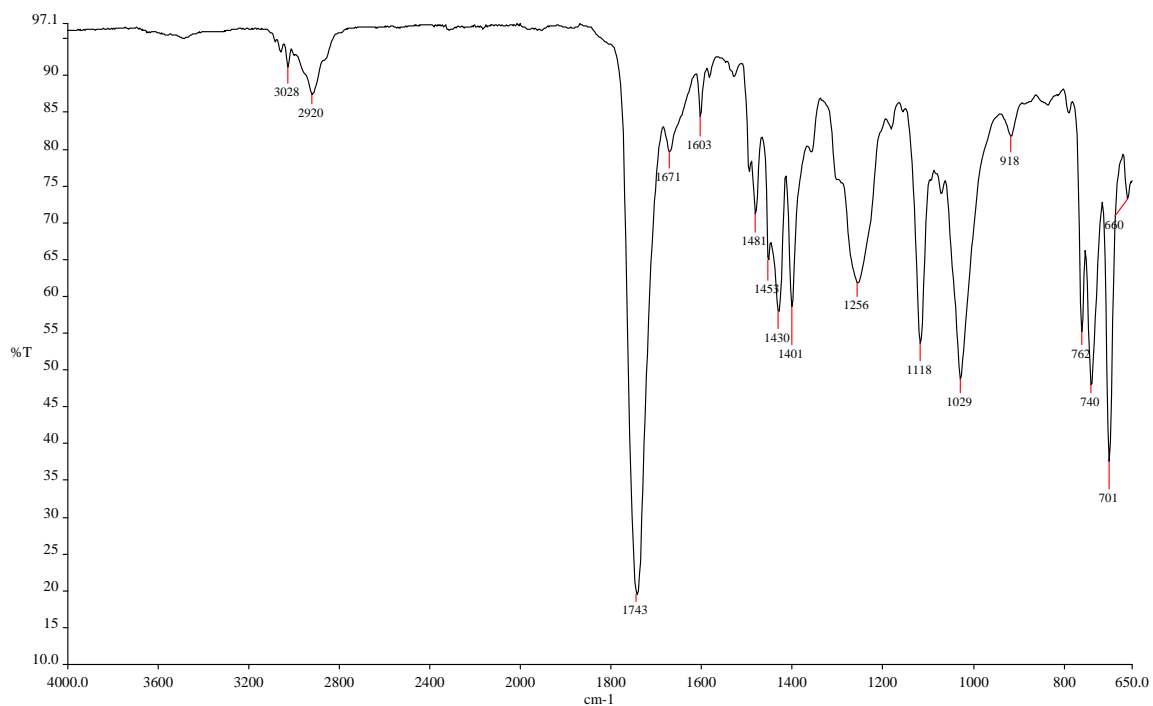


Figure 28: IR spectrum of crude obtained in conditions described Table 2, entry 9

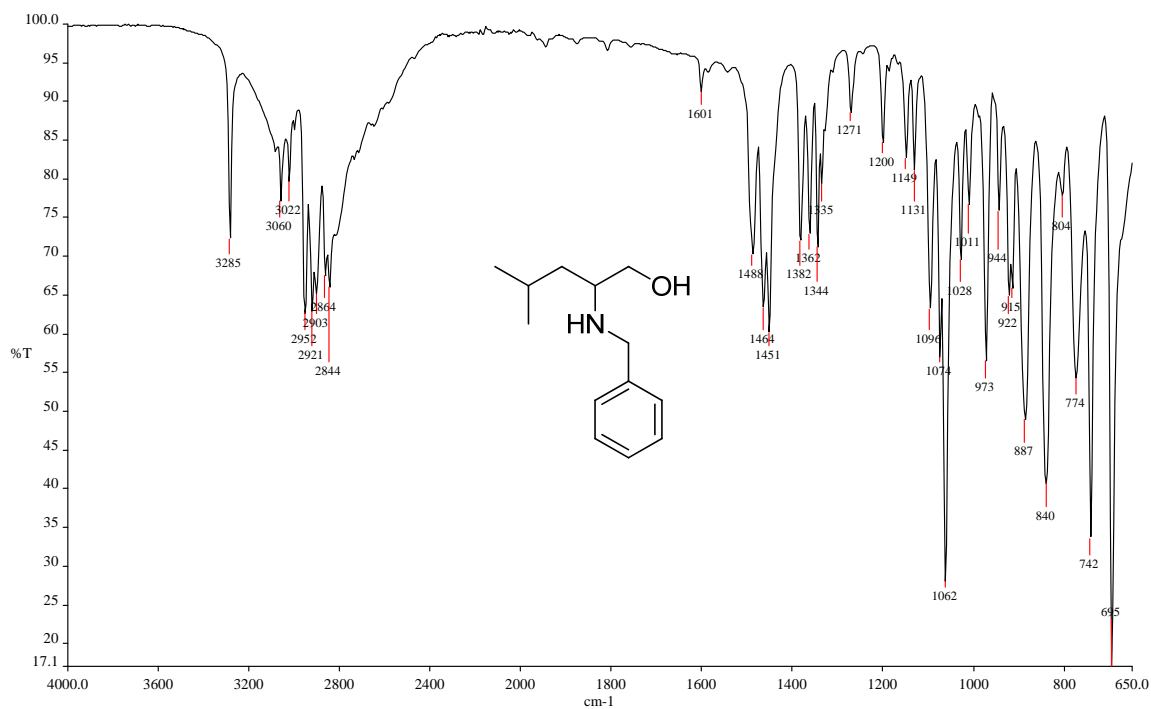


Figure 29: IR spectrum of 2-(benzylamino)-4-methylpentan-1-ol (**18**)

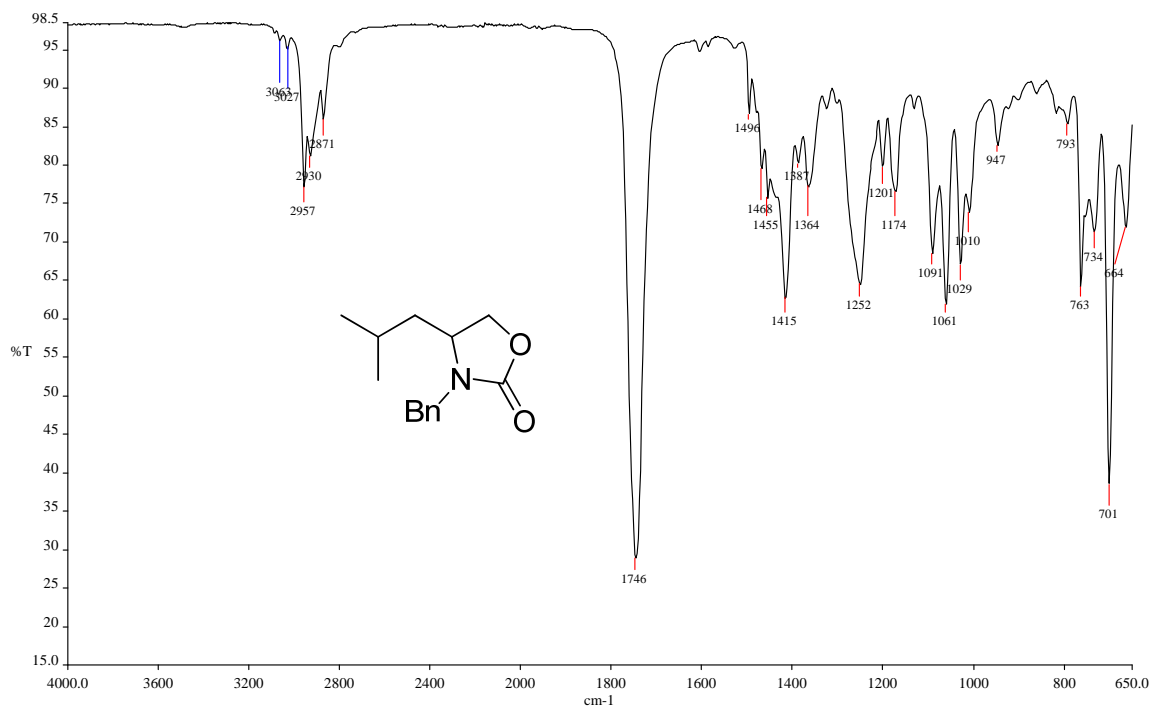


Figure 30: IR spectrum of crude obtained in conditions described in Table 2, entry 10

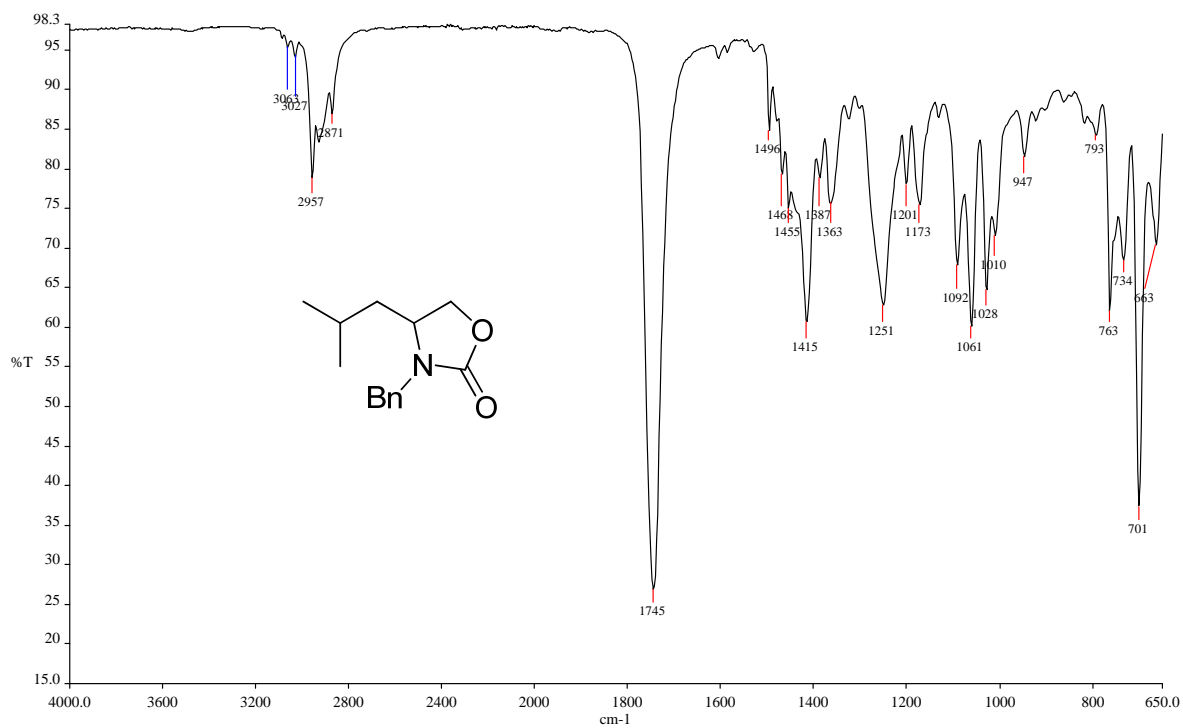


Figure 31: IR spectrum of crude obtained in conditions described in Table 2, entry 11

File :C:\MSDCHEM\1\DATA\OCHOA\Snapshot\ADRIAN72.d
Operator : D. CHAVEZ
Acquired : 12 Aug 2014 13:43 using AcqMethod VOLATILESPESADOS.M
Instrument : Numero 1
Sample Name: TETRABUTILAMINA
Misc Info :
Vial Number: 5

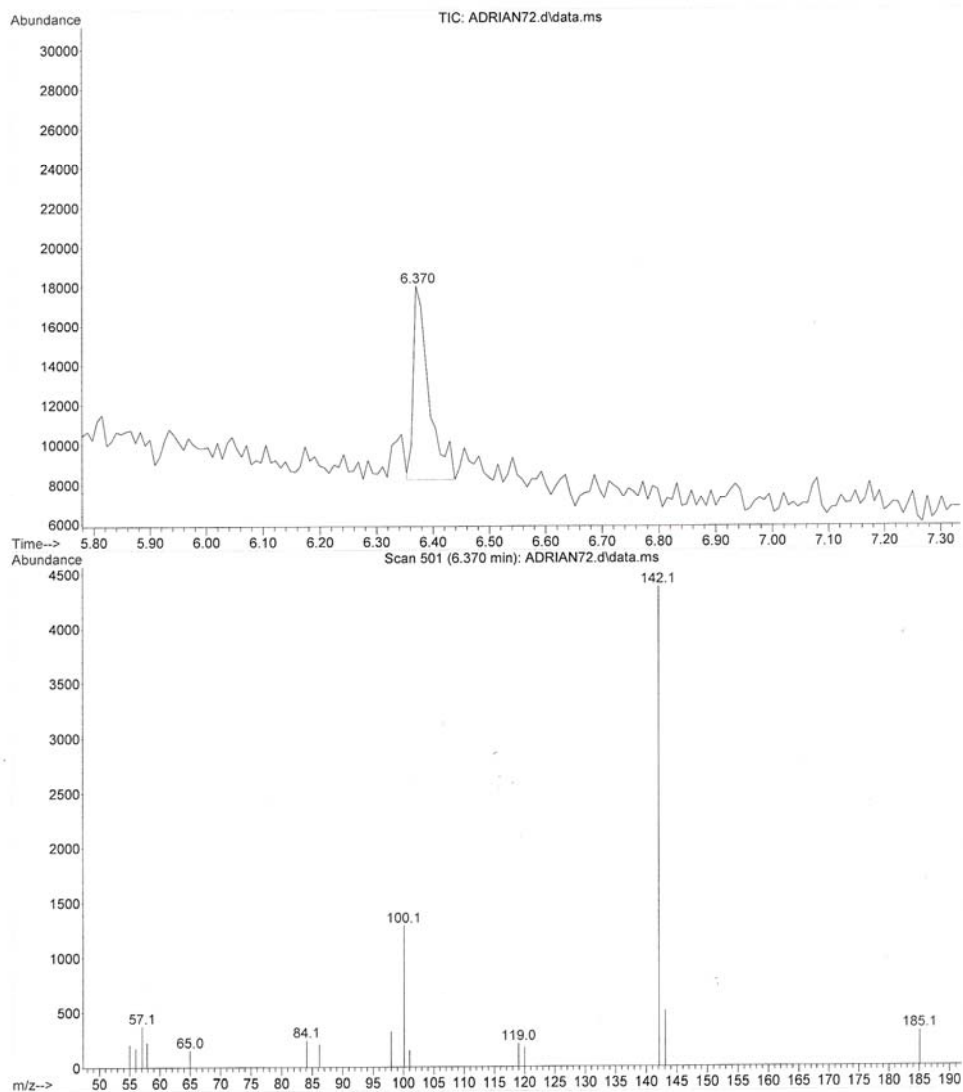
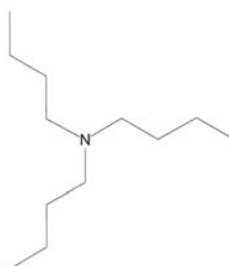
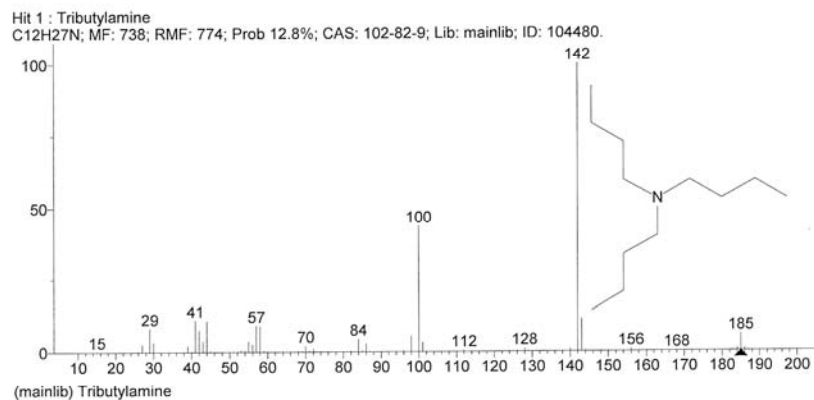


Figure 32:GC-MS of TBAC in solution (part 1).



Name: Tributylamine
 Formula: C₁₂H₂₇N
 MW: 185 CAS#: 102-82-9 NIST#: 291265 ID#: 104480 DB: mainlib
 Other DBs: Fine, TSCA, RTECS, HODOC, EINECS, IRDB
 Contributor: NIST Mass Spectrometry Data Center, 1998.
 10 largest peaks:
 142 999 | 100 435 | 143 112 | 41 110 | 44 107 | 57 91 | 58 90 | 29 82 | 42 75 | 185 58 |
 Synonyms:
 1.1-Butanamine, N,N-dibutyl-
 2.Tri-n-butylamine
 3.Tris[N-butylamine]
 4.(n-C₄H₉)₃N
 5.Tributylamina
 6.UN 2542
 7.N,N-Dibutyl-1-butanamine #

Figure 33:GC-MS of TBAC in solution (part 2).

File : C:\MSDCHEM\1\DATA\OCHOA\Snapshot\ADRIAN73.d
 Operator : D. CHAVEZ
 Acquired : 12 Aug 2014 14:19 using AcqMethod VOLATILESPESADOS.M
 Instrument : Numero 1
 Sample Name: DET. DE ACETATO DE ETILO
 Misc Info :
 Vial Number: 4

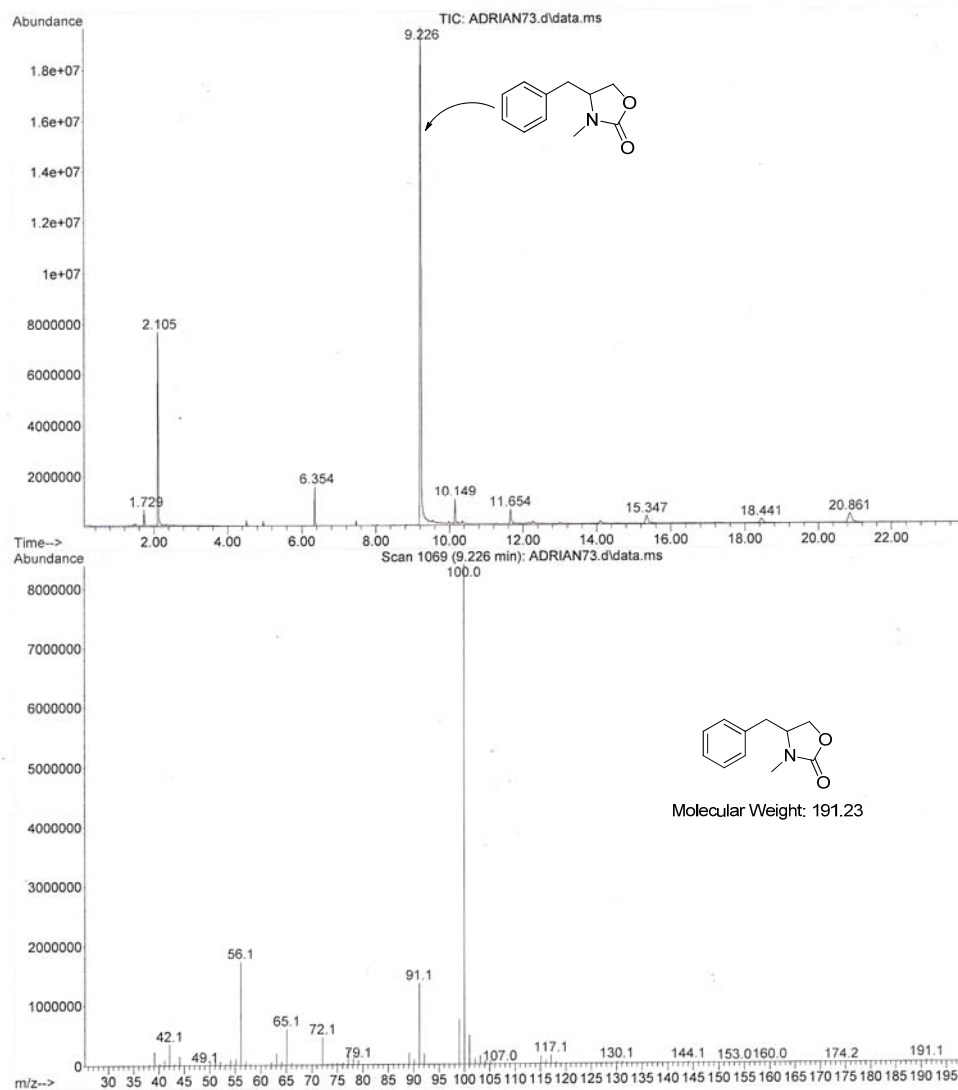


Figure 34:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 4 (part 1)

File :C:\MSDCHEM\1\DATA\OCHOA\Snapshot\ADRIAN73.d
Operator : D. CHAVEZ
Acquired : 12 Aug 2014 14:19 using AcqMethod VOLATILESPESADOS.M
Instrument : Numero 1
Sample Name: DET. DE ACETATO DE ETILO
Misc Info :
Vial Number: 4

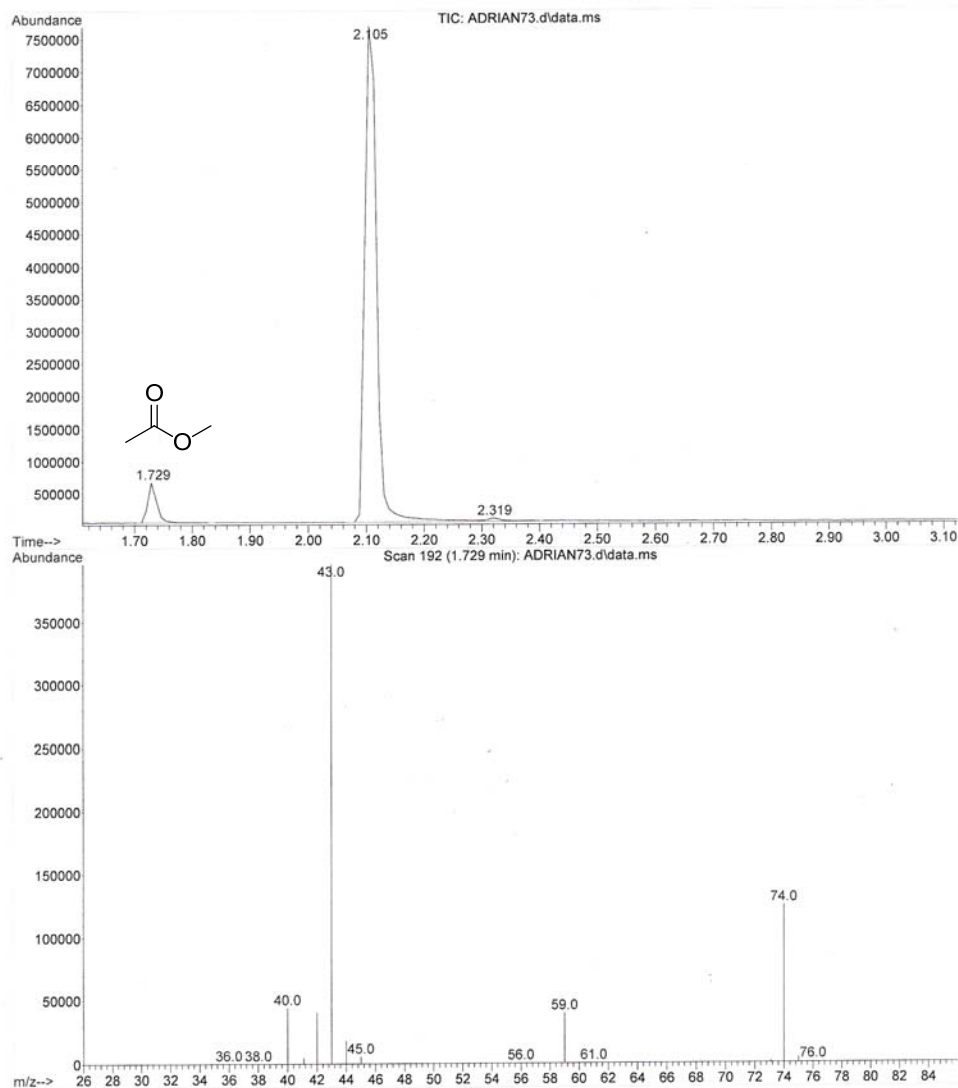
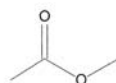
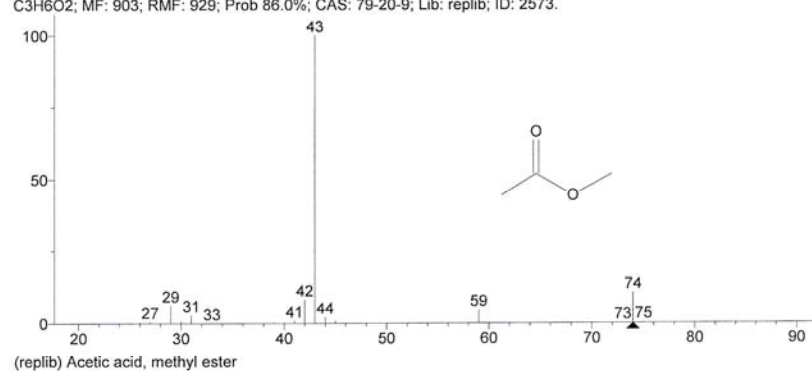


Figure 35:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 4 (part 2)

Hit 1 : Acetic acid, methyl ester
 C3H6O2; MF: 903; RMF: 929; Prob 86.0%; CAS: 79-20-9; Lib: replib; ID: 2573.



Name: Acetic acid, methyl ester
 Formula: C₃H₆O₂
 MW: 74 CAS#: 79-20-9 NIST#: 154747 ID#: 2573 DB: replib
 Other DBs: Fine, TSCA, RTECS, HODOC, NIH, EINECS, IRDB
 Contributor: Chemical Concepts
 10 largest peaks:
 43 999 | 74 105 | 42 82 | 29 62 | 59 46 | 31 29 | 44 22 | 41 9 | 30 8 | 45 8 |
 Synonyms:
 1.Methyl acetate
 2.Devoton
 3.Tereton
 4.CH₃COOCH₃
 5.Methyl ethanoate
 6.Acetate de methyle
 7.Methyl acetic ester
 8.Methylacetaat

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Figure 36:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 4 (part 3)

File :C:\MSDCHEM\1\DATA\OCHOA\Snapshot\ADRIAN73.d
 Operator : D. CHAVEZ
 Acquired : 12 Aug 2014 14:19 using AcqMethod VOLATILESPESADOS.M
 Instrument : Numero 1
 Sample Name: DET. DE ACETATO DE ETILO
 Misc Info :
 Vial Number: 4

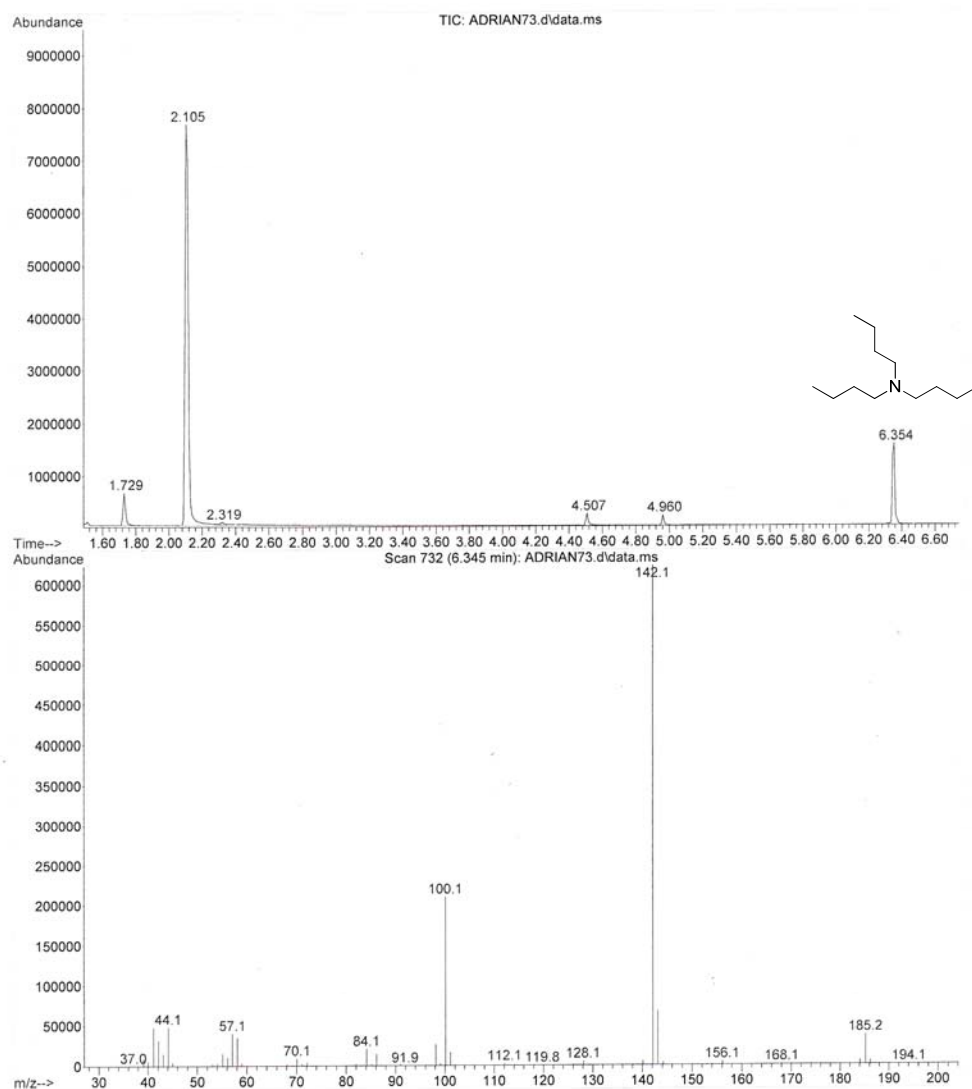
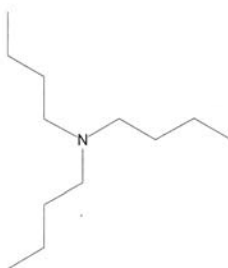
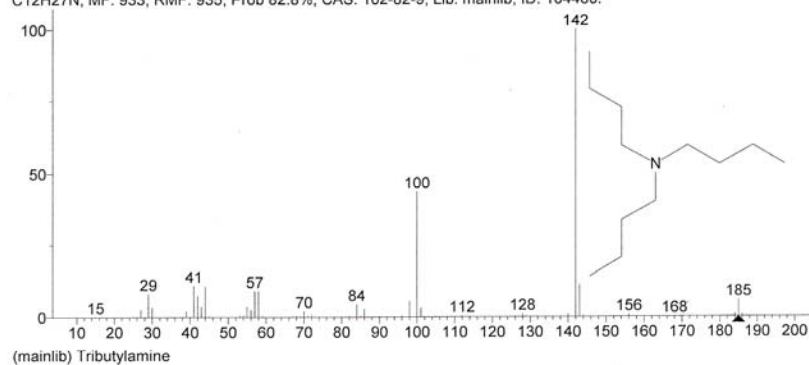


Figure 37:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 4 (part 4)

Hit 1 : Tributylamine
 C₁₂H₂₇N; MF: 933; RMF: 935; Prob 82.8%; CAS: 102-82-9; Lib: mainlib; ID: 104480.



Name: Tributylamine
 Formula: C₁₂H₂₇N
 MW: 185 CAS#: 102-82-9 NIST#: 291265 ID#: 104480 DB: mainlib
 Other DBs: Fine, TSCA, RTECS, HODOC, EINECS, IRDB
 Contributor: NIST Mass Spectrometry Data Center, 1998.
 10 largest peaks:
 142 999 | 100 435 | 143 112 | 41 110 | 44 107 | 57 91 | 58 90 | 29 82 | 42 75 | 185 58 |
 Synonyms:
 1.1-Butanamine, N,N-dibutyl-
 2.Tri-n-butylamine
 3.Tris[N-butylamine]
 4.(n-C₄H₉)₃N
 5.Tributylamina
 6.UN 2542
 7.N,N-Dibutyl-1-butanamine #

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Figure 38:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 4 (part 5)

File :C:\msdchem\1\data\OCHOA\ADRIAN73.D
Operator : D. CHAVEZ
Acquired : 12 Aug 2014 14:19 using AcqMethod VOLATILESPESADOS.M
Instrument : Numero 1
Sample Name: DET. DE ACETATO DE ETILO
Misc Info :
Vial Number: 4

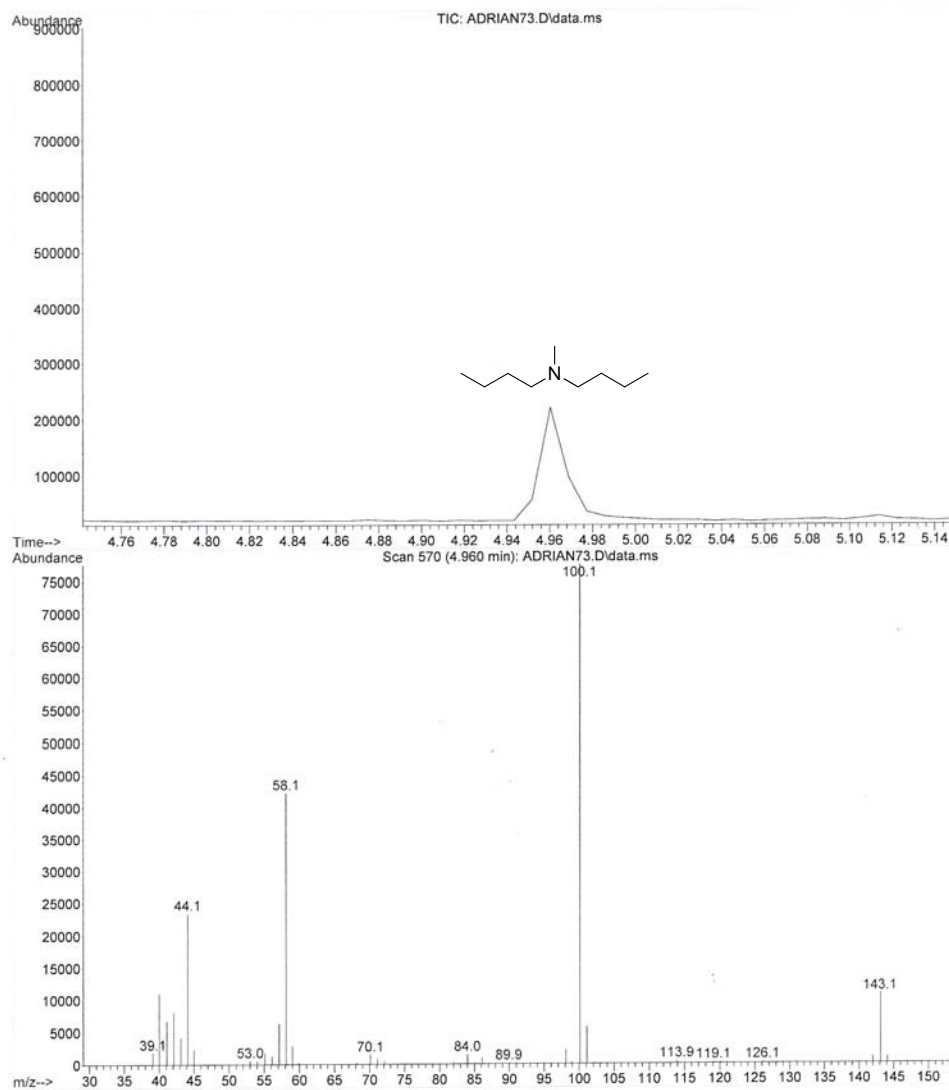
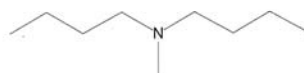
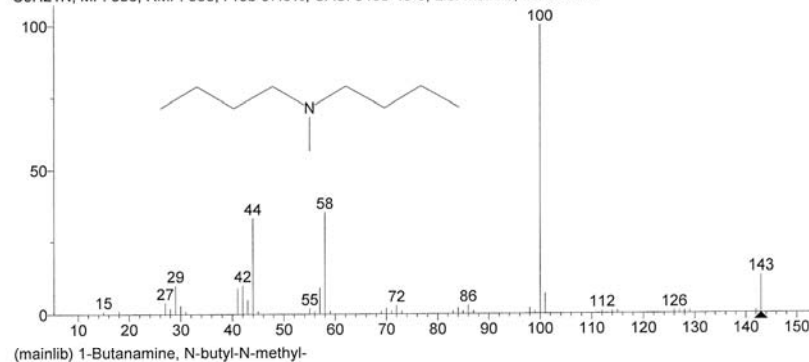


Figure 39:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 4 (part 6)

Hit 1 : 1-Butanamine, N-butyl-N-methyl-
 C₉H₂₁N; MF: 836; RMF: 866; Prob 67.0%; CAS: 3405-45-6; Lib: mainlib; ID: 62561.



Name: 1-Butanamine, N-butyl-N-methyl-
 Formula: C₉H₂₁N
 MW: 143 CAS#: 3405-45-6 NIST#: 46649 ID#: 62561 DB: mainlib
 Other DBs: Fine, TSCA, RTECS, EINECS
 Contributor: CARL DJERASSI DEPT OF CHEM STANFORD UNIV STANFORD CALIF 94305
 10 largest peaks:
 100 999 | 58 350 | 44 330 | 143 130 | 29 100 | 42 100 | 41 90 | 57 90 | 101 70 | 43 50 |
 Synonyms:
 1. Dibutylamine, N-methyl-
 2. Methanamine, N,N-dibutyl-
 3. Methyl dibutylamine
 4. N-Methyl dibutylamine
 5. N,N-Dibutylmethylamine
 6. di-n-Butylmethylamine
 7. Methyl di-n-butylamine
 8. N,N-di-n-Butylmethylamine

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Figure 40: GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 4 (part 7)

File :C:\MSDCHEM\1\DATA\OCHOA\Snapshot\ADRIAN74.d
 Operator : D. CHAVEZ
 Acquired : 12 Aug 2014 14:58 using AcqMethod VOLATILESPESADOS.M
 Instrument : Numero 1
 Sample Name: REACCION 14
 Misc Info : DET. DE ACETATO DE ETILO
 Vial Number: 6

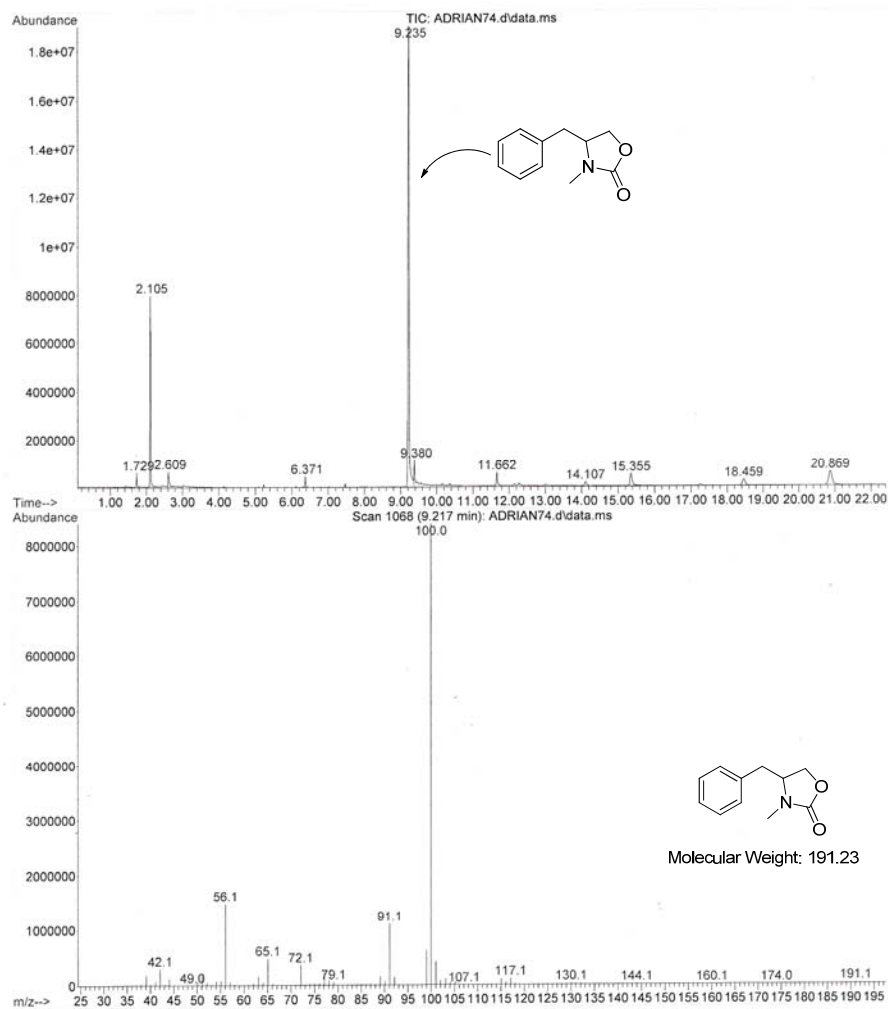


Figure 41:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 9 (part 1)

File :C:\MSDCHEM\1\DATA\OCHOA\Snapshot\ADRIAN74.d
 Operator : D. CHAVEZ
 Acquired : 12 Aug 2014 14:58 using AcqMethod VOLATILESPESADOS.M
 Instrument : Numero 1
 Sample Name: REACCION 14
 Misc Info : DET. DE ACETATO DE ETILO
 Vial Number: 6

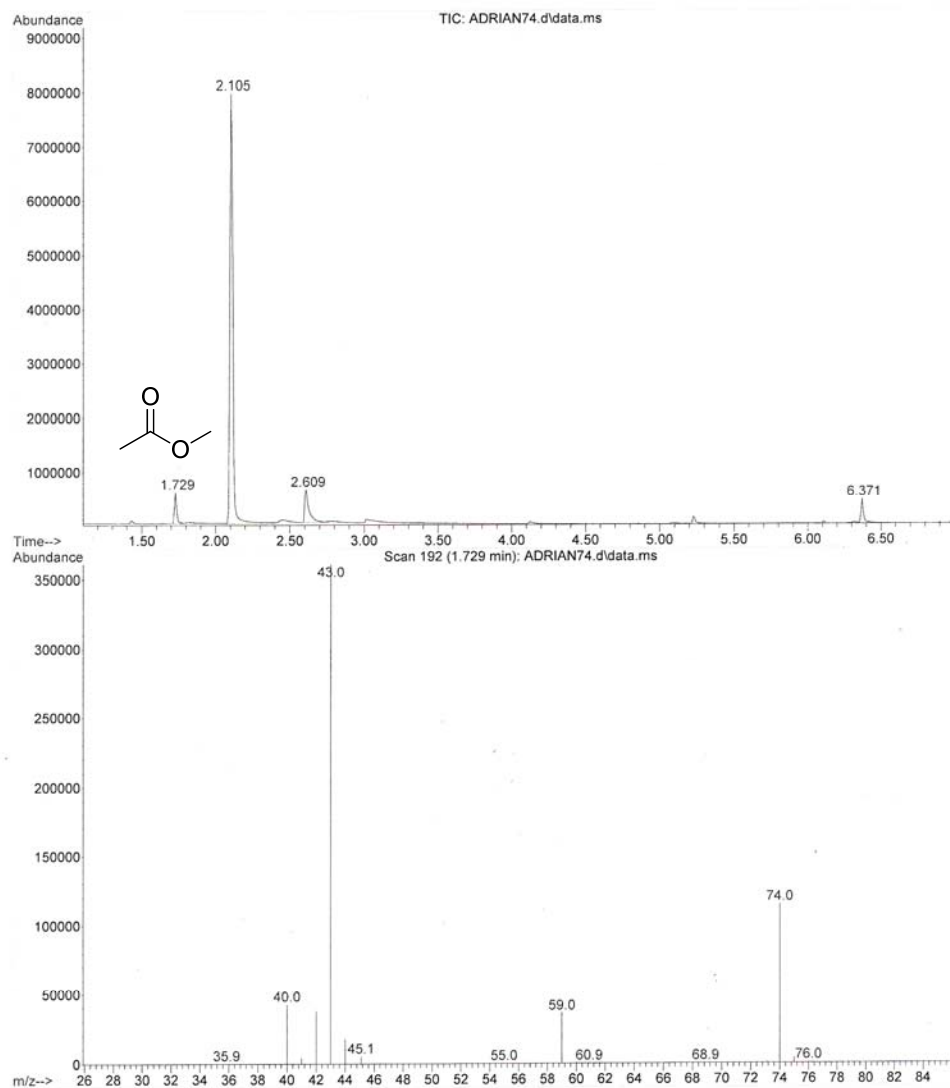
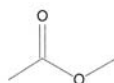
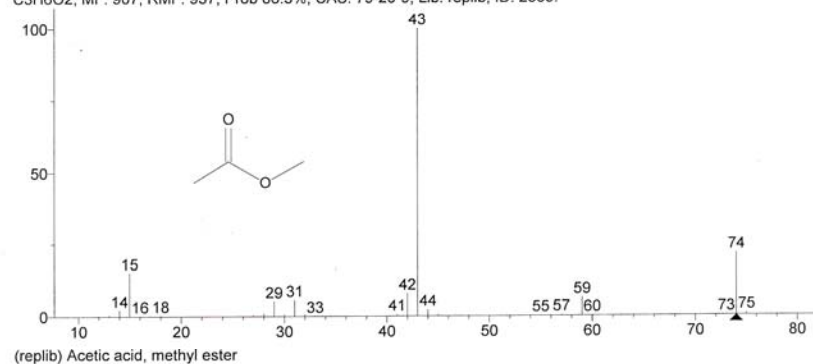


Figure 42:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 9 (part 2)

Hit 1 : Acetic acid, methyl ester
 C3H6O2; MF: 907; RMF: 937; Prob 86.3%; CAS: 79-20-9; Lib: replib; ID: 2569.



Name: Acetic acid, methyl ester
 Formula: C₃H₆O₂
 MW: 74 CAS#: 79-20-9 NIST#: 229449 ID#: 2569 DB: replib
 Other DBs: Fine, TSCA, RTECS, HODOC, NIH, EINECS, IRDB
 Contributor: Japan AIST/NIMC Database- Spectrum MS-NW- 27
 10 largest peaks:
 43 999 | 74 214 | 15 150 | 42 79 | 59 64 | 31 57 | 29 52 | 44 23 | 14 21 | 28 10 |
 Synonyms:
 1.Methyl acetate
 2.Devoton
 3.Tereton
 4.CH₃COOCH₃
 5.Methyl ethanoate
 6.Acetate de methyle
 7.Methyl acetic ester
 8.Methylacetaat

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Figure 43:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 9 (part 3)

File :C:\MSDCHEM\1\DATA\OCHOA\Snapshot\ADRIAN74.d
Operator : D. CHAVEZ
Acquired : 12 Aug 2014 14:58 using AcqMethod VOLATILESPESADOS.M
Instrument : Numero 1
Sample Name: REACCION 14
Misc Info : DET. DE ACETATO DE ETILO
Vial Number: 6

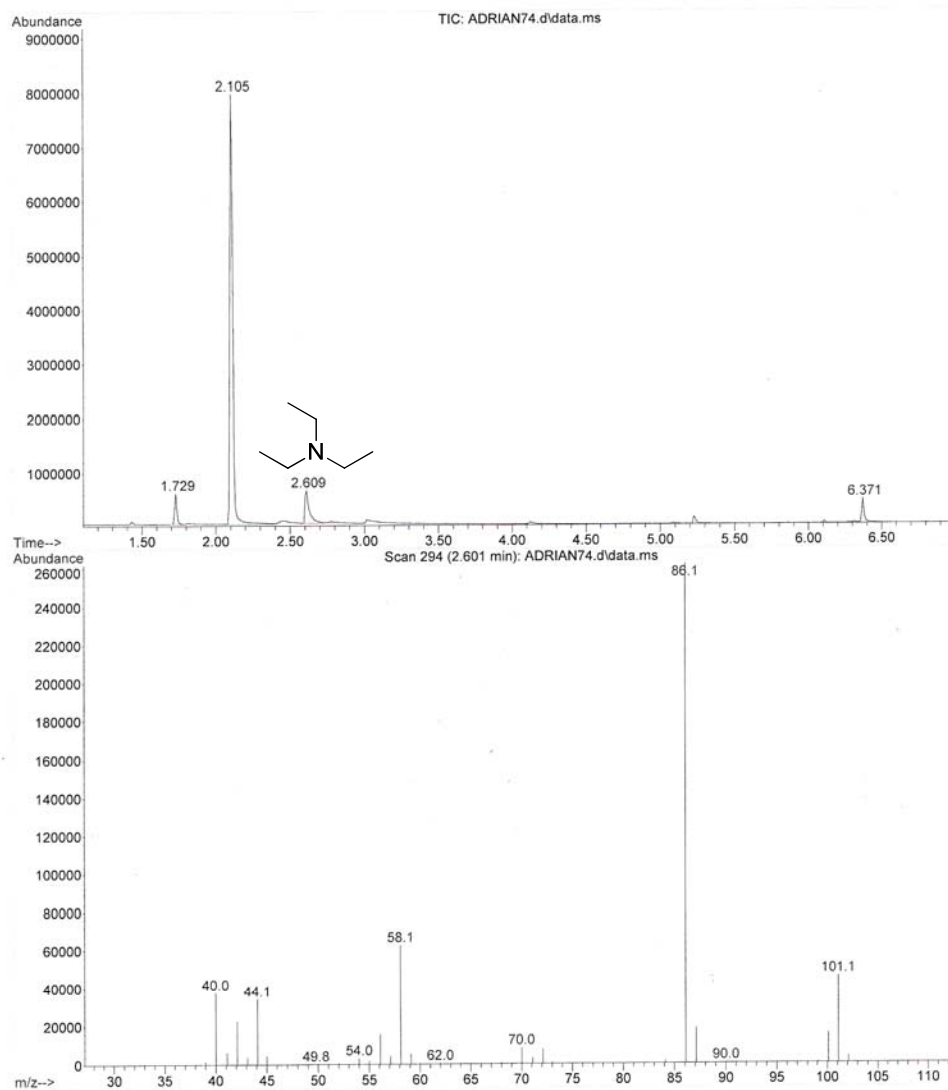
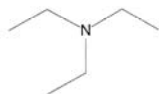
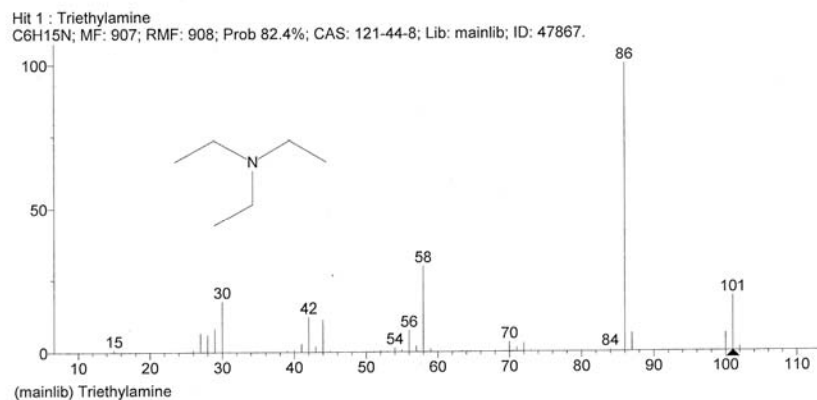


Figure 44:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 9 (part 4)



Name: Triethylamine
 Formula: C₆H₁₅N
 MW: 101 CAS#: 121-44-8 NIST#: 291545 ID#: 47867 DB: mainlib
 Other DBs: Fine, TSCA, RTECS, EPA, HODOC, EINECS, IRDB
 Contributor: NIST Mass Spectrometry Data Center, 1998.
 10 largest peaks:
 86 999 | 58 296 | 101 188 | 30 174 | 42 121 | 44 113 | 29 82 | 56 75 | 27 66 | 87 63 |
 Synonyms:
 1.Ethanamine, N,N-diethyl-
 2.(C₂H₅)₃N
 3.(Diethylamino)ethane
 4.N,N-Diethylethanamine
 5.TEN
 6.Triaethylamin
 7.Trietilamina
 8.UN 1296

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Figure 45:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 9 (part 5)

File :C:\msdchem\1\data\OCHOA\ADRIAN74.D
 Operator : D. CHAVEZ
 Acquired : 12 Aug 2014 14:58 using AcqMethod VOLATILESPESADOS.M
 Instrument : Numero 1
 Sample Name: REACCION 14
 Misc Info : DET. DE ACETATO DE ETILO
 Vial Number: 6

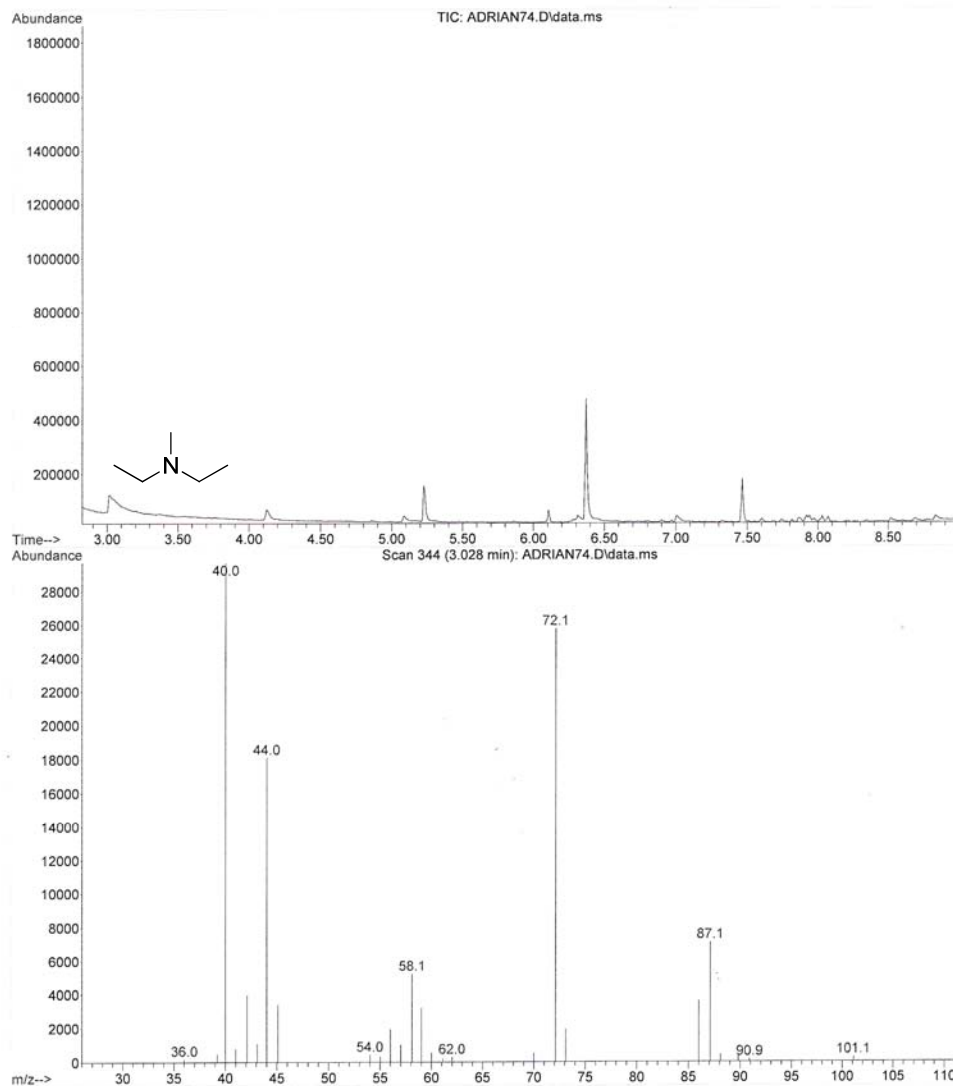
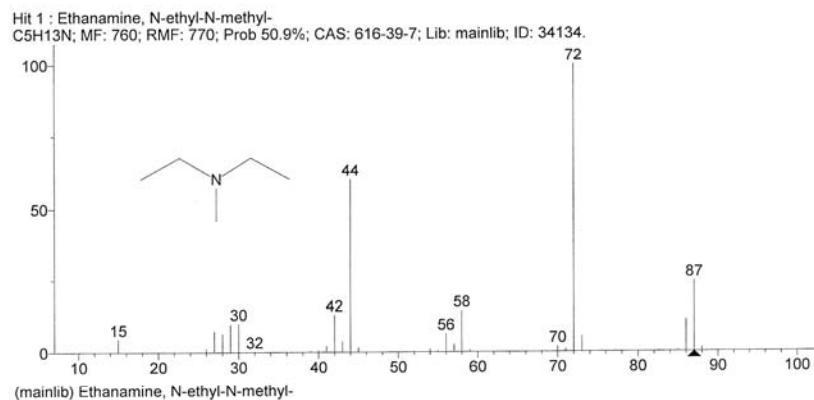


Figure 46:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 9 (part 6)



Name: Ethanamine, N-ethyl-N-methyl-
 Formula: C₅H₁₃N
 MW: 87 CAS#: 616-39-7 NIST#: 233768 ID#: 34134 DB: mainlib
 Other DBs: Fine, TSCA, HODOC, EINECS
 Contributor: Japan AIST/NIMC Database- Spectrum MS-NW-2121
 10 largest peaks:
 72 999 | 44 602 | 87 244 | 58 143 | 42 129 | 86 110 | 30 100 | 29 95 | 27 72 | 56 64 |
 Synonyms:
 1.Diethylamine, N-methyl-
 2.Diethylmethylaniline
 3.Methyldiethylamine
 4.N-Methyldiethylamine
 5.N,N-Diethylmethylaniline
 6.(C₂H₅)₂(CH₃)N
 7.N-Ethyl-N-methyl-ethanamine

Figure 47:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 9 (part 7)

File :C:\msdchem\1\data\OCHOA\ADRIAN75.D
 Operator : D. CHAVEZ
 Acquired : 14 Aug 2014 10:45 using AcqMethod GENERAL.M
 Instrument : Numero 1
 Sample Name: REACCION 12
 Misc Info :
 Vial Number: 2

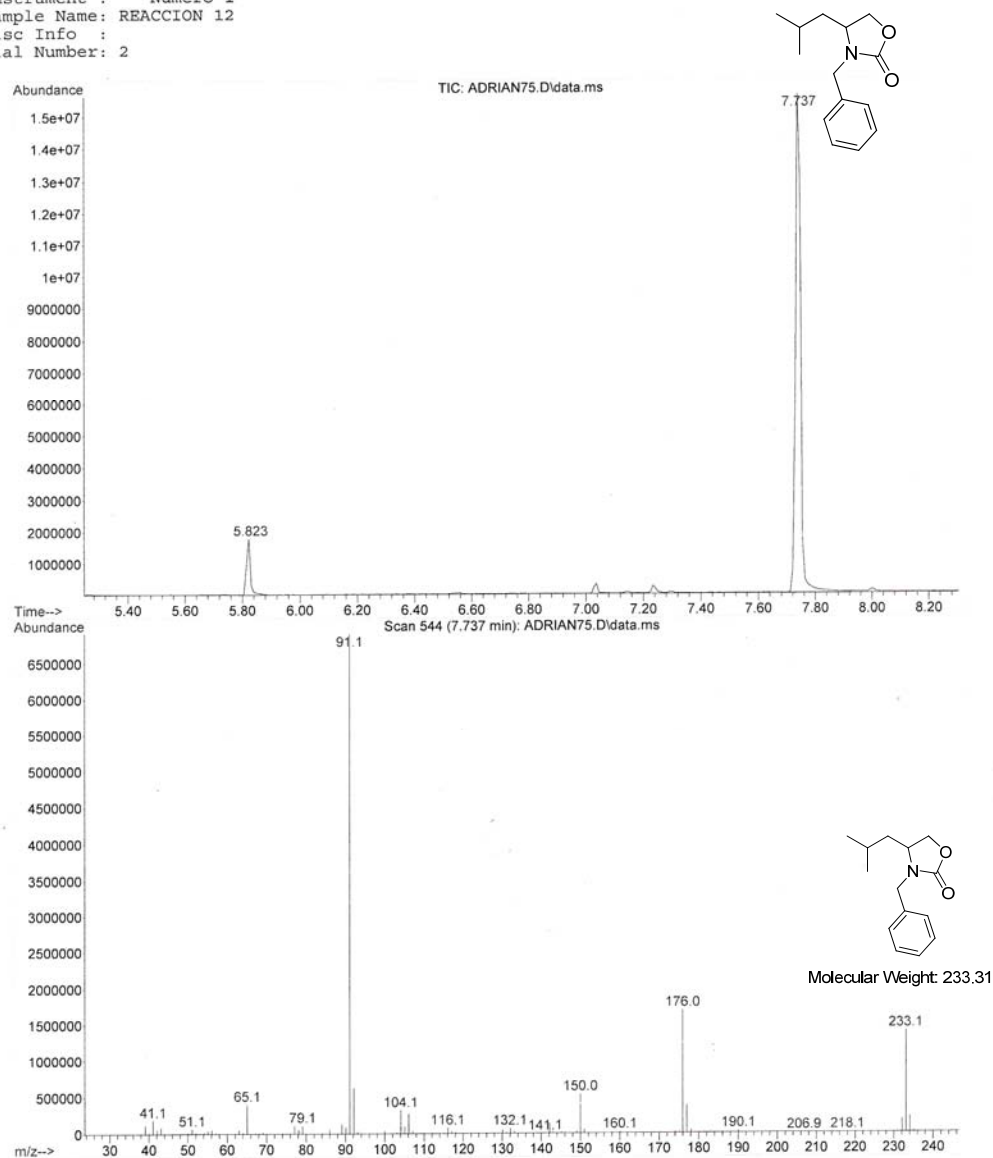


Figure 48:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 10 (part 1)

File :C:\msdchem\1\data\OCHOA\ADRIAN75.D
 Operator : D. CHAVEZ
 Acquired : 14 Aug 2014 10:45 using AcqMethod GENERAL.M
 Instrument : Numero 1
 Sample Name: REACCION 12
 Misc Info :
 Vial Number: 2

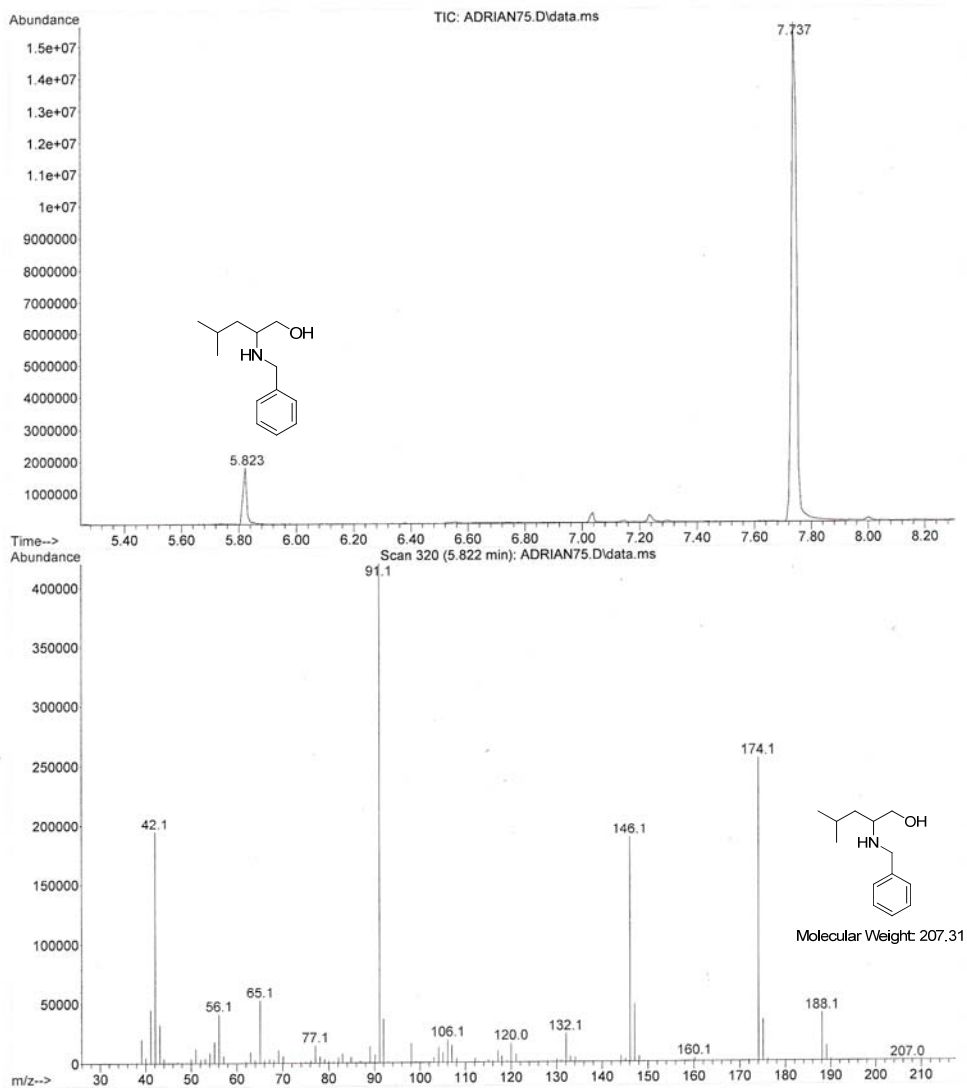


Figure 49:GC-MS of the crude of reaction obtained in conditions described in Table 2, entry 10 (part 2)