

Supplementary Information

Isolation of cytotoxic cycloartane triterpenoids from *Dysoxylum malabaricum*

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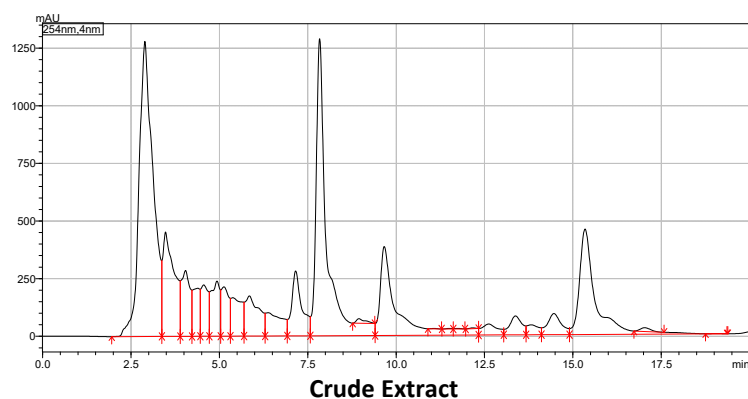
Figure S.44 HRESIMS spectrum of compound 4

Figure S.45 IR spectrum of compound 4

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Table S.47 MTT assay after 24 hours.

Figure S.1 A. Chromatogram of crude extract using reverse phase cartridge column at 254nm.



Fraction 1	2.5-5min
Fraction 2	5.1-7.5min
Fraction 3	7.55-9.5min
Fraction 4	9.55-12min
Fraction 5	12.5-15min
Fraction 6	15.1-18min

B. HPLC chromatogram of compounds isolated from active fraction 2 using analytical column at 220nm.

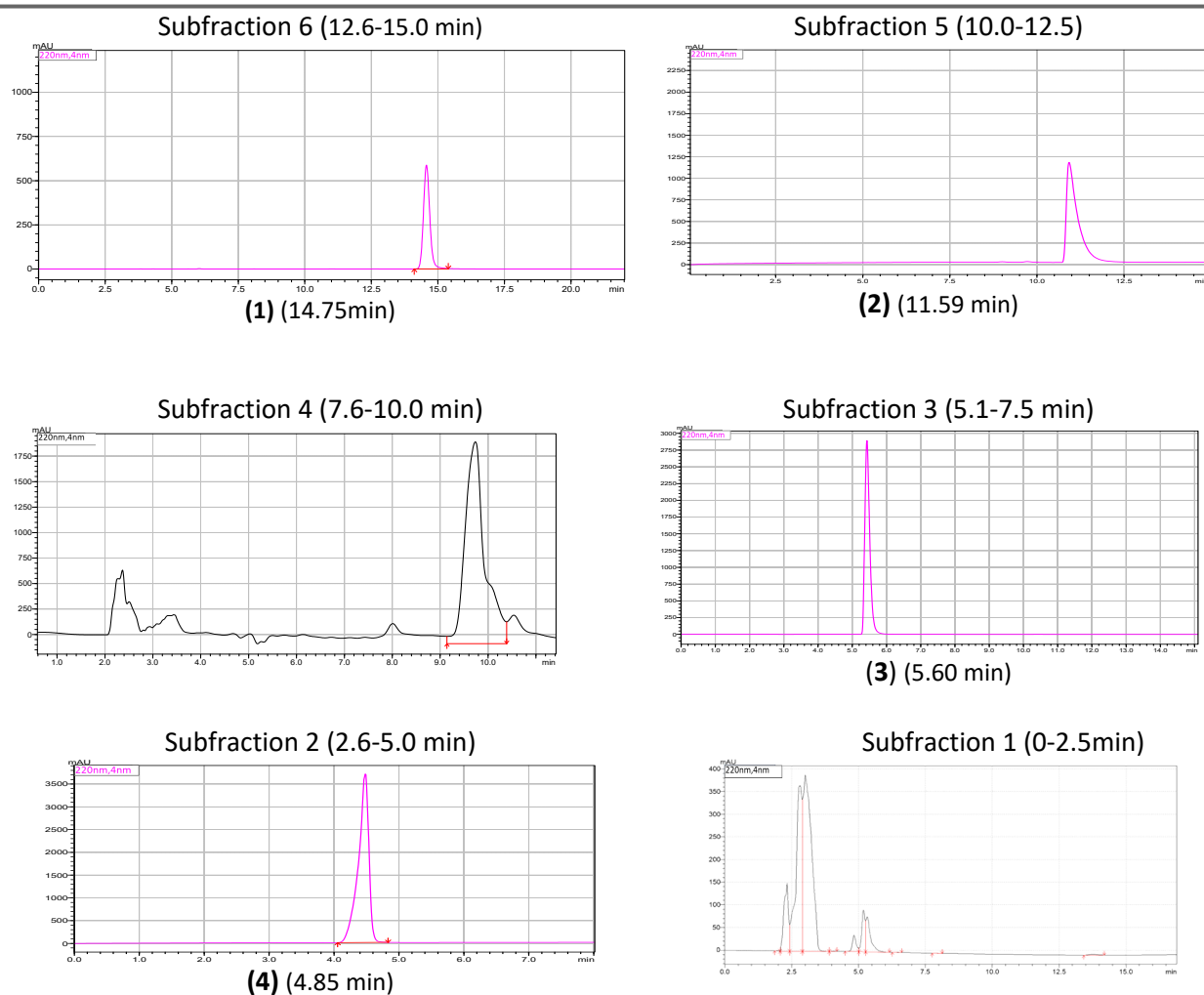


Table S.2. MS/MS fragmentation peak present in crude fraction 2.

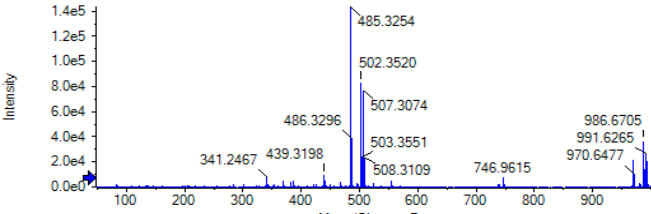
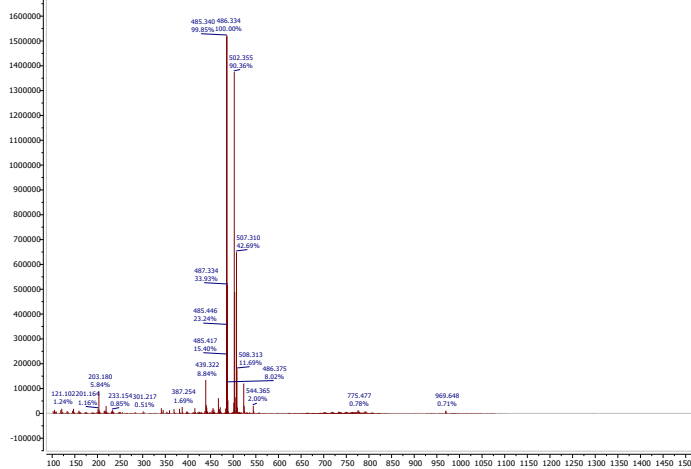
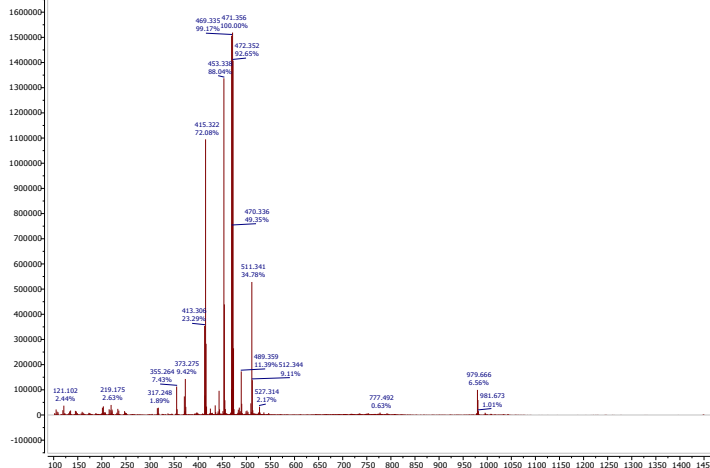
LCMS spectra of cytotoxic fraction 2 at 12.09min	Abundant m/z Peaks
Spectrum from 27M.wiff (sample 1) - 27M, Experiment 1, +TOF MS (50 - 1000) from 12.099 min 	485 486 502
LCMS spectra of fraction 2 at 18.16min	
C:\Users\Nived... \NP502-S82935.d Injection 1 ESI (+) MS centroid MS + spectrum 18.16 	486 502
LCMS spectra of ethylacetate fraction at 17.52min	
C:\Users\Nived... \NP502-S82935.d Injection 1 ESI (+) MS centroid MS + spectrum 17.52 	472

Figure S.3. DNP search for identification of compound 1

Boolean	Property	Comparison	Value
AND	Chemical Name		
AND	Molecular Formula		
AND	Molecular Formula by Element	= C	
AND	CAS Registry Nos.		
AND	All Text		
AND	Melting Point	=	
AND	Boiling Point	=	
AND	Biological Source		Dysoxylum
AND	Accurate Mass	Range	min. 484.000 max. 484.999

Chemical Search Results
Total Hits: 7

< Page 1 of 1 > Hits Per Page: 40

Tr	Chemical Name	Tl Molecular Formula
☐	Beddomelactone	C ₃₀ H ₄₄ O ₅
☐	2,7-Dihydroxydymma-13(17),24-dien-26-oic acid (3i,7i,20S,24Z)-form, 3-Ketone, 26-Me ester	C ₃₁ H ₄₈ O ₆
☐	24,25-Epoxy-3,23-dioxotricala-7-en-21-oic acid (24R)-form	C ₃₀ H ₄₄ O ₅
☐	21,23-Epoxytricala-7,24-diene-3,21-diol (3i,21R,23R)-form, 24,515-Epoxyde, 3-ketone, 21-Me ether	C ₃₁ H ₄₈ O ₆
☐	3-Hydroxy-6,23-dioxotricala-7,25(27)-dien-26-oic acid (3i)-form	C ₃₀ H ₄₄ O ₅
☐	3-Hydroxy-6-oxotricala-7,24-dien-26-oic acid (3i),24Z)-form, Me ester	C ₃₁ H ₄₈ O ₆
☐	3,4-Seco-4(23),12-oleanadiene-3,30-dioic acid 3-Me ester	C ₃₁ H ₄₈ O ₆

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Searched properties:

- Biological source: Dysoxylum
- Accurate mass range: 484.000 to 484.999

Results:

- 7 compounds reported from Dysoxylum genus of mol wt 484
- only 3 compounds having C30

Search Chemicals

Q Search

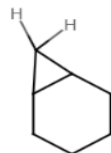
X Tautomers

Substructure

Exact

Not Substr.

Clear Structure



Boolean	Property	Comparison	Value	Remove
AND	Biological Source		Dysoxylum	
AND	Accurate Mass	Range	min. 484.000 max. 484.999	

Q Search

Searched properties:

Biological source:

Dysoxylum

Accurate mass range:

484.000 to 484.999

Cyclopropane fused ring

Chemical Search Results

Total Hits: 1

< Page 1 of 1 >

Hits Per Page: 40

Chemical Name

Molecular Formula



Beddomeilactone

C₃₀H₄₄O₅

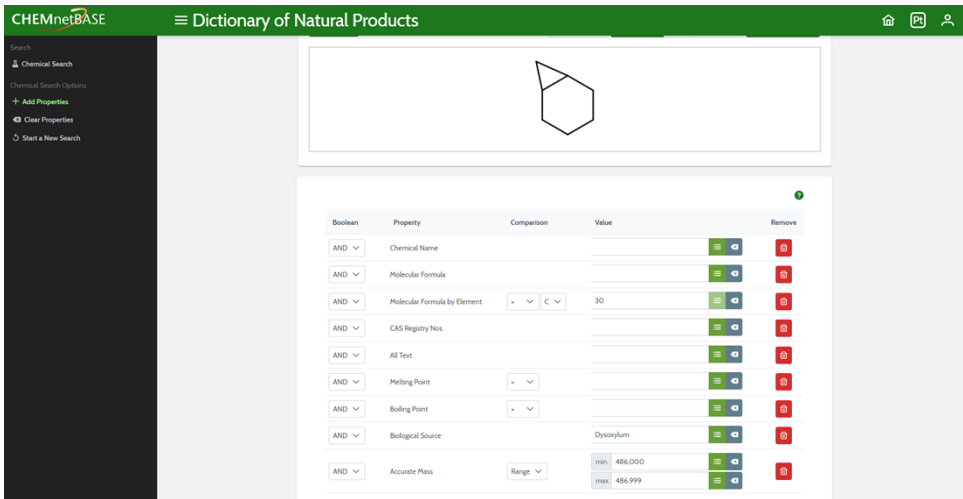
< Page 1 of 1 >

Hits Per Page: 40

Results:

1 compound reported from
Dysoxylum genus of mol wt 484

Figure S.4. DNP search for identification of compound 2

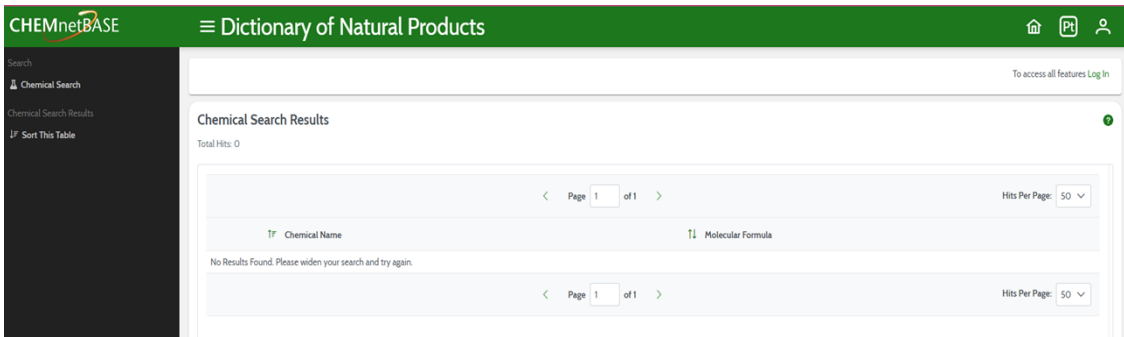


The screenshot shows the CHEMnetBASE search interface. The search criteria are as follows:

Boolean	Property	Comparison	Value	Remove
AND	Chemical Name			
AND	Molecular Formula			
AND	Molecular Formula by Element	C	30	
AND	CAS Registry Nos.			
AND	All Text			
AND	Melting Point			
AND	Boiling Point			
AND	Biological Source		Dysoxylum	
AND	Accurate Mass	Range	min: 486.000 max: 486.999	

Searched properties:

- Biological source:
Dysoxylum
- Accurate mass range:
486.000 to 486.999
- Number of carbon=30



The screenshot shows the CHEMnetBASE search results page. The search criteria are as follows:

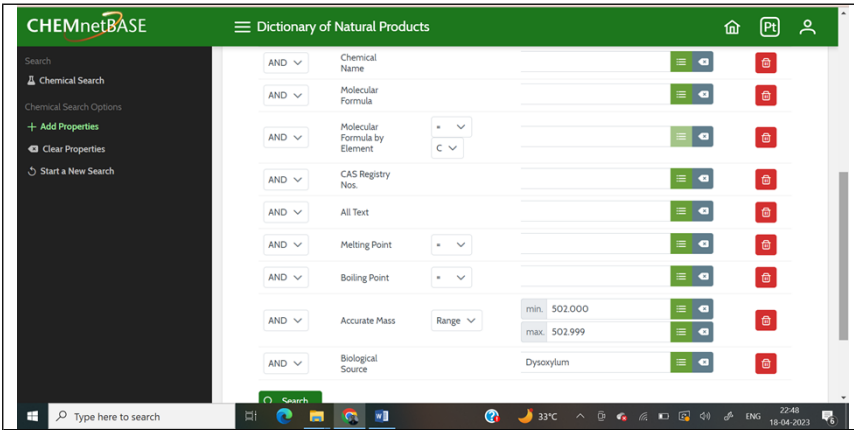
Property	Value
Chemical Name	
Molecular Formula	

No Results Found. Please widen your search and try again.

Results:

- No compounds reported from Dysoxylum genus of mol wt 486

Figure S.5. DNP search for identification of compound 3

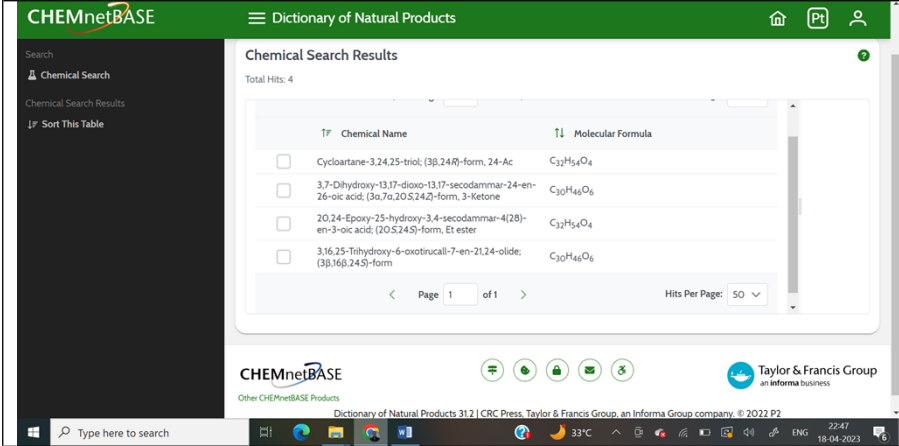


The screenshot shows the CHEMnetBASE search interface. The search criteria are as follows:

- AND Chemical Name
- AND Molecular Formula
- AND Molecular Formula by Element (C)
- AND CAS Registry Nos.
- AND All Text
- AND Melting Point
- AND Boiling Point
- AND Accurate Mass (Range: min. 502.000, max. 502.999)
- AND Biological Source (Dysoxylum)

Searched properties:

- Biological source: Dysoxylum
- Accurate mass range: 502.000 to 502.999



The screenshot shows the CHEMnetBASE search results page. The results are as follows:

TF	Chemical Name	Molecular Formula
☐	Cycloartane-3,24,25-triol; (3β,24R)-form, 24-Ac	C ₃₂ H ₅₄ O ₄
☐	3,7-Dihydroxy-13,17-dioxo-13,17-secodammar-24-en-26-oic acid; (3α,7α,20S,24Z)-form, 3-ketone	C ₃₀ H ₄₆ O ₆
☐	20,24-Epoxy-25-hydroxy-3,4-secodammar-4(28)-en-3-oic acid; (20S,24S)-form, Et ester	C ₃₂ H ₅₄ O ₄
☐	3,16,25-Trihydroxy-6-oxotrucali-7-en-21,24-olide; (3β,16β,24S)-form	C ₃₀ H ₄₆ O ₆

Page 1 of 1, Hits Per Page: 50

Results:

- 4 compounds reported from Dysoxylum genus of mol wt 502

Figure S.6. DNP search for identification of compound 4

CHEMnetBASE

Dictionary of Natural Products

Search

Chemical Search

Chemical Search Options

+ Add Properties

Clear Properties

Start a New Search

Boolean	Property	Comparison	Value	Remove
AND	Chemical Name			
AND	Molecular Formula			
AND	Molecular Formula by Element	* C		
AND	CAS Registry Nos.			
AND	All Text			
AND	Melting Point	=		
AND	Boiling Point	=		
AND	Biological Source		Dysoxylum	
AND	Accurate Mass	Range	min. 472.000 max. 472.999	

Searched properties:

Biological source:
Dysoxylum

Accurate mass range:
472.000 to 472.999

CHEMnetBASE

Dictionary of Natural Products

Search

Chemical Search

Chemical Search Results

Sort This Table

Chemical Search Results

Total Hits: 17

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Hits Per Page: 10

	Chemical Name	Molecular Formula
☐	21,24-Cyclotriacall-7-ene-3,16,21,25-tetrol- (3 β ,16 β ,21S,24S)-form, 3-Ketone	C ₃₀ H ₄₈ O ₄
☐	3,7-Dihydroxyapotriacalla-14,24-dien-26- oic acid, (3 α ,7 α ,24Z)-form	C ₃₀ H ₄₈ O ₄
☐	3,7-Dihydroxydammar-13(17),24-dien-26- oic acid, (3 α ,7 α ,20S,24Z)-form	C ₃₀ H ₄₈ O ₄
☐	21,24-Epoxy-23,25-dihydroxycycloartan-3- one, (23S,24S)-form	C ₃₀ H ₄₈ O ₄
☐	21,25-Epoxy-23,24-dihydroxycycloartan-3- one, (23R,24S)-form	C ₃₀ H ₄₈ O ₄

Results:

17 compounds reported
from Dysoxylum genus of
mol wt 472 having
different mol formula

CHEMnetBASE

Dictionary of Natural Products

Search

Chemical Search

Chemical Search Options

+ Add Properties

Clear Properties

Start a New Search

Boolean	Property	Comparison	Value	Remove
AND	Chemical Name			
AND	Molecular Formula			
AND	Molecular Formula by Element	= C	30	
AND	CAS Registry Nos.			
AND	All Text			
AND	Melting Point	*		
AND	Boiling Point	*		
AND	Biological Source		Dysoxylum	
AND	Accurate Mass	Range	min. 472.000 max. 472.999	

Search

Searched properties:

Biological source:
Dysoxylum

Accurate mass range:
472.000 to 472.999

Number of carbon=30

CHEMnetBASE

Dictionary of Natural Products

Search

Chemical Search

Chemical Search Results

Sort This Table

To access all features L

Chemical Search Results

Total Hits: 14

Page 1 of 2

Hits Per Page: 10

Chemical Name	Molecular Formula
☐ 2124-Cyclotriacall-7-ene-3,16,21,25-tetrol; (3S,16R,21S,24S)-form, 3-Ketone	C ₃₀ H ₄₈ O ₄
☐ 3,7-Dihydroxyapotiucalla-14,24-dien-26-oic acid; (3S,7S,24Z)-form	C ₃₀ H ₄₈ O ₄
☐ 3,7-Dihydroxydammar-13(17),24-dien-26-oic acid; (3S,7S,20S,24Z)-form	C ₃₀ H ₄₈ O ₄
☐ 2124-Epoxy-23,25-dihydroxycycloartan-3-one; (23S,24S)-form	C ₃₀ H ₄₈ O ₄
☐ 2125-Epoxy-23,24-dihydroxycycloartan-3-one; (23R,24S)-form	C ₃₀ H ₄₈ O ₄

Results:

14 compounds reported from Dysoxylum genus of mol wt 472

CHEMnetBASE Dictionary of Natural Products

Search Chemicals

Q Search Tautomers Substructure Exact Not Subst Clear Structure



Boolean	Property	Comparison	Value	Remove
AND	Chemical Name			
AND	Molecular Formula			
AND	Molecular Formula by Element		30	
AND	CAS Registry Nos.			
AND	All Text			
AND	Melting Point			
AND	Boiling Point			
AND	Biological Source		Dysoxylum	
AND	Accurate Mass	Range	min: 472.000 max: 472.999	

Q Search

Searched properties:

Biological source:
Dysoxylum

Accurate mass range:
472.000 to 472.999

Number of carbon=30

Cycloartane ring

CHEMnetBASE Dictionary of Natural Products

Search

Chemical Search

Chemical Search Results

Sort This Table

Chemical Search Results

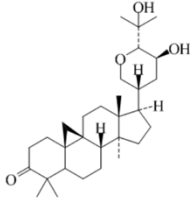
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Page 1 of 1 Hits Per Page: 50

Chemical Name	Molecular Formula
☒ 21,24-Epoxy-23,25-dihydroxycycloartan-3-one; (23S,24S)-form	C ₃₀ H ₄₈ O ₄
☐ 21,25-Epoxy-23,24-dihydroxycycloartan-3-one; (23R,24S)-form	C ₃₀ H ₄₈ O ₄

Page 1 of 1 Hits Per Page: 50

Entry Name: 21,24-Epoxy-23,25-dihydroxycycloartan-3-one



CRC Number: RLO89-A

Molecular Formula: C₃₀H₄₈O₄

Results:

2 compounds reported from
Dysoxylum genus of mol wt
472 having 30 carbon and
cycloartane ring

Spectral information of compound 1

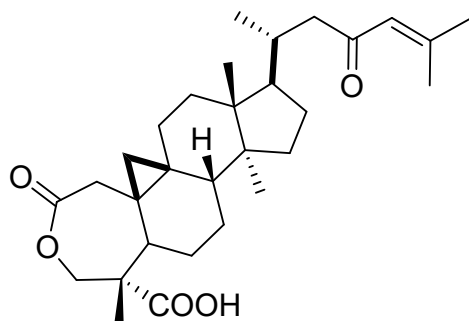


Figure S.7. ^1H NMR spectrum of compound 1 in CDCl_3

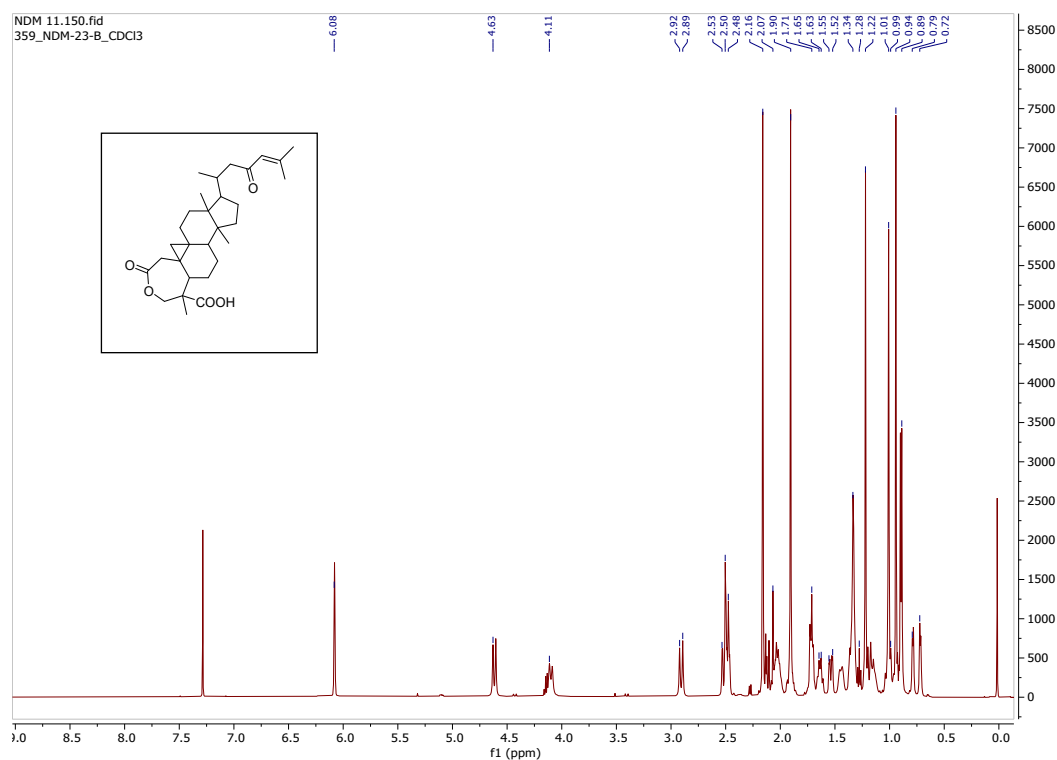


Figure S.8. ^{13}C NMR spectrum of compound 1 in CDCl_3

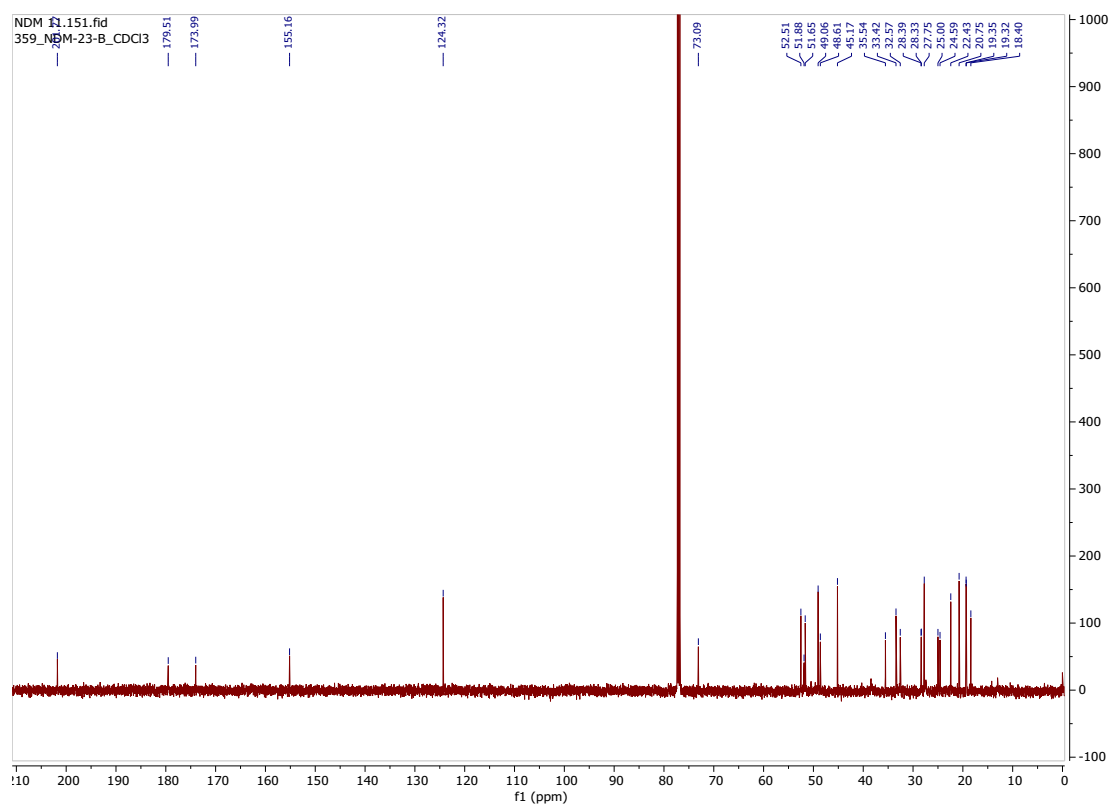


Figure S.9. DEPT-135 NMR spectrum of compound 1 in CDCl₃

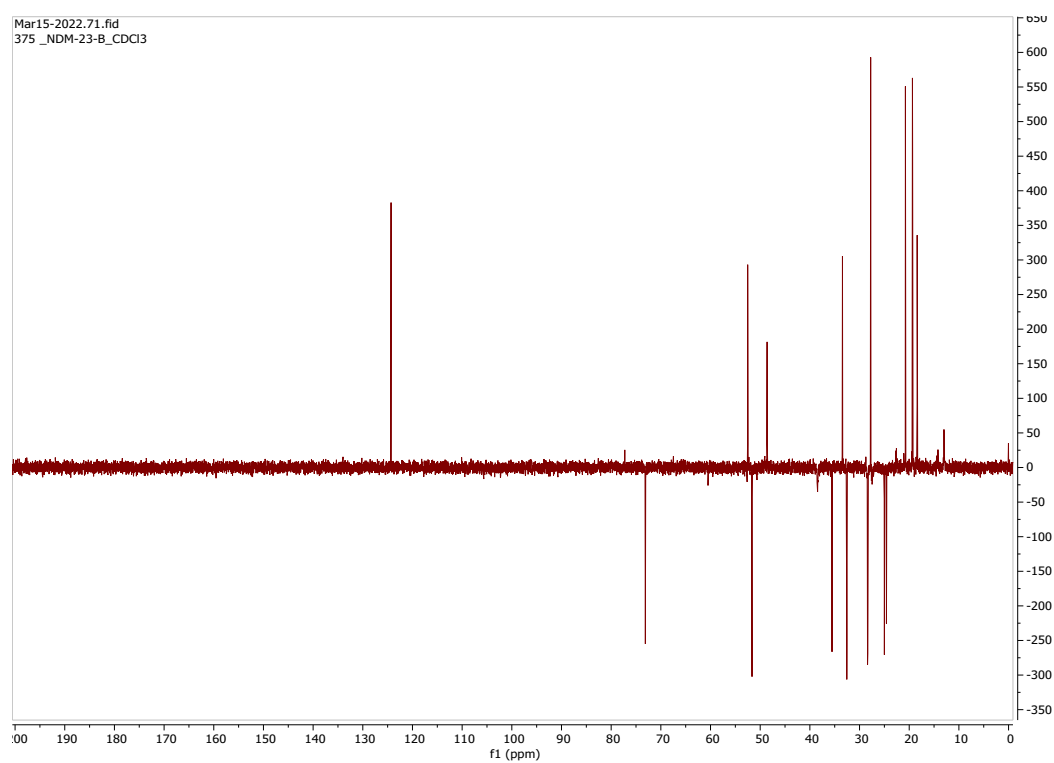


Figure S.10. HSQC spectrum of compound 1 in CDCl₃

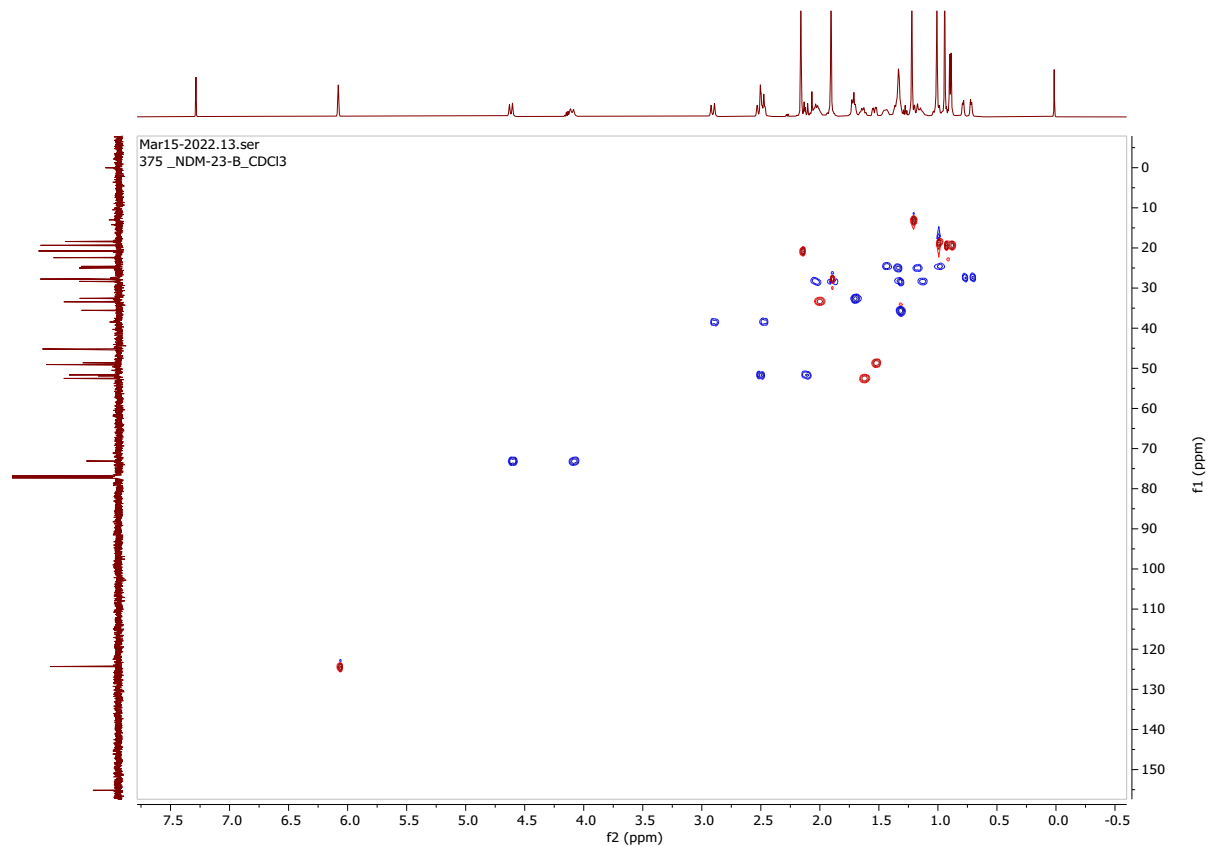


Figure S.11. HMBC spectrum of compound 1 in CDCl₃

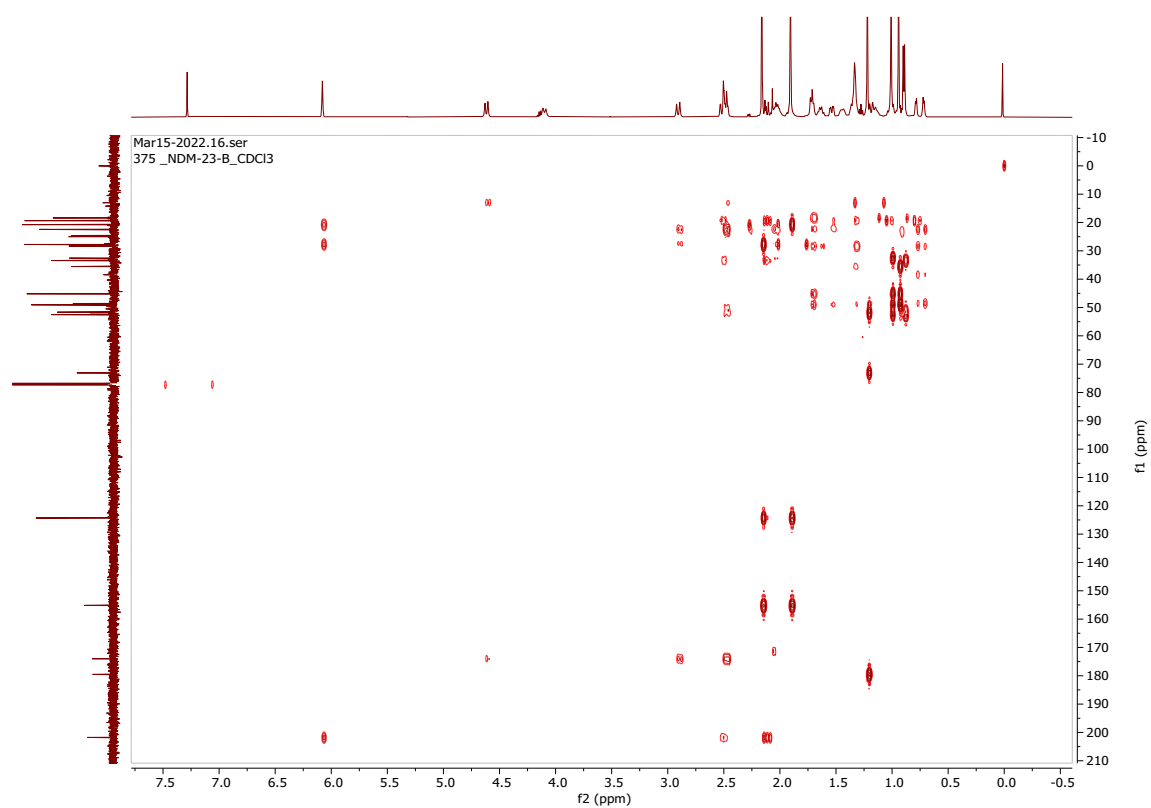


Figure S.12. COSY NMR spectrum of compound 1 in CDCl₃

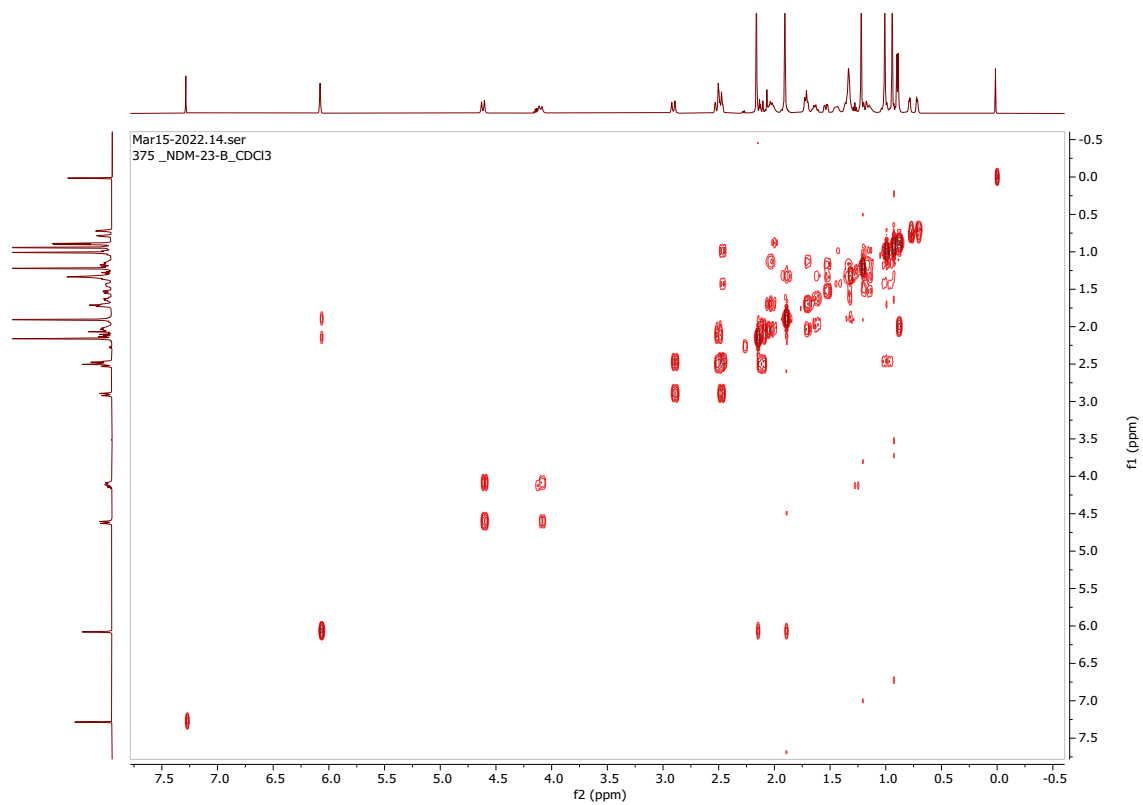


Figure S.13. NOESY spectrum of compound 1 in CDCl₃

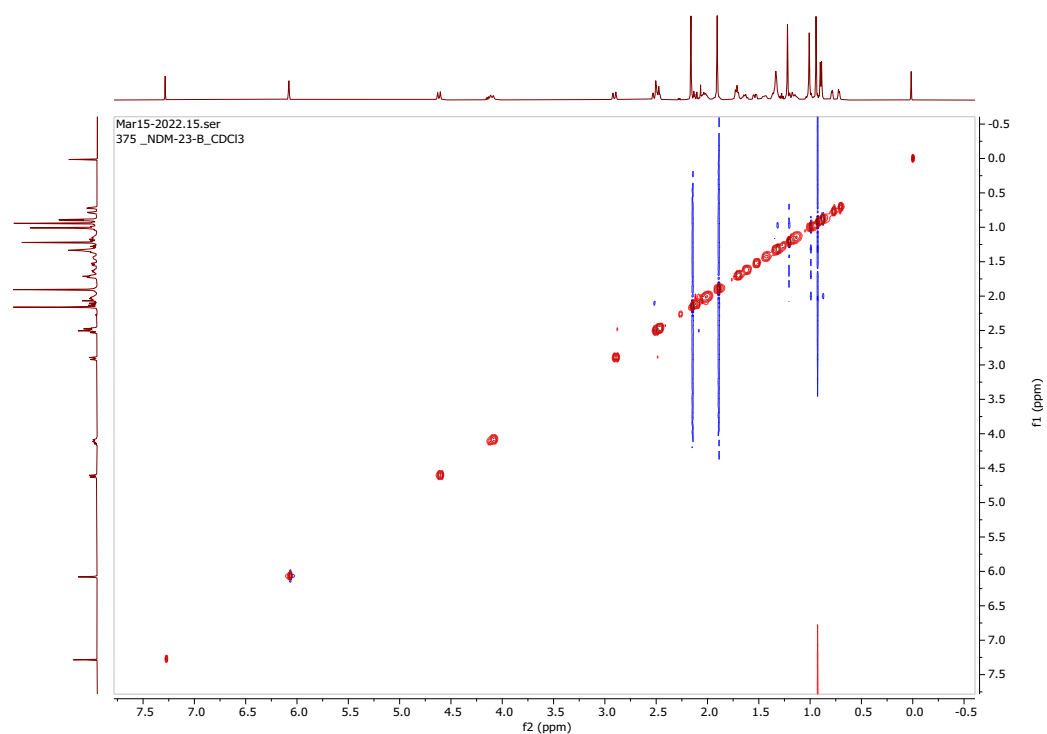


Figure S.14. HRMS spectrum of compound 1

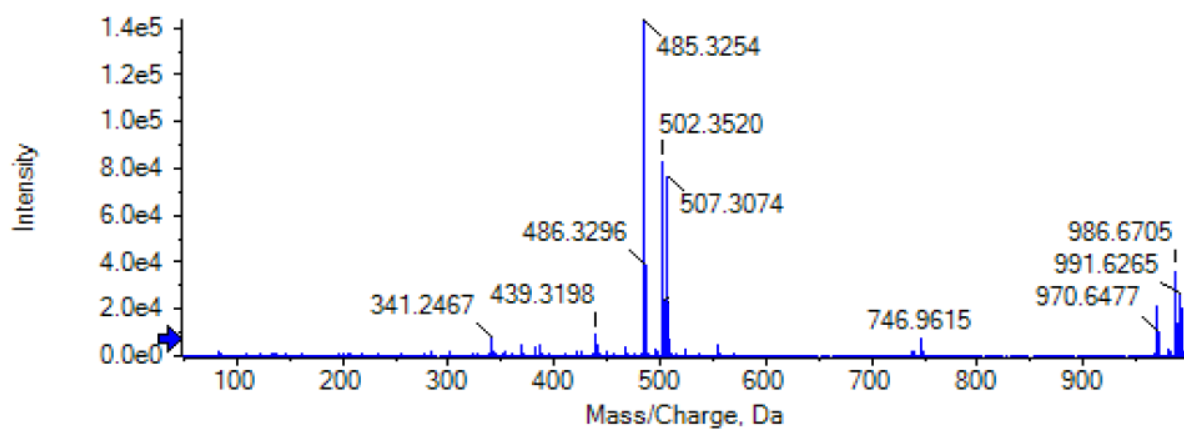


Figure S.15. IR spectrum of compound 1

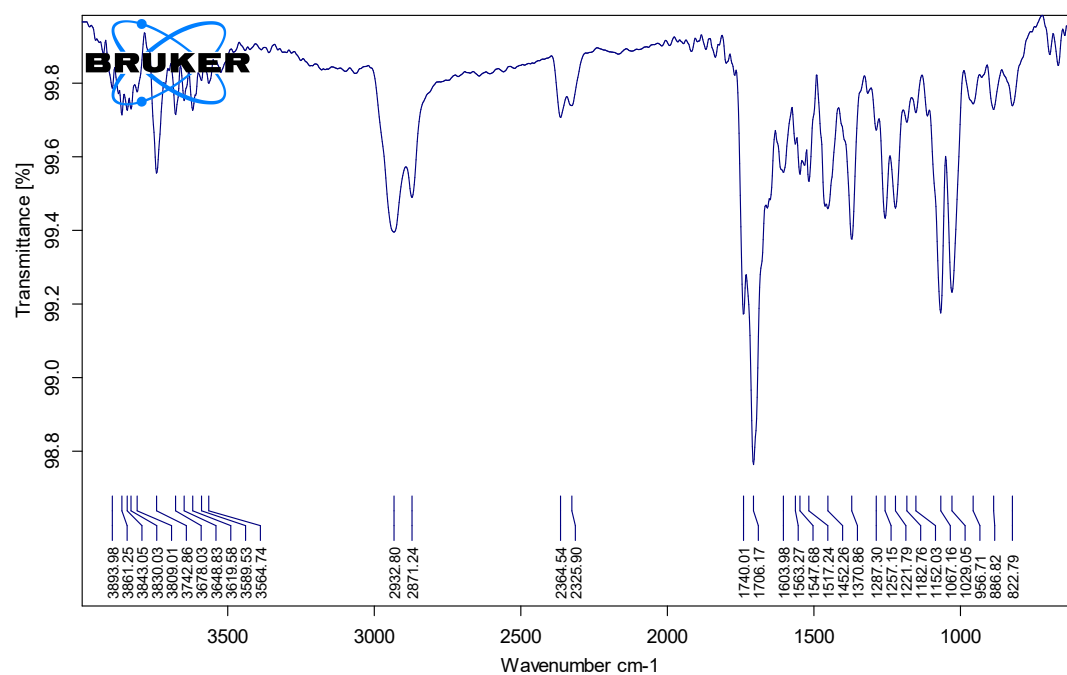
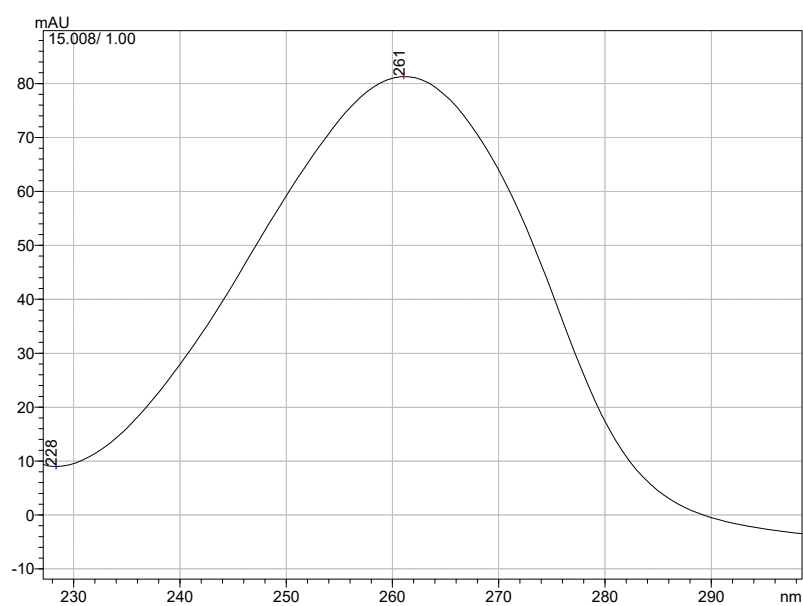
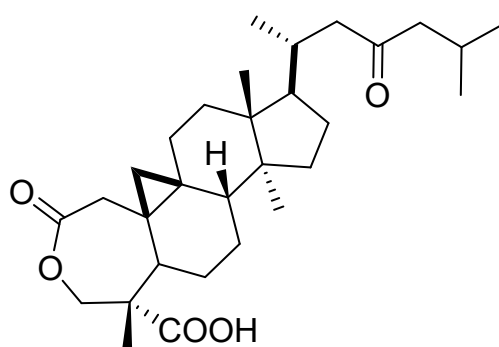


Figure S.16: UV spectrum of compound 1 in methanol



Spectral information of Compound 2



21-11-22.10.fid
199_NB-DM-4/11_CDCl3

CC(C)CC(=O)C1C(C)CC2(C)CC3C(C1)C(=O)OCC3C2
Dihydrobiddemallalactone
(2)

Chemical shift (ppm): 13.5, 13.0, 12.5, 12.0, 11.5, 11.0, 10.5, 10.0, 9.5, 9.0, 8.5, 8.0, 7.5, 7.0, 6.5, 6.0, 5.5, 5.0, 4.5, 4.0, 3.5, 3.0, 2.5, 2.0, 1.5, 1.0, 0.5, 0.0

Integration values (from left to right): 4.35, 3.84, 2.22, 2.18, 2.02, 1.90, 1.81, 1.79, 1.77, 1.65, 1.61, 1.57, 1.47, 1.46, 1.39, 1.37, 1.30, 1.28, 1.25, 1.15, 1.12, 1.10, 1.08, 1.03, 1.03, 0.93, 0.76, 0.69, 0.68, 0.54, 0.47

11-19-22.51.fid
174j-NB-DM-4/11_CDCl3

210.0
179.23
173.74
72.85
52.39
52.03
51.63
48.81
48.34
44.90
35.55
32.49
28.10
28.07
24.31
22.44
22.32
22.18
19.13
19.09
18.14
12.75

f1 (ppm)

Figure S.19 : DEPT-135 NMR spectrum of compound 2 in CDCl₃

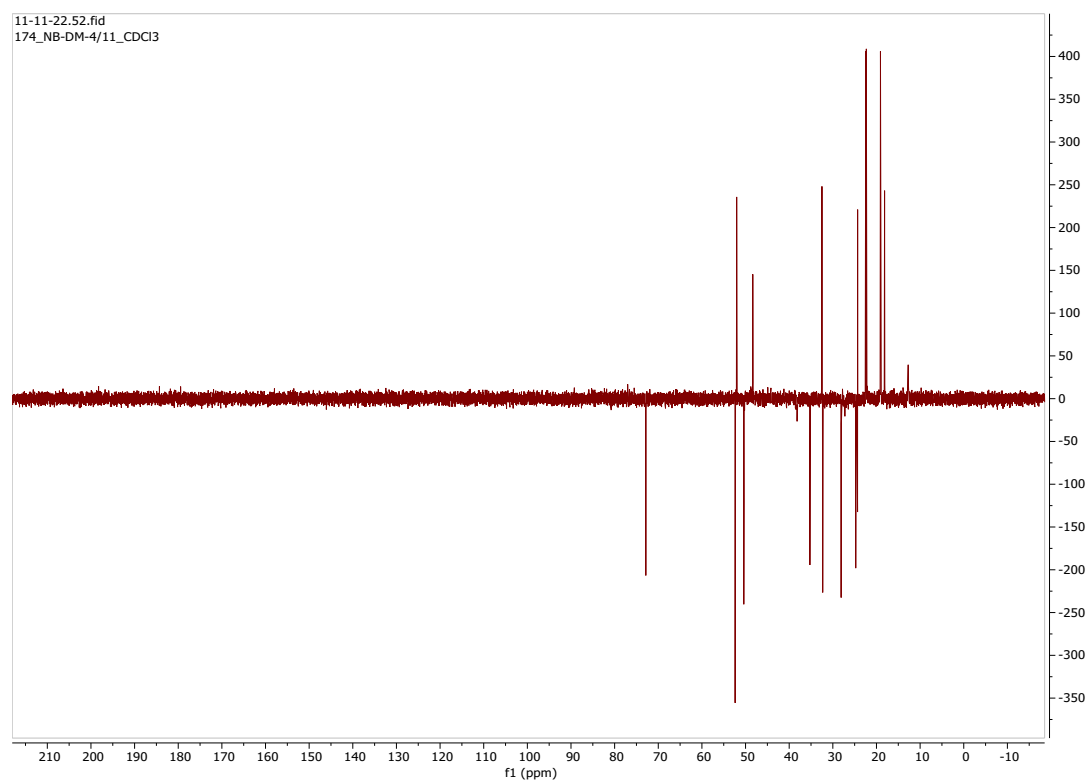


Figure S.20: HSQC NMR spectrum of compound 2 in CDCl₃

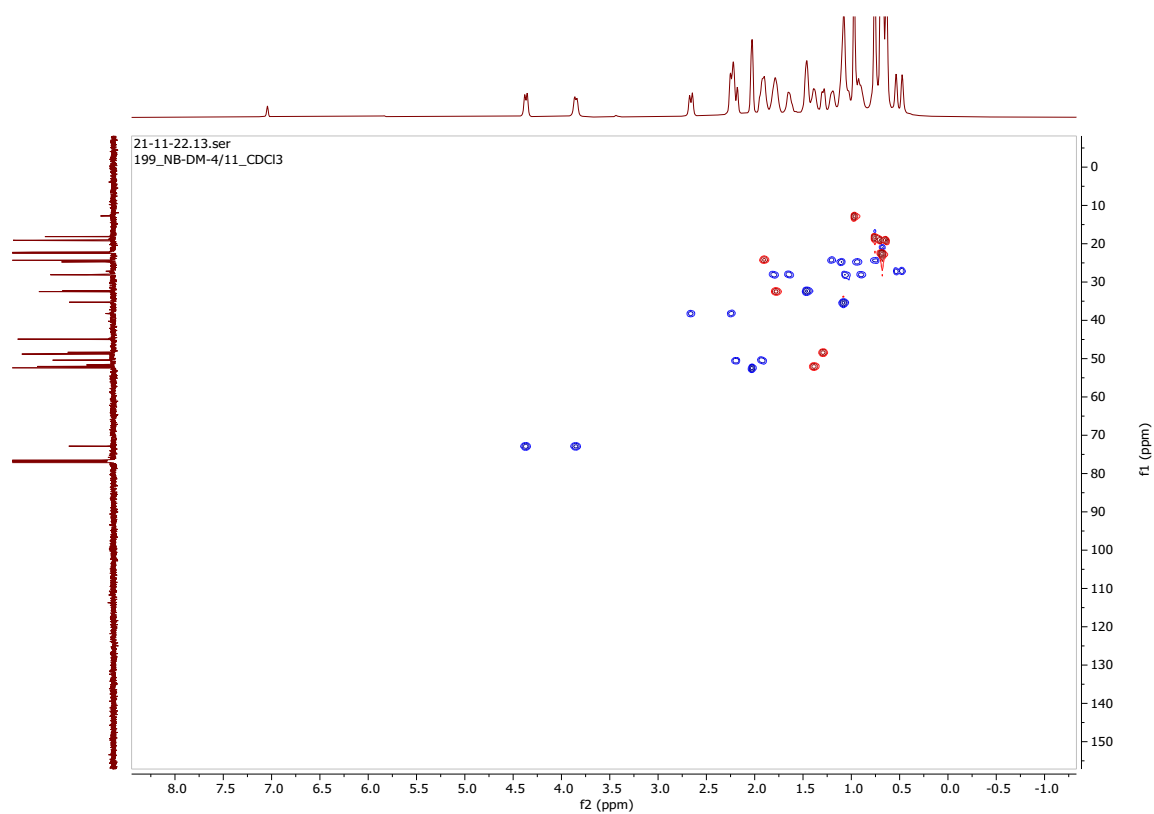


Figure S.21 : HMBC NMR spectrum of compound 2 in CDCl₃

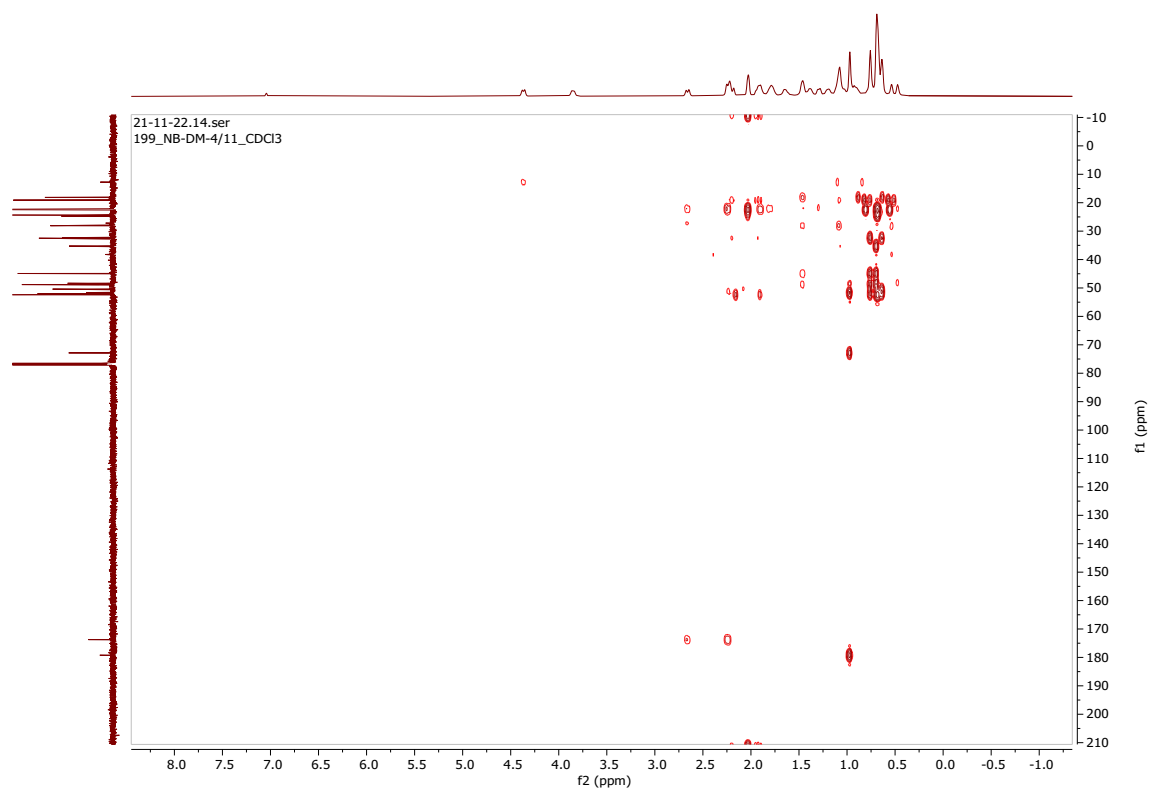


Figure S.22: ¹H-¹H COSY NMR spectrum of compound 2 in CDCl₃

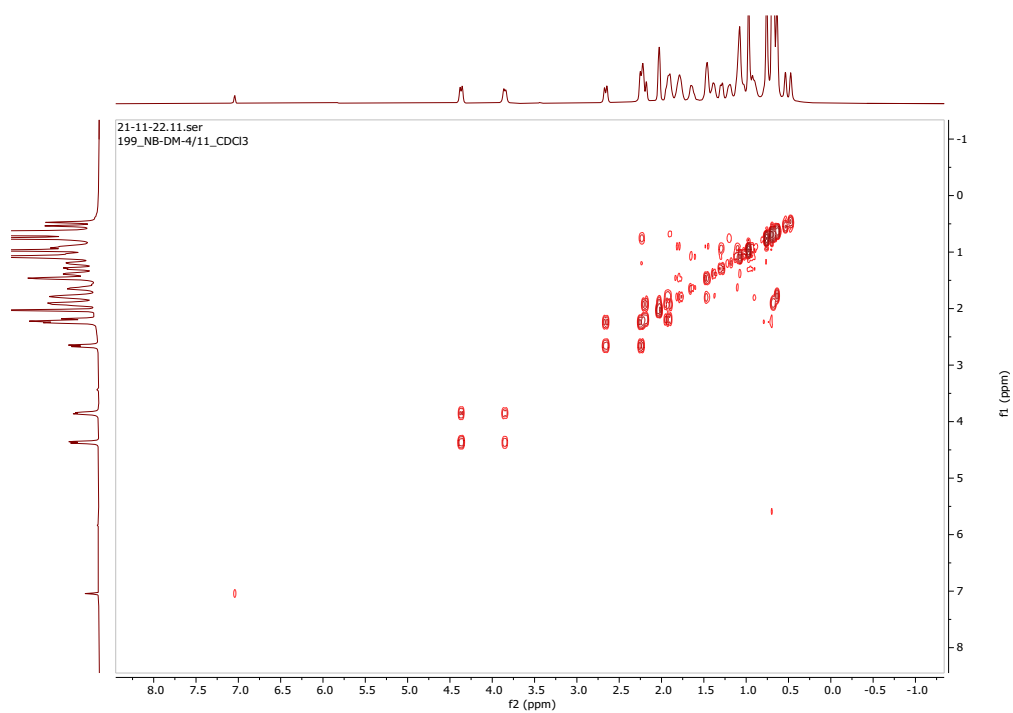


Figure S.23: NOESY NMR spectrum of compound 2 in CDCl_3

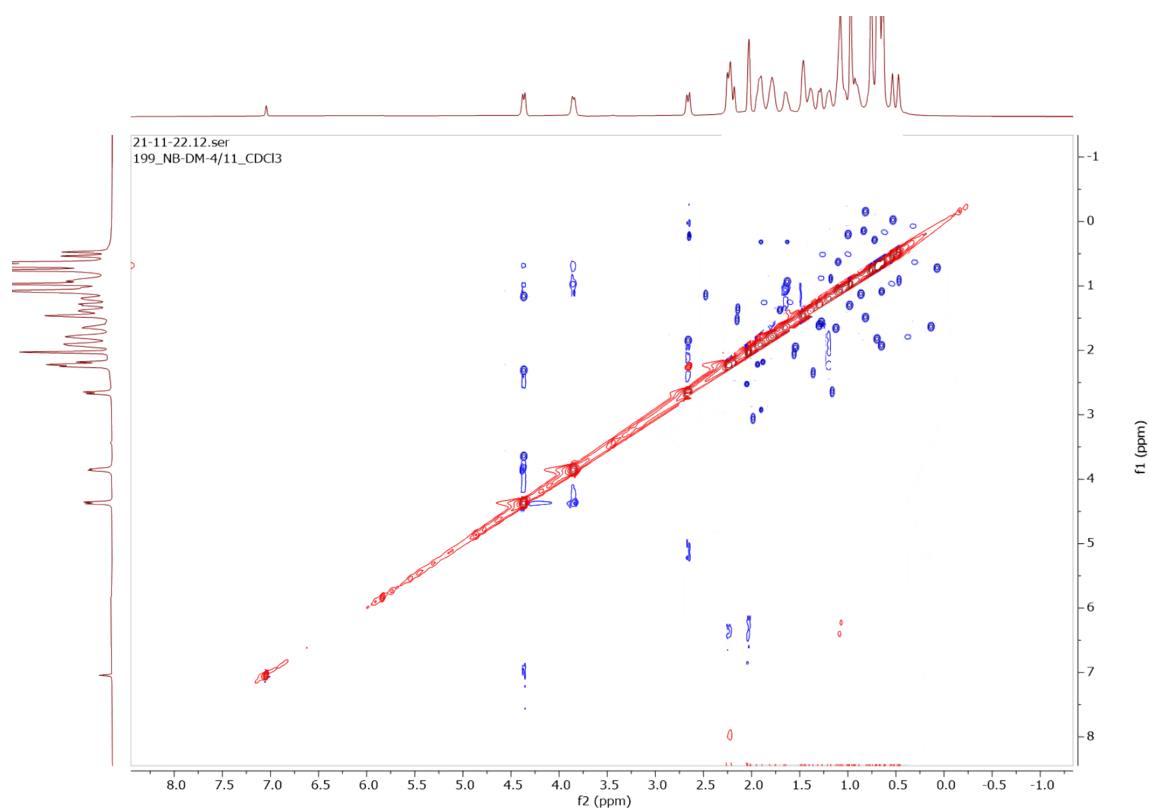


Figure S.24: HRESIMS spectrum of compound 2

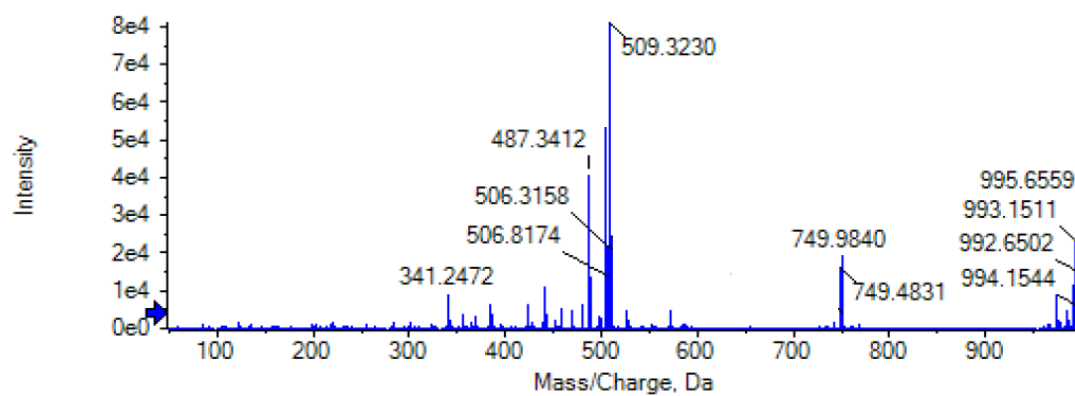


Figure S.25: IR spectrum of compound 2

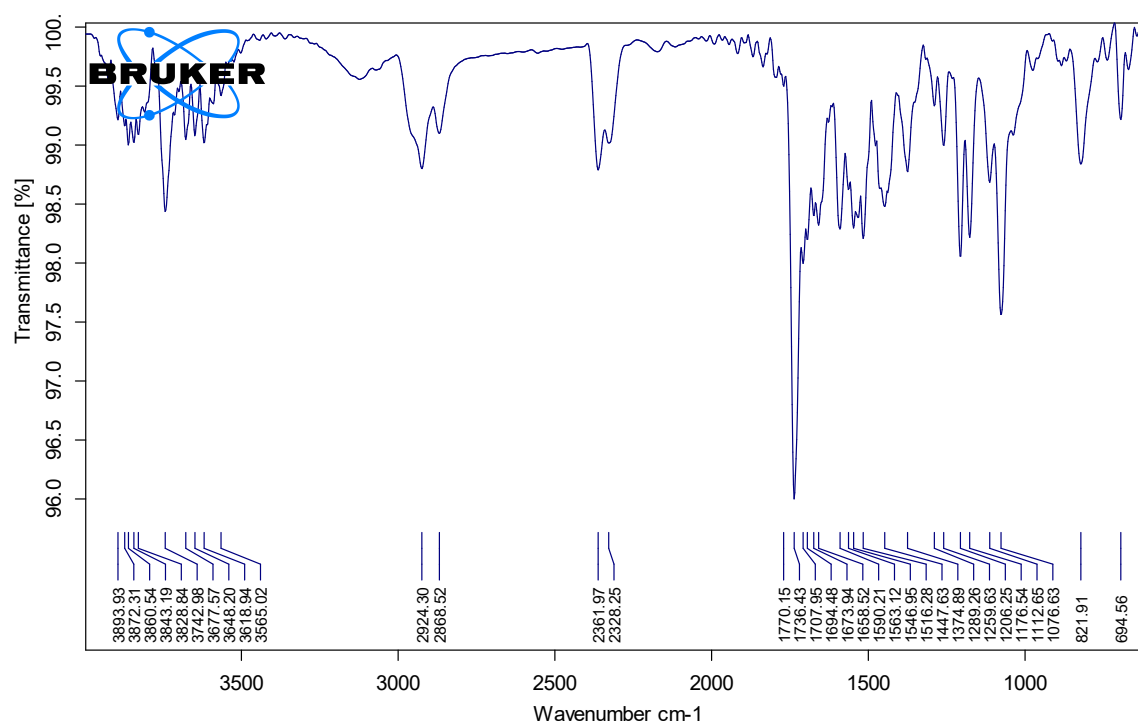
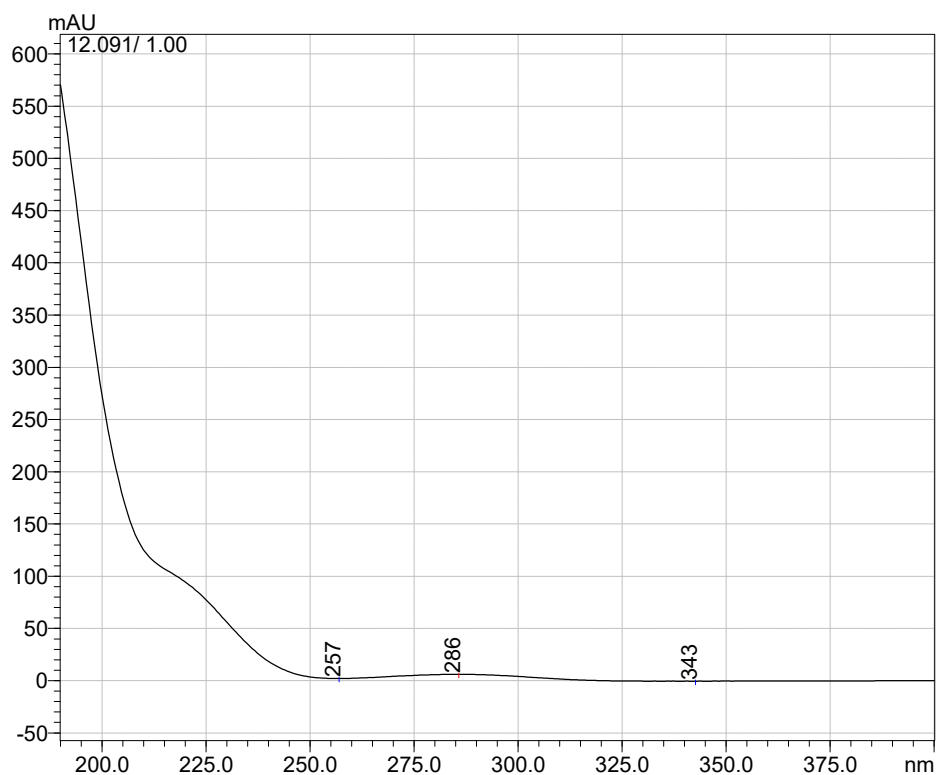


Figure S.26: UV spectrum of compound 2 in methanol



(Note: Compound 2 is UV inactive)

Spectral information of Compound 3

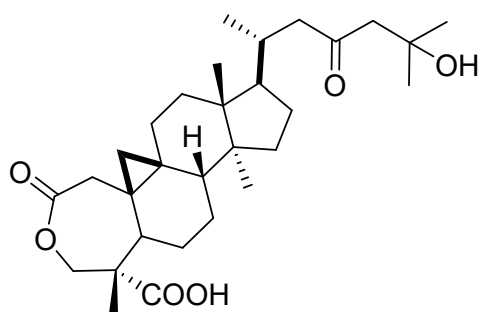


Figure S.27: ^1H NMR spectrum of compound 3 in CD_3OD

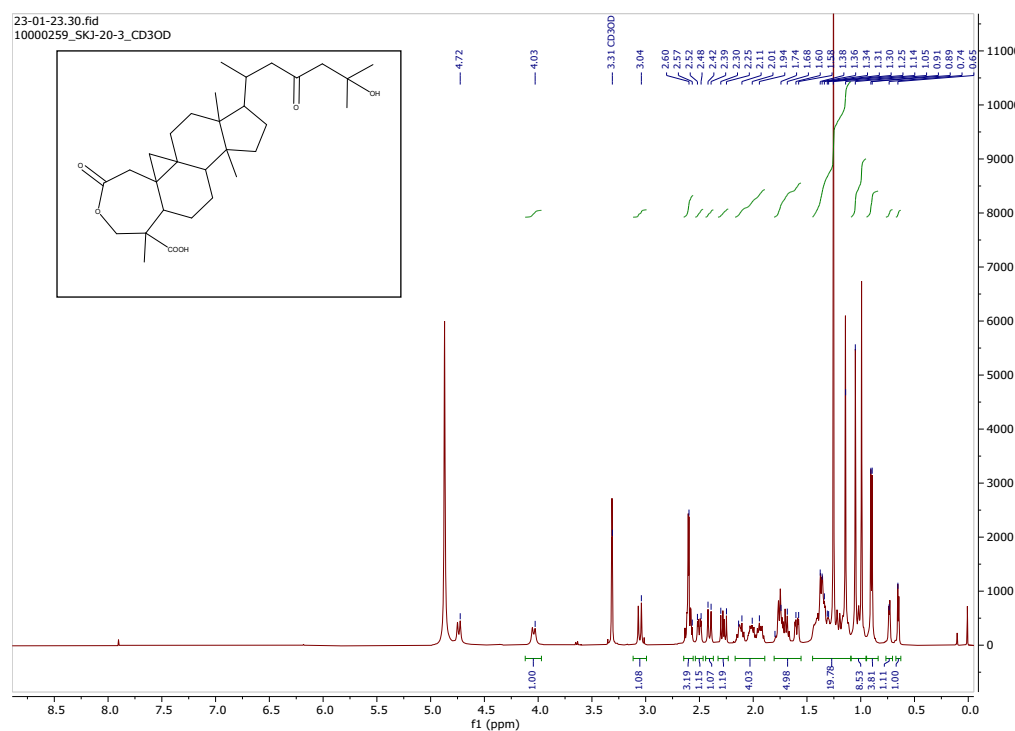


Figure S.28: ^{13}C NMR spectrum of compound 3 in CD_3OD

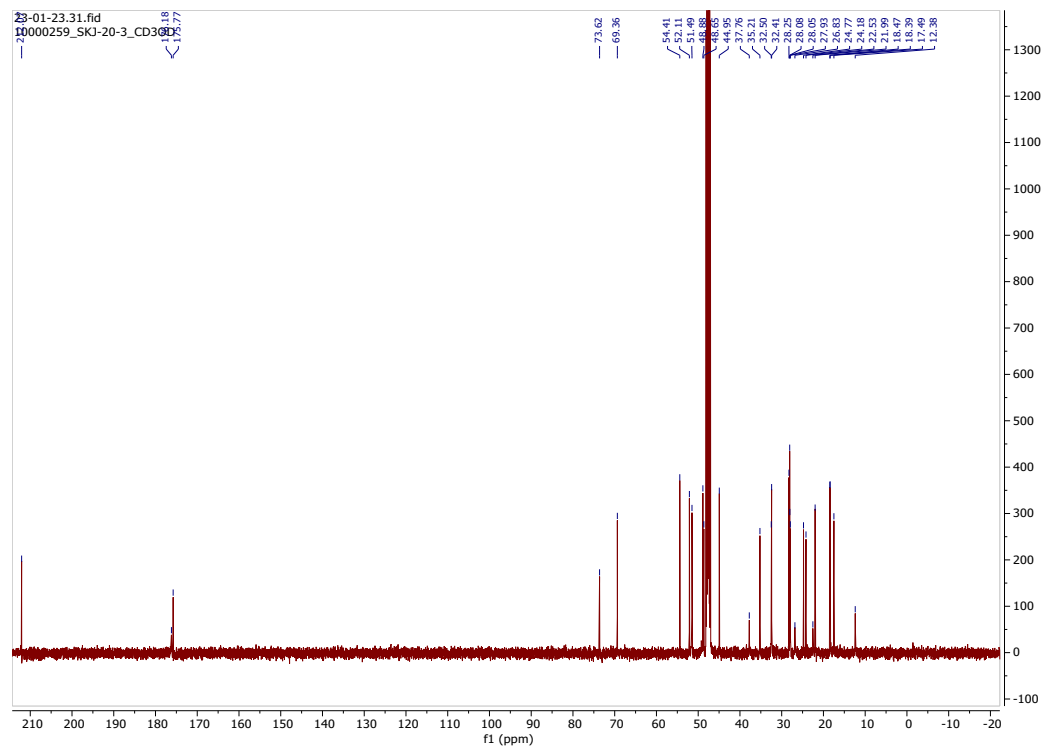


Figure S.29: DEPT-135 NMR spectrum of compound 3 in CD₃OD

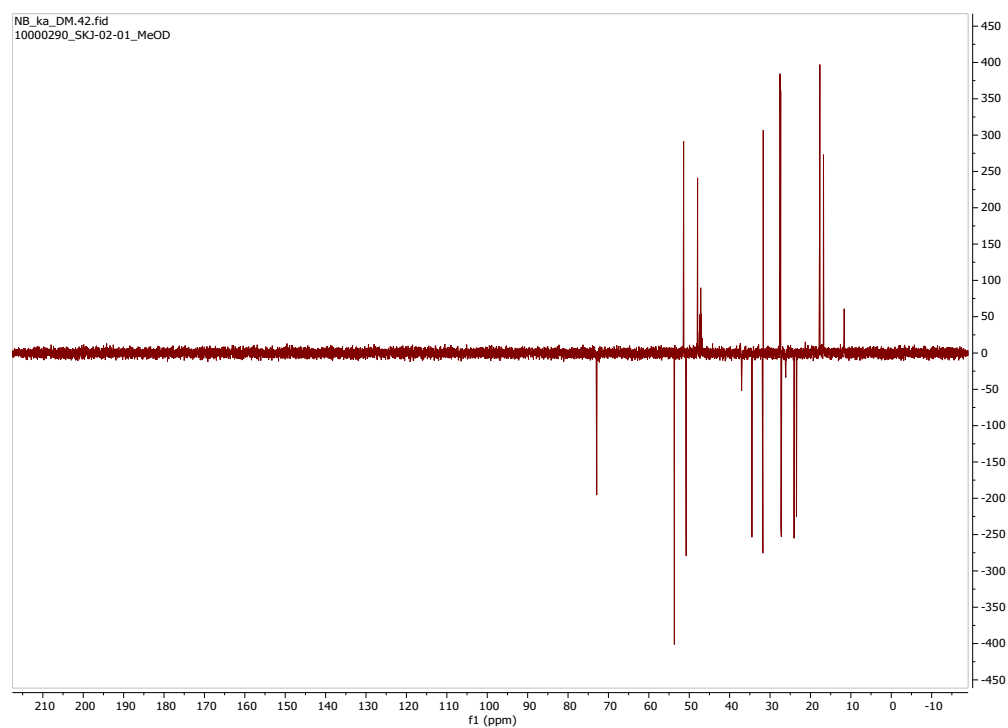


Figure S.30: HSQC NMR spectrum of compound 3 in CD₃OD

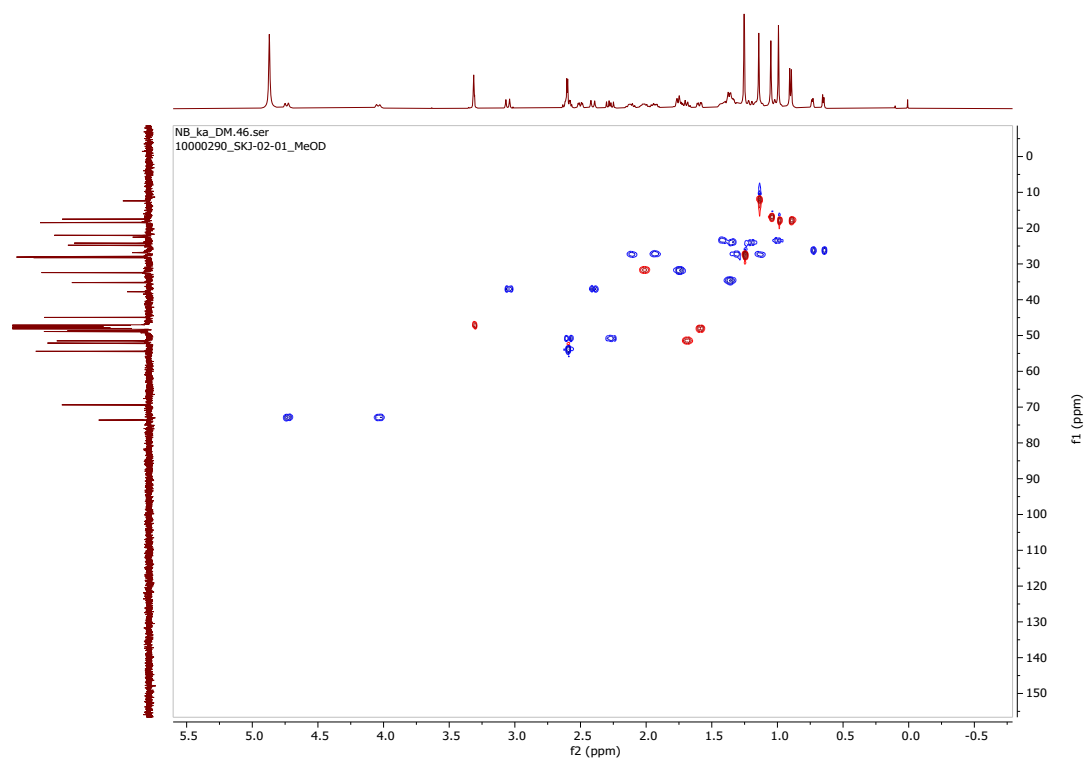


Figure S.31: HMBC NMR spectrum of compound 3 in CD₃OD

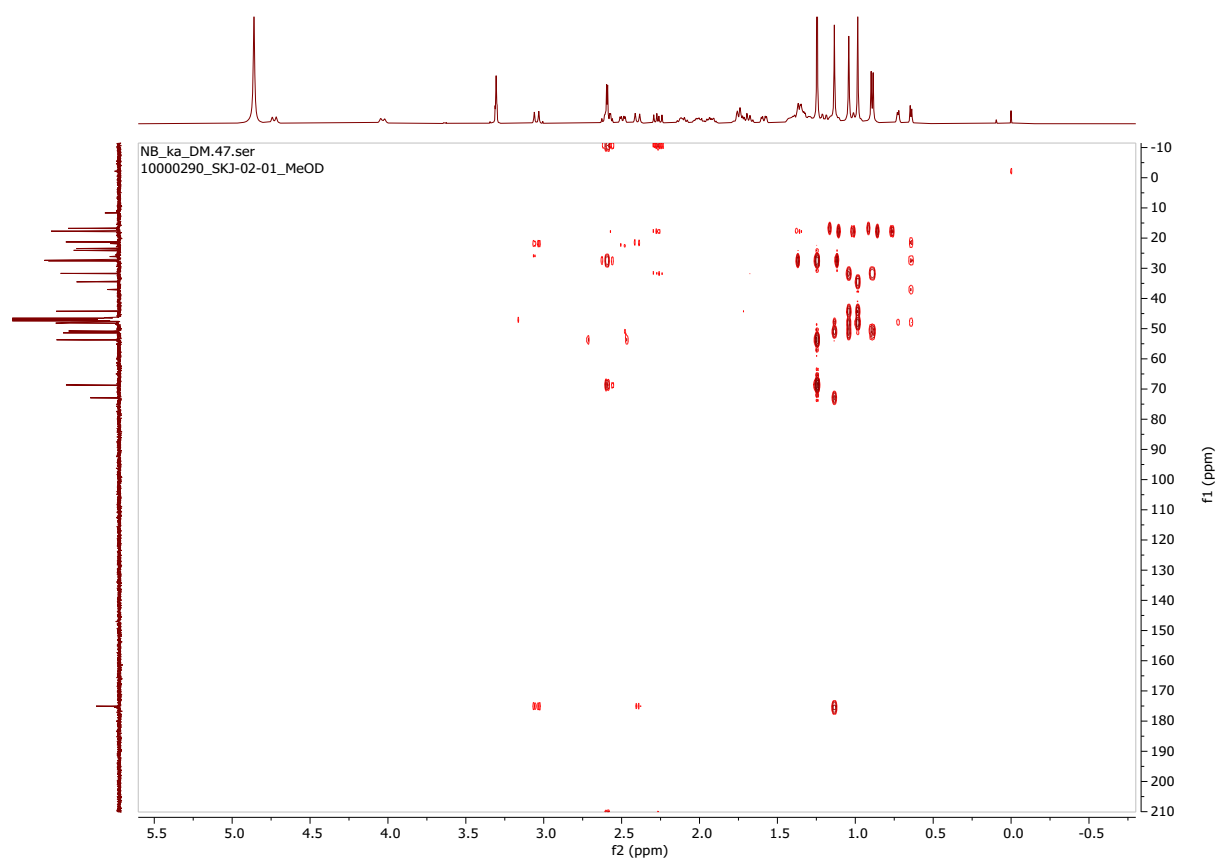


Figure S.32: ¹H-¹H COSY NMR spectrum of compound 3 in CD₃OD

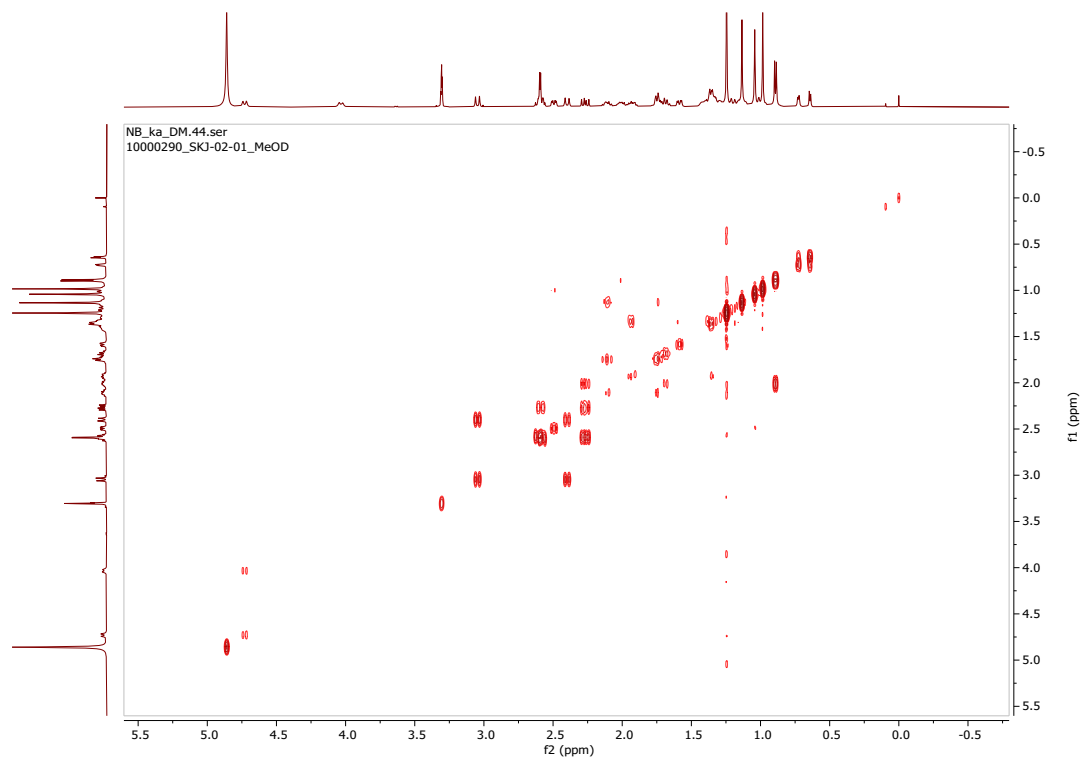


Figure S.33: NOESY NMR spectrum of compound 3 in CD₃OD

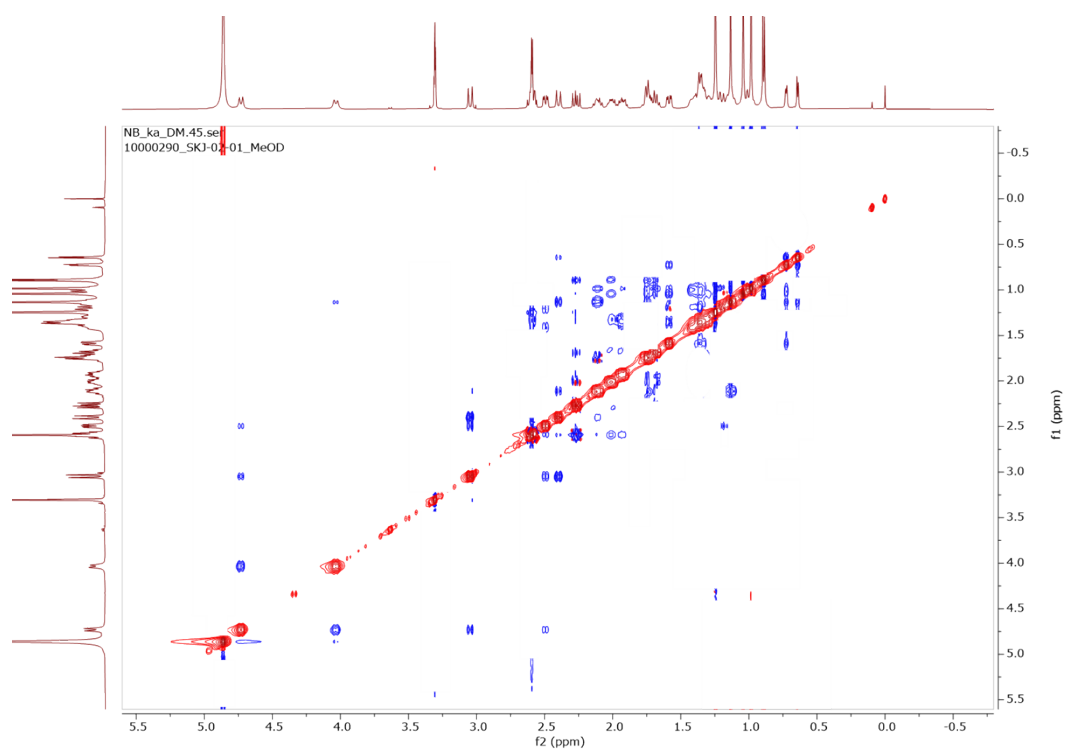


Figure S.34: HRMS mass spectrum of compound 3

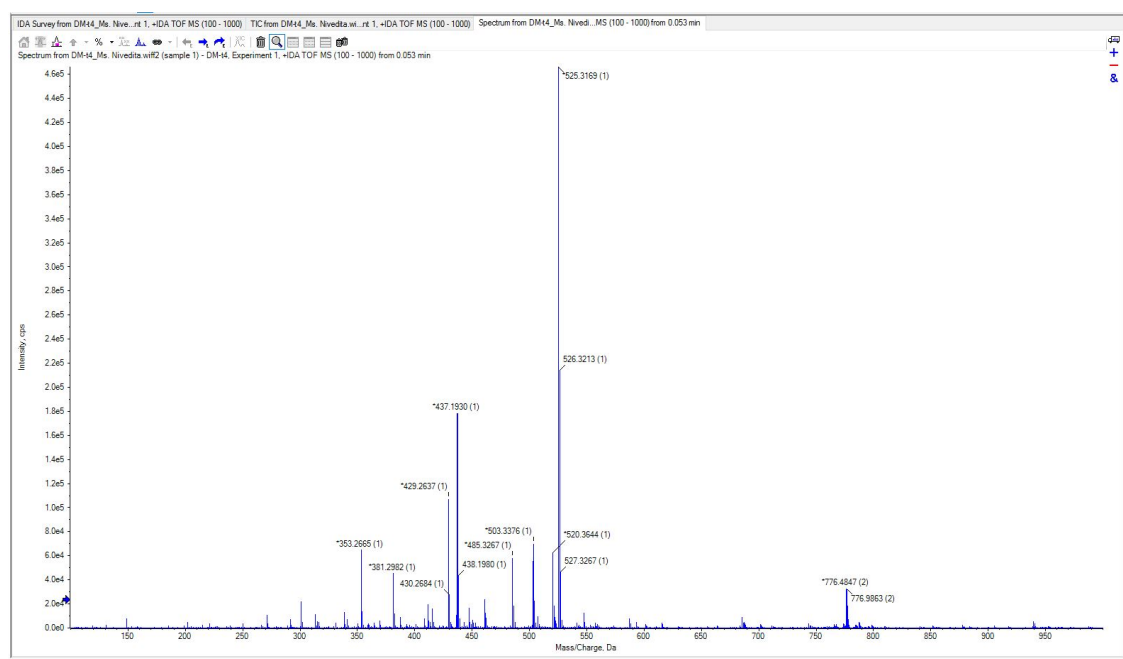


Figure S.35: IR spectrum of compound 3

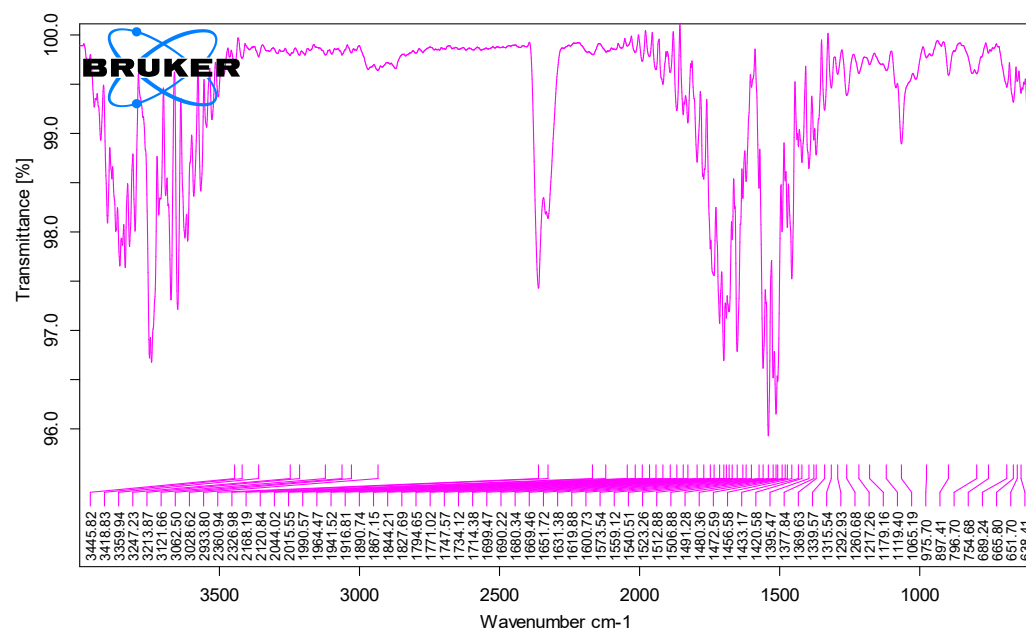
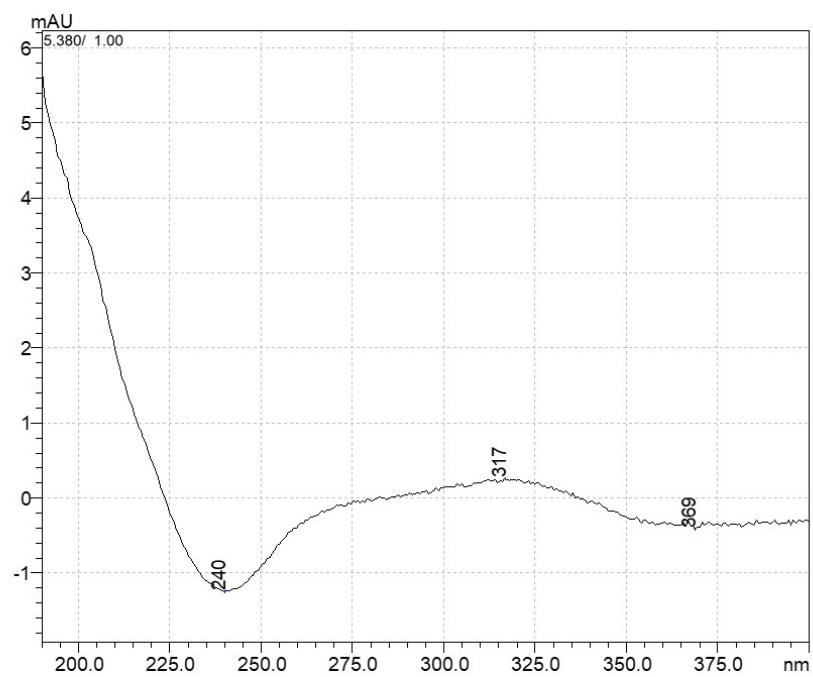


Figure S.36: UV spectrum of compound 3



(Note: Compound 3 is UV inactive)

Spectral information of Compound 4

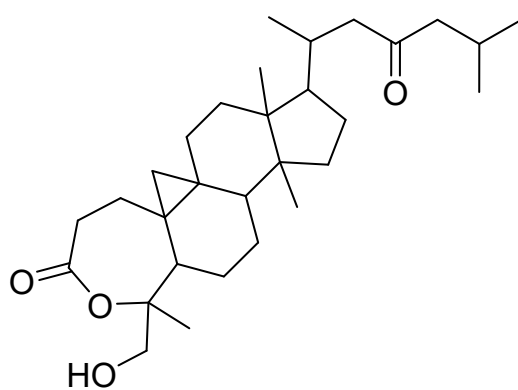


Figure S.37: ^1H NMR spectrum of compound 4 in CD_3OD

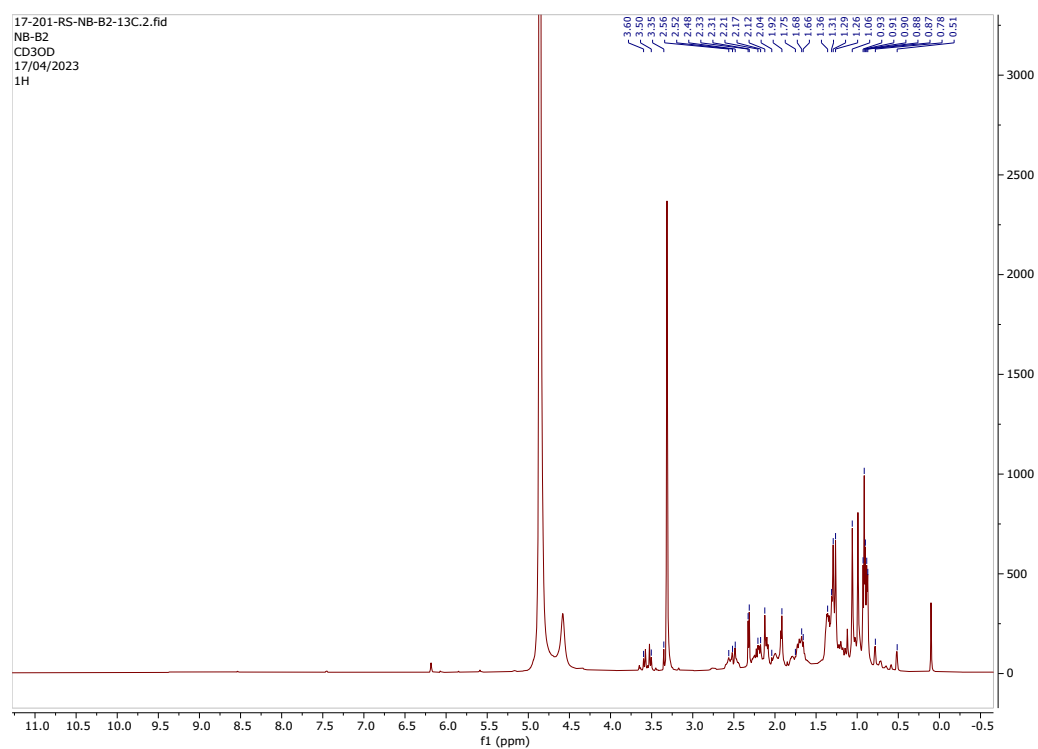


Figure S.38: ^{13}C NMR spectrum of compound 4 in CD_3OD

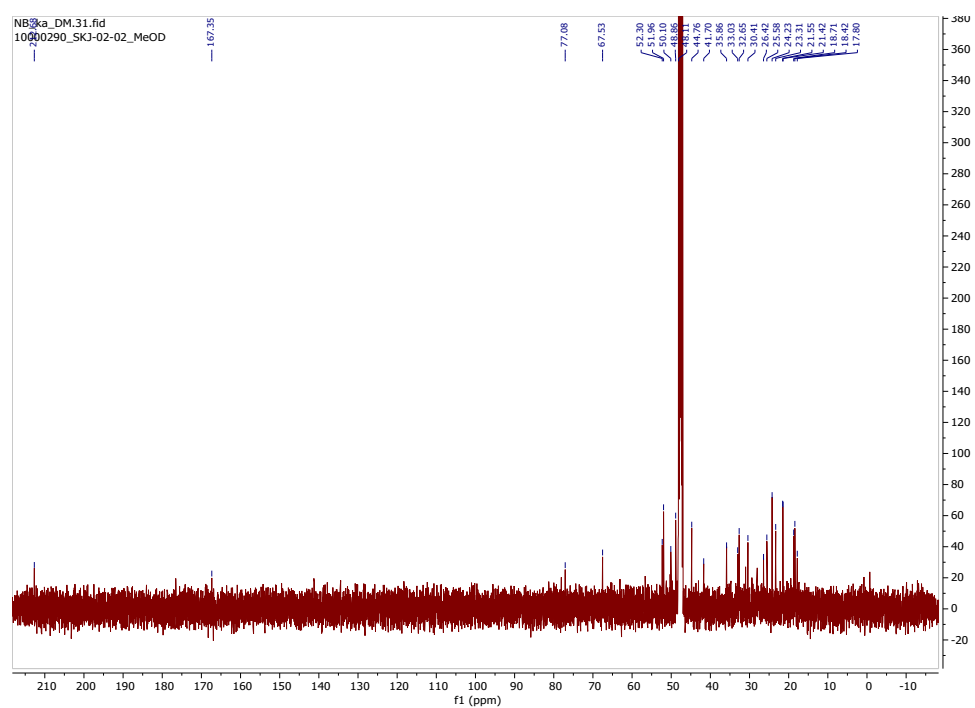


Figure S.39: DEPT-135 NMR spectrum of compound 4 in CD₃OD

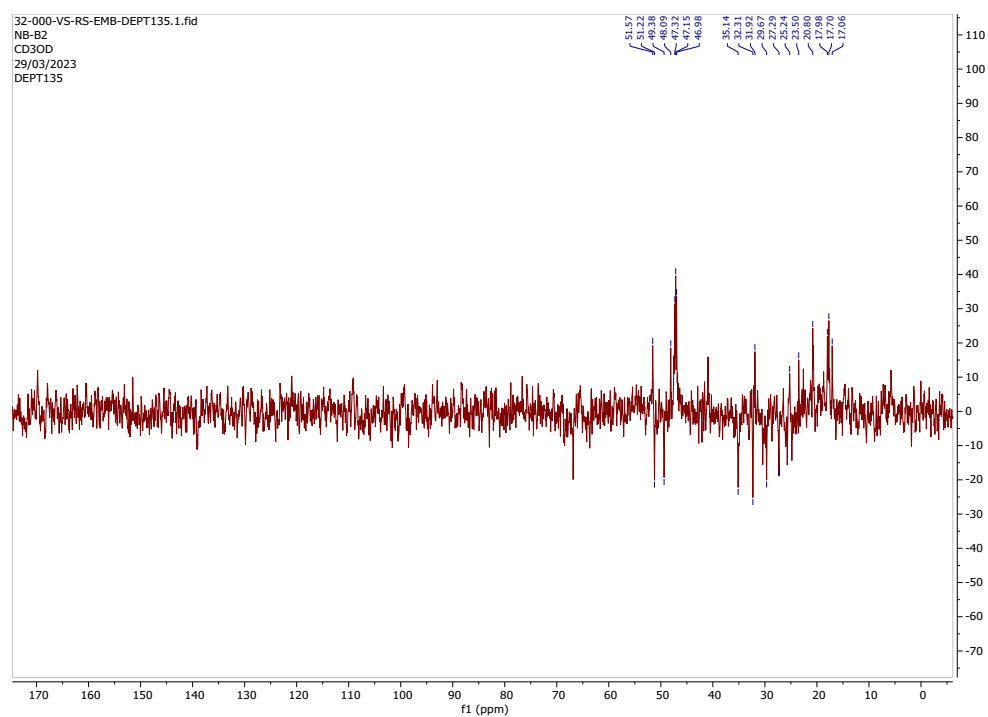


Figure S.40: HSQC NMR spectrum of compound 4 in CD₃OD

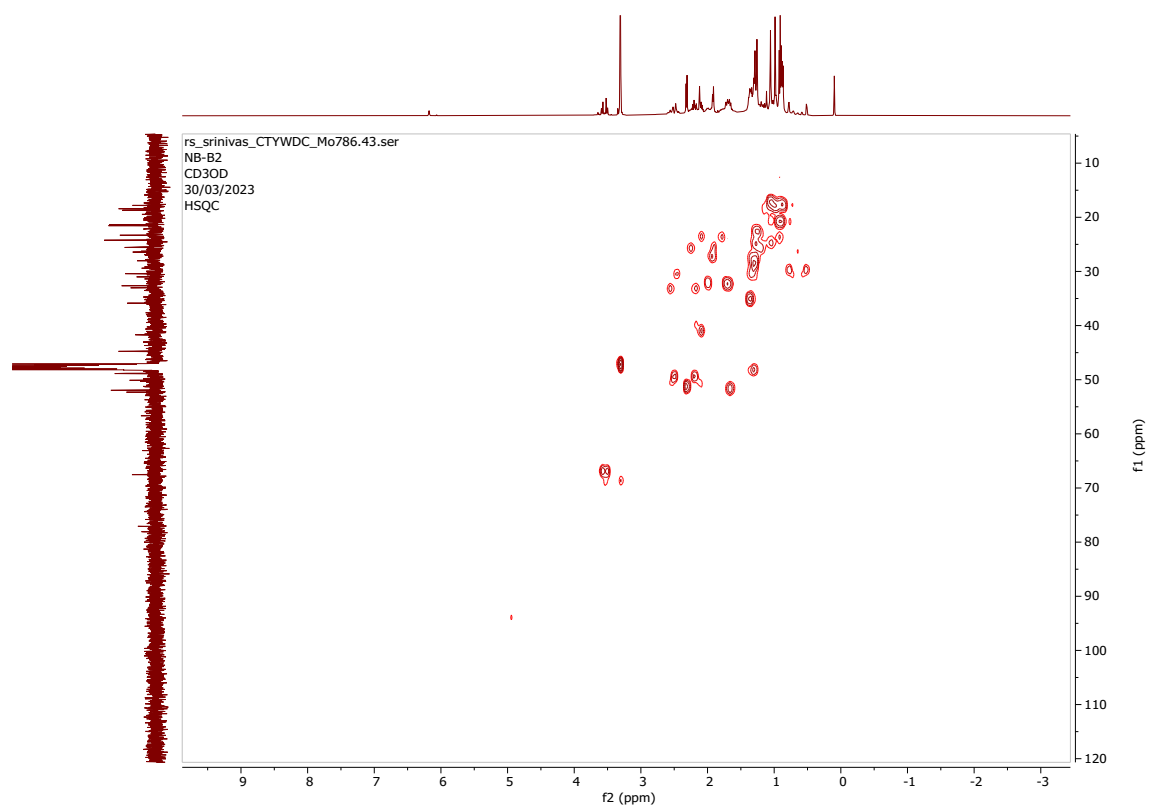


Figure S.41: HMBC NMR spectrum of compound 4 in CD₃OD

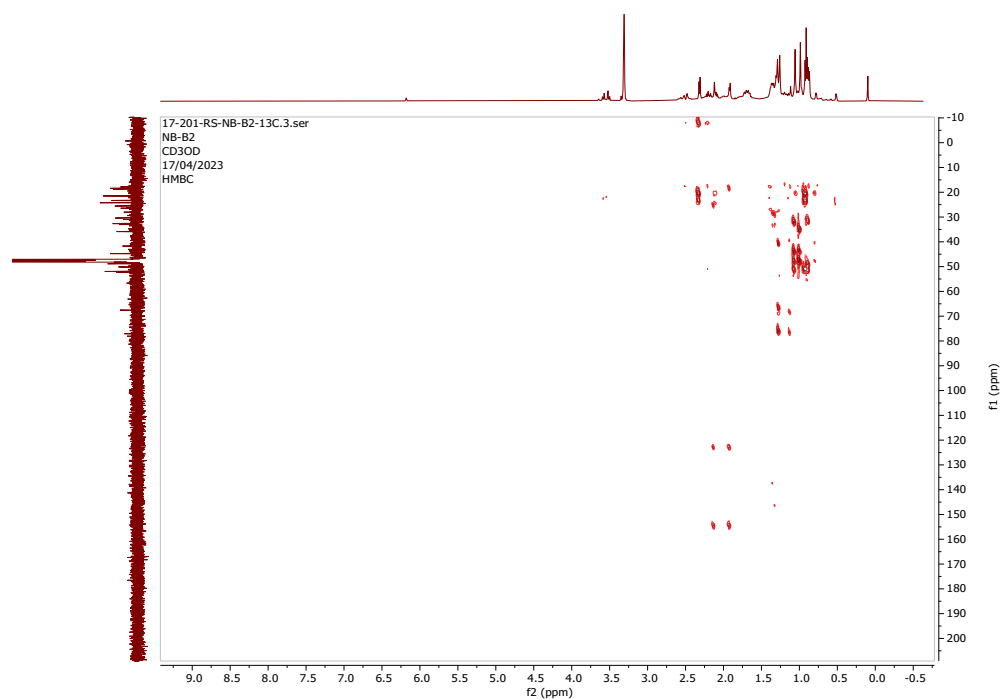


Figure S.42: ¹H-¹H COSY NMR spectrum of compound 4 in CD₃OD

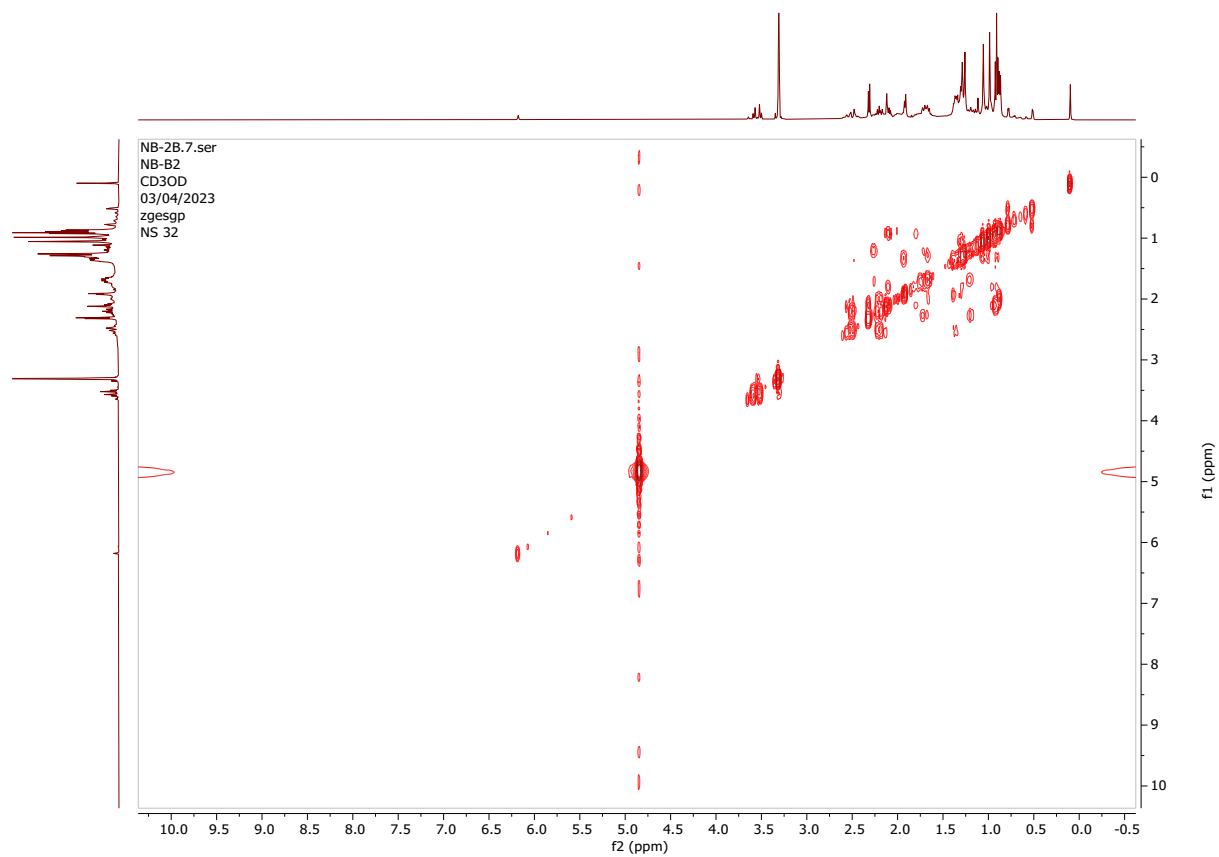


Figure S.43: NOESY NMR spectrum of compound 4 in CD₃OD

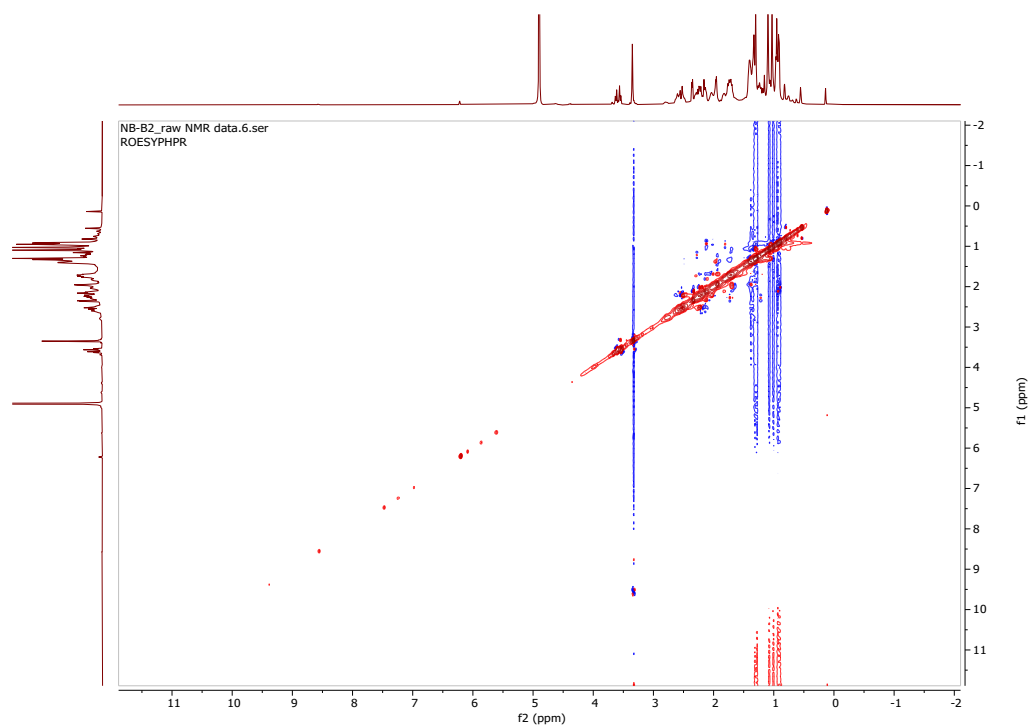


Figure S.44: HRESIMS spectrum of compound 4

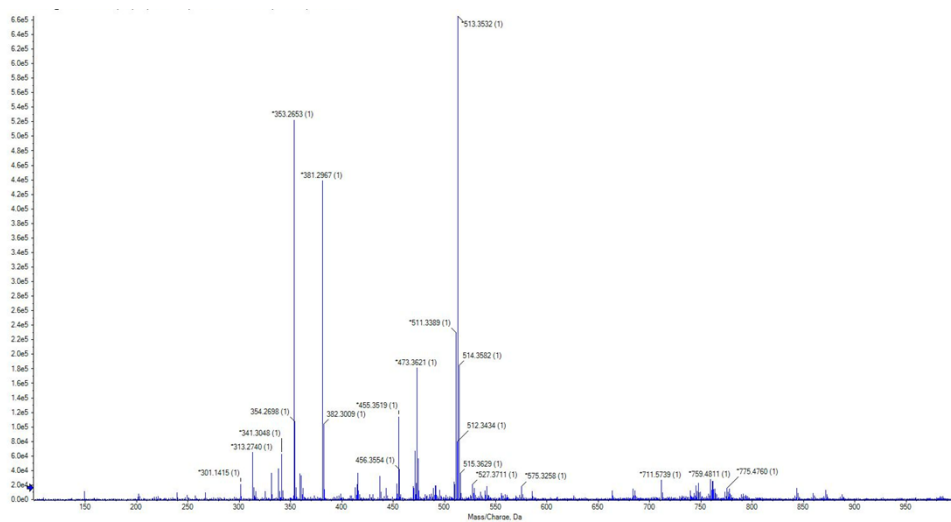


Figure S.45: IR spectrum of compound 4

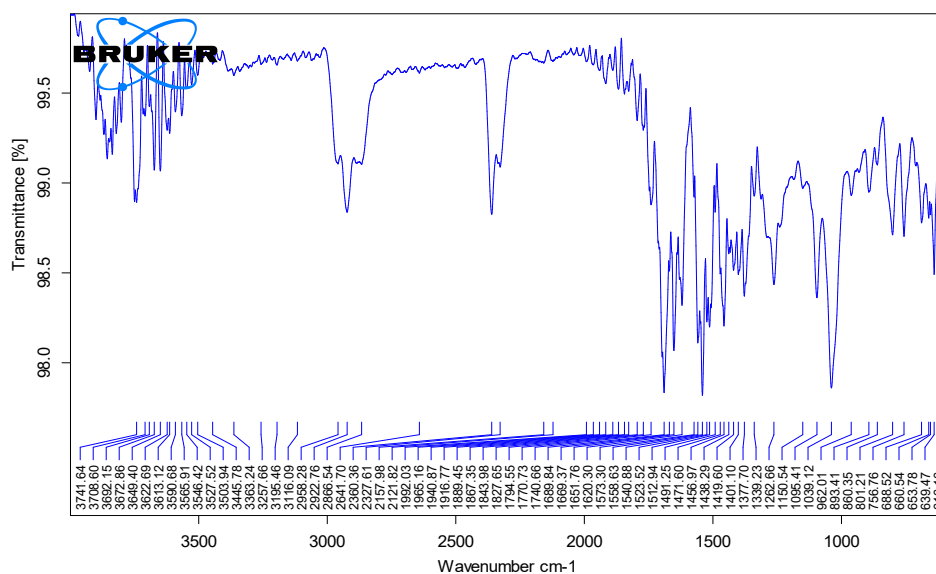


Figure S.46: UV spectrum of compound 4

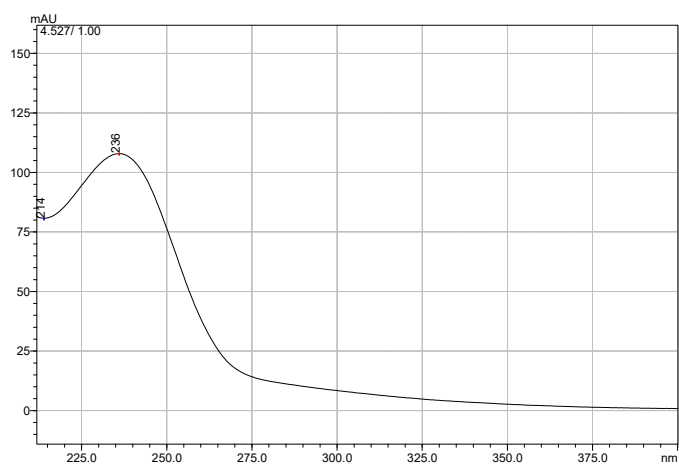


Figure S.47: Table representing MTT assay after 24 hours.

Compounds	T47D	MCF-7	MDAMB-231	HEK-293
1	20.07±1.16	24.20±1.68	66.13±1.23	-
2	18.42±1.09	22.39±1.21	40.20±2.29	-
3	36.31±1.31	42.22±1.97	-	-
4	76.40±2.09	88.43±2.14	-	-