Supporting Information

***Rec. Nat. Prod*. 16:6 (2022) 633-638**

A New Indole Glucoside and Other Constituents from the Sea Cucumber-Derived *Aspergillus fumigatus* M580 and Their Biological Activities

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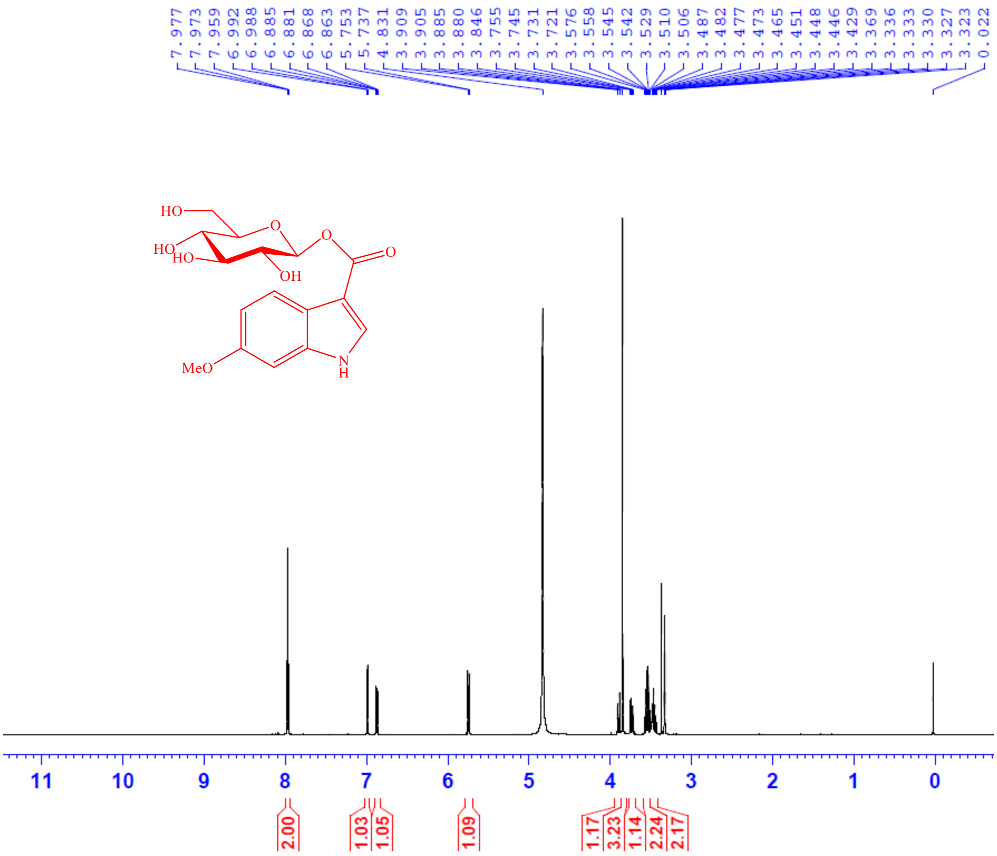


Figure S1: ^{1}H-NMR spectrum of compound **1**

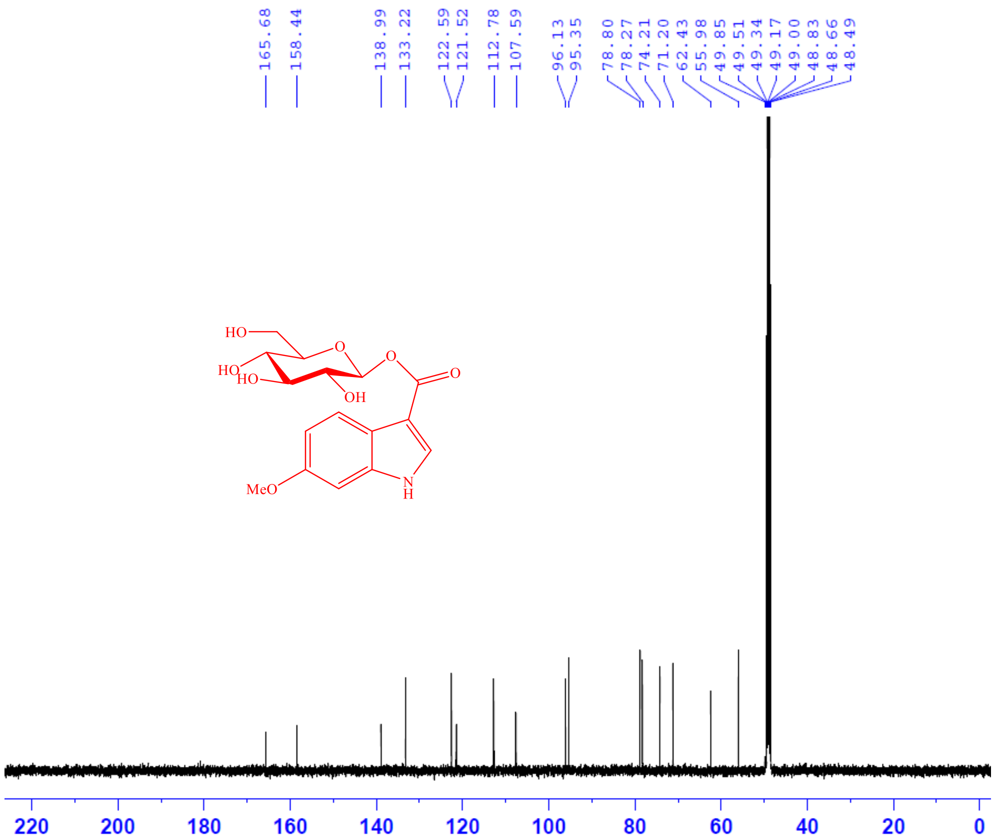


Figure S2: ^{13}C-NMR spectrum of compound **1**

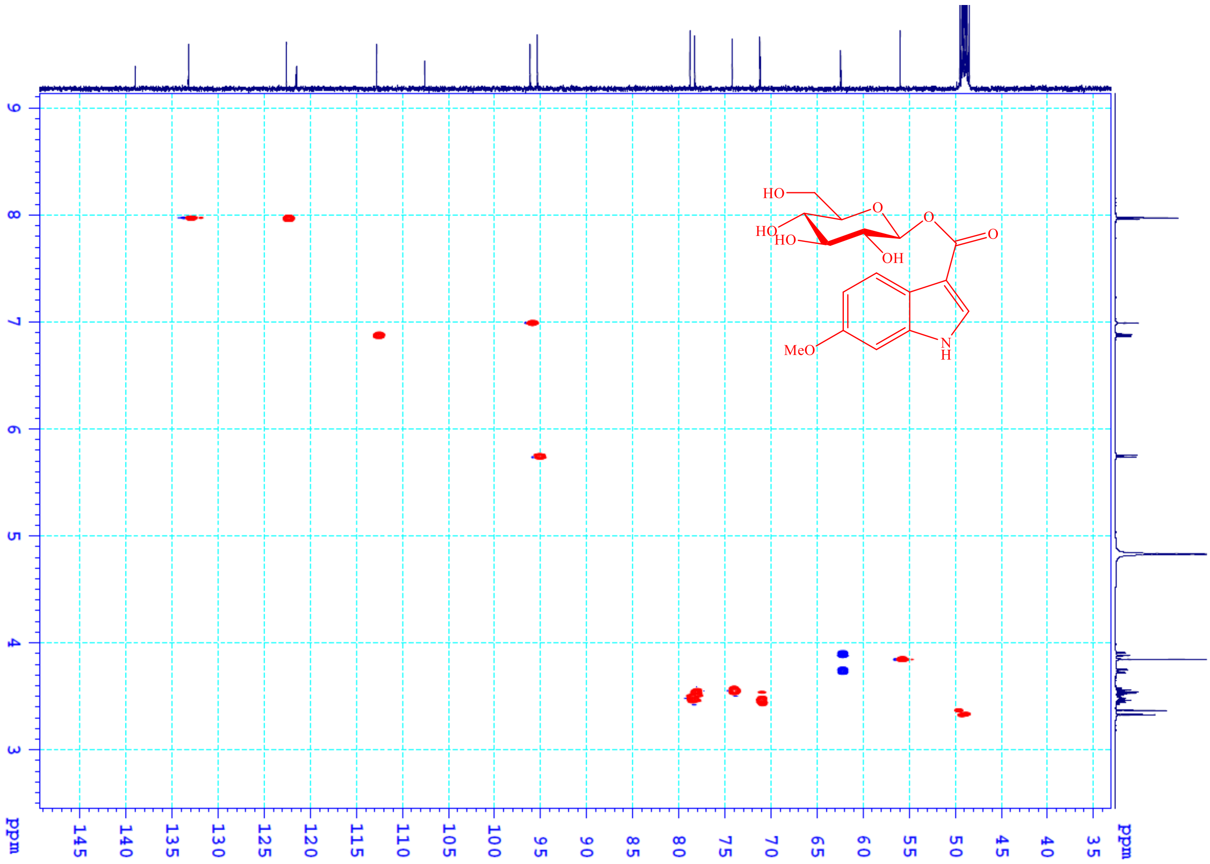


Figure S3: HSQC spectrum of compound **1**

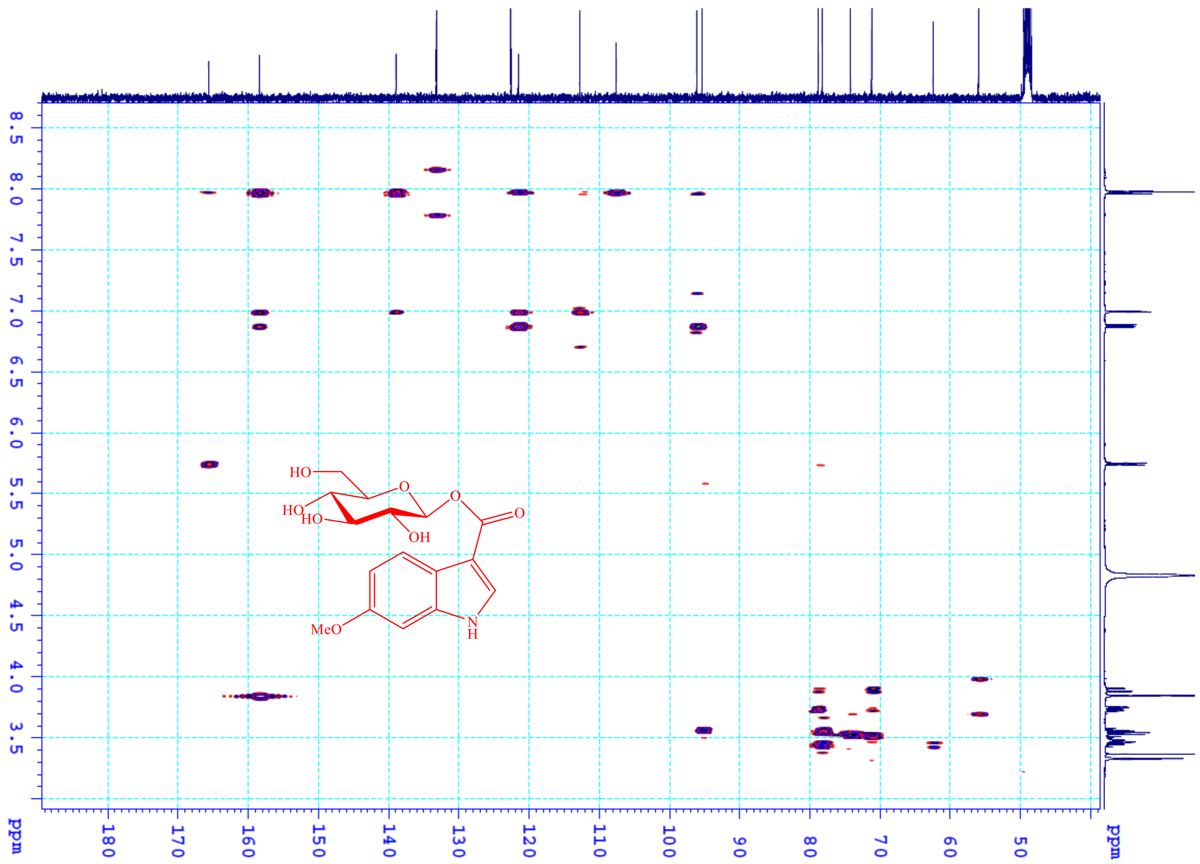


Figure S4: HMBC spectrum of compound **1**

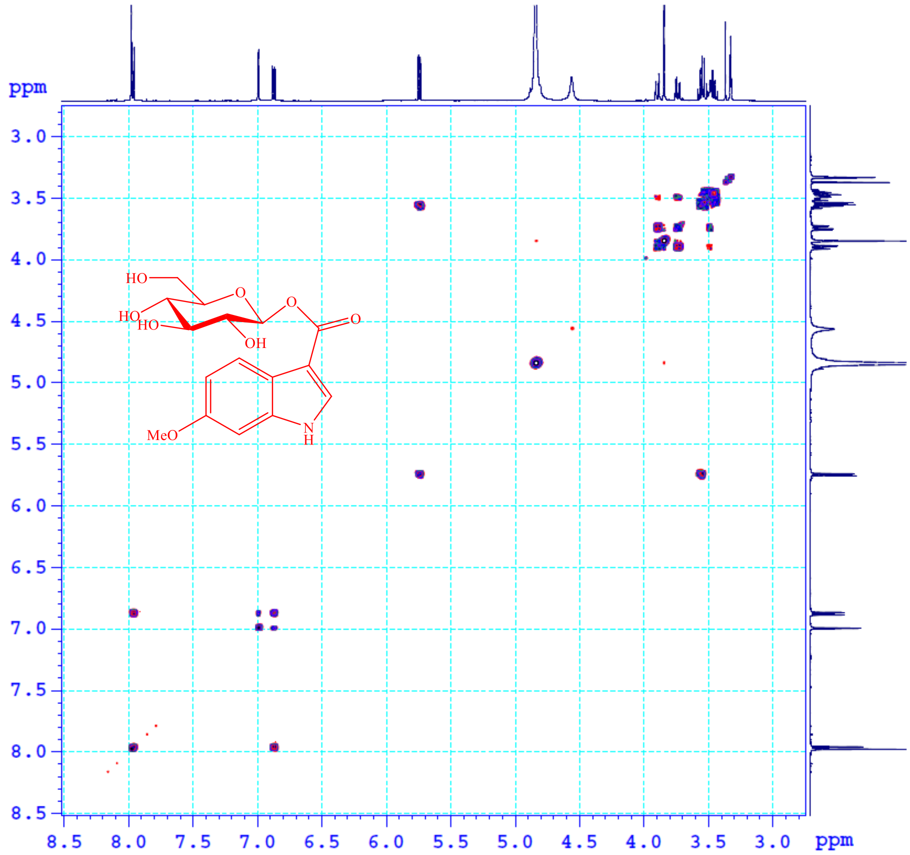


Figure S5: COSY spectrum of compound 1

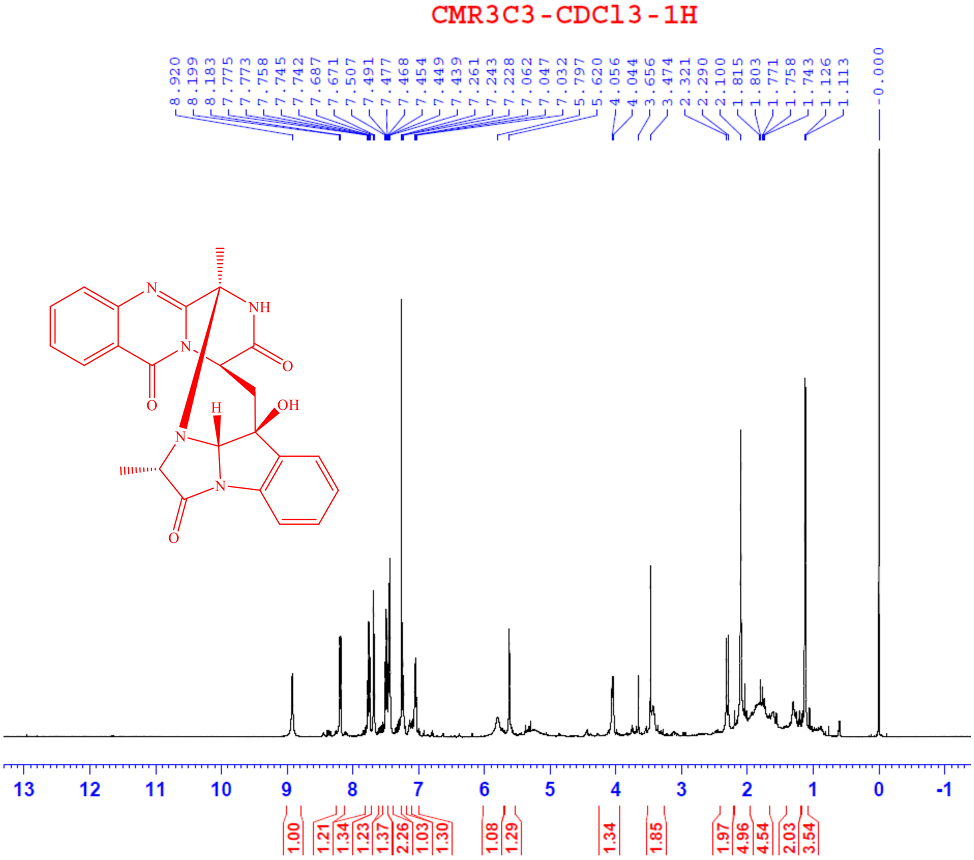


Figure S6: [1]H-NMR spectrum of compound 2

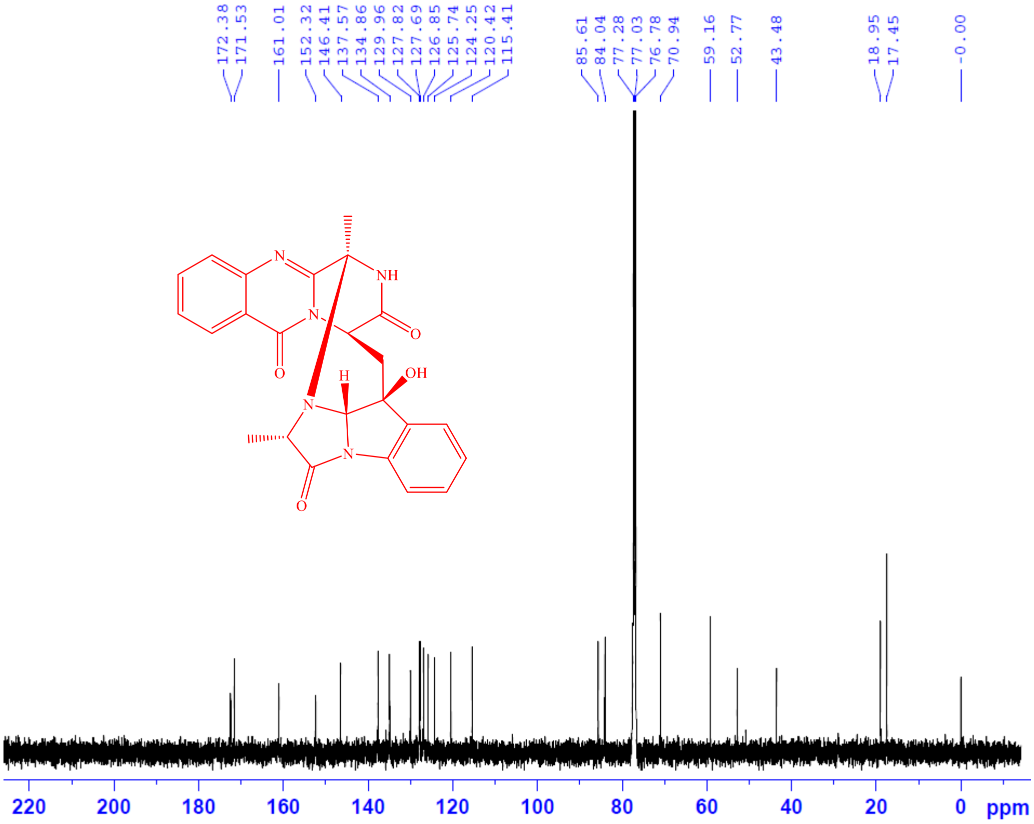


Figure S7: ^{13}C-NMR spectrum of compound **2**

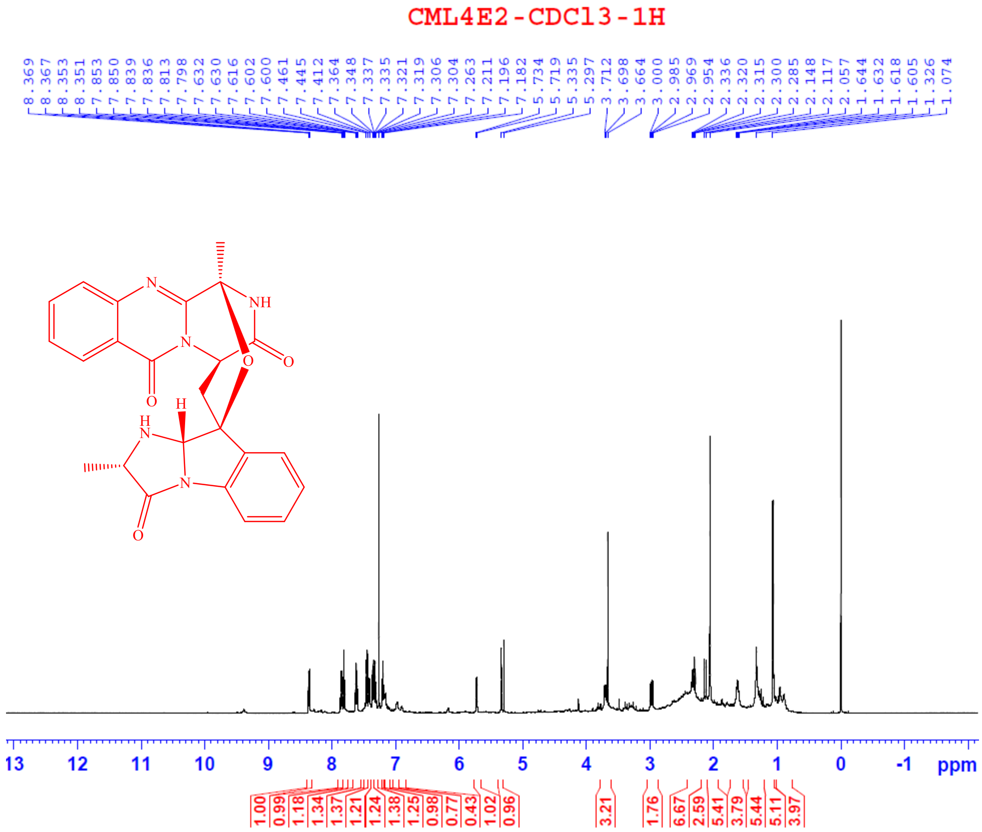


Figure S8: ^{1}H-NMR spectrum of compound **3**

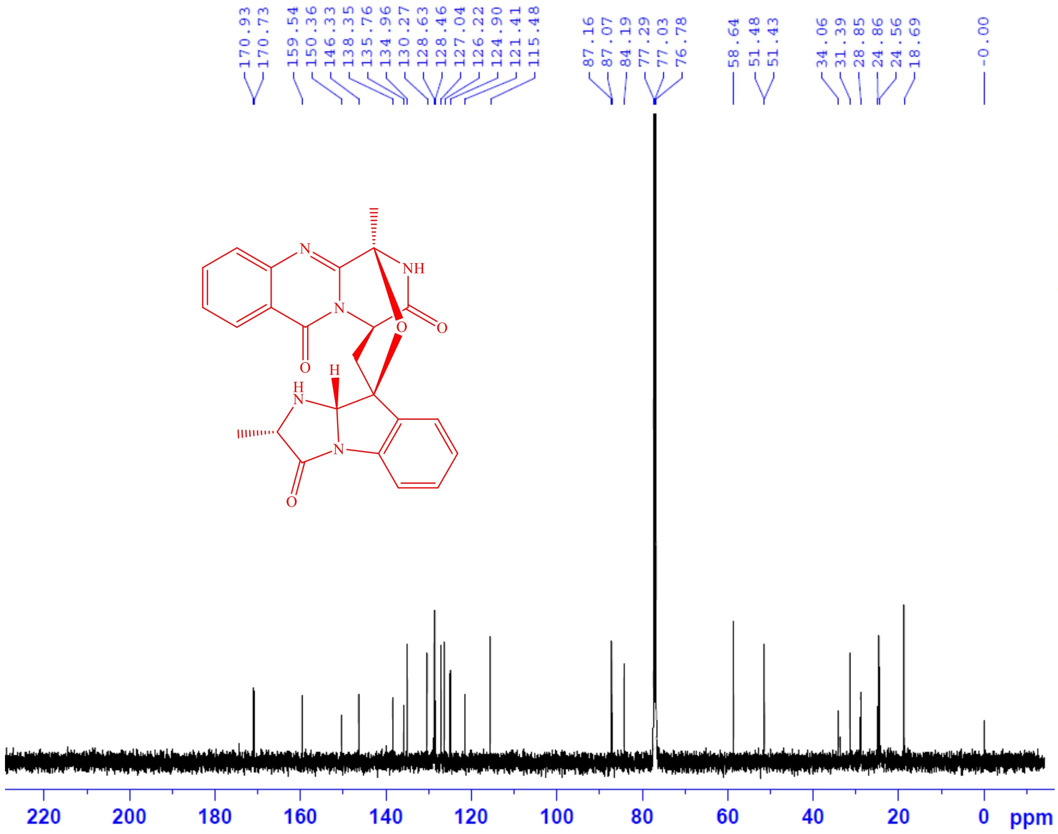


Figure S9: ^{13}C-NMR spectrum of compound **3**

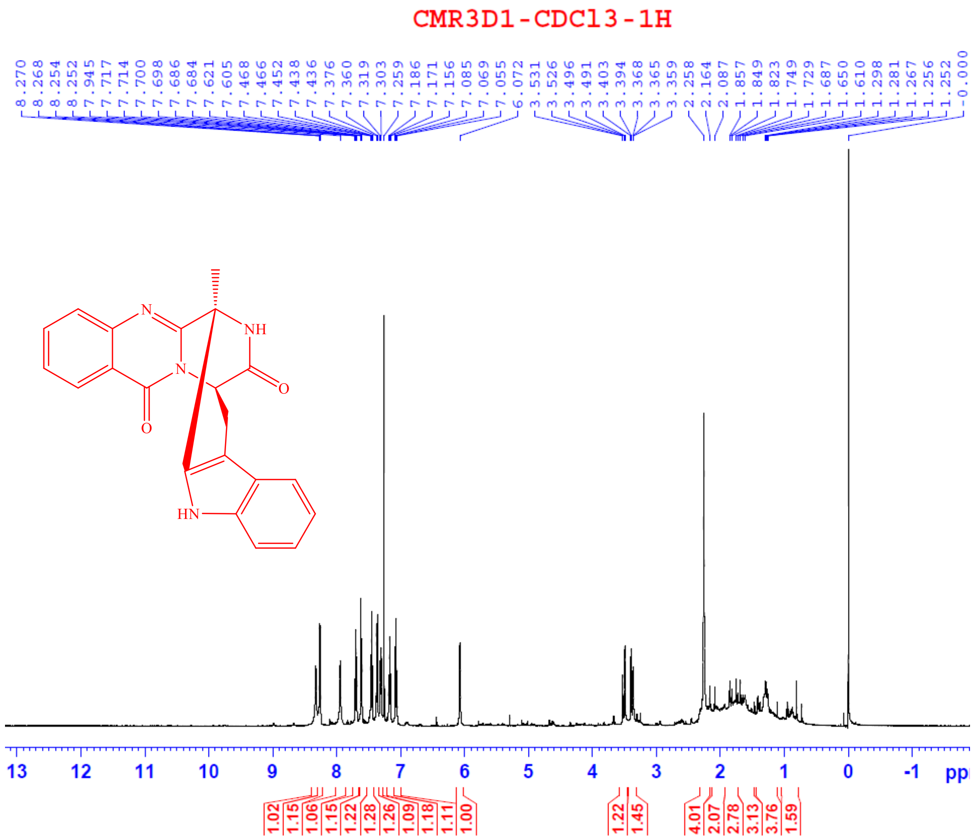


Figure S10: ^{1}H-NMR spectrum of compound **4**

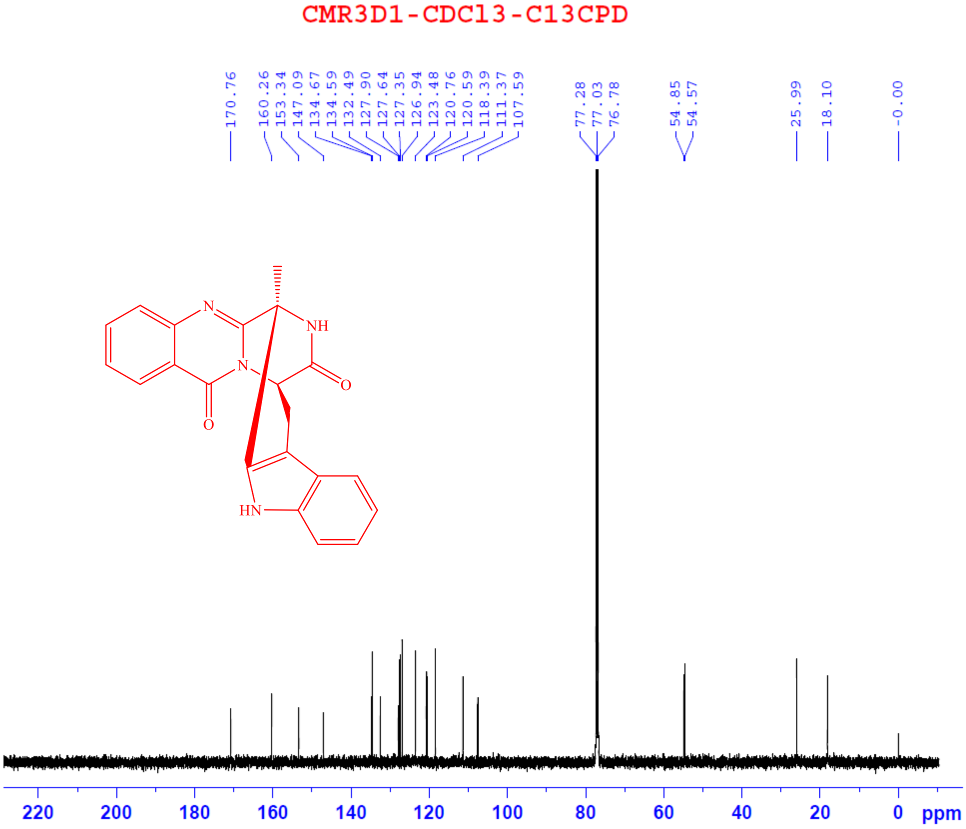


Figure S11: ^{13}C-NMR spectrum of compound **4**

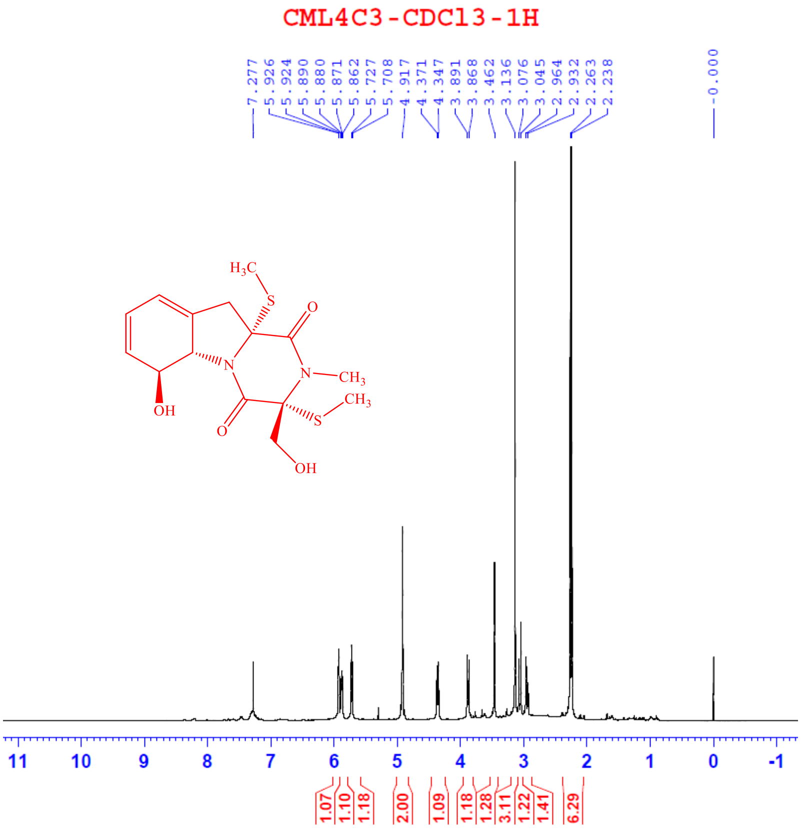


Figure S12: ^{1}H-NMR spectrum of compound **5**

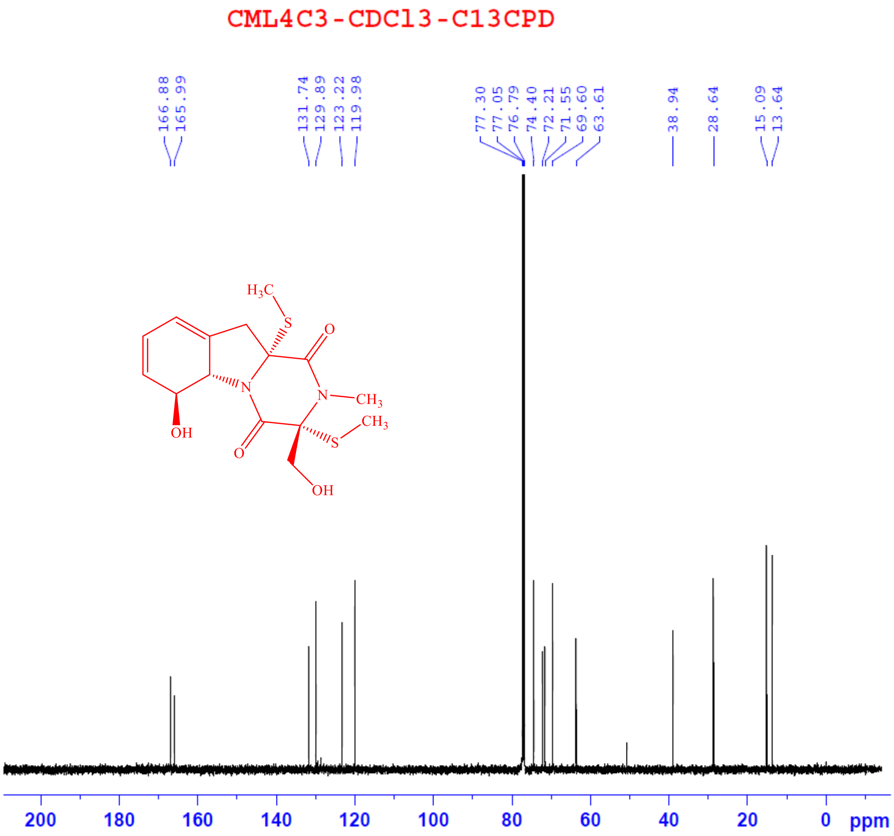


Figure S13: ^{13}C-NMR spectrum of compound **5**

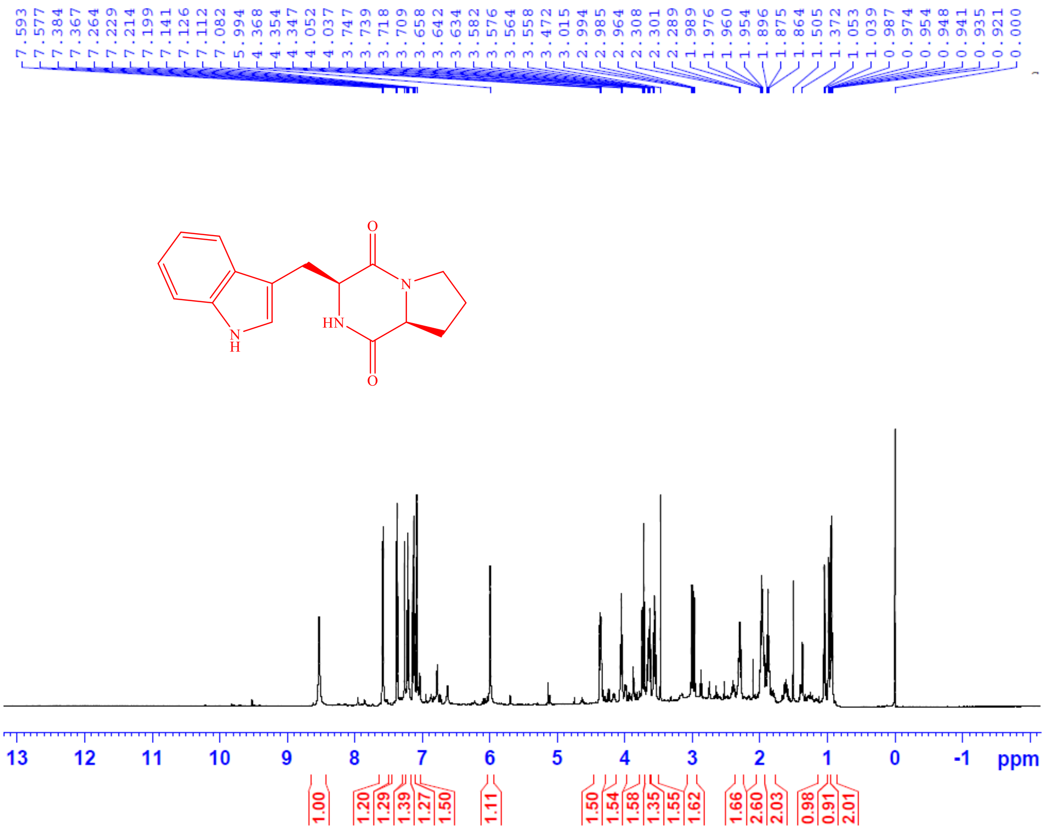


Figure S14: ^{1}H-NMR spectrum of compound **6**

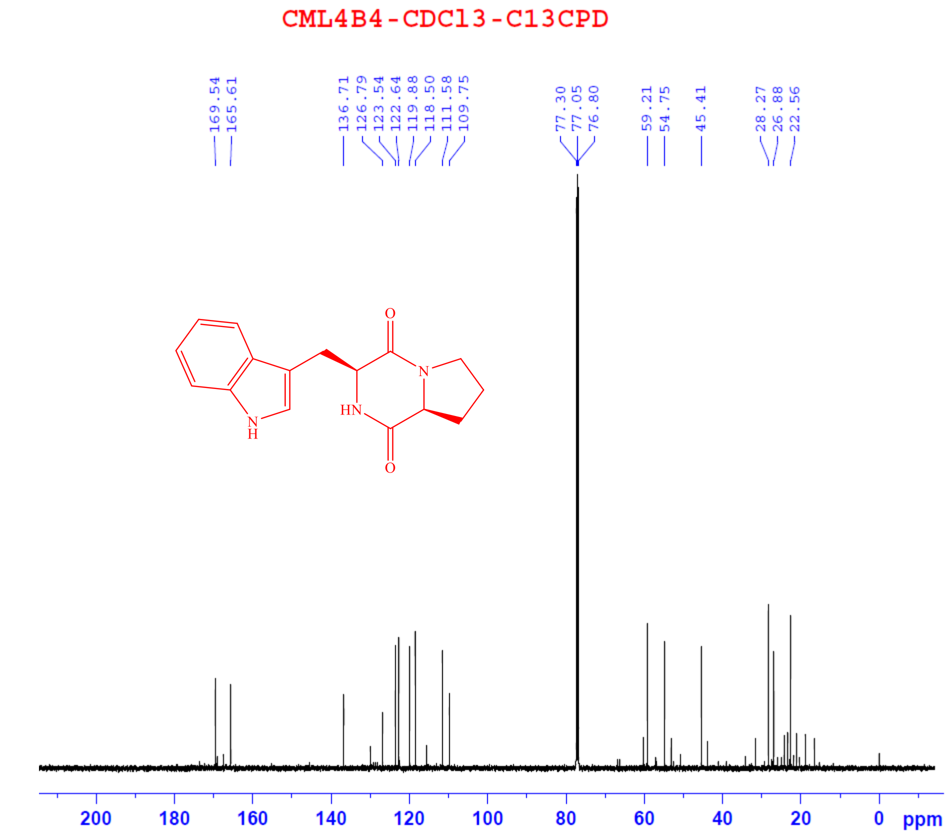


Figure S15: ^{13}C-NMR spectrum of compound **6**

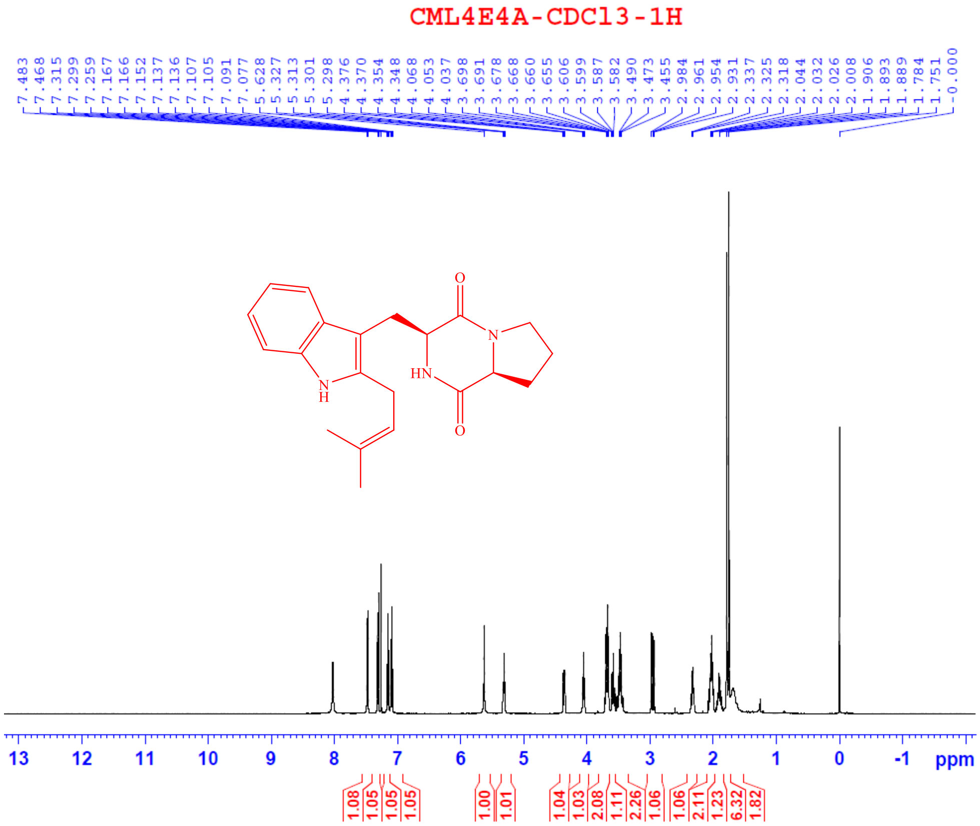


Figure S16: ^{1}H-NMR spectrum of compound **7**

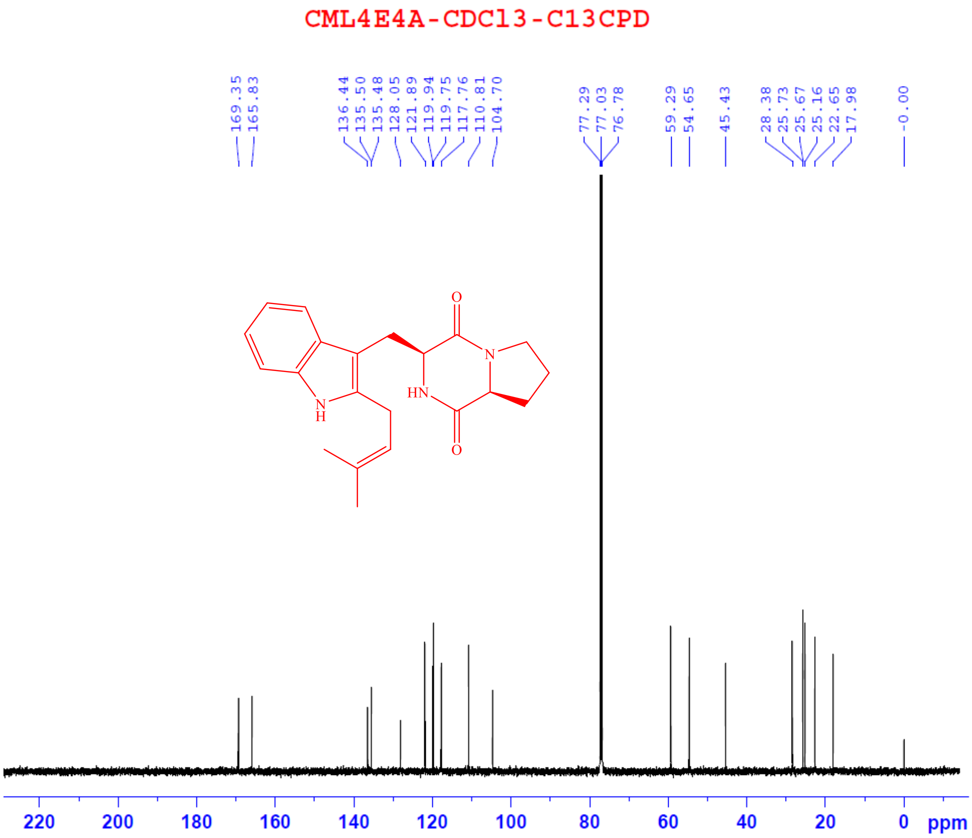


Figure S17: ^{13}C-NMR spectrum of compound **7**

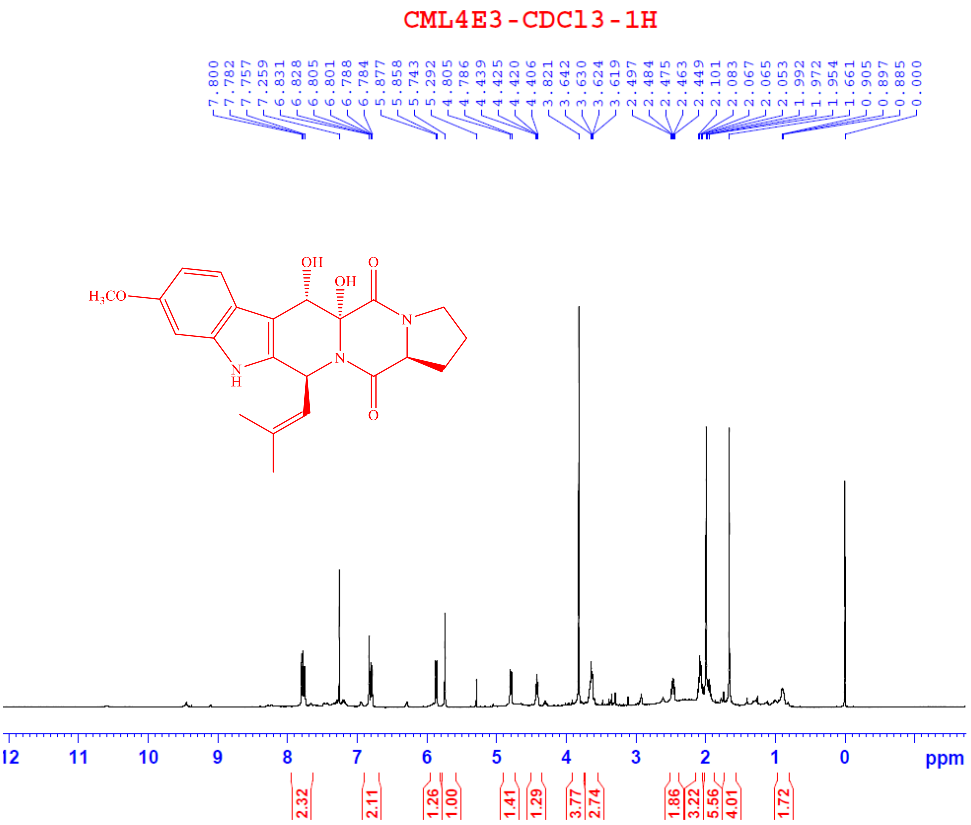


Figure S18: ^{1}H-NMR spectrum of compound **8**

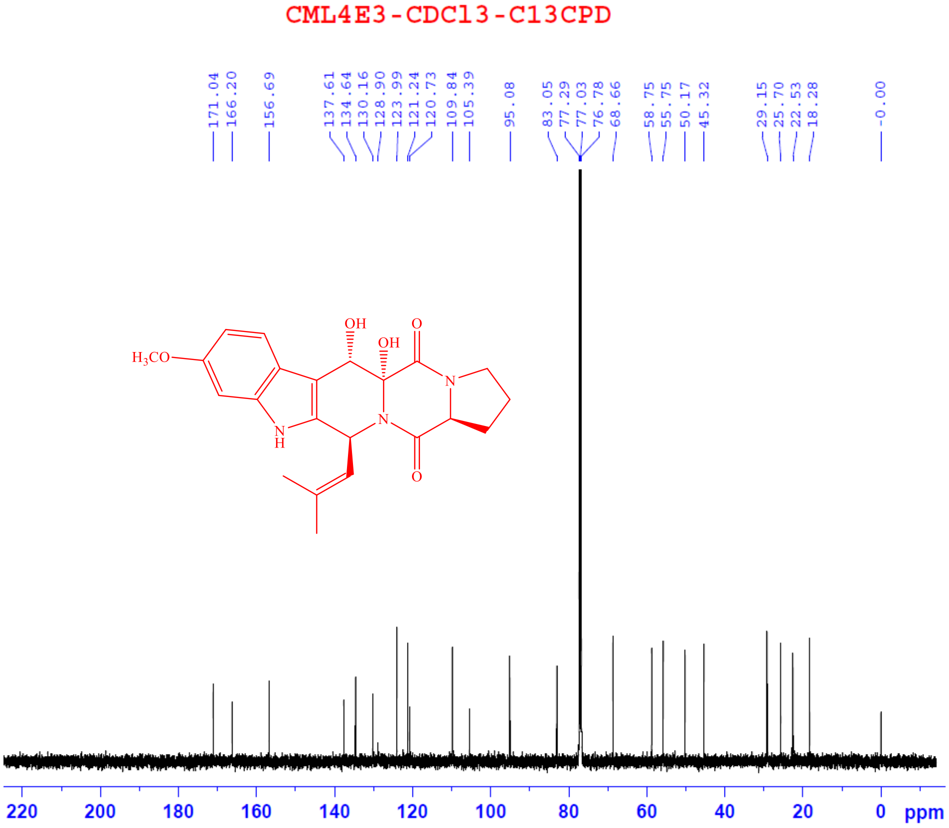


Figure S19: ^{13}C-NMR spectrum of compound **8**

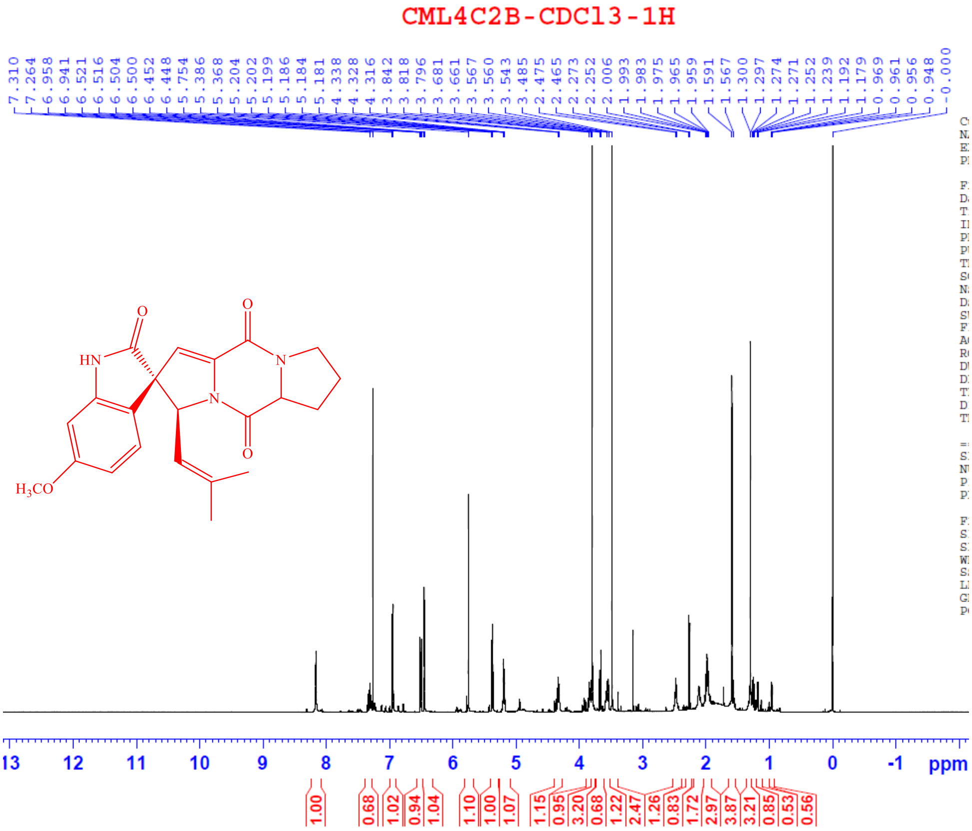


Figure S20: ^{1}H-NMR spectrum of compound **9**

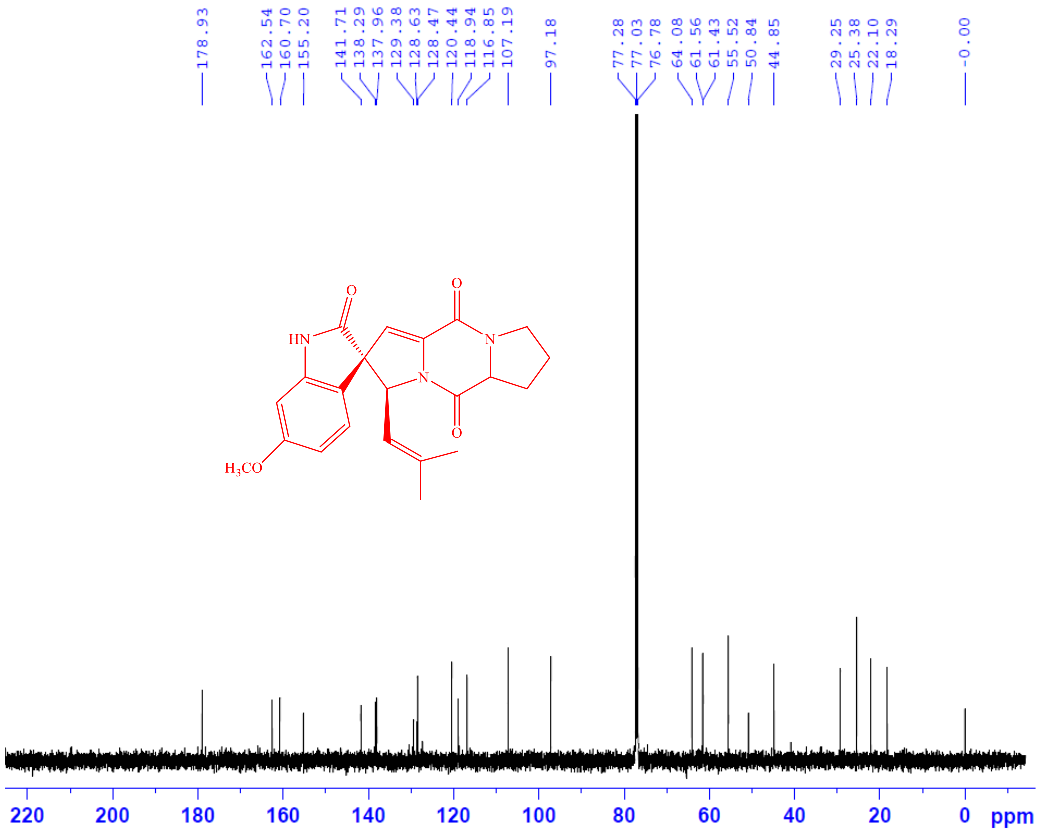


Figure S21: ^{13}C-NMR spectrum of compound **9**

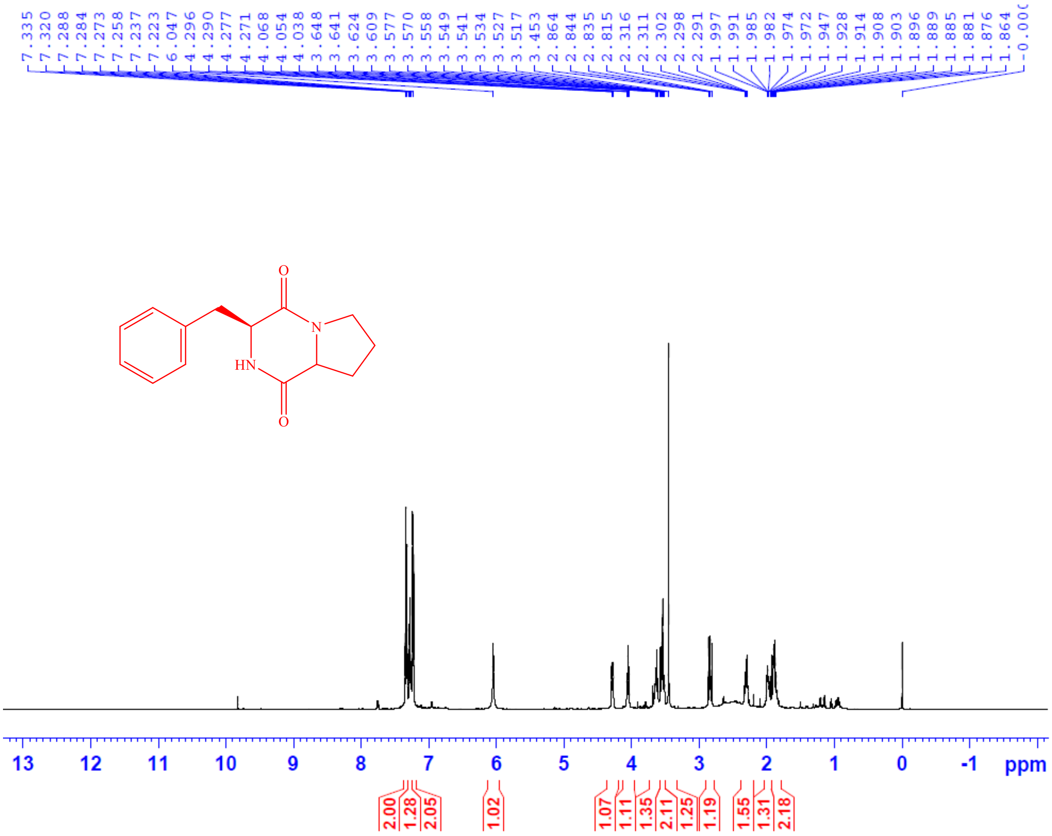


Figure S22: ^{1}H-NMR spectrum of compound **10**

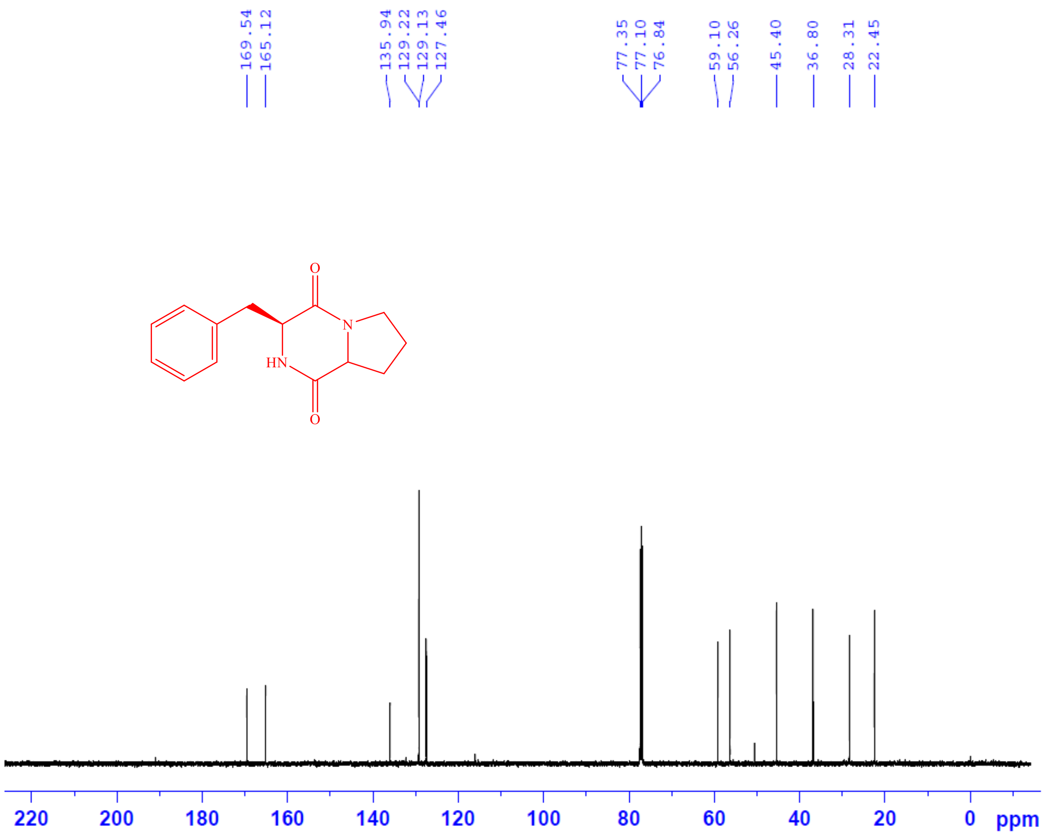


Figure S23: ^{13}C-NMR spectrum of compound **10**

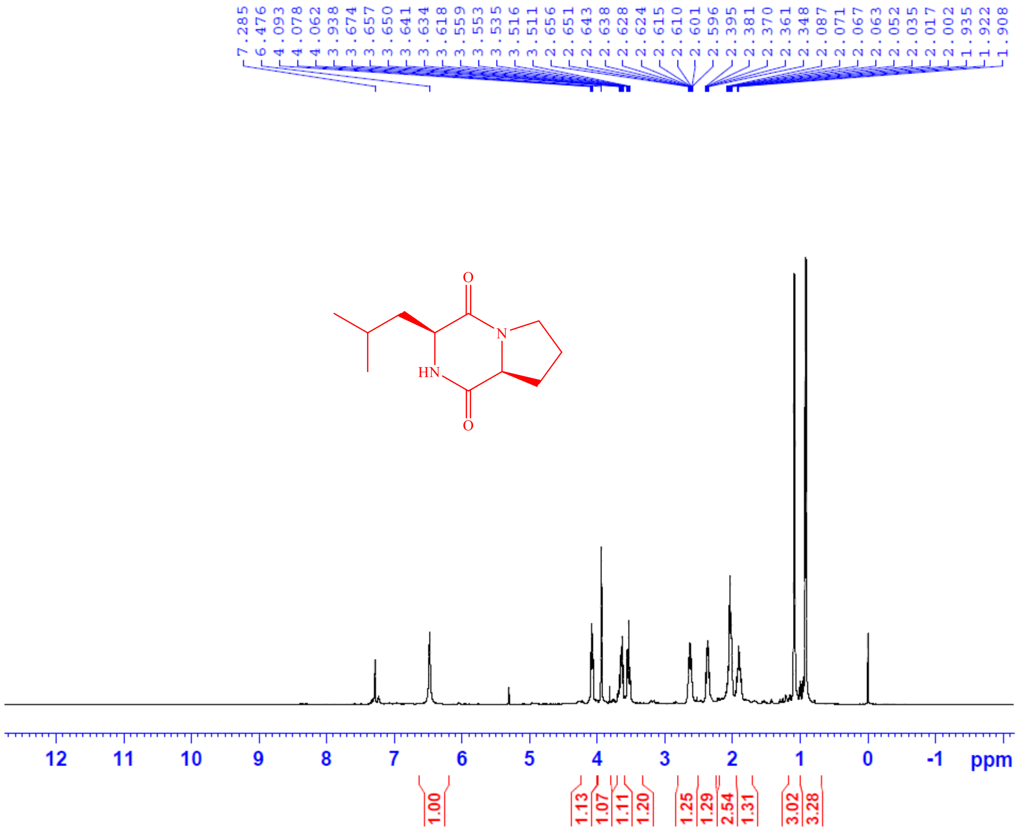


Figure S24: ^{1}H-NMR spectrum of compound **11**

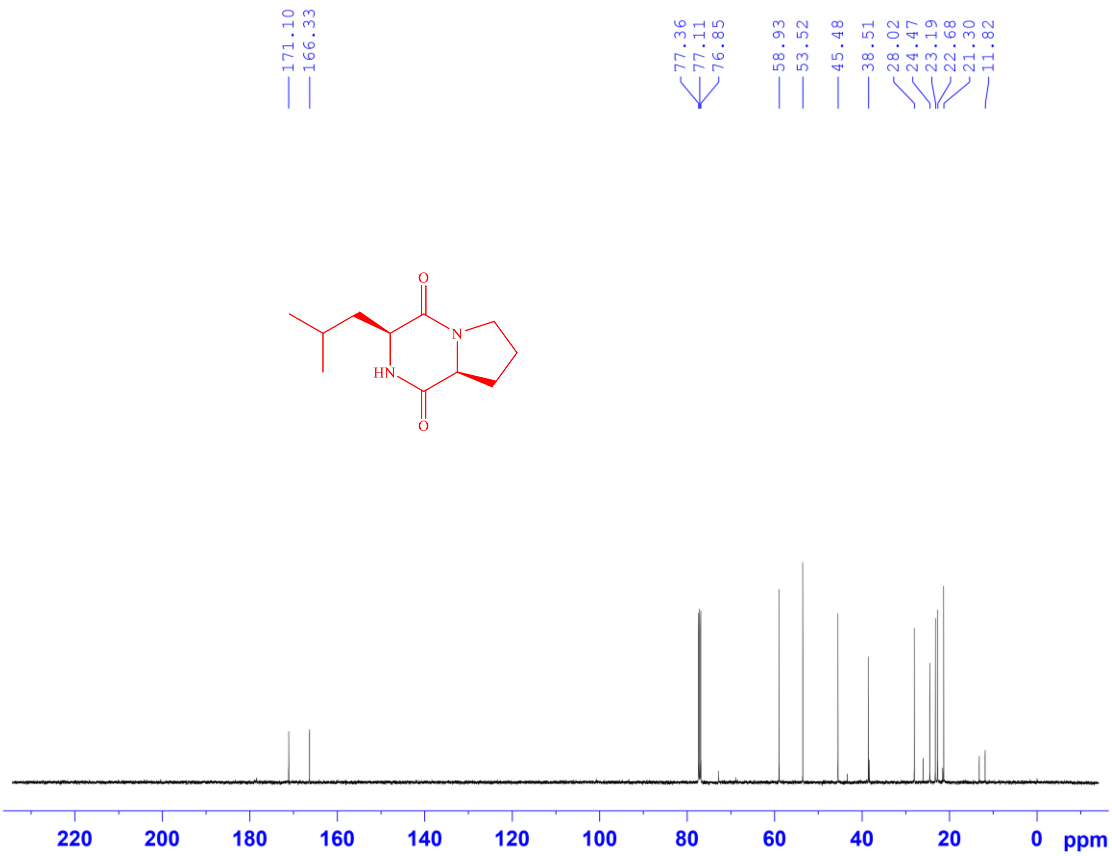


Figure S25: ^{13}C-NMR spectrum of compound **11**

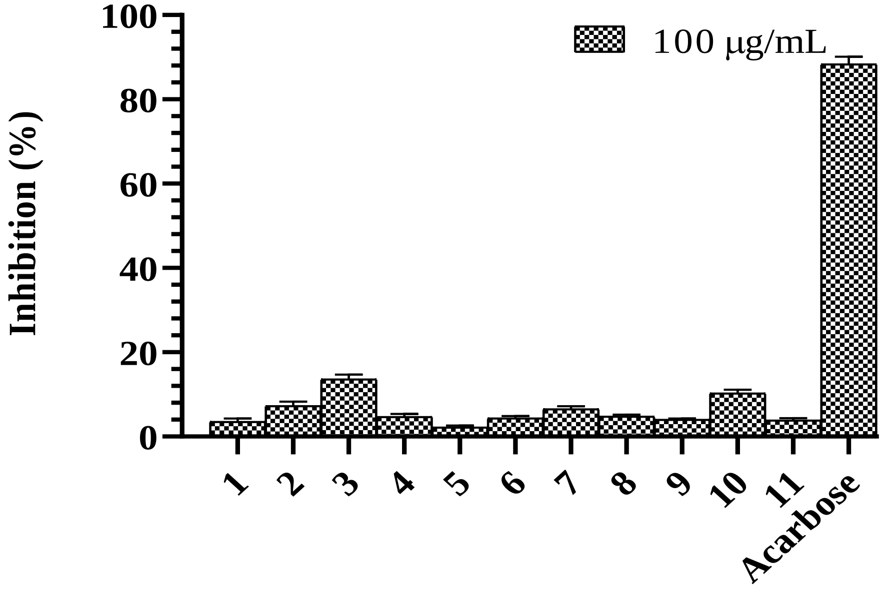


Figure S26: *α*-Glucosidase inhibitory activity of compounds **1**–**10**.

Colochirus quadrangularis Troschel, 1846 identification: Method and Data

Figure S27: Photos of collected sea cucumber

The identification of collected sea cucumber was done base on the analysis of its spicule morphology and skeleton structure followed published methods (Conand 1990; Hooper and Van Soest 2002). Briefly, thin slides at various body parts of the sea cucumber were prepared. The slides were then soak with sodium hypochlorite for 10-15 minutes to remove all contained tissues. The spicule and skeleton structure was observed under microscope (Figure S28-S30).

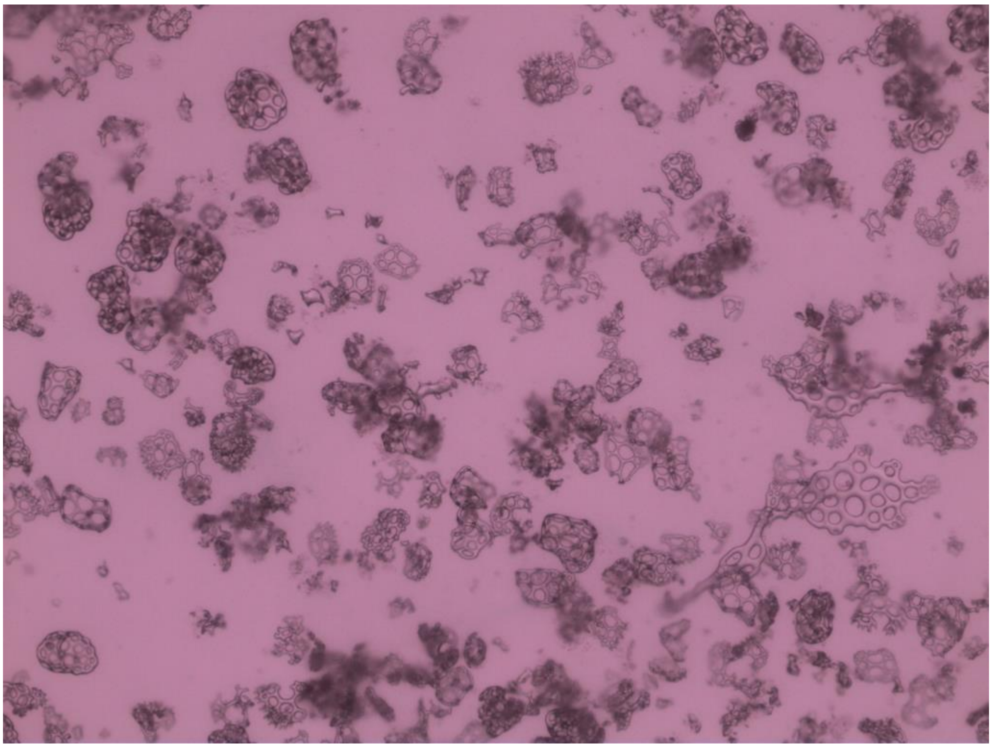


Figure S28: Figure: Popular bond types of the collected sea cucumber (10X)

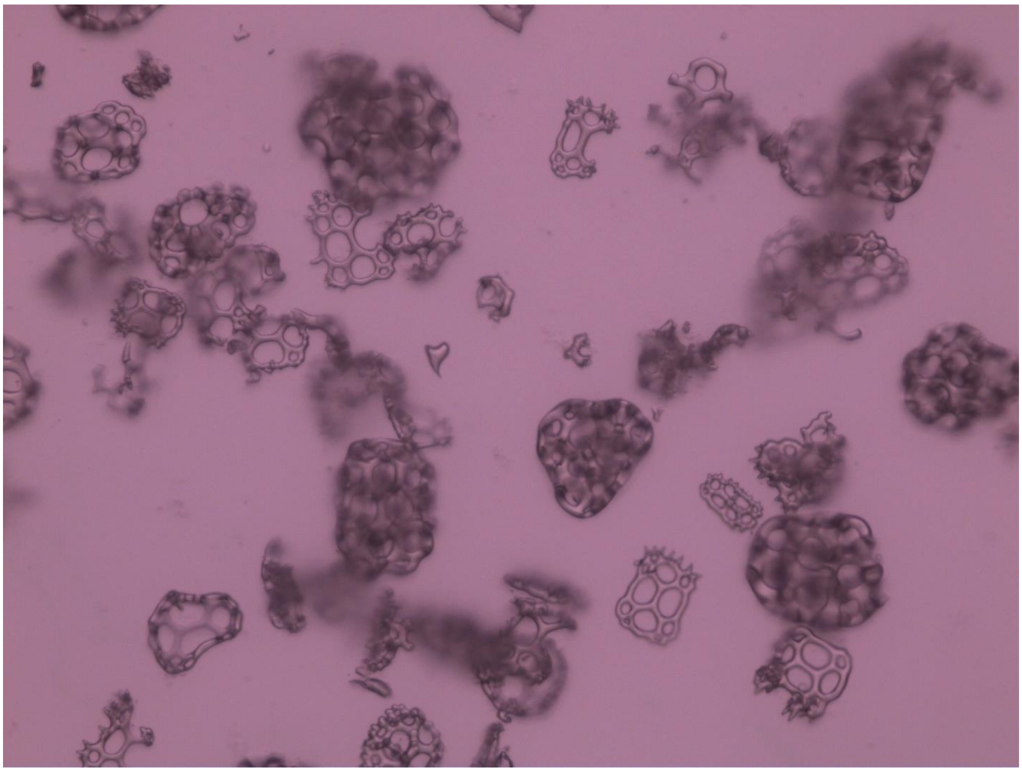


Figure S29: Bowls and ellipsoids bonds of collected sea cucumber (20X)

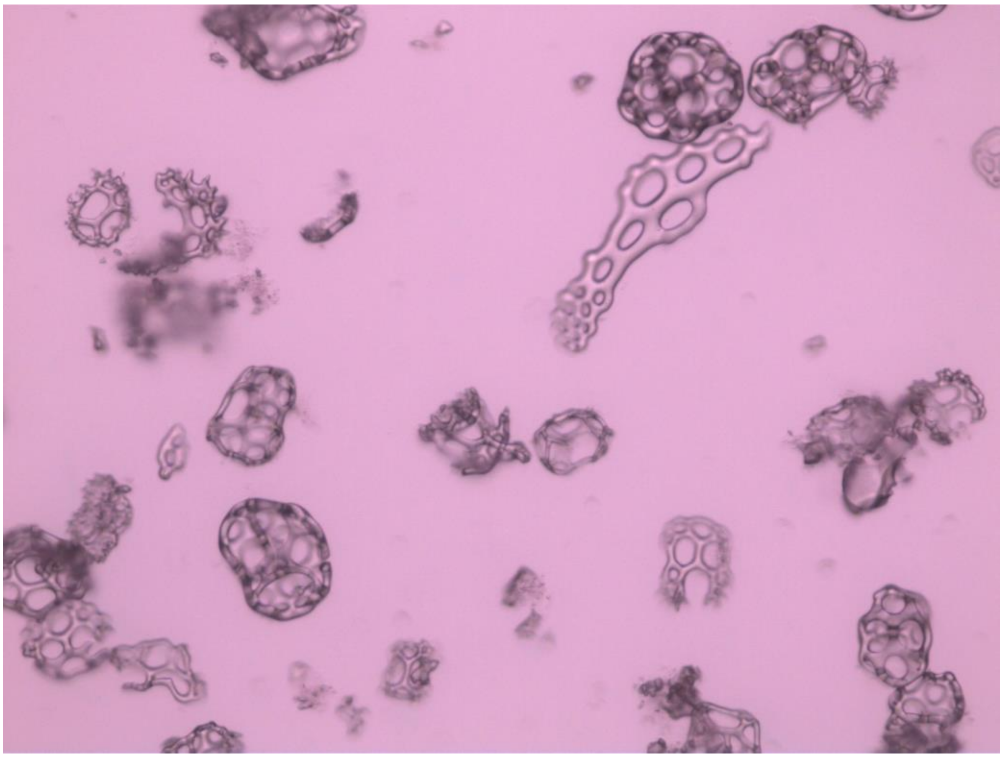


Figure S30: Figure: Ellipsoids, Rosettes and Rods bonds of collected sea cucumber (20X)

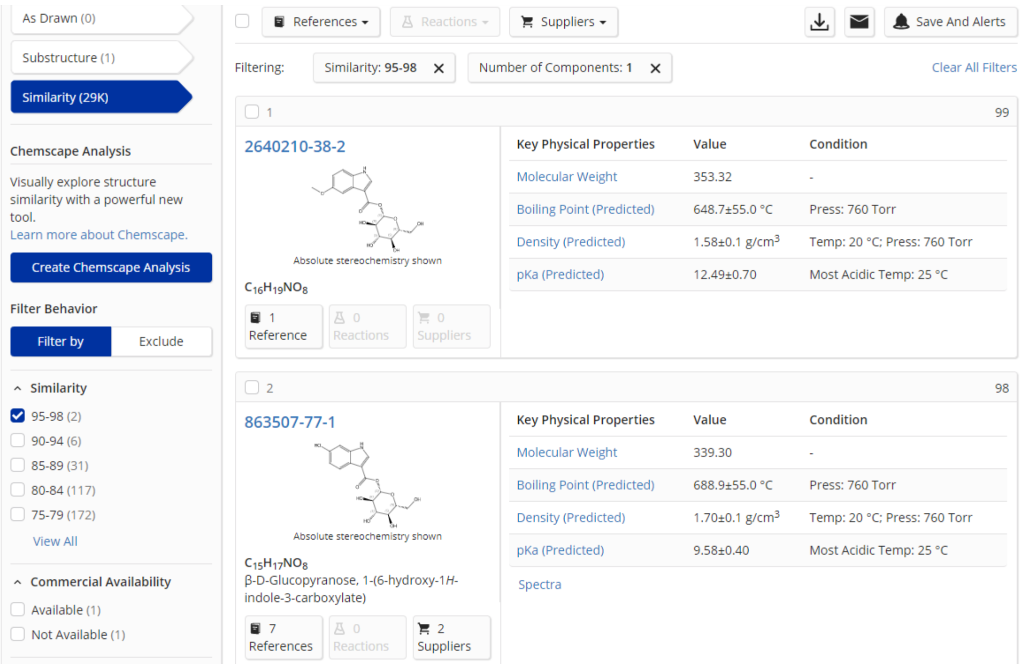


Figure S31: The results of SciFinder searching for new compound

Table S1: ^{1}H and ^{13}C-NMR data of compound **2** and Fumiquinazoline D

Position	**2**		**Fumiquinazoline D** [1]
	δ_H^{a,b} mult. (J in Hz)	**δ_C ^{a,c}**	**$\delta_C$$^{\#}$**
1	-	172.4	172.4
3	-	70.9	70.9
4	-	152.3	152.3
6	-	146.4	146.4
7	7.68 d (8.5)	127.8	127.8
8	7.76 t (8.5)	134.9	134.9
9	7.49 t (8.5)	127.7	127.7
10	8.19 d (8.5)	126.9	126.9
11	-	120.4	120.5
12	-	161.0	161.0
14	5.80 m	52.8	52.8
15	2.30 d (15.5)/3.45 d (15.5)	43.5	43.5
16	2.10 s	19.0	19.0
17	-	84.0	84.1
18	5.62 s	85.6	85.7
20	4.05 q (6.5)	59.2	59.2
21	-	171.5	171.5
23	-	137.6	137.6
24	7.45 d (8.0)	115.4	115.5
25	7.24 t (8.0)	130.0	130.1
26	7.05 t (8.0)	125.8	125.8
27	7.45 d (8.0)	124.3	124.3
28	-	137.6	137.6
29	1.12 d (6.5)	17.5	17.5

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$ $CDCl_3$

Table S2: ^{1}H and ^{13}C-NMR data of compound **3** and Fumiquinazoline C

Position	**3**		**Fumiquinazoline C** [1]
	δ_H^{a,b} mult. (J in Hz)	**δ_C ^{a,c}**	**$\delta_C$$^{\#}$**
1	-	170.9	170.8
3	-	84.2	84.2
4	-	150.4	150.4
6	-	146.3	146.3
7	7.80 d (8.0)	128.5	128.4
8	7.85 t (8.0)	135.0	134.9
9	7.62 t (8.0)	128.6	128.6
10	8.36 d (8.0)	127.0	127.0
11	-	121.4	121.4
12	-	159.5	159.5
14	5.72 d (7.5)	51.4	51.5
15	2.13 d (15.5)/2.97 dd (15.5, 7.5)	31.4	31.5
16	2.06 s	24.6	24.6
17	-	87.2	87.2
18	5.34 s	87.1	87.0
20	3.70 d (7.0)	58.6	58.6
21	-	170.7	170.0
23	-	135.8	135.8
24	7.45 d (8.0)	115.5	115.4
25	7.33 t (8.0)	130.3	130.2
26	7.20 t (8.0)	126.2	126.2
27	7.35 d (8.0)	124.9	124.8
28	-	138.4	138.3
29	1.07 d (7.0)	18.7	18.7

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$ $CDCl_3$

Table S3: ^{1}H and ^{13}C-NMR data of compound **4** and Fumiquinazoline J

Position	**4**		**Fumiquinazoline J** [2]
	δ_H^{a,b} mult. (J in Hz)	**δ_C ^{a,c}**	**$\delta_C$$^{\#}$**
1		170.8	170.8
3		54.9	54.8
4		153.3	153.3
6		147.1	147.0
7	7.61 d (8.0)	127.6	127.6
8	7.70 d (8.0)	134.6	134.4
9	7.45 t (8.0)	172.4	172.4
10	8.26 d (8.0)	126.9	126.9
11		120.8	120.7
12		160.3	160.3
14	6.07 br s	54.6	54.5
15	3.37 dd (17.5, 4.5)	26.0	25.9
	3.52 dd (17.5, 2.5)		
16	2.26 s	18.1	18.0
17		107.6	107.5
18		132.5	132.4
20		134.7	134.6
21	7.31 d (8.0)	111.4	111.3
22	7.17 t (8.0)	123.5	123.5
23	7.07 t (8.0)	120.6	120.6
24	7.37 d (8.0)	118.4	118.4
25		127.9	127.8

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$recoded in

Table S4: ^{1}H and ^{13}C-NMR data of compound **5** and bisdethiobis(methylthio)gliotoxin

Position	**5**		**bisdethiobis(methylthio)gliotoxin** [1]
	δ_H^{a,b} mult. (J in Hz)	**δ_C ^{a,c}**	**$\delta_C$$^{\#}$**
1		166.0	165.9
3		72.2	71.9
4		166.9	166.8
5a	4.92 (m)	69.6	69.6
6	4.92 m	74.4	74.4
7	5.88 d (9.5)	123.2	123.1
8	5.72 dd (9.5, 1.0)	129.9	130.1
9	5.92 d (1.0)	120.0	120.0
9a		131.7	131.6
10	2.95 d (16.0)	38.9	38.9
	3.06 d (16.0)		
11		71.6	71.5
12	2.24 s	15.1	14.6
13	3.14 s	28.6	28.7
14	2.26 s	13.6	13.7
15	3.88 d (12.0)	63.6	63.6
	4.36 d (12.0)		

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$ $CDCl_3$

Table S5: ^{1}H and ^{13}C-NMR data of compound **6** and cyclo(L-prolyl-L-tryptophane)

Position	6		cyclo(L-prolyl-L-tryptophane) [3]
	δ_H^{a,b} mult. (J in Hz)	δ_C ^{a,c}	$\delta_C$$^{\#}$
1	8.60 s	-	-
2	-	165.6	168.23
3	4.35 dd (4.0, 10.5)	54.8	59.18
5	-	169.5	171.89
6	4.05 t (7.5)	59.2	59.51
7	1.98 m 2.30 m	28.3	29.74
8	1.88 m 1.94 m	22.6	22.26
9	3.55 m 3.63 m	45.4	46.04
10	2.99 dd (10.5, 15.0) 3.72 dd (4.0, 15.0)	28.3	31.19
11	-	109.8	109.31
12	-	126.8	128.66
13	7.58 d (8.0)	118.5	119.55
14	7.13 t (8.0)	119.9	120.04
15	7.21 t (8.0)	122.6	122.73
16	7.38 d (8.0)	111.6	112.37
17	-	136.7	138.01
18	5.99 s	-	-
19	7.08 s	123.5	126.21

a) recorded in $CDCl_3$, **b)** 500 MHz, c)125 MHz, **#)**CD_3OD

Table S6: ^{1}H and ^{13}C-NMR data of compound **7** and tryprostatin B

Position	7		Tryprostatin B [4]
	δ_H^{a,b} mult. (*J* in Hz)	δ_C ^{a,c}	$\delta_C$$^{\#}$
1(NH)	8.02 br s		-
2	-	136.4	136.4
3	-	104.7	104.6
3a	-	128.1	128.0
4	7.47 d (7.5)	117.8	117.7
5	7.09 t (7.5)	119.9	119.9
6	7.16 t (7.5)	121.9	121.9
7	7.30 d (7.5)	110.8	110.8
7a	-	135.5	135.4
8	2.99 dd (10.5, 15.0)/3.72 dd (4.0, 15.0)	25.7	25.6
9	4.36 dd (1.5, 11.0)	54.7	54.6
10 (NH)	5.62 s	-	-
11	-	169.4	169.4
12	4.05 t (7.5)	59.3	59.3
13	2.02 m 2.33 m	28.4	28.3
14	1.91 m 2.03 m	22.7	22.6
15	3.60 m 3.68m	45.4	45.4
17	-	165.8	165.8
18	3.49 m	25.2	25.1
19	5.30 t (8.0)	119.8	119.7
20	-	135.5	135.5
21	1.78 s	25.7	25.7
22	1.75 s	18.0	18.0

a) recorded in $CDCl_3$, **b)** 500 MHz, c)125 MHz, **#)** $CDCl_3$

Table S7: ^{1}H and 13C-NMR data of compound **8** and 12,13-dihydroxy-fumitremorgin C

Position	8		12,13-Dihydroxy-fumitremorgin C [5]
	δ_H ^{a,b} mult. (J in Hz)	δ_C ^{a,c}	δ_C $^{\#}$
2	-	130.2	130.3
3	5.87 d (9.5)	58.8	58.8
5	-	166.2	166.3
6	4.42 dd (2.0, 9.5)	50.2	50.2
7	2.09 m/2.48 m	29.2	29.2
8	1.95 m/2.10 m	22.5	22.6
9	3.64 m	45.3	45.3
11	-	171.0	171.1
12	-	83.1	83.1
13	5.74 m	68.7	68.8
14	-	105.4	105.6
15	-	120.7	120.9
16	6.79 d (9.5)	121.2	121.4
17	7.79 d (9.5)	109.8	109.9
18	-	156.7	156.9
19	6.83 s	95.1	95.3
20	-	137.6	137.7
21	1.99 m	124.0	124.1
22	-	134.6	134.6
23	4.79 d (9.5)	25.7	25.7
24	1.66 m	18.3	18.3
25	3.82 s	55.8	55.8

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$ $CDCl_3$

Table S8: ^{1}H and ^{13}C-NMR data of compound **9** and 6-methoxyspirotryprostatin B

Position	9		6-methoxyspirotryprostatin B [6]
	δ_H ^{a,b} mult. (J in Hz)	δ_C ^{a,c}	δ_C $^{\#}$
1(NH)	8.16 s	-	-
2	-	178.9	179.0
3	-	61.4	61.4
3a	-	118.9	118.8
4	6.95 d (8.5)	128.5	128.4
5	6.51 dd (8.5, 2.0)	107.2	107.1
6	-	160.7	160.6
7	6.45 d (2.0)	97.2	97.1
7a	-	141.7	141.7
8	5.75 s	116.9	116.9
9	-	138.0	137.9
11	-	162.5	162.5
12	4.33 dd (11.0, 5.5)	61.6	61.5
13	1.99 m/2.48 m	29.3	29.2
14	1.99 m/2.11 m	22.1	22.1
15	3.58 m/3.83 m	44.9	44.8
17	-	155.2	155.1
18	5.38 d (9.0)	64.1	64.0
19	5.20 d (9.0)	120.4	120.4
20	-	138.3	138.3
21	1.59 s	25.4	25.4
22	1.30 s	18.3	18.3
6-OCH_3	3.80 s	55.5	55.5

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$ $CDCl_3$

Table S9: ^{1}H and ^{13}C-NMR data of compound **10** and cyclo(L-prolinyl-L-phenylalanine)

Position	10		cyclo(L-prolinyl-L-phenylalanine) [7]
	δ_H^{a,b} mult. (J in Hz)	δ_C ^{a,c}	$\delta_C$$^{\#}$
1	-	169.5	169.4
3	3.55 m/3.64 m	45.4	45.2
4	1.88 m/1.97 m	22.5	22.2
5	1.99 m/2.31 m	28.3	28.3
6	4.29 dd (9.5, 3.0)	59.1	58.9
7	-	165.1	164.9
9	4.27 d (3.0)	56.3	56.1
10	2.84 dd (14.5, 10.0)/3.54 m	36.8	36.6
1'	-	135.9	135.8
2'	7.33 m	129.1	128.9
3'	7.24 m	127.5	127.3
4'	7.28 m	129.2	129.1

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$ $CDCl_3$

Table S10. ^{1}H and 13C-NMR data of compound **11** and cyclo(L-prolinyl-L-valine)

Position	11		**cyclo(L-prolinyl-L-valine)** [8]
	δ_H^{a,b} mult. (J in Hz)	δ_C ^{a,c}	$\delta_C$$^{\#}$
1	-	171.1	170.7
3	3.51 m/3.60 m	45.5	45.9
4	1.90 m/2.09 m	22.7	23.1
5	2.13 m/2.34 m	28.0	28.5
6	4.13 t (8.5)	58.9	59.4
7	-	166.3	166.6
9	4.02 d (9.0)	53.5	53.8
10	1.56 m/2.02 m	38.5	39.0
11	1.85 m	24.5	25.1
12	0.95 d (6.5)	23.2	23.7
13	1.00 d (6.5)	21.3	21.6

$^{a)}$ recorded in $CDCl_3$, $^{b)}$ 500 MHz, $^{c)}$125 MHz, $^{\#)}$ $CDCl_3$

Table S11: Cytotoxic activity of compounds **1-11**

Compounds	IC_{50} (μg/mL)	
	Huh-7	HT-29
2	70.9±2.1	60.9±6.2
3	66.2±3.3	61.7±2.4
4	9.7±0.9	10.3±0.9
Ellipticine	0.4± 0.1	0.4± 0.1

*Compounds **1**, **4-10** did not showed cytotoxic activity (IC_{50} > 100 (μg/mL). Ellipticine was used as a positive control. Data are presented as mean ± SD of experiments performed in triplicate.*

S1. Sea cucumber Identification

The obtained spicule morphology and skeleton structure were compared with reference data (Mark O'Loughlin 2016) revealed that the collected sea cucumber belongs to *Colochirus quadrangularis* Troschel, 1846, with detail classification as follow:

Phylum: Echinodermata

Class: Holothuroidea

Order: Dendrochirotida

Family: Cucumariidae

Genus: *Colochirus*

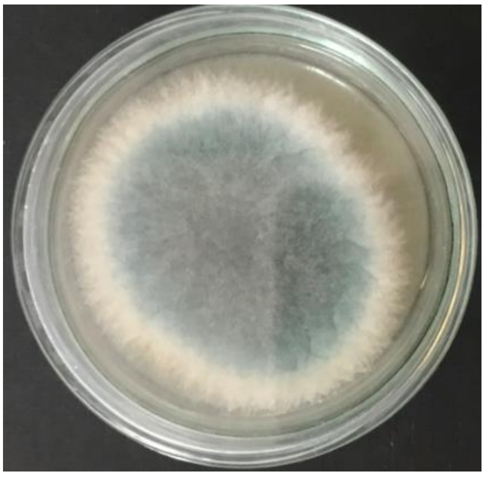
Species: *Colochirus quadrangularis* Troschel, 1846

Reference source:

O'Loughlin, P.M., Harding, C. & Paulay, G., 2016. The sea cucumbers of Camden Sound in northwest Australia, including four new species (Echinodermata: Holothuroidea). Memoirs of Museum Victoria 75: 7-52. http://doi.org/10.24199/j.mmv.2016.75.02.

S2. Aspegillus *fumigatus* M580 Isolation and Characterization: Method and Data

0.5 g of *Colochirus quadrangularis* Troschel, 1846 was suspended in 4.5 ml of sterile distilled water, homogenized by vortexing for 1 min, and the suspension was treated at 60 ºC for 6 min. Next, 0.5 ml of the heat-treated suspension was used for serial dilution in sterile distilled water to 10^{-3}. 50 µl of the final dilution were spread on the petri dishes containing solid media, PDA - potato dextrose agar (30 g/L potato extract, 20g/L dextrose 5g/L soluble starch, 30g/L instant ocean, 15g/L agar). Plates were incubated at 28 °C for 7 days. Single colonies of fungi were transferred onto new petri dishes of PDA medium for further purification steps (Figure).



Colony morphological characteristics of the strain M580 grown on PDA medium for 10 days at 28°C

S3. Fungal Identification

Genomic DNA of strain M580 was extracted by Wizard® Genomic DNA Purification Kit (Promega, USA). Sequences of 18S rRNA was used for taxonomical identification of the fungal strain. Gene amplifications were performed in a 25.0 µl mixture containing 10 µl of sdH_2O, 12.5 µl of 2× PCR Master mix (Thermo Scientific), 1.0 µl of 0.05 mM for both primers NS3F (5'-GCAAGTCTGGTGCCAGCAGCC-3') and NS8R (5'-TCCGCAGGTTCACCTACGGA-3') and 0.5 µl of genomic DNA. The thermocycling was performed on MJ Thermal cycler (Bio - Rad), with a preheating step at 94 °C for 3 min, followed by 35 cycles of denaturation at 94oC for 1 min, annealing at 60°C for 30s and extension at 72 °C for 45s before a final extension of 72 °C for 10 minus. The PCR product size was about 1300 bp. PCR products were purified by DNA purification kit (Invitrogen) and sequenced by DNA Analyzer (ABI PRISM 3100, Applied Bioscience). Gene sequences were handled by BioEdit v.2.7.5. and compared with fungal 18S rDNA sequences available in GenBank database using NBCI Blast program. The alignment was manually verified and adjusted prior to the reconstruction of a phylogenetic neighbour-joining tree by using the MEGA program version 4.1. The results showed that strain M580 belonged to species *Aspegillus fumigatus* (Table S1). Strain M580 was registered with GenBank code: **MW015802**.

S4. M580 strain's 18S rRNA gene sequence comparison results on GenBank

Descriptions | Graphic Summary | Alignments | Taxonomy

Sequences producing significant alignments Download Manage Columns Show 100

select all *100 sequences selected* GenBank Graphics Distance tree of results

Description	Max Score	Total Score	Query Cover	E value	Per. Ident	Accession
Aspergillus fumigatus strain TMS-26 18S ribosomal RNA gene, partial sequence	2130	2130	98%	0.0	100.00%	KJ746594.1
Aspergillus fumigatus strain YuZhu2 18S ribosomal RNA gene, partial sequence	2128	2128	98%	0.0	100.00%	KU512836.1
Aspergillus fumigatus ATCC 13073 gene for 18S ribosomal RNA, partial sequence	2124	2124	98%	0.0	99.91%	LC485158.1
Aspergillus fumigatus strain UPSC 1771 18S ribosomal RNA gene, partial sequence	2124	2124	98%	0.0	99.91%	AF548061.1
Aspergillus fumigatus gene for 18S rRNA, partial sequence	2124	2124	98%	0.0	99.91%	AB008401.1
Aspergillus fumigatus small subunit ribosomal RNA	2124	2124	98%	0.0	99.91%	M60300.1
Aspergillus fumigatus small subunit ribosomal RNA	2124	2124	98%	0.0	99.91%	M55626.1
Aspergillus fumigatus strain MJ-X6 18S ribosomal RNA gene, complete sequence	2122	2122	98%	0.0	99.91%	HM590663.1
Aspergillus sp. ISSFT-021 18S ribosomal RNA gene, partial sequence	2121	2121	97%	0.0	100.00%	KT832787.1
Aspergillus fumigatus strain CY018 18S ribosomal RNA gene, partial sequence; internal transcribed spacer 1, 5.8S ribosomal RNA gene	2119	2119	98%	0.0	99.83%	KJ809565.1
Aspergillus fumigatus strain WL002 18S ribosomal RNA gene, partial sequence	2119	2119	98%	0.0	99.83%	KJ528402.1
Aspergillus fumigatus strain FS160 18S ribosomal RNA gene, partial sequence	2119	2119	98%	0.0	99.83%	FJ840490.1

S5. The 18S rRNA gene sequence of strain M580 is displayed on GenBank

GenBank: MW015802.1

FASTA Graphics

Go to:

```
LOCUS       MW015802                1175 bp    DNA     linear   PLN 23-SEP-2020
DEFINITION  Aspergillus fumigatus isolate M580 small subunit ribosomal RNA
            gene, partial sequence.
ACCESSION   MW015802
VERSION     MW015802.1
KEYWORDS    .
SOURCE      Aspergillus fumigatus
  ORGANISM  Aspergillus fumigatus
            Eukaryota; Fungi; Dikarya; Ascomycota; Pezizomycotina;
            Eurotiomycetes; Eurotiomycetidae; Eurotiales; Aspergillaceae;
            Aspergillus; Aspergillus subgen. Fumigati.
REFERENCE   1  (bases 1 to 1175)
  AUTHORS   Tuan,C.D., Khai,N.V., Hung,N.V., Anh,N.M., Minh,L.T., Huong,D.T.,
            Cuong,P.V. and Lien,H.T.
  TITLE     18s rRNA of fungi
  JOURNAL   Unpublished
REFERENCE   2  (bases 1 to 1175)
  AUTHORS   Tuan,C.D., Khai,N.V., Hung,N.V., Anh,N.M., Minh,L.T., Huong,D.T.,
            Cuong,P.V. and Lien,H.T.
  TITLE     Direct Submission
  JOURNAL   Submitted (18-SEP-2020) biotechnology, Institute of marine
            biochemistry, hoang quoc viet, ha noi, 0243 084, Viet Nam
COMMENT     ##Assembly-Data-START##
            Sequencing Technology :: Sanger dideoxy sequencing
            ##Assembly-Data-END##
FEATURES             Location/Qualifiers
```

S6. Physical properties of compounds 2-10

Fumiquinazoline D (**2**)*:* Pale yellow amorphous powder; $[\alpha]_D^{25}$ +62.5 (c 0.1, $CHCl_3$); $C_{24}H_{21}N_5O_4$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S1.

Fumiquinazoline C (**3**)*:* Pale yellow amorphous powder; $[\alpha]_D^{25}$ -150.0 (c 0.1, $CHCl_3$); $C_{24}H_{21}N_5O_4$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S2.

Fumiquinazoline J (**4**)*:* Pale yellow amorphous powder; $[\alpha]_D^{25}$ -72.4 (c 0.1, $CHCl_3$); $C_{21}H_{16}N_4O_2$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S3.

Bisdethiobis(methylthio)gliotoxin (**5**)*:* White amorphous powder; $[\alpha]_D^{25}$ -42.0 (c 0.1, $CHCl_3$); $C_{15}H_{20}N_2O_4S_2$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S4.

Cyclo(L-Pro-L-Trp) (**6**)*:* Pale yellow amorphous powder; $[\alpha]_D^{25}$ –58.3 (c 0.1, $CHCl_3$); $C_{16}H_{17}N_3O_2$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S5.

Tryprostatin B (**7**)*:* Pale yellow amorphous powder; $[\alpha]_D^{25}$ –55.9 (c 0.1, $CHCl_3$); $C_{21}H_{25}N_3O_2$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S6.

12,13-dihydroxy-fumitremorgin C (**8**)*:* Pale yellow amorphous powder; $[\alpha]_D^{25}$ +20.5 (c 0.1, $CHCl_3$); $C_{22}H_{25}N_3O_5$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S7.

6-Methoxyspirotryprostatin B (**9**)*:* Pale yellow amorphous powder; $[\alpha]_D^{25}$ -37.0 (c 0.1, $CHCl_3$); $C_{22}H_{23}N_3O_4$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S8.

Cyclo(D-Pro-D-Phe) (**10**)*:* White amorphous powder; $[\alpha]_D^{25}$ +75.0 (c 0.1, $CHCl_3$); $C_{14}H_{16}N_2O_2$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S9.

Cyclo(S-Pro-S-Leu) (**11**)*:* White amorphous powder; $[\alpha]_D^{25}$ +75.0 (c 0.1, $CHCl_3$); $C_{14}H_{16}N_2O_2$; ^1H NMR ($CDCl_3$) and ^{13}C NMR ($CDCl_3$) data: see Table S10.

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