Substance Identity Report

Name : **PI1525**
Batch: Ho 154 262 - MM

Structure:

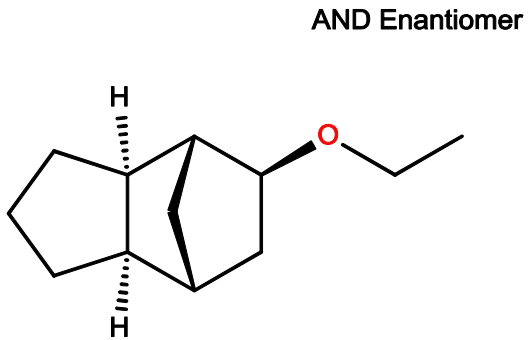

Table of Contents :

I. Name, structure(s), additional information
II. NMR (spectra, method, interpretation)
a) ^{1}H-NMR
b) ^{13}C-NMR
III. MS (spectra, method, interpretation)
a) GC-MS
b) LC-MS (optional)
IV. FT-IR (spectra, method, interpretation)
V. UV-VIS (spectra, method)
VI. GC (chromatogram, method, assignment)
VII. optical activity (optional)
VIII. additional information and references
IX. conclusion
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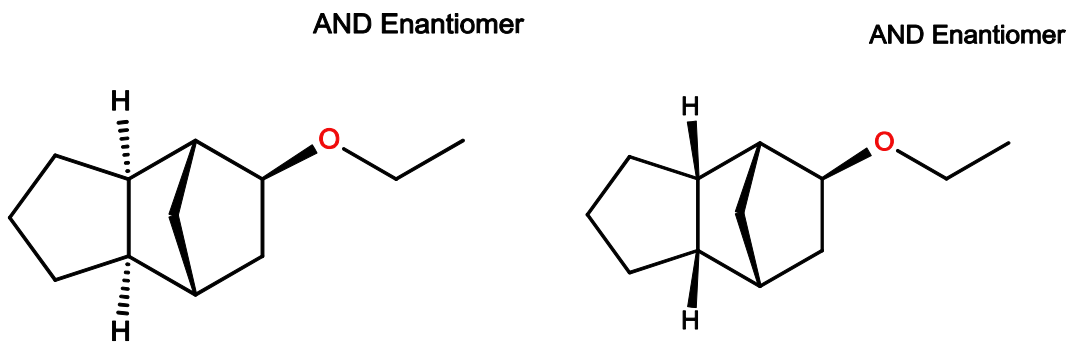
I. Name, structure, additional information

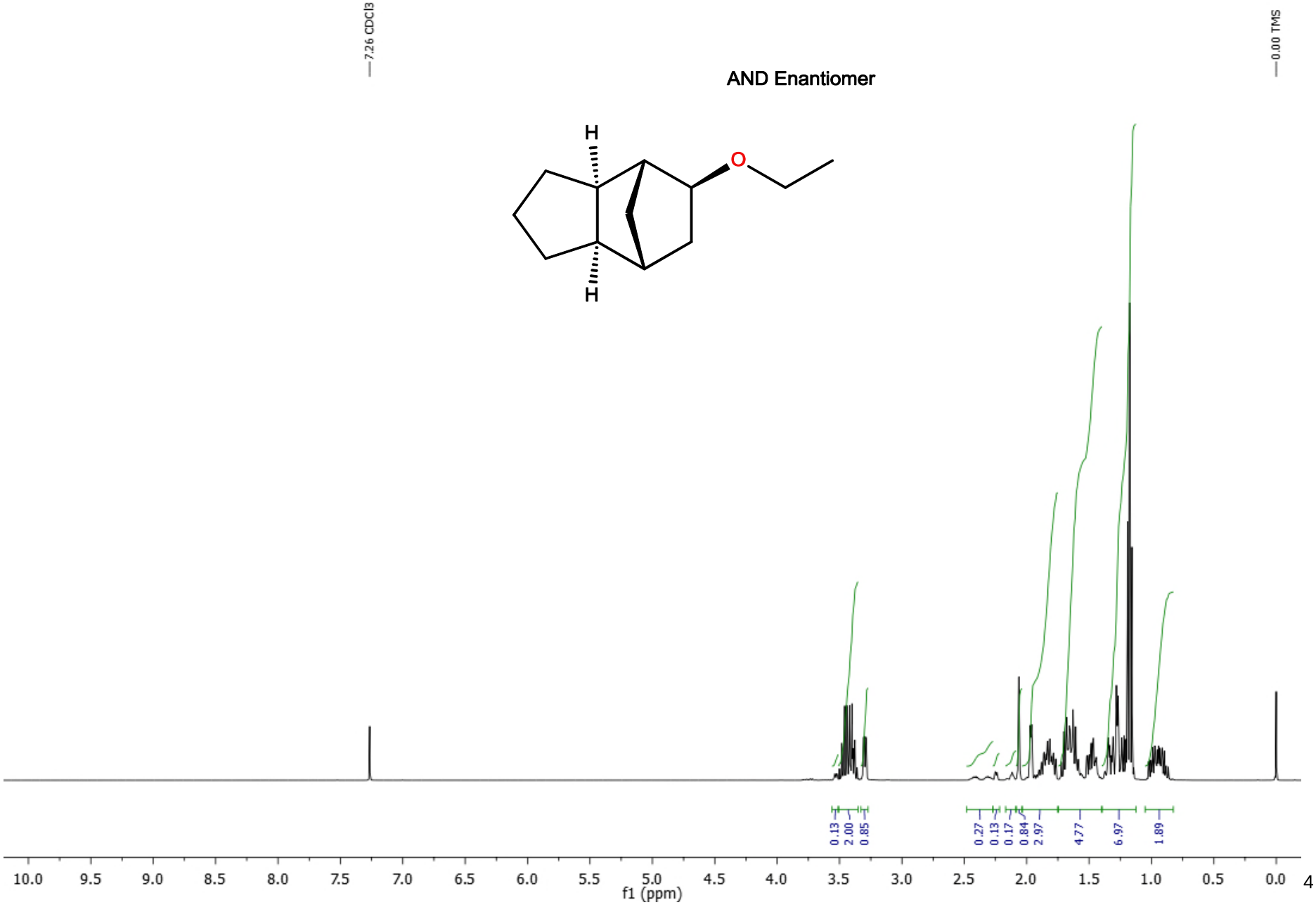
Structure:



Name:	Inden, (3aR*,4R*,5S*,7R*,7aR*)-5-ethoxy-4,7-methano -2,3,3a,4,5,6,7,7a-octahydro-1H-; Inden, (3aS*,4R*,5S*,7R*,7aS*) -5-ethoxy-4,7-methano-2,3,3a,4,5,6,7,7a-octahydro-1H-
Molweight[1] :	*both 180.29*
Exact Mass[1] :	*both 180.151415265*
Formula[1] :	*both* $C_{12}H_{20}O$
further information[3] :	
Product Name:	PI1525
Batch:	Ho 154 262 - MM
Synonyms:	4,7-Methano-1H-indene, 5-ethoxyoctahydro-,(3aR,4R,5S,7R,7aR)-rel-; 4,7-Methano-1H-indene, 5-ethoxyoctahydro-,(3aS,4R,5S,7R,7aS)-rel-
CAS No.:	1897392-68-5; 13213-08-6

II.a ^{1}H-NMR spectrum

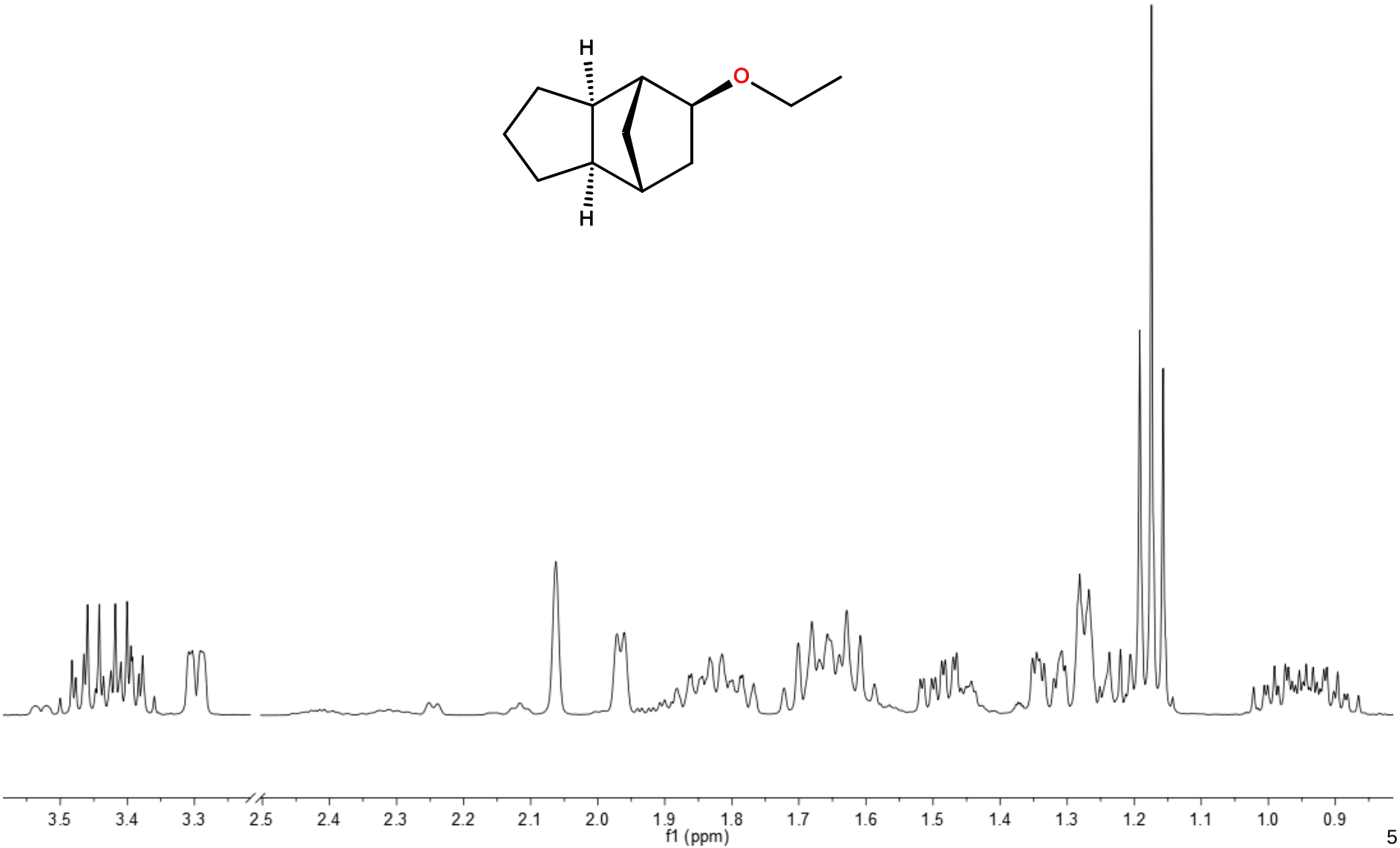
Name: PI1525
Batch: Ho 154 262 - MM



II.a ^{1}H-NMR extensions

Name: PI1525
Batch: Ho 154 262 - MM

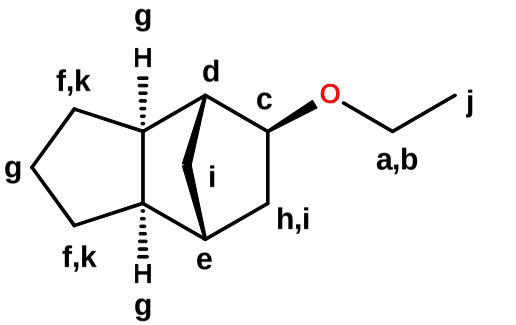
AND Enantiomer



II.a ^{1}H-NMR interpretation

Name: PI1525
Batch: Ho 154 262 - MM

AND Enantiomer



	Shift in ppm	Multiplicity	Functionality	Number of Protons
a	3.46	Doublet of quadruplets	aliphatic proton	1
b	3.40	Doublet of quadruplets	aliphatic proton	1
c	3.30	Doublet of doublets	aliphatic proton	1
d	2.06	Singlet	aliphatic proton	1
e	1.97	Doublet	aliphatic proton	1
f	1.82	Multiplet	aliphatic protons	2
g	1.65	Multiplet	aliphatic protons	4
h	1.49	Doublet of doublets of doublets	aliphatic proton	1
i	1.30	Multiplet	aliphatic protons	3
j	1.17	Triplet	aliphatic protons	3
k	0.94	Multiplet	aliphatic protons	2

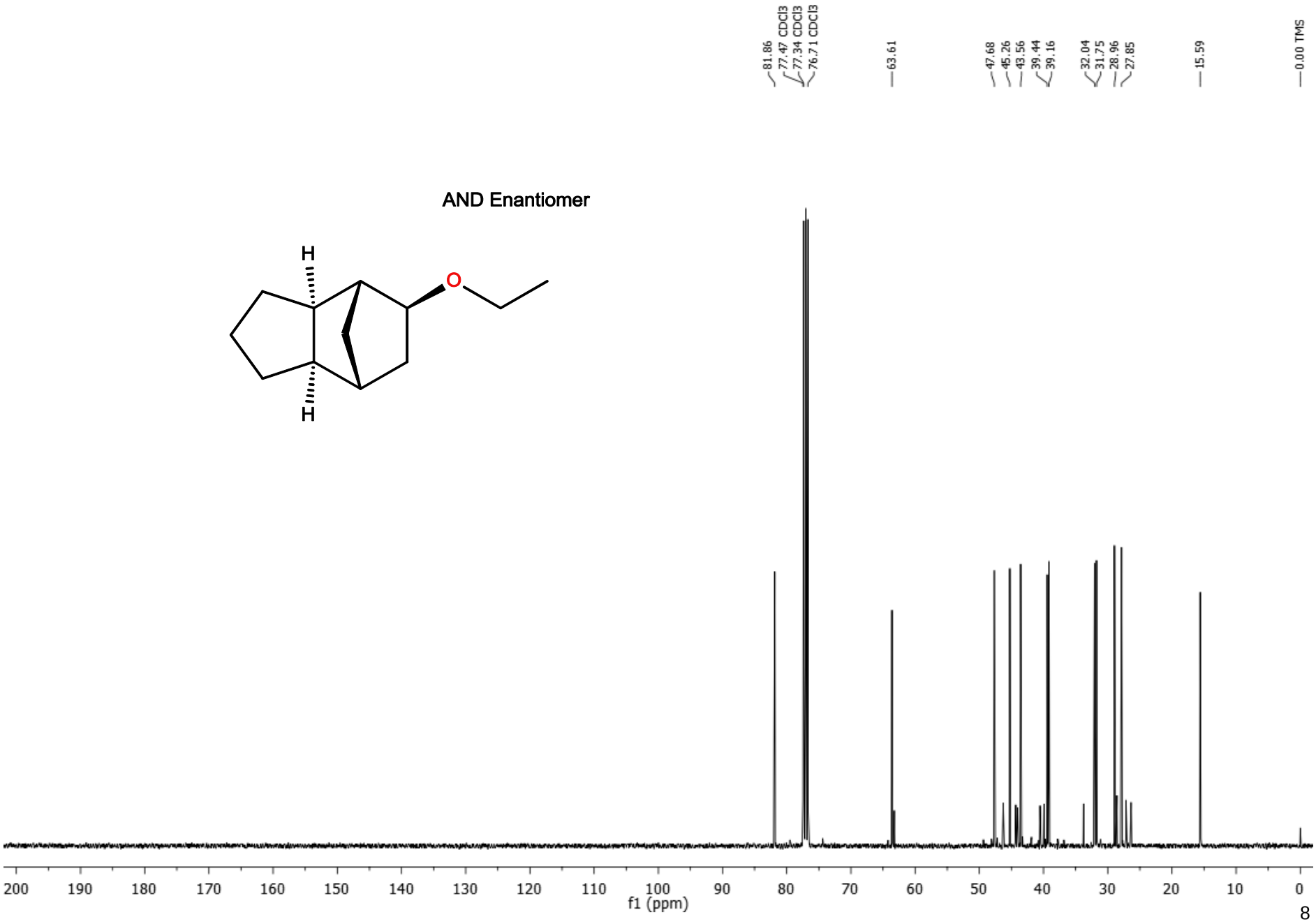
II.a ^{1}H-NMR method

Name: PI1525
Batch: Ho 154 262 - MM

Instrument	400 MHz-NMR-Spectrometer
Typ	Avance 400 - Bruker BioSpin GmbH
Frequency	400.412 MHz
Range	8012.8 Hz
Scans	64
Acquisition Time	4.089 sec
Concentration	10 mg / 0.8 mL
Solvent	Deuterochloroform (CDCl3)
Reference	Tetramethylsilane (TMS)
Reference Shift	set to 0.00 ppm
Temperature	25 °C (298 K)
Measurement Identifier	3602246
Date of Measurement	January/06/2016

II.b ^{13}C-NMR spectrum

Name: PI1525
Batch: Ho 154 262 - MM

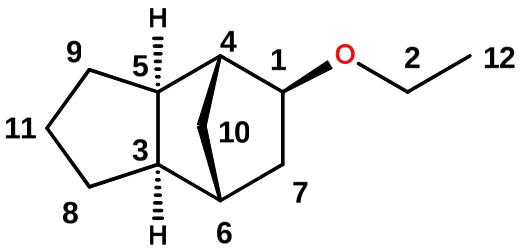


AND Enantiomer

II.b ^{13}C-NMR interpretation

Name: PI1525
Batch: Ho 154 262 - MM

AND Enantiomer



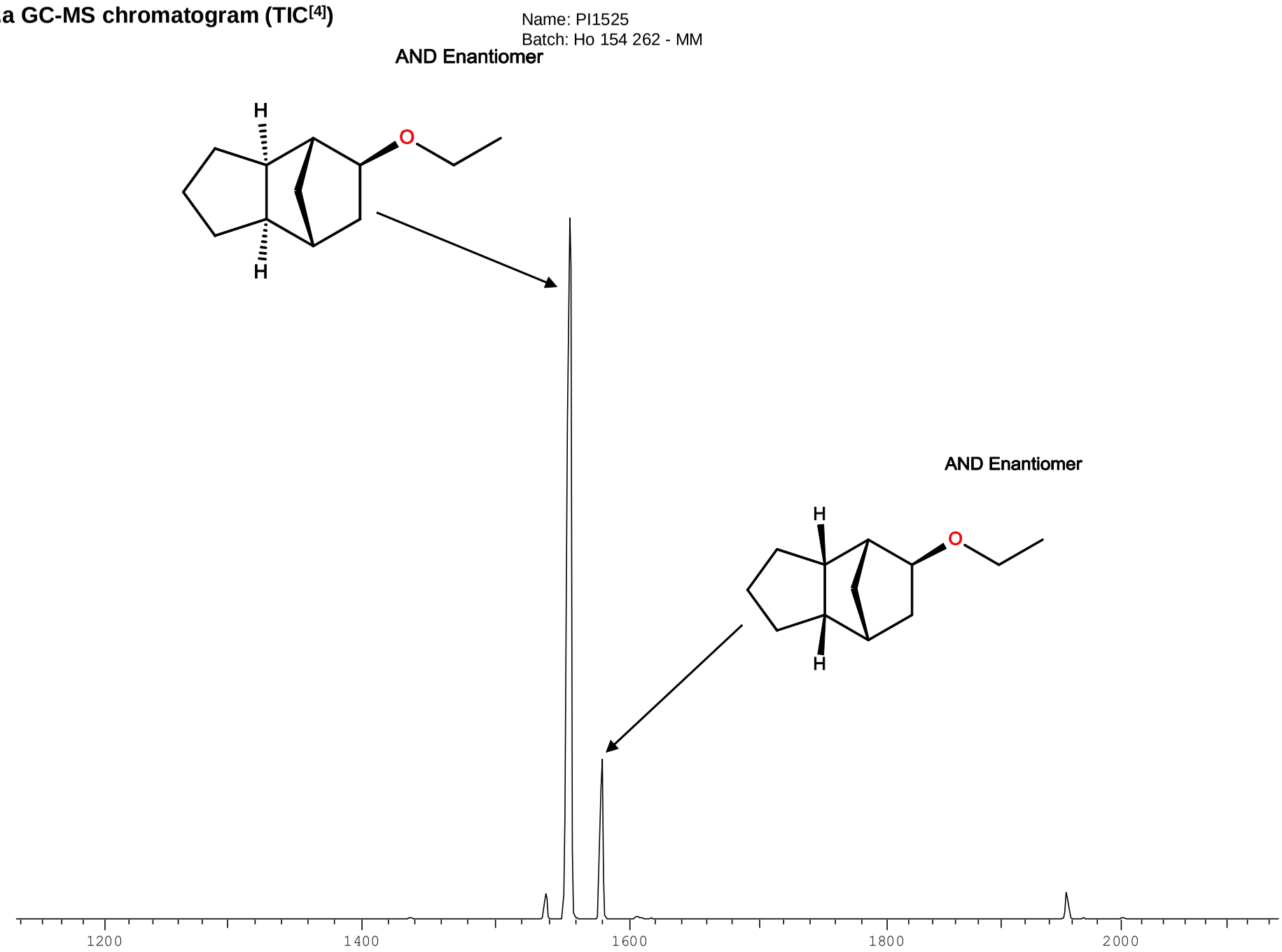
Pos.	ppm	Number of Carbons
1	81.86	1
2	63.61	1
3	47.68	1
4	45.26	1
5	43.56	1
6	39.44	1
7	39.16	1
8	32.04	1
9	31.75	1
10	28.96	1
11	27.85	1
12	15.59	1

II.b ^{13}C-NMR method

Name: PI1525
Batch: Ho 154 262 - MM

Instrument	400 MHz-NMR-Spectrometer
Typ	Avance 400 - Bruker BioSpin GmbH
Frequency	100.69 MHz
Range	24038.5 Hz
Scans	1024
Acquisition Time	1.36 sec
Concentration	10mg / 0.8mL
Solvent	Deuterochloroform (CDCl3)
Reference	Tetramethylsilane (TMS)
Reference Shift	set to 0.00 ppm
Temperature	25 °C (298 K)
Measurement Identifier	3602246
Date of Measurement	January/01/2016

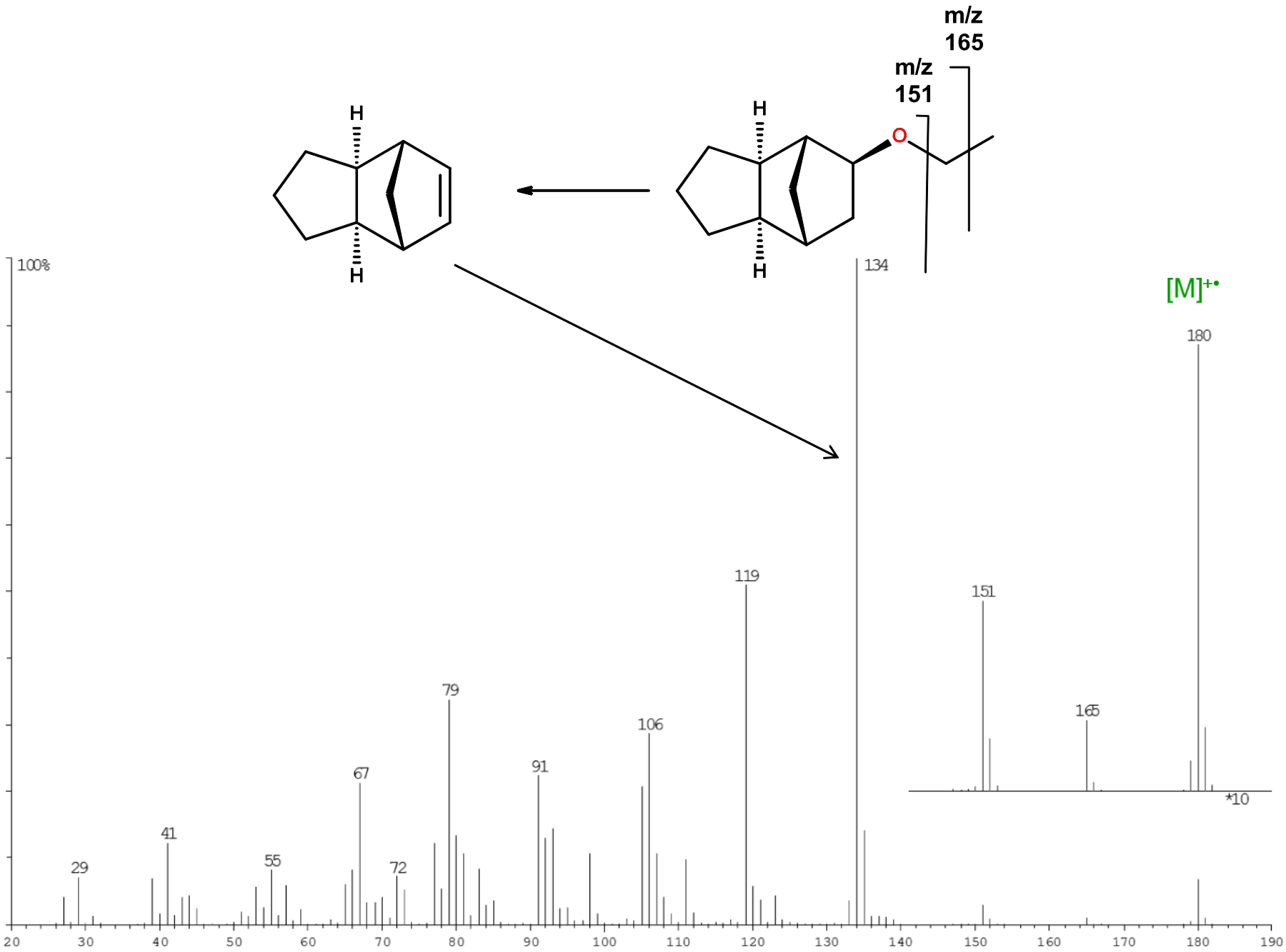
III.a GC-MS chromatogram (TIC[4])



Scale: retention index corresponding to GC data in section VI.

III.a GC-MS spectrum and interpretation

Name: PI1525
Batch: Ho 154 262 - MM



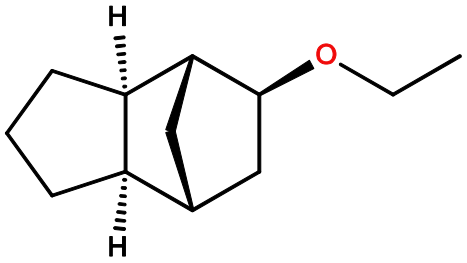
III.a GC-MS method

Instrument:	Gas Chromatograph - Mass Spectrometer
GC Chromatograph:	Agilent 7890A
Mass Spectrometer:	Agilent 5975
Detector:	Quadrupol
GC-Column[9]:	30 m FFAP, id 0.25 mm, film 0.25µm
Carrier Gas/ Flow:	Helium, 2.2mL/min (Linear Velocity Mode)
Sample Preparation	solved 1:300 in MTBE[5]
Injection Value :	1µL (Split 1:40)
Heating Rate:	60°C (Start) – 9°C/min. (Rate) – 230°C
Injector:	KAS4 split-/splitless
Electron Energy:	70 eV
Ionization Mode:	electron impact (EI)
Scan Range:	m/z 25 – 500
Data System:	MSD Chemstation, Agilent
Measurement Identity	MS60870A15
Date of Measurement	December/12/2015

III.b LC-MS chromatogram (optional)

Name: PI1525
Batch: Ho 154 262 - MM

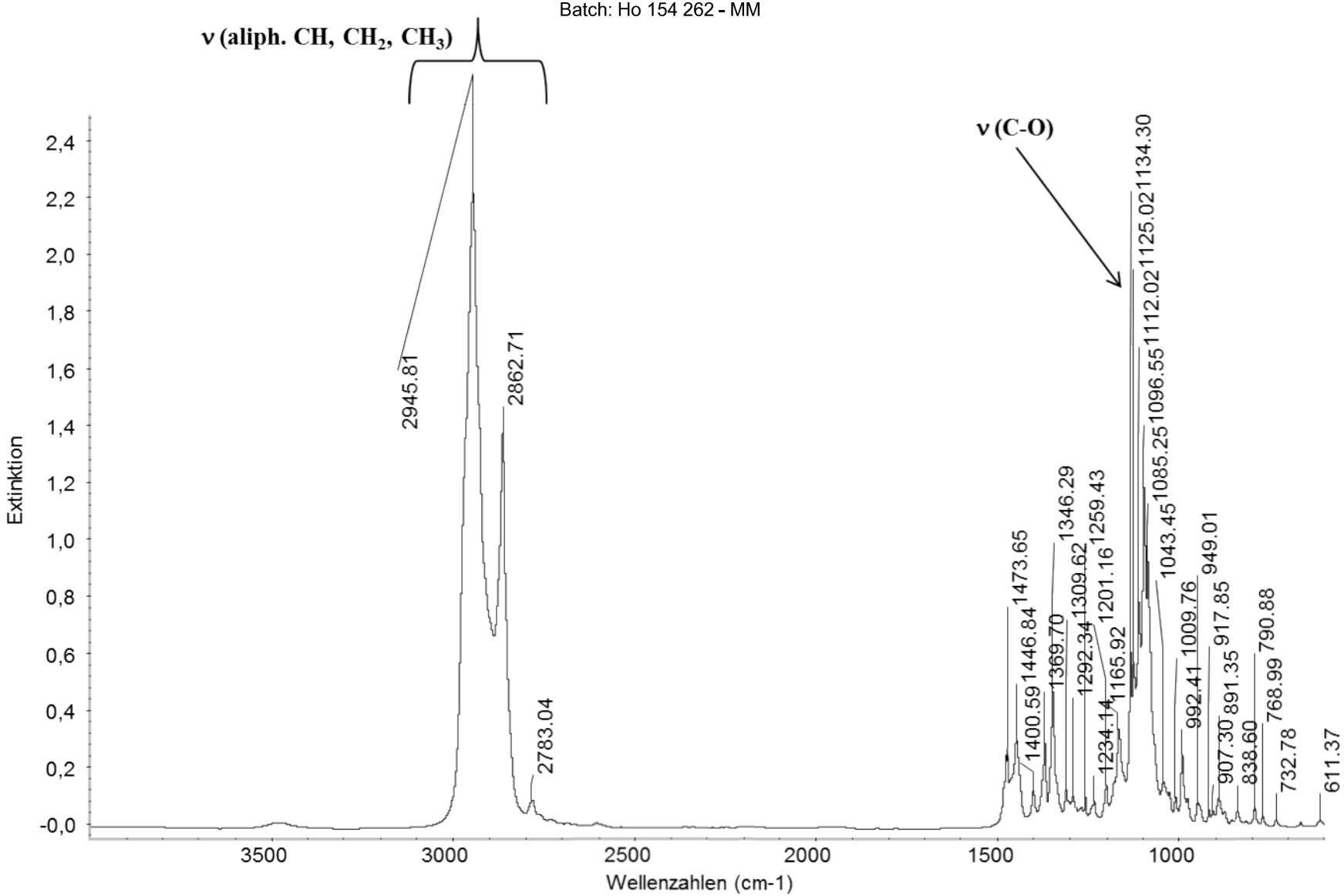
AND Enantiomer



LC-MS was not performed. There are no additional information expected from this method. For the identification of possible impurities >1.0% GC, GC-MS and NMR was used. Higher molecular and non-volatile by-products are not present as a result of the production process and purification of the product.

IV. FT-IR spectrum and interpretation

Name: PI1525
Batch: Ho 154 262 - MM



Instrument : Nicolet 6700

Resolution : 1 cm^{-1}

Technique: FTIR - NaCl Film

IV. FT-IR frequency list

Name: PI1525
Batch: Ho 154 262 - MM

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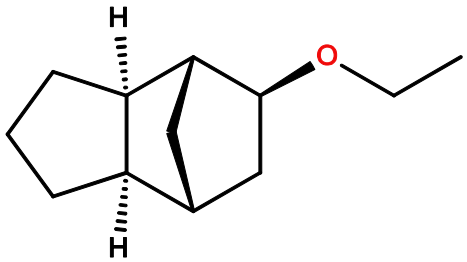


number	frequency	intensity
1	768,99	0,0195
2	790,88	0,0509
3	838,60	0,0374
4	891,35	0,0877
5	907,30	0,0394
6	917,85	0,0396
7	949,01	0,0694
8	992,41	0,241
9	1009,76	0,0867
10	1043,45	0,140
11	1085,25	0,919
12	1096,55	1,190
13	1112,02	0,779
14	1125,02	0,565
15	1134,30	0,596
16	1165,92	0,328
17	1201,16	0,131
18	1234,14	0,0705
19	1259,43	0,0846
20	1292,34	0,0881
21	1309,62	0,110
22	1346,29	0,457

Measuring range	600 – 4000 cm^{-1}
resolution	4,000
abs. threshold	0,018
sensitivity	74
scans	32
background scans	32
signal gain	1,0

number	frequency	intensity
23	1369,70	0,278
24	1400,59	0,114
25	1446,84	0,285
26	1473,65	0,258
27	2783,04	0,0782
28	2862,71	1,382
29	2945,81	2,215

IV. FT-IR method and parameters

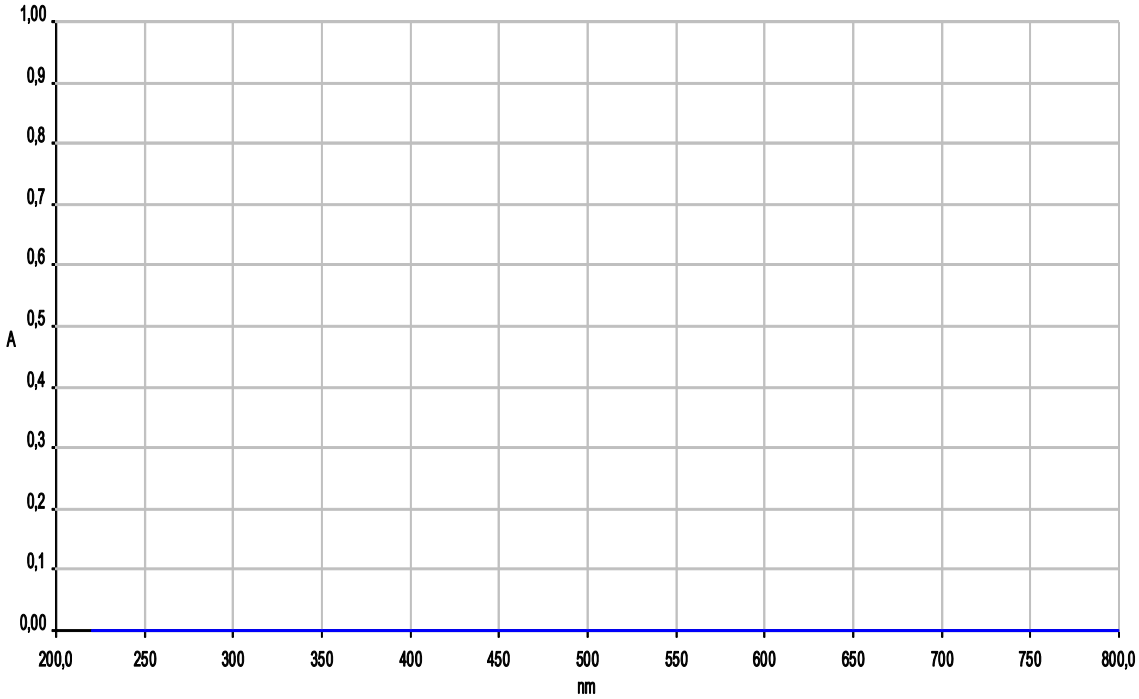
Name: PI1525
Batch: Ho 154 262 - MM

Manufacturer:	Thermo Scientific
Spectrometer:	GC/FT-IR Interface Nicolet 6700
Preparation:	NaCl - Film
Sampling:	wavelength range: 4000-600 cm^{-1} resolution: 4 cm^{-1} number of scans: 32
Calibration:	Polystyrene[6]
Sample:	PI1525 Batch : Ho 154 262 - MM
Measurement Identity:	4803762
Date of Measurement:	January/05/2016

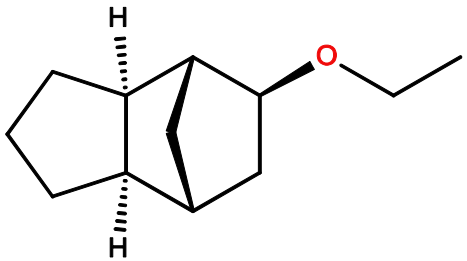
IR spectra are measured for characterization of the product. Impurities < 5% show no significant bands.
Characteristic bands of the functional groups of the product are assigned. A complete interpretation of the fingerprint region (<1600 cm^{-1}) was desisted, due to the complexity of overlapping bending vibration bands in this area.

V. UV-VIS spectrum

Name: PI1525
Batch: Ho 154 262 - MM



AND Enantiomer



MAXIMA/MINIMALISTE

Daten-File: 161001A Information: 10,00mg PI 1525 Ho154 262-MM
Schwellwert: 0,0030
Abszissen-Einheit: nm
Ordinaten-Einheit: A

Nr.	Abszisse	Ordinate	Typ

V. UV-VIS method and result

Name: PI1525
Batch: Ho 154 262 - MM

Method for determination of UV-absorption

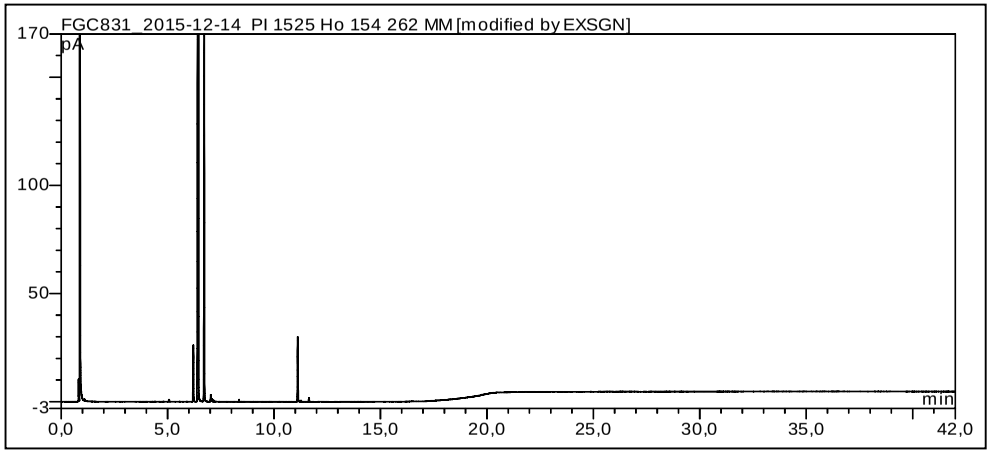
Instrument	UV/ Vis spectrometer
Spectrometer	Perkin Elmer Lambda 12
Spectrometertyp	Dual beam spectrometer
Cuvette material	quartz glass
Measuring range	200 – 800 nm
Scan speed	120 nm/min
Concentration	10.00mg / 100ml
Solvent	Ethanol
Pathlength	1 mm
Extinction / Absorption maximum	-
lower limit	$E^{1\%}/_{1cm}$ 10
upper limit	$E^{1\%}/_{1cm}$ 1000
Date of Measurement	January/08/2016

VI. GC chromatogram (FID[7]) , assignment, method[9]

Name: PI1525
Batch: Ho 154 262 - MM

PI 1525 Ho 154 262 MM

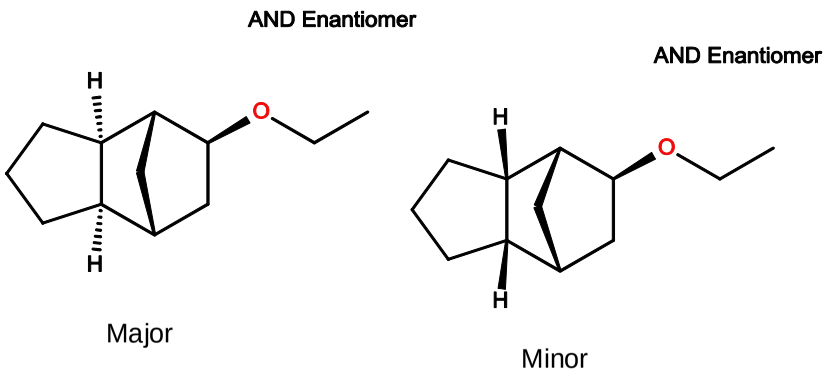
Operator:	UISZF	*Path:*	RD\CHROM\FGCTB03\FGC831
Vial Number:	19	*Injection Volume:*	1.0µl
Control Program:	FGC831	*Sample Type:*	unknown
Quantif. Method:	FGC831_PROBEN	*Run Time:*	42min
Recording Time:	16/12/2015 09:20	*Column Info:*	20m DS 0.20µm df:0,20mm / 60-9-240° H
Channel:	Channel_A	(Channel_A = DB-WAX	Channel_B = DB-1)



No.	Ret.Time min	Ret.Index	Area pA*min	Type	Rel.Area %	Peak Name
1	4.408	1373.77	0.003	BMB	0.010	n.a.
2	5.067	1429.04	0.026	BMB	0.076	n.a.
3	5.560	1469.64	0.003	BMB	0.010	n.a.
4	5.895	1496.89	0.003	BMB	0.009	n.a.
5	6.208	1522.38	0.643	BMB	1.897	n.a.
6	6.448	1541.88	28.086	BMB	82.855	Major isomer
7	6.720	1563.94	4.145	BMB	12.228	Minor isomer
8	7.030	1589.14	0.075	BM	0.220	n.a.
9	7.073	1592.67	0.042	MB	0.123	n.a.
10	7.160	1599.73	0.019	BMB	0.055	n.a.
11	7.257	1607.63	0.006	BMB	0.017	n.a.
12	8.360	1698.61	0.006	BMB	0.018	n.a.
13	11.113	1938.14	0.774	BMB	2.283	n.a.
14	11.267	1952.08	0.016	BMB	0.047	n.a.
15	11.640	1986.38	0.051	BMB	0.151	n.a.
Total:			33.898		100.000	

Retention index[8] PI1525 in ZB-Wax

Major isomer 1542 (6.45 min)
Minor isomer 1564 (6.72 min)



Variation in the retention indices on GC and GC-MS systems are depending on the different carrier gas (H_2, He) and on different gas pressures used.

VI. GC method[9]

Name: PI1525
Batch: Ho 154 262 - MM

Chromatographic Conditions

GC Instrument	HP 6890 (Agilent)
Column Type[9]	ZB-Wax
Phase	Polyethylenglycole
Carrier Gas	Hydrogen
Flow	1.3 mL/min (17 min) – 0.5 mL/min – 3.0 mL/min
Detector Type	FID
Detector Temperature	275°C
Injection Volume	1µL (Split 1:70; 1:300 in MTBE[5])
Column Length	20 m
Column Diameter (i. D.)	0.18 mm
Film	0.18 µm
Injector	CIS
Injector Temperature	80°C – 12°C/s – 250 °C
Oven Temperature and Heating Rate	60°C – 9°C/min – 240 °C
Date of Measurement	December/14/2015

Variation in the retention indices on GC and GC-MS systems are depending on the different carrier gas (H_2, He) and on different gas pressures used.

[1]: Name and additional information was generated by Accelrys Draw 4.1 .NET
[2]: SMILES-Code was generated by Accelrys Draw 4.1 .NET
[3]: further information :
[4]: TIC= Total Ion Chromatogram
[5]: MTBE = Methyl-*t*-butylether
[6]: Polystyrene : Pharmacopoeia Eur. 4.Edition Grunwerk 2002
[7]: FID = Flame Ionisation Detector
[8]: Kovats Indices e.g.: http://goldbook.iupac.org/R05360.html
[9]: GC-column phases: polyethylene glycol coating = DB-Wax = ZB-Wax = FFAP
polydimethylsiloxane coating = DB-1 = ZB-1

IX. conclusion

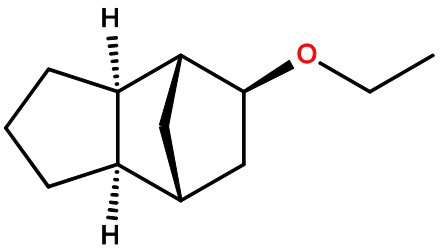
Name: PI1525
Batch: Ho 154 262 - MM

The results of the different analytical techniques are in agreement with the composition of the product

PI1525

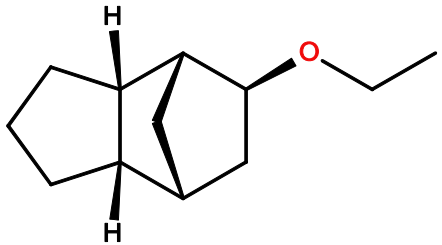
and the described chemical structures of the major and minor compounds are,

AND Enantiomer



Major

AND Enantiomer



Minor

The different analytical measurements were performed in specific laboratories by skilled lab technicians.

Data combined by:

Data checked by:

.

Date : February/03/2016

Certificate:

XI. appendix

CERTIFICATE

This is to certify that

Scope:
Research, development, application, production and sales of fragrance compounds, flavors for the foodstuff and beverage industry, tobacco flavors, ingredients for the cosmetic and toiletry industry, aroma molecules, colors, sophisticated biochemicals such as bioflavonoids, rare sugars and standardised or highly purified plant extracts as well as food additives and seasonings

Through an audit, documented in a report, it was verified that the management system fulfills the requirements of the following standard:

ISO 9001 : 2008

Certificate registration no.	283116 QM08
Date of certification	2014-07-30
Valid until	2017-07-29

DAkkS Deutsche Akkreditierungsstelle D-ZM-18402-01-00

DQS-UL CFSGmbH

Dr. Sied Sadek
Managing Director

Accredited Body: DQS-UL CFS GmbH, August-Schanz-Straße 21, 60433 Frankfurt am Main 1 / 8

Annex to Certificate
Registration No. 283116 QM08

This annex (edition: 2014-07-30) is only valid in connection with the above-mentioned certificate. 2 / 8

XI. appendix

Annex to Certificate
Registration No. 283116 QM08

Annex to Certificate
Registration No. 283116 QM08

XI. appendix

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Registration No. 283116 QM08

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Registration No. 283116 QM08

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Registration No. 283116 QM08