HIMC-RE072401

결과보고서

(GC-MSMS, HPLC screening)

제주대학교 휴먼인터페이스미디어센터

분 석 개 요

의뢰내용	GC-MSMS 및 HPLC를 통한 의뢰시료 screening 분석

의뢰기관	기관명	제주 자하연	사업자번호	890-06-02489
	의뢰자	김희용	부서/직함	
	전화번호	010-3775-7701	이메일	yong7598@hanmail.net
	주소	제주특별자치도 서귀포시 표선면 번영로 3113		
	의뢰일	2023-07-24		

시험기관	기관명/소속	제주대학교 / 휴먼인터페이스미디어센터
	소재지	제주특별자치도 제주시 제주대학로 102
	센터장	이봉규
	시험담당자	김건윤
	시험책임자	이정아
	연락처	064-754-8340

시험기간	2023-07-24 ~ 2023-07-31
보 고 일	2023-08-01

제주대학교 휴먼인터페이스미디어센터장

목 차

1. 요약

GC-MSMS 및 HPLC를 이용하여 시료 2종에 대한 Screening 분석을 실시함

2. 시약 및 시액

▶ 디클로로메탄(Dichloromethane), 아세토니트릴(Acetonitrile), 증류수, 메탄올(Methanol), 트리플루오로아세트산(Trifluoroacetic acid)

▶ 분석시료 : 감귤 생체수, 가지 생체수

3. 시험용액의 조제

▶ 시험용액의 조제

- 의뢰시료를 분액깔때기에 옮겨 Dichloromethane을 가하고 강하게 흔들어 혼합한 후 1시간 이상 정치하여 분리된 유기용매층을 취하고, 이를 40 ℃ 이하에서 감압 농축으로 용매를 모두 날려버린 다음 재용해(Dichloromethane), 여과(PVDF filter, 0.2μm)하여 GC-MSMS 분석에 사용함.
- HPLC 시험용액 의뢰시료를 PTFE filter(0.45μm)로 여과하여 사용함.

4. 시험조작

▶ GC-MSMS(Shimadzu, TQ-8050NX) 분석조건

- 컬럼 : SH-Rxi™-5Sil MS (30 m × 0.25 ㎜ × 0.25 ㎛)
- 이동상 가스 및 유속 : He, 1.56 mL/min
- 온도 : Injector = 280 ℃
  Ion source = 250 ℃
  Interface = 300 ℃
- 주입부 : split(10:1)

▶ 액체크로마토그래피의 측정조건

- 검출기 : PDA detector (272 nm)
- 칼럼 : Shim-pack GIS C18 (4.6 mm×250 mm, 5μm)
- 이동상 = 아세토니트릴 : 0.1% 트리플루오로아세트산(water) (Gradient)
- 이동상 유량 : 1.0 mL/min
- 주입량 : 10 μL

▶ 시험용액을 위 조건에 따라 각각의 분석기기에 주입함

- GC-MSMS : 검출된 Peak 간의 면적비율 확인 및 Library searching을 실시
- HPLC : Peak의 Retention Time 과 Wavelength로부터 검체 중의 유효성분 함유 여부 유추

5. 시험결과

▶ 의뢰 시료 분석결과(GC-MSMS)

- Chromatograph 내 Area 면적값 상위 Peak 중 유효한 것을 선택하여 Library searching을 진행함

<감귤 생체수>

NO.	Ingredient	%	비고
1	Linalool	1.9	
2	*p*-Menthone	14.8	
3	Isomethone	3.4	
4	*neo*-Menthol	2.3	
5	L-(-)-Menthol	33.7	
6	.alpha.-Terpineol	1.4	
7	Dihydrocarvone	1.7	
8	D-Carvone	19.6	
9	Piperitone	3.4	
10	Menthyl acetate	11.1	
11	Veridiflorol	0.6	
12	9-Octadecenamide, (Z)-	6.0	
	Total	100	

※ Library searching을 통해 확인한 성분명이므로 실제 정성분석시 결과가 바뀔 수 있음.

※ 나타난 PEAK의 단순 면적으로 산출한 (%)이므로 실제 정량분석시 수치가 바뀔 수 있음.

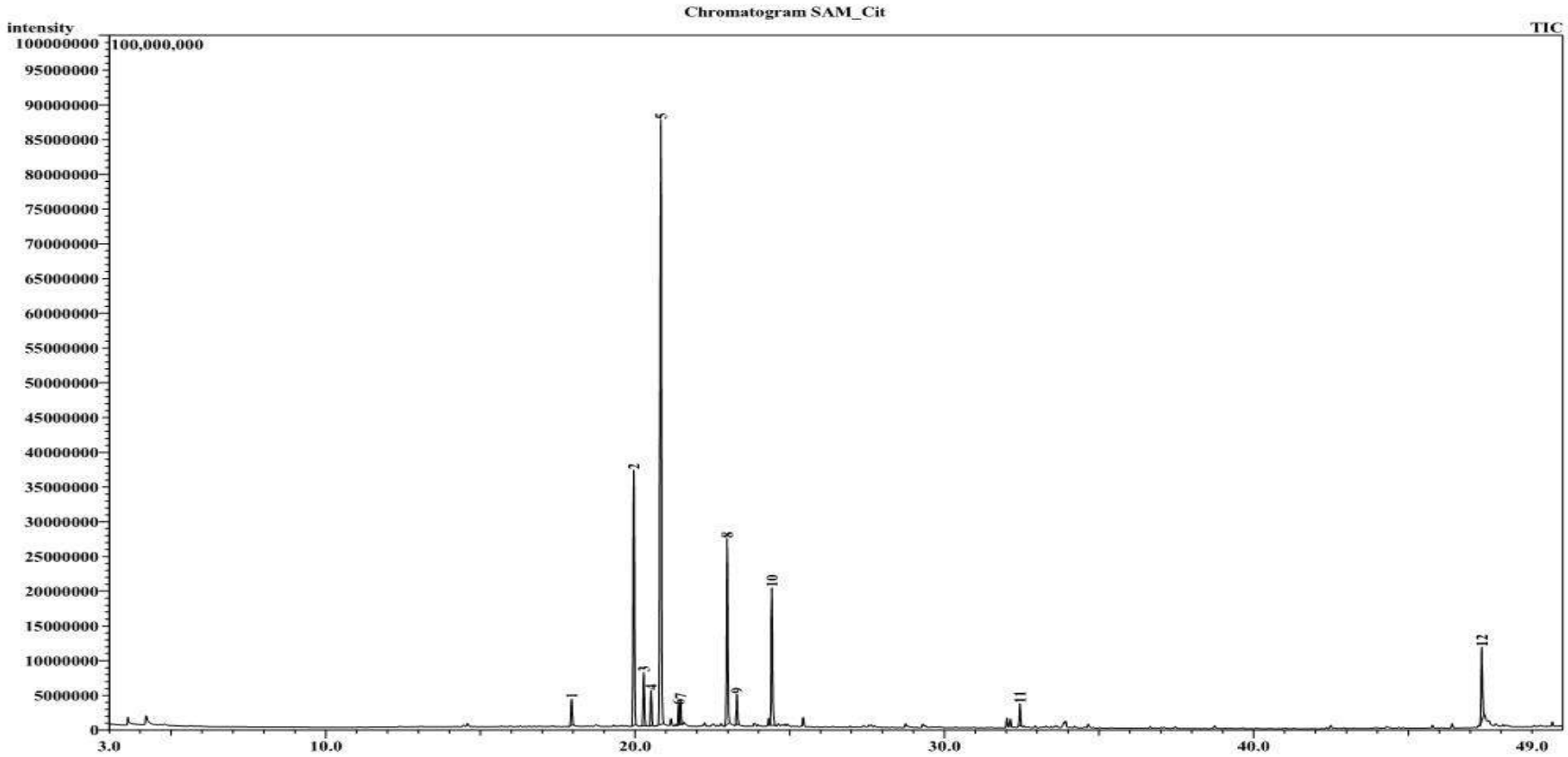
<가지 생체수>

NO.	Ingredient	%	비고
1	Benzeneethanol (CAS)	15.6	
2	*p*-Menthone	3.6	
3	L-(-)-Menthol	5.6	
4	(-)-Carvone	1.9	
5	Piperitone	2.3	
6	Cinnamaldehyde, (E)-	54.6	
7	Menthyl acetate	3.1	
8	9-Octadecenamide, (Z)-	13.1	
	Total	100	

※ Library searching을 통해 확인한 성분명이므로 실제 정성분석시 결과가 바뀔 수 있음.

※ 나타난 PEAK의 단순 면적으로 산출한 (%)이므로 실제 정량분석시 수치가 바뀔 수 있음.

<감귤 생체수>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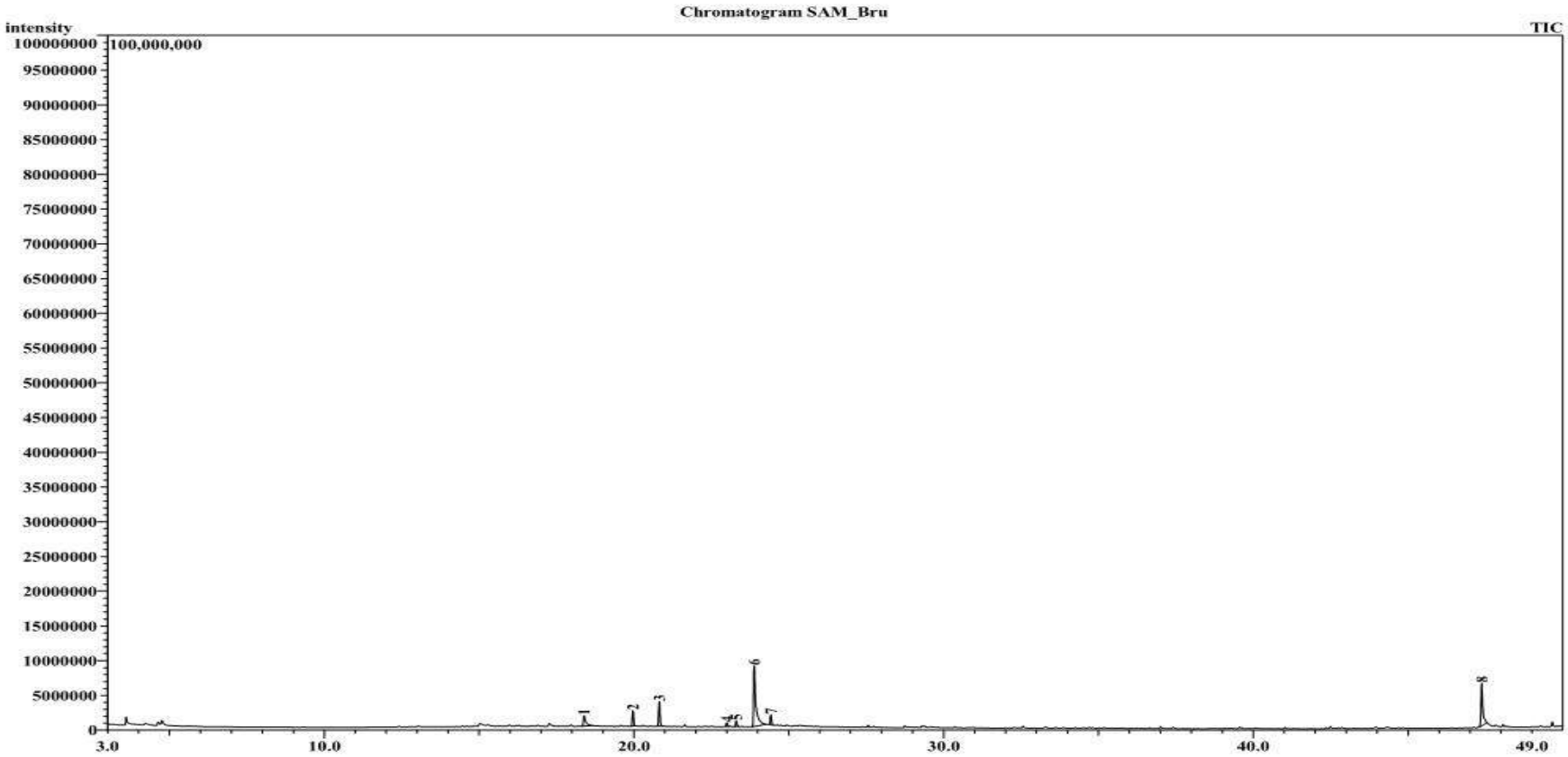
12

<가지 생체수>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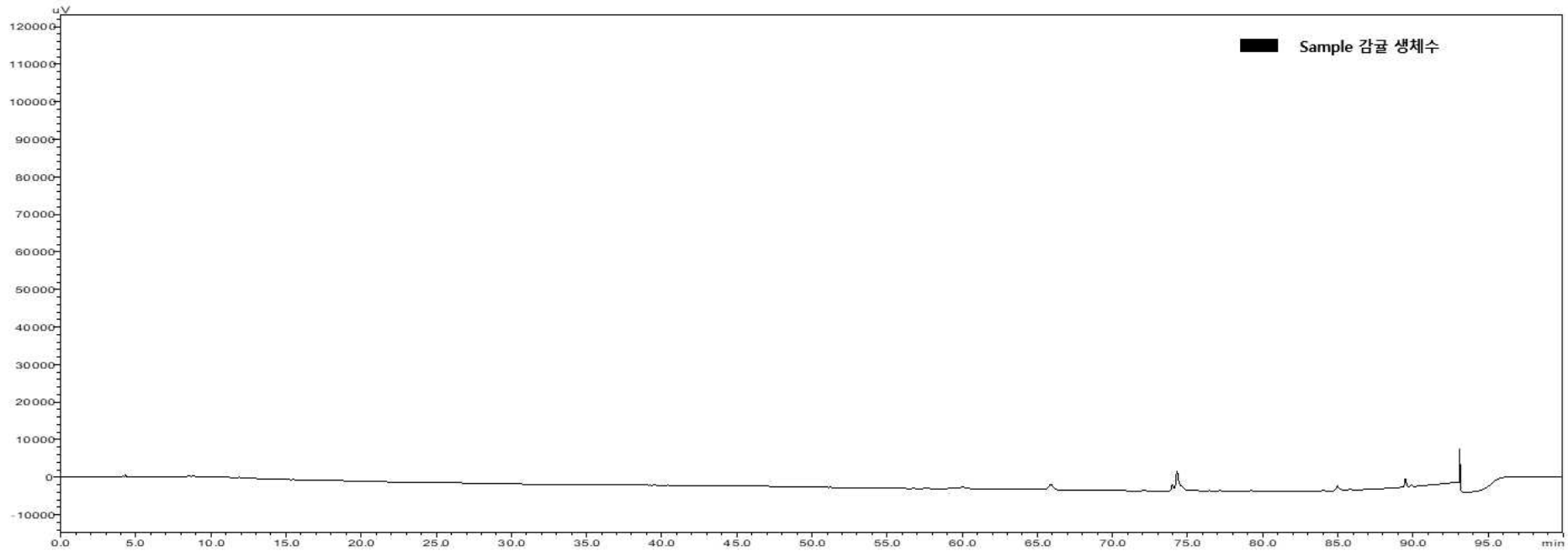
40000000
35000000
30000000
25000000
20000000
15000000
10000000
5000000
0
3.0
10.0
20.0
30.0
40.0
49.0

▶ 의뢰 시료 분석결과(HPLC)

<감귤 생체수>

- Chromatograph에서 보유 STD와 비교 가능한 수준의 유효성분 peak가 검출되지 않음

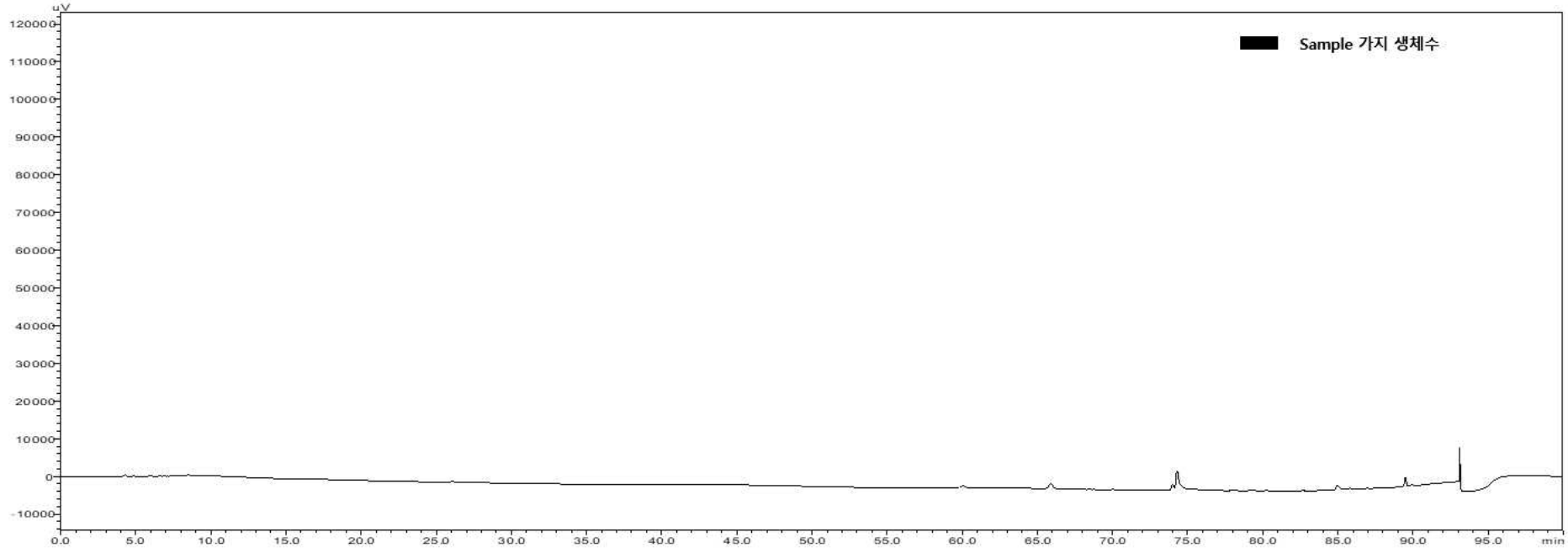
※ 본 데이터는 단순 Screening Method를 적용한 실험결과이므로 정밀, 정량분석 시 결과와 다를 수 있음



<가지 생체수>

- Chromatograph에서 보유 STD와 비교 가능한 수준의 유효성분 peak가 검출되지 않음

※ 본 데이터는 단순 Screening Method를 적용한 실험결과이므로 정밀, 정량분석 시 결과와 다를 수 있음



6. 첨부

<감귤 생체수_GC-MSMS>

==== HIMC GCMS Report ====

Sample Information

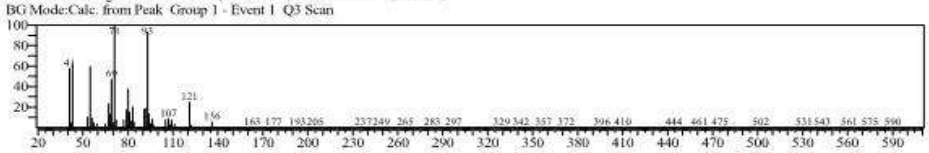
Analyzed by	: Admin
Analyzed	: 7/29/2023 6:07:23 AM
Sample Type	: Unknown
Level #	: 1
Sample Name	: SAM_Cit
Sample ID	: SAM_Cit
$EndIfSIS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 14
Injection Volume	: 1.00

Peak#	Name	%
1	Linalool	1.91
2	p-Menthone	14.85
3	Isomethone	3.41
4	neo-Menthol	2.28
5	L-(-)-Menthol	33.66
6	.alpha.-Terpineol	1.44
7	Dihydrocarvone	1.73
8	D-Carvone	19.55
9	Piperitone	3.45
10	Menthyl acetate	11.08
11	Veridiflorol	0.63
12	9-Octadecenamide, (Z)-	6.01

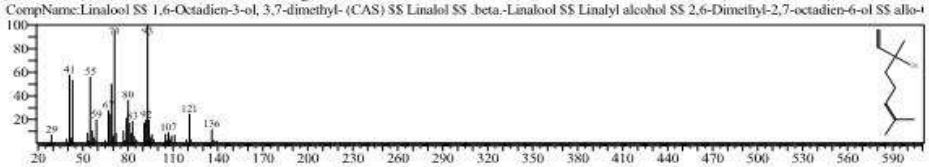
<Library search>

Library

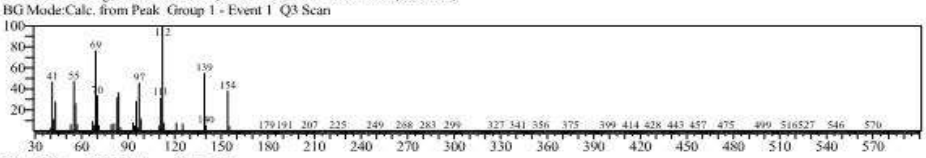
<< Target >>
Line#:1 R.Time:17.950(Scan#:2991) MassPeaks:331
RawMode:Averaged 17.945-17.955(2990-2992) BasePeak:71.05(509994)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



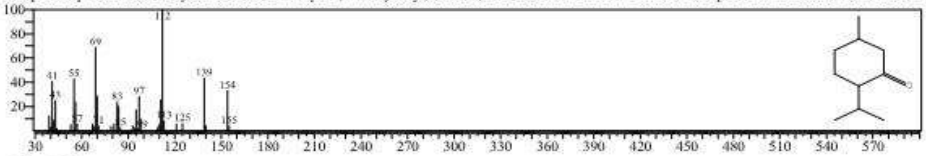
Hit#:1 Entry:57962 Library:Wiley9.lib
SI:96 Formula:C10H18O CAS:78-70-6 MolWeight:154 RetIndex:0
CompName:Linalool $$ 1,6-Octadien-3-ol, 3,7-dimethyl- (CAS) $$ Linalol $$ .beta.-Linalool $$ Linalyl alcohol $$ 2,6-Dimethyl-2,7-octadien-6-ol $$ allo-



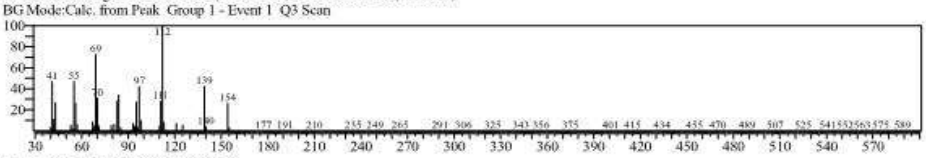
<< Target >>
Line#:2 R.Time:19.960(Scan#:3393) MassPeaks:329
RawMode:Averaged 19.955-19.965(3392-3394) BasePeak:112.10(4958252)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



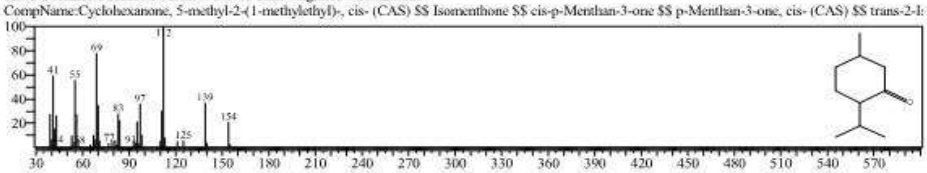
Hit#:2 Entry:58181 Library:Wiley9.lib
SI:95 Formula:C10H18O CAS:89-80-5 MolWeight:154 RetIndex:0
CompName:p-Menthone $$ Cyclohexanone, 5-methyl-2-(1-methylethyl)-, trans- (CAS) $$ trans-Menthan-3-one $$ trans-p-Menthan-3-one $$ Menthone $$



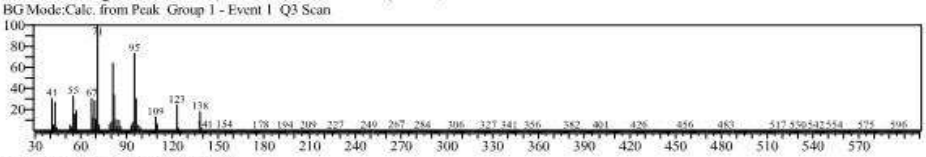
<< Target >>
Line#:3 R.Time:20.280(Scan#:3457) MassPeaks:309
RawMode:Averaged 20.275-20.285(3456-3458) BasePeak:112.10(1117801)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



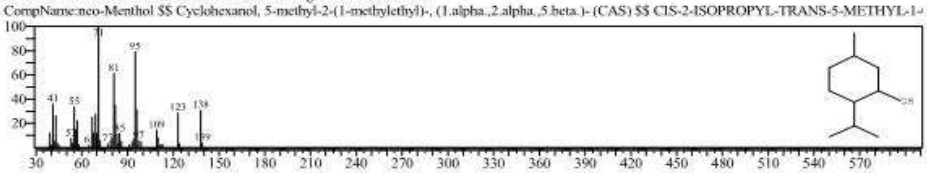
Hit#:1 Entry:58164 Library:Wiley9.lib
SI:96 Formula:C10H18O CAS:491-07-6 MolWeight:154 RetIndex:0
CompName:Cyclohexanone, 5-methyl-2-(1-methylethyl)-, cis- (CAS) $$ Isomenthone $$ cis-p-Menthan-3-one $$ p-Menthan-3-one, cis- (CAS) $$ trans-2-I



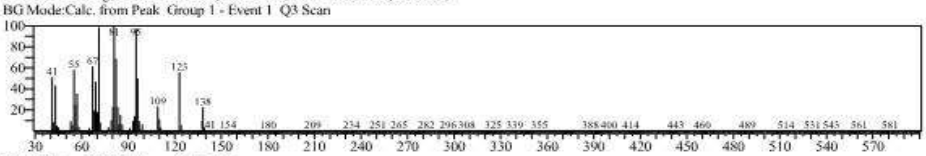
<< Target >>
Line#:4 R.Time:20.520(Scan#:3505) MassPeaks:319
RawMode:Averaged 20.515-20.525(3504-3506) BasePeak:71.05(780306)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



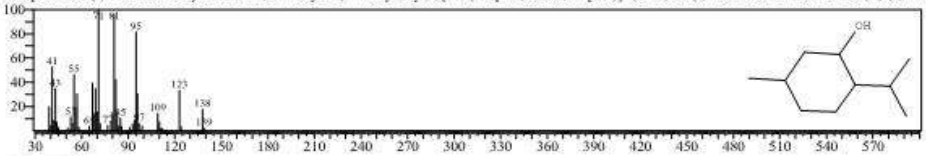
Hit#:1 Entry:61823 Library:Wiley9.lib
SI:97 Formula:C10H20O CAS:491-01-0 MolWeight:156 RetIndex:0
CompName:neo-Menthol $$ Cyclohexanol, 5-methyl-2-(1-methylethyl)-, (1.alpha.,2.alpha.,5.beta.)- (CAS) $$ CIS-2-ISOPROPYL-TRANS-5-METHYL-1-



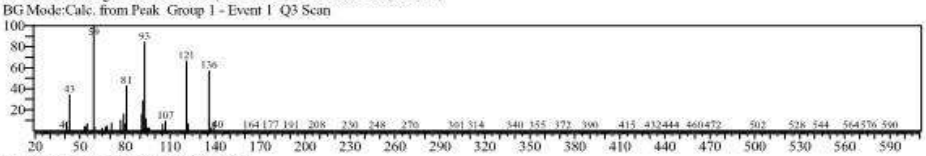
<< Target >>
Line#:5 R.Time:20.835(Scan#:3568) MassPeaks:357
RawMode:Averaged 20.830-20.840(3567-3569) BasePeak:81.05(8302681)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



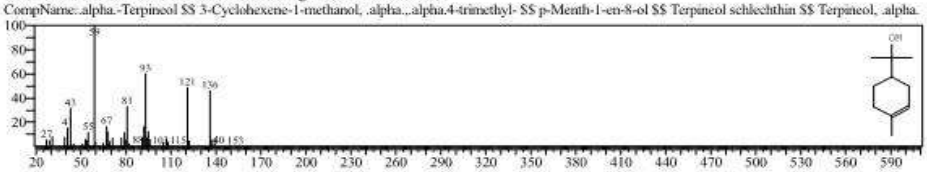
Hit#:3 Entry:61809 Library:Wiley9.lib
SI:94 Formula:C10H20O CAS:2216-51-5 MolWeight:156 RetIndex:0
CompName:L-(-)-Menthol $$ Cyclohexanol, 5-methyl-2-(1-methylethyl)-, [1R-(1.alpha.,2.beta.,5.alpha.)]- (CAS) $$ (-)-Menthol $$ l-Menthol $$ (R)-(-)-M



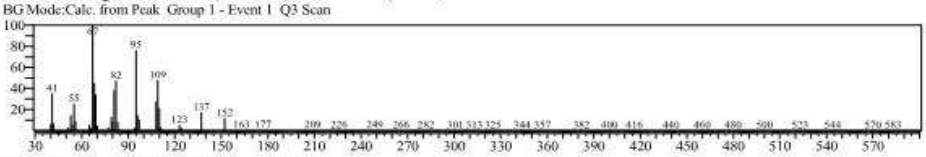
<< Target >>
Line#:6 R.Time:21.405(Scan#:3682) MassPeaks:280
RawMode:Averaged 21.400-21.410(3681-3683) BasePeak:59.05(400771)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



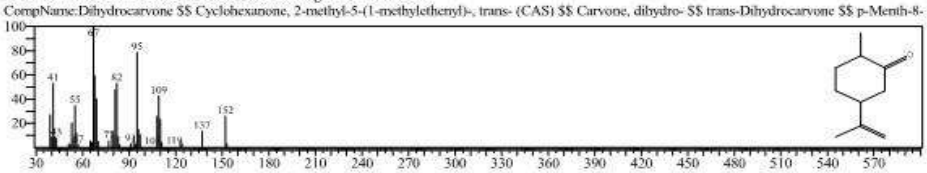
Hit#:4 Entry:10327 Library:NIST14s.lib
SI:91 Formula:C10H18O CAS:98-55-5 MolWeight:154 RetIndex:1143
CompName:.alpha.-Terpineol $$ 3-Cyclohexene-1-methanol, .alpha.,.alpha.4-trimethyl- $$ p-Menth-1-en-8-ol $$ Terpineol schlechthin $$ Terpineol, .alpha.



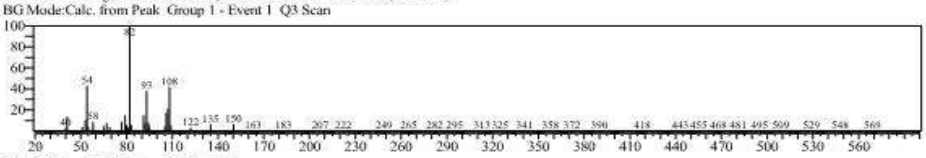
<< Target >>
Line#:7 R.Time:21.470(Scan#:3695) MassPeaks:289
RawMode:Averaged 21.465-21.475(3694-3696) BasePeak:67.05(416189)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



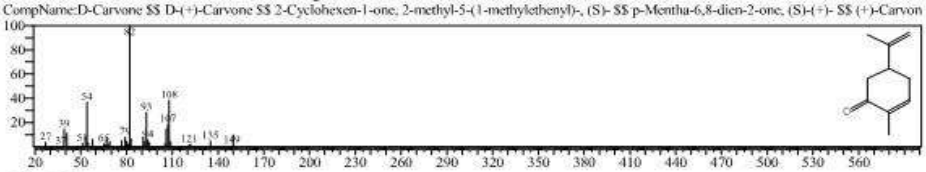
Hit#:2 Entry:54319 Library:Wiley9.lib
SI:94 Formula:C10H16O CAS:5948-04-9 MolWeight:152 RetIndex:0
CompName:Dihydrocarvone $$ Cyclohexanone, 2-methyl-5-(1-methylethenyl)-, trans- (CAS) $$ Carvone, dihydro- $$ trans-Dihydrocarvone $$ p-Menth-8-



<< Target >>
Line#:8 R.Time:22.980(Scan#:3997) MassPeaks:339
RawMode:Averaged 22.975-22.985(3996-3998) BasePeak:82.05(6440711)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



Hit#:1 Entry:9293 Library:NIST14s.lib
SI:95 Formula:C10H14O CAS:2244-16-8 MolWeight:150 RetIndex:1190
CompName:D-Carvone $$ D-(+)-Carvone $$ 2-Cyclohexen-1-one, 2-methyl-5-(1-methylethenyl)-, (S)- $$ p-Mentha-6,8-dien-2-one, (S)-(+)- $$ (+)-Carvon



<< Target >>
Line#:9 R.Time:23.295(Scan#:4060) MassPeaks:344
RawMode:Averaged 23.290-23.300(4059-4061) BasePeak:82.05(1000010)
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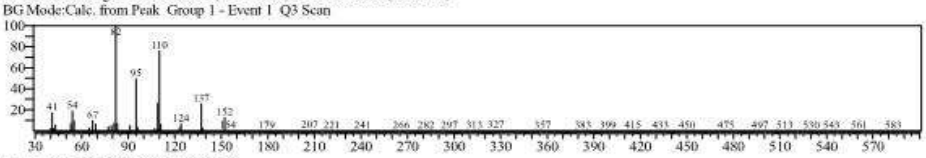
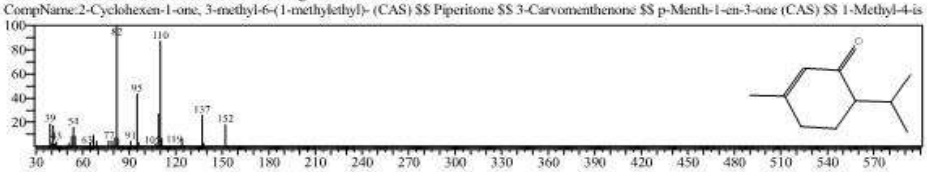
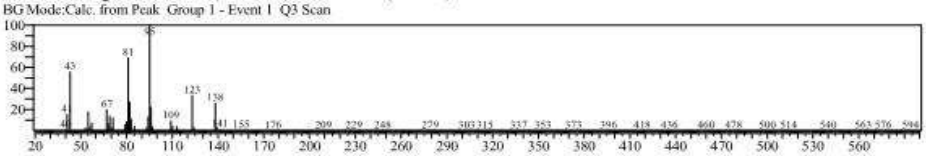


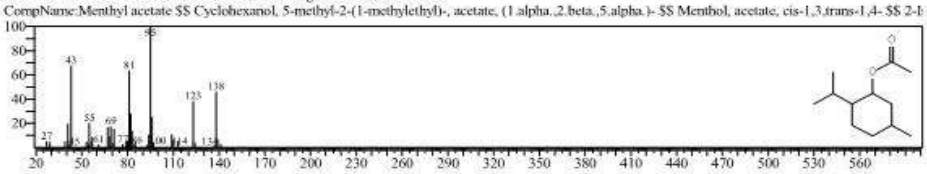
Hit#:1 Entry:54226 Library:Wiley9.lib
SI:97 Formula:C10H16O CAS:89-81-6 MolWeight:152 RetIndex:0
CompName:2-Cyclohexen-1-one, 3-methyl-6-(1-methylethyl)- (CAS) $$ Piperitone $$ 3-Carvomenthenone $$ p-Menth-1-en-3-one (CAS) $$ 1-Methyl-4-is

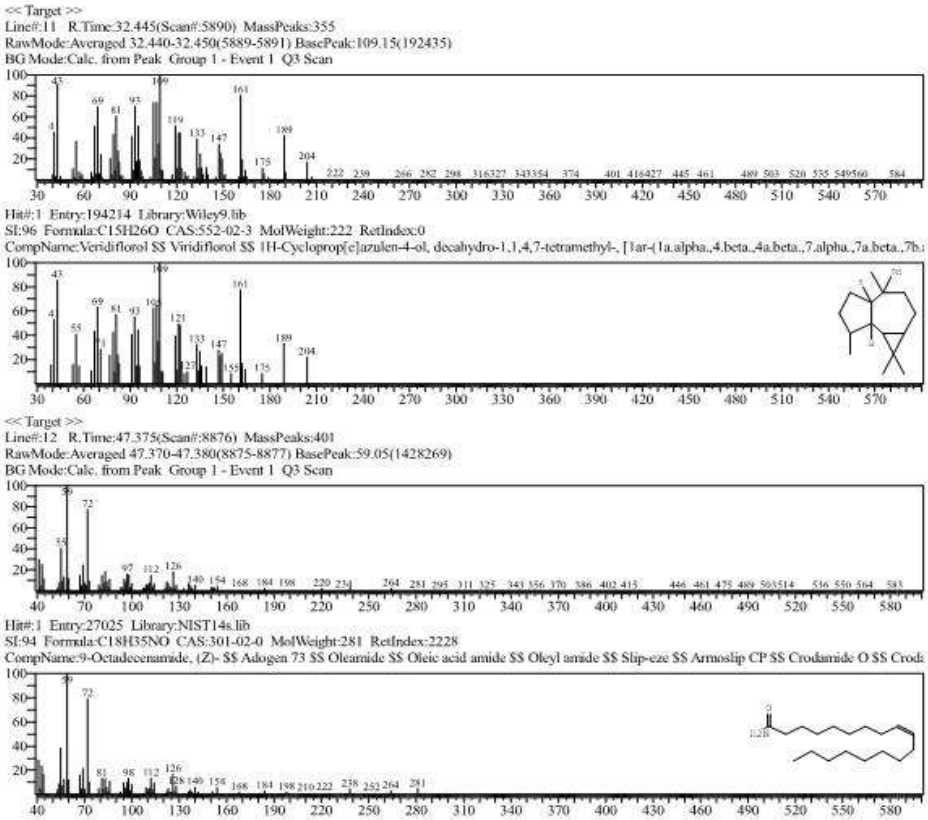


<< Target >>
Line#:10 R.Time:24.425(Scan#:4286) MassPeaks:351
RawMode:Averaged 24.420-24.430(4285-4287) BasePeak:95.10(3950752)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



Hit#:2 Entry:18172 Library:NIST14s.lib
SI:95 Formula:C12H22O2 CAS:89-48-5 MolWeight:198 RetIndex:1304
CompName:Menthyl acetate $$ Cyclohexanol, 5-methyl-2-(1-methylethyl)-, acetate, (1.alpha.,2.beta.,5.alpha.)- $$ Menthol, acetate, cis-1,3,trans-1,4- $$ 2-I

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Line#:11 R.Time:32.445(Scan#:5890) MassPeaks:355
RawMode:Averaged 32.440-32.450(5889-5891) BasePeak:109.15(192435)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan
Hit#:1 Entry:194214 Library:Wiley9.lib
SI:96 Formula:C15H26O CAS:552-02-3 MolWeight:222 RetIndex:0
CompName:Veridiflorol $$ Viridiflorol $$ 1H-Cycloprop[e]azulen-4-ol, decahydro-1,1,4,7-tetramethyl-, [1ar-(1a.alpha.,4.beta.,4a.beta.,7.alpha.,7a.beta.,7b.
<< Target >>
Line#:12 R.Time:47.375(Scan#:8876) MassPeaks:401
RawMode:Averaged 47.370-47.380(8875-8877) BasePeak:59.05(1428269)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan
Hit#:1 Entry:27025 Library:NIST14s.lib
SI:94 Formula:C18H35NO CAS:301-02-0 MolWeight:281 RetIndex:2228
CompName:9-Octadecenamide, (Z)- $$ Adogen 73 $$ Oleamide $$ Oleic acid amide $$ Oleyl amide $$ Slip-eze $$ Armoslip CP $$ Crodamide O $$ Crod

<감귤 생체수_HPLC>

8/1/2023 4:52:08 PM Page 1 / 1

SHIMADZU LabSolutions Analysis Report

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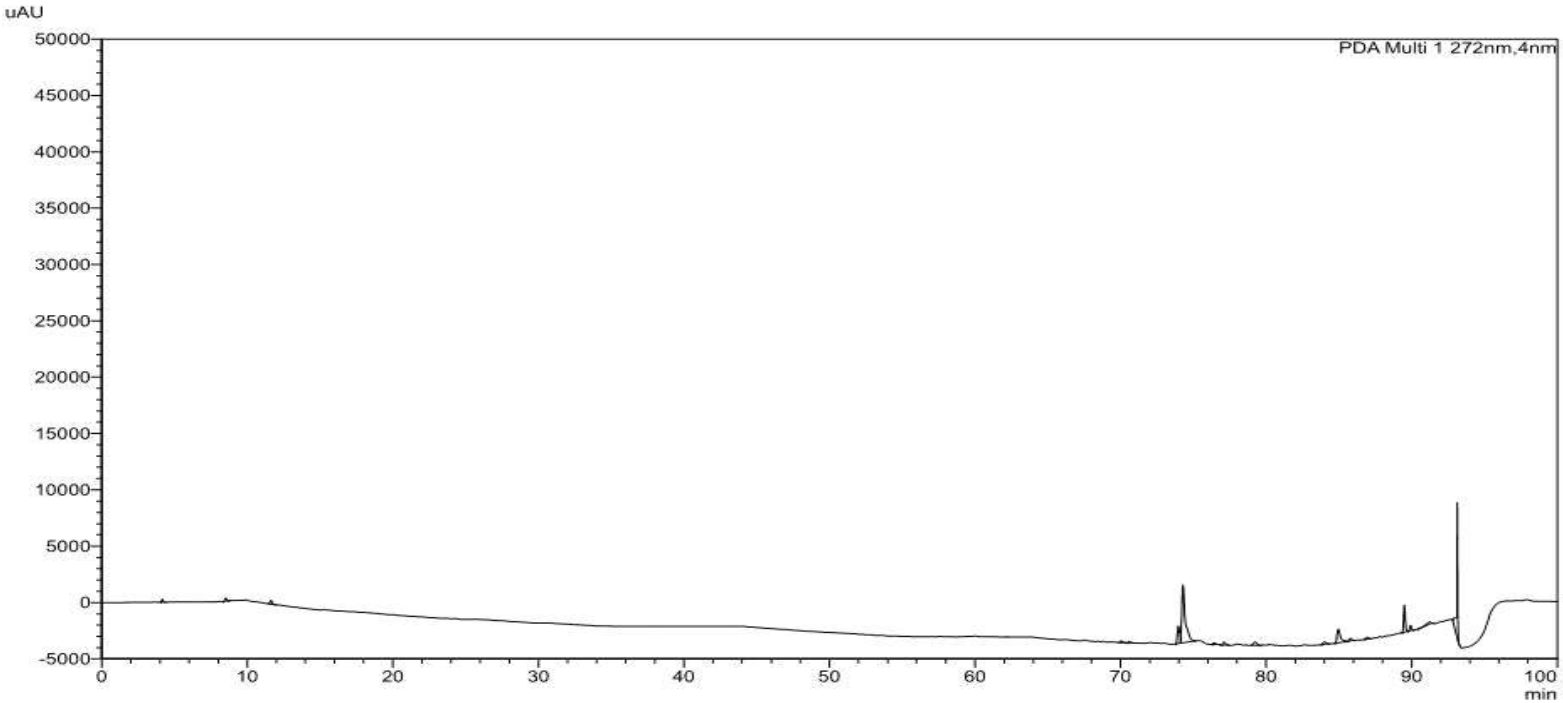


C:\HIMC\DuRae\Chlorogenic(+Neo) acid\bipayeob\230724\SAM Gamgyul0731.lcd

<가지 생체수>

===== HIMC GCMS Report =====

Sample Information

Analyzed by	: Admin
Analyzed	: 7/29/2023 4:59:11 AM
Sample Type	: Unknown
Level #	: 1
Sample Name	: SAM_Bru
Sample ID	: SAM_Bru
$EndIf$IS Amount	: [1]=1
Sample Amount	: 1
Dilution Factor	: 1
Vial #	: 13
Injection Volume	: 1.00

Peak#	Name	%
1	Benzeneethanol (CAS)	15.63
2	p-Menthone	3.64
3	L-(-)-Menthol	5.61
4	(-)-Carvone	1.89
5	Piperitone	2.33
6	Cinnamaldehyde, (E)-	54.64
7	Menthyl acetate	3.13
8	9-Octadecenamide, (Z)-	13.13

<Library search>

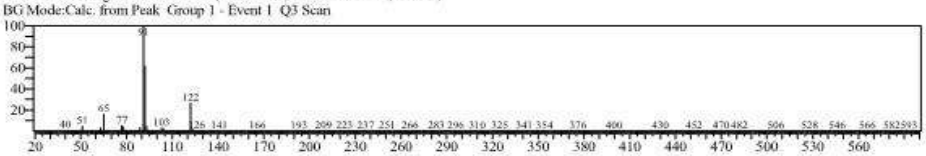
Library

<< Target >>

Line#:1 R.Time:18.385(Scan#:3078) MassPeaks:292

RawMode:Averaged 18.380-18.390(3077-3079) BasePeak:91.05(625920)

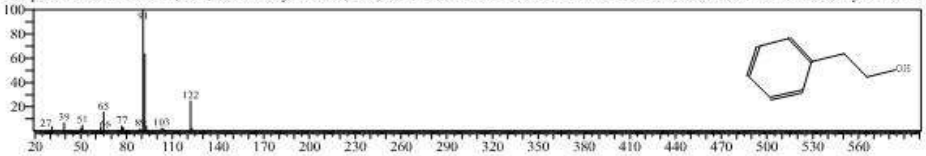
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



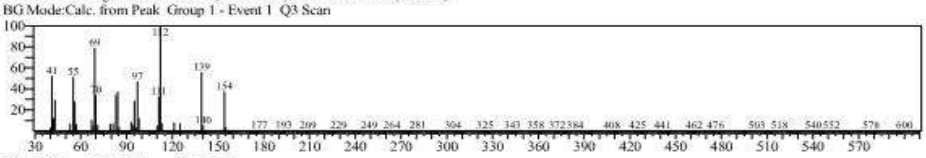
Hit#:1 Entry:20539 Library:Wiley9.lib

SI:98 Formula:C8H10O CAS:60-12-8 MolWeight:122 RetIndex:0

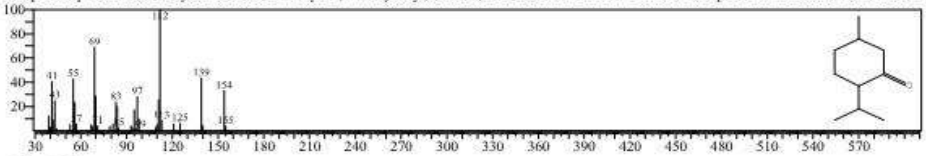
CompName:Benzeneethanol (CAS) $$ Phenethyl alcohol (CAS) $$ PEA $$ PHENETHYL ALCOLHOL $$ Phenethanol $$ .beta.-PEA $$ Benzyl carbinol $



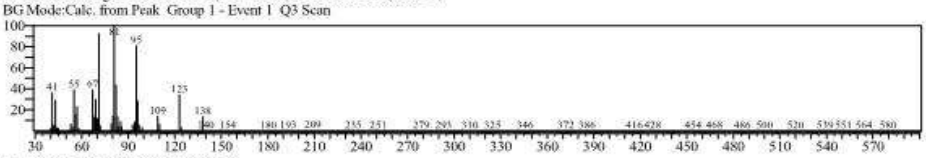
<< Target >>
Line#:2 R.Time:19.965(Scan#:3394) MassPeaks:318
RawMode:Averaged 19.960-19.970(3393-3395) BasePeak:112.10(294623)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



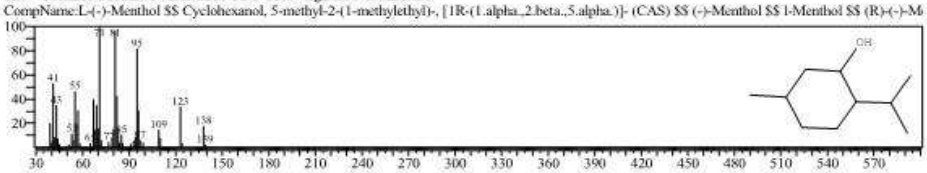
Hit#:6 Entry:58181 Library:Wiley9.lib
SI:94 Formula:C10H18O CAS:89-80-5 MolWeight:154 RetIndex:0
CompName:p-Menthone $$ Cyclohexanone, 5-methyl-2-(1-methylethyl)-, trans- (CAS) $$ trans-Menthan-3-one $$ trans-p-Menthan-3-one $$ Menthone $$



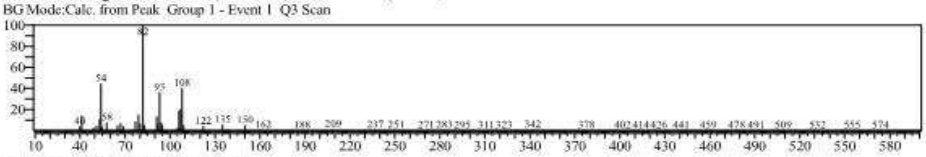
<< Target >>
Line#:3 R.Time:20.820(Scan#:3565) MassPeaks:321
RawMode:Averaged 20.815-20.825(3564-3566) BasePeak:81.10(480047)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



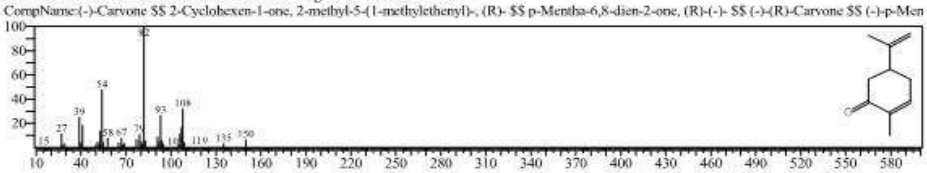
Hit#:2 Entry:61809 Library:Wiley9.lib
SI:97 Formula:C10H20O CAS:2216-51-5 MolWeight:156 RetIndex:0
CompName:L-(-)-Menthol $$ Cyclohexanol, 5-methyl-2-(1-methylethyl)-, [1R-(1.alpha.,2.beta.,5.alpha.)]- (CAS) $$ (-)-Menthol $$ l-Menthol $$ (R)-(-)-M



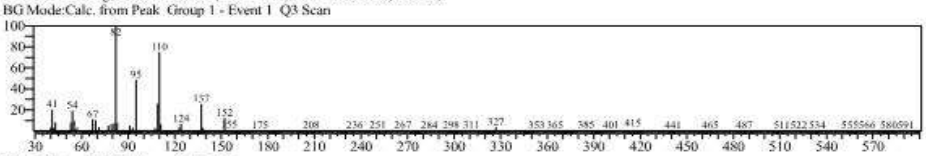
<< Target >>
Line#:4 R.Time:23.000(Scan#:4001) MassPeaks:288
RawMode:Averaged 22.995-23.005(4000-4002) BasePeak:82.05(136603)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



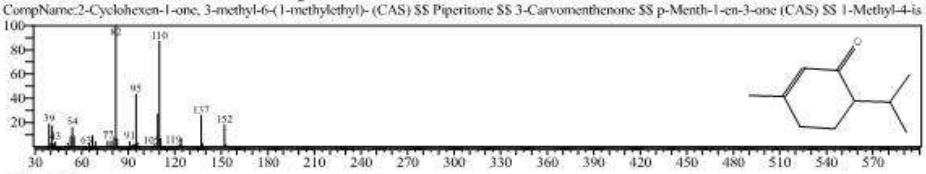
Hit#:1 Entry:9291 Library:NIST14s.lib
SI:95 Formula:C10H14O CAS:6485-40-1 MolWeight:150 RetIndex:1190
CompName:(-)-Carvone $$ 2-Cyclohexen-1-one, 2-methyl-5-(1-methylethenyl)-, (R)- $$ p-Mentha-6,8-dien-2-one, (R)-(-)- $$ (-)-(R)-Carvone $$ (-)-p-Men



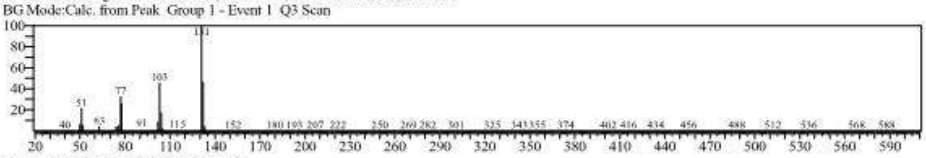
<< Target >>
Line#:5 R.Time:23.305(Scan#:4062) MassPeaks:358
RawMode:Averaged 23.300-23.310(4061-4063) BasePeak:82.05(183584)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



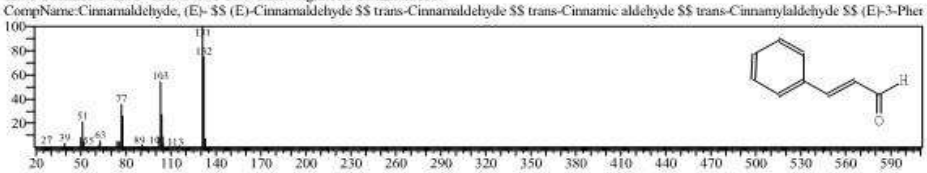
Hit#:1 Entry:54226 Library:Wiley9.lib
SI:94 Formula:C10H16O CAS:89-81-6 MolWeight:152 RetIndex:0
CompName:2-Cyclohexen-1-one, 3-methyl-6-(1-methylethyl)- (CAS) $$ Piperitone $$ 3-Carvomenthenone $$ p-Menth-1-en-3-one (CAS) $$ 1-Methyl-4-is



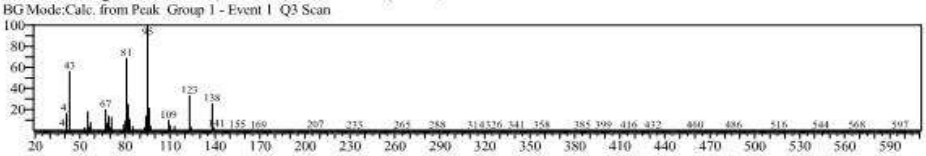
<< Target >>
Line#:6 R.Time:23.885(Scan#:4178) MassPeaks:358
RawMode:Averaged 23.880-23.890(4177-4179) BasePeak:131.05(2571951)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



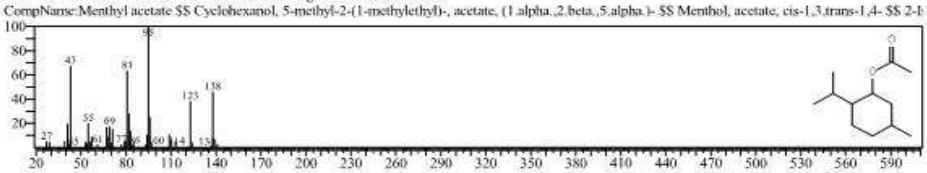
Hit#:1 Entry:6043 Library:NIST14s.lib
SI:96 Formula:C9H8O CAS:14371-10-9 MolWeight:132 RetIndex:1189
CompName:Cinnamaldehyde, (E)- $$ (E)-Cinnamaldehyde $$ trans-Cinnamaldehyde $$ trans-Cinnamic aldehyde $$ trans-Cinnamylaldehyde $$ (E)-3-Pher

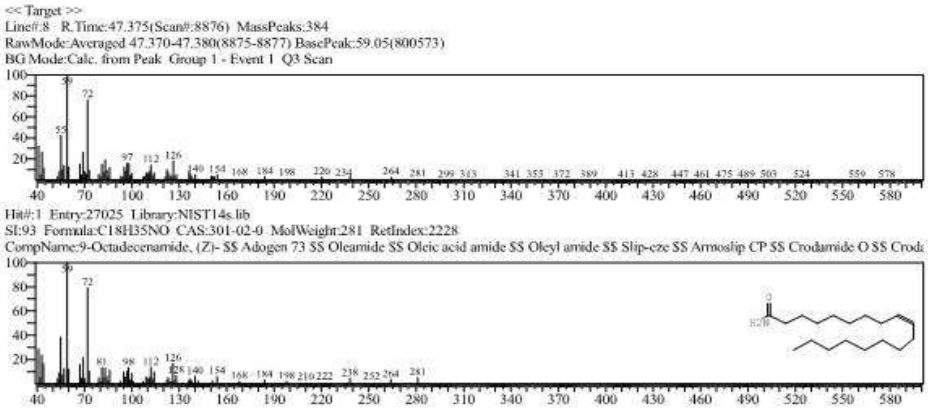


<< Target >>
Line#:7 R.Time:24.425(Scan#:4286) MassPeaks:325
RawMode:Averaged 24.420-24.430(4285-4287) BasePeak:95.10(290551)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan



Hit#:2 Entry:18172 Library:NIST14s.lib
SI:95 Formula:C12H22O2 CAS:89-48-5 MolWeight:198 RetIndex:1304
CompName:Menthyl acetate $$ Cyclohexanol, 5-methyl-2-(1-methylethyl)-, acetate, (1.alpha.,2.beta.,5.alpha.)- $$ Menthol, acetate, cis-1,3,trans-1,4- $$ 2-I

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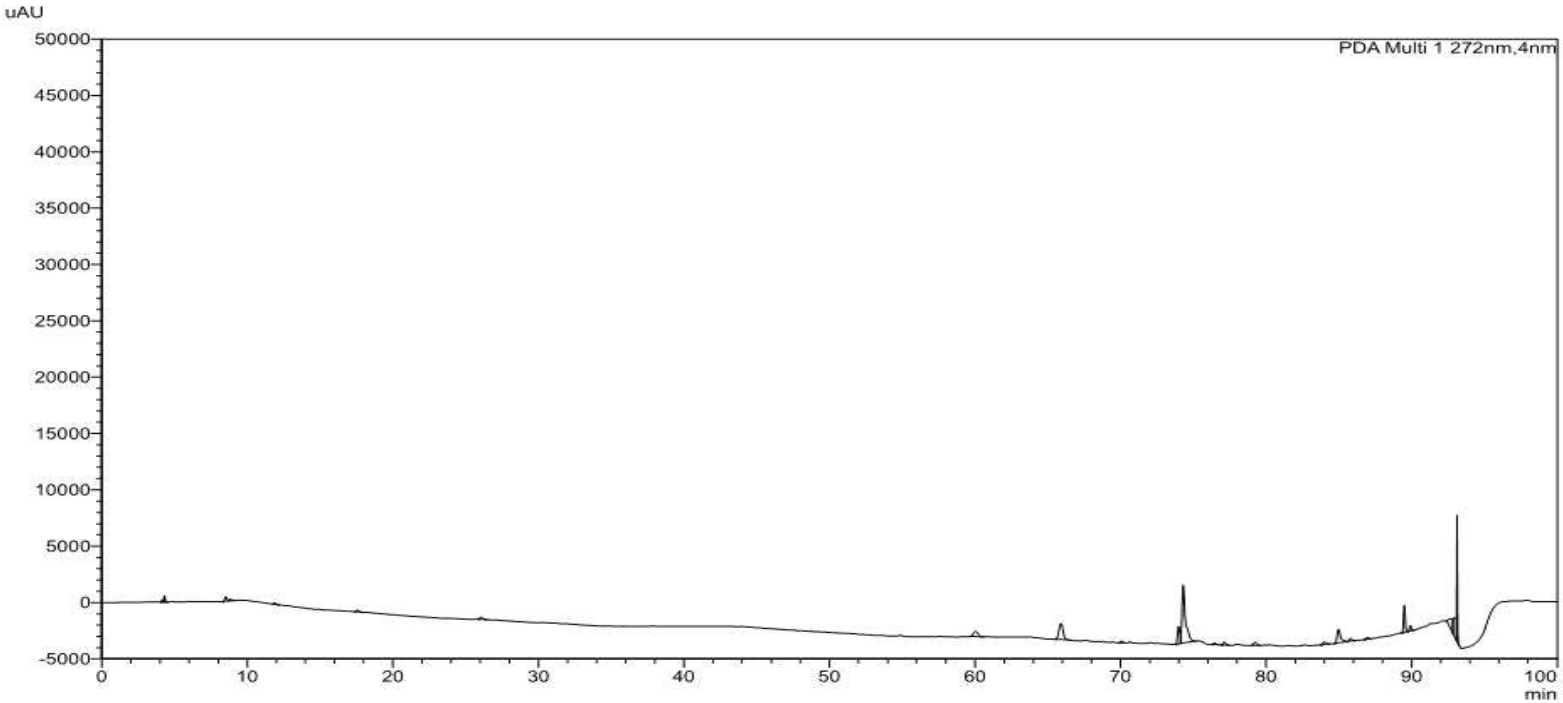
Line#:8 R.Time:47.375(Scan#:8876) MassPeaks:384
RawMode:Averaged 47.370-47.380(8875-8877) BasePeak:59.05(800573)
BG Mode:Calc. from Peak Group 1 - Event 1 Q3 Scan
Hit#:1 Entry:27025 Library:NIST14s.lib
SI:93 Formula:C18H35NO CAS:301-02-0 MolWeight:281 RetIndex:2228
CompName:9-Octadecenamide, (Z)- $$ Adogen 73 $$ Oleamide $$ Oleic acid amide $$ Oleyl amide $$ Slip-eze $$ Armoslip CP $$ Crodamide O $$ Croda

<가지 생체수_HPLC>

8/1/2023 4:51:40 PM Page 1 / 1

SHIMADZU LabSolutions Analysis Report

<Chromatogram>



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본 보고서의 결과는 마케팅 등 상업적 용도로 사용할 수 없으며, 법적 증거 자료로서의 효력 또한 없으므로 내부 참고자료로만 활용하시기 바라며, 본 보고서의 결과 공개 및 유출로 인한 분쟁 발생 시 제주대학교에서는 어떠한 책임도 지지 않습니다.