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REVIEW

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Marine natural products‡

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This review covers the literature published in 2017 for marine natural products (MNPs), with 740 citations (723 for the period January to December 2017) referring to compounds isolated from marine microorganisms and phytoplankton, green, brown and red algae, sponges, cnidarians, bryozoans, molluscs, tunicates, echinoderms, mangroves and other intertidal plants and microorganisms. The emphasis is on new compounds (1490 in 477 papers for 2017), together with the relevant biological activities, source organisms and country of origin. Reviews, biosynthetic studies, first syntheses, and syntheses that led to the revision of structures or stereochemistries, have been included. Geographic distributions of MNPs at a phylogenetic level are reported.

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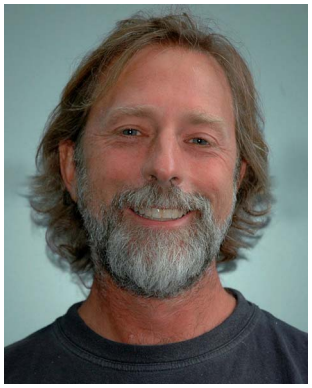
1 Introduction

This review is of the literature for 2017 and describes 1490 new compounds from 477 papers, a 17% increase from the 1277 new compounds in 432 papers reported for 2016.[1] As in previous reviews, the structures are shown only for new MNPs, or for previously reported compounds where there has been a structural revision or a newly established stereochemistry. Previously reported compounds for which first syntheses or new bioactivities are described are referenced, but separate structures are generally not shown. Where the absolute configuration has been determined for all stereocentres in a compound, the identifying diagram number is distinguished by addition of the † symbol. The review retains the format introduced in 2016, with only a selection of highlighted structures (172) now shown in the review.[2] Compound numbers for structures not highlighted in the review are italicised, and all structures are available for viewing, along with their names, taxonomic origins, locations for collections, and biological activities, in an ESI‡ document associated with this review. The Reviews section (2) contains selected highlighted reviews, with all other reviews referenced in a section of the ESI.‡

2 Reviews

The number of MNP-related reviews continued to increase significantly in 2017 compared to previous years. Of the 158 reviews and general articles relating to MNPs published in this

period, 24% focussed on specific compounds or compound classes, 30% related to the bioactivities of MNPs, 24% had an organism or geographic focus, while the remainder were more general. Some of the reviews with a broad relevance to the field of MNPs are highlighted here (15) while the remainder (143) can be found in the ESI section.‡ MNPs reported in 2016 have been comprehensively reviewed.[3] The open access inventory, the World Register of Marine Species (WoRMS), is now ten years old and provides a valuable resource for all marine biodiversity researchers. A timely review discusses the importance of expert taxonomic knowledge, consistent data entry, and nomenclature as they relate to the WoRMS.[4] Two reviews discuss the chemistry and chemical ecology of nudibranchs[5] and the structures and structure determination challenges associated with the chemistry of crinoids.[6] Analysis of trends in chemical diversity of microbial and MNPs reported between 1941 and 2015 illustrates that the discovery of new scaffolds has plateaued but also that there is still a high likelihood for novel chemical discoveries.[7] Emerging cheminformatics tools that can be used to identify potential protein targets for natural products (NPs) has been reviewed.[8] MNPs possessing inhibitory activity towards drug resistant viral, fungal and parasitic infections, *Leishmania*, protein tyrosine phosphatase 1B (PTP1B) and quorum sensing have been reviewed.[9–12] Tricyclic sesquiterpenes and compounds containing guanidine moieties are highly represented in MNPs and both of these structure classes have been reviewed.[13,14] Halogenated MNPs are incredibly abundant and the mechanism for their biosynthesis has been reviewed.[15] A paper has provided detailed insights in to various metabolic aspects of the life cycle of uncultivatable bacteria associated with the sponge *Theonella swinhoei*.[16] A free platform (DEREP-NP) incorporating almost 230 000 NPs derived from the non-proprietary Universal Natural Product Database (UNPD) has been developed. This platform allows users to rapidly identify compounds through matching of



Anthony (Tony) Carroll initially studied the alkaloid and lignan chemistry of rainforest plants (BSc (Hons) and PhD, Prof Wal Taylor, Sydney University) but marine natural products became a major focus after postdoctoral fellowships at the University of Hawaii with Paul Scheuer and at James Cook University, Australia with John Coll and Bruce Bowden. Fifteen years as head of natural products chemistry for the AstraZeneca/Griffith University drug discovery project expanded his interests to include high throughput purification and structure determination techniques and cheminformatics. Since 2008 he has held a faculty position at Griffith University, Gold Coast where he is currently a Professor.

Rohan Davis received a BSc (Hons) degree in chemistry and biochemistry from the University of Melbourne (1992). He worked as a research assistant on the AstraZeneca/Griffith University natural product drug discovery program (1994–1996) before undertaking PhD studies (1997–2000) under the supervision of Professors Ronald Quinn and Anthony Carroll. After 2 years of postdoctoral research at University of Utah with Chris Ireland, he returned to Griffith University in 2003 where he is currently an Associate Professor. His current research involves the discovery and development of new bioactive natural products from plants and marine invertebrates and is currently the Academic Lead for NatureBank, an Australian-based drug discovery platform.

Brent Copp received his BSc (Hons) and PhD degrees from the University of Canterbury, where he studied the isolation, structure elucidation and structure–activity relationships of biologically active marine natural products under the guidance of Professors John Blunt and Murray Munro. Two postdoctoral positions with Jon Clardy at Cornell and Chris Ireland at the University of Utah were then followed with a period spent working in industry as an isolation chemist with Xenova Plc. In 1993 Brent returned to New Zealand to take a lectureship at the University of Auckland, where he is currently a Professor.

Rob Keyzers carried out his BSc (Hons) and PhD studies at Victoria University of Wellington. His thesis research, carried out under the guidance of Assoc. Prof. Peter Northcote, a former contributor to this review, focused on spectroscopy-guided isolation of sponge metabolites. He then carried out post-doctoral research with Mike Davies-Coleman (Rhodes University, South Africa) and Raymond Andersen (University of British Columbia, Canada) before a short role as a flavour and aroma chemist at CSIRO in Adelaide, Australia. He was appointed to the faculty at his alma mater in 2009 where he is currently a Senior Lecturer.

fragments derived from ^{1}H, HSQC, HMBC NMR and MS data of purified compounds and semi-purified mixtures with compounds in the database.[17] An essential requirement for the preparation of this review is the continuous updating of the structures and literature in the MarinLit database.[18] The data derived from MarinLit forms the basis of this review.

3 Marine microorganisms and phytoplankton

The upward trend in the discovery of new MNPs sourced from marine microorganisms continues unabated and now represents 57% of the total new MNPs reported in 2017. Fig. 1 shows the change in discovery efforts across the broad groups of microorganisms, invertebrates, algae and mangroves over the last six years and this clearly indicates that microbial chemistry will dominate future MNP research.

3.1 Marine-sourced bacteria

The 242 new NPs from this microbial source is a significant increase from those reported in the previous three years (2014, 167; 2015, 187; 2016, 161); indicating the field of MNPs has fully embraced the chemistry of the marine microbes in general, at the expense of the macro organisms.

As in previous years, the actinomycete genus *Streptomyces* continues to be the predominant source of new chemistry, with 137 metabolites reported during 2017. This is >50% of the marine-sourced bacterial metabolite numbers; the next closest genera are *Pseudoalteromonas*, *Nocardiopsis* and *Bacillus* with 16, 15, and 14 new compounds, respectively.

Fermentation of a strain of *Streptomyces pratensis* (Bohai Sea, China) yielded three pairs of enantiomeric metabolites, (+)- and (−)-pratensilins A–C **1–6**, which all contained an unprecedented spiro indolinone-naphthofuran motif. Chiral HPLC enabled resolution of the racemic mixtures; all pure enantiomers were shown to racemise when stored in solution. Theoretical density functional theory (DFT) and chemical derivatisation studies indicated that **1** and **2** racemise *via* a keto–enol type tautomerism.[19]

Michèle Prinsep received her BSc (Hons) and PhD degrees from the University of Canterbury, where she studied the isolation and structural elucidation of biologically active secondary metabolites from sponges and bryozoans under the supervision of Professors Blunt and Munro. She undertook postdoctoral research on cyanobacteria with Richard Moore at the University of Hawaii before returning to New Zealand to take up a lectureship at the University of Waikato, where she is currently an Associate Professor.

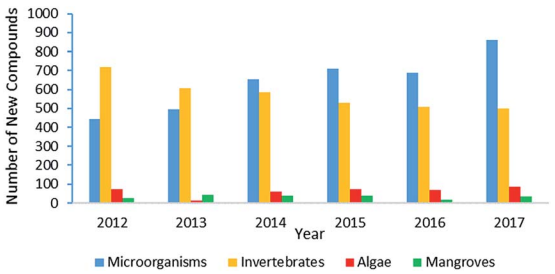
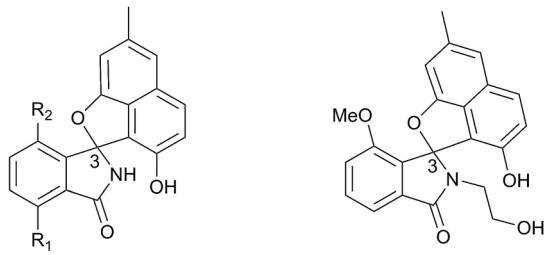


Fig. 1 Sources of new compounds over the period 2012–2017.



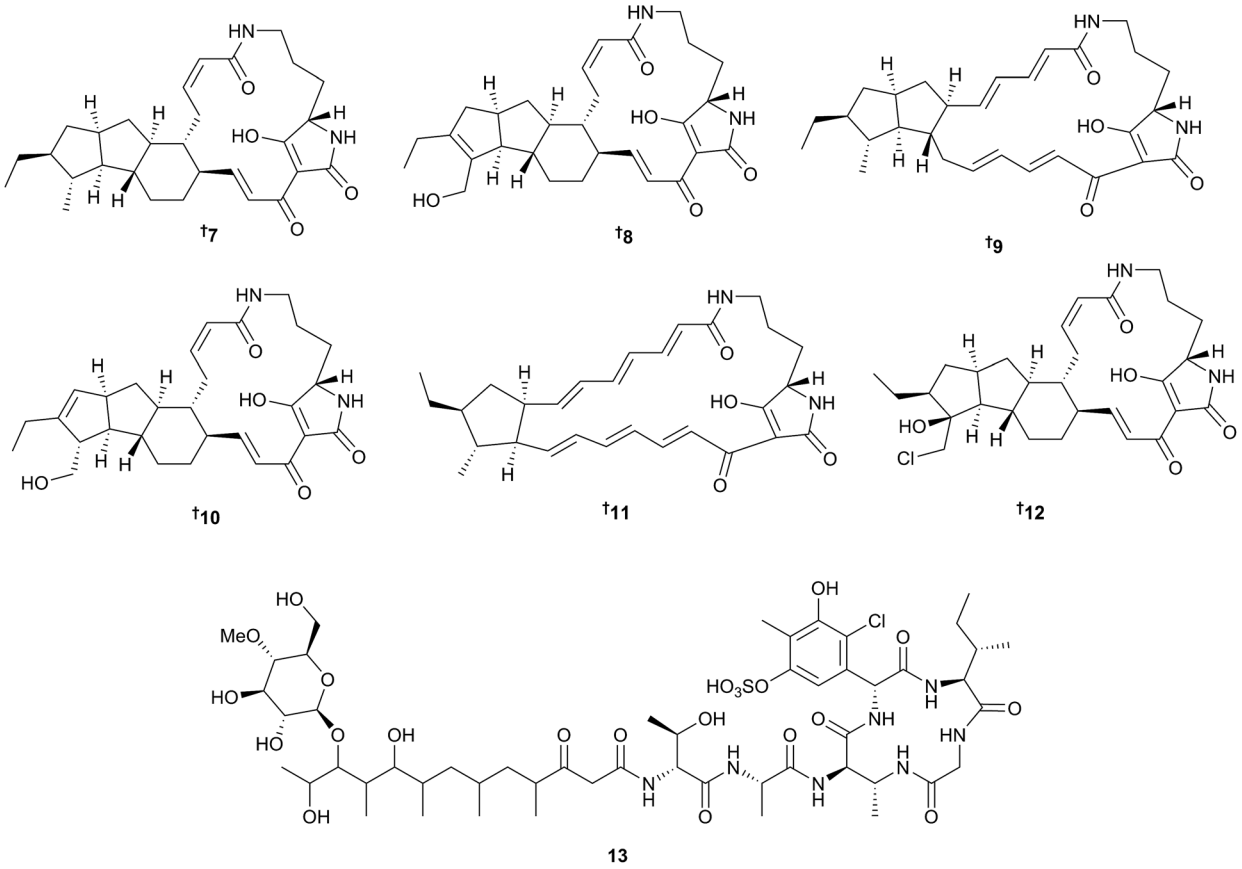
†**1** R_1 = OMe, R_2 = H (*3S*)
†**2** R_1 = OMe, R_2 = H (*3R*)
†**3** R_1 = H, R_2 = OMe (*3S*)
†**4** R_1 = H, R_2 = OMe (*3R*)

†**5** (*3S*)
†**6** (*3R*)

Polycyclic tetramate macrolactams (PTM) are generated *via* a hybrid polyketide synthase/non-ribosomal peptide synthetase pathway. The biosynthetic gene clusters responsible for this class of NP are conserved and widely distributed in bacteria, but most remain silent. The activation of these gene clusters in *Streptomyces pactum* SCSIO 02999 (South China Sea) by promoter engineering and heterologous expression, led to the production of six new PTMs, pactamides A–F (**7–12**). These compounds displayed potent to moderate cytotoxic activity against several human tumour cell lines (HTCLs).[20] Additional genome-based studies on the same *Streptomyces* strain showed that activation of this silent gene cluster by alteration of several regulatory genes (*totR5*, *totR3*, *totR1*) directed biosynthesis towards the known metabolites totopotensamides A and B, along with a novel sulfonate-analogue totopotensamide C **13**.[21] These studies exemplify the many benefits of genome mining for the discovery of new bioactive NPs.

The majority of the 16 known ammosamides contain a rare chlorinated pyrroloquinoline scaffold. Treatment of an extract from an ammosamide-producing *Streptomyces* culture with a cysteine-based reagent designed to label electrophilic compounds produced an ammosamide C-thiol adduct.[22] Additional experimentation revealed that most of the previously reported ammosamides are actually artefacts that are derived from ammosamide C *via* nonenzymatic processes involving exposure to air, light and nucleophilic reagents.[22]

Streptomyces sp. P11-238, isolated from mud on a marine tidal flat (East China Sea), yielded two cyclodepsipeptides streptodepsipeptides P11A ***14*** and P11B ***15***. Both compounds inhibited proliferation of different glioma cell lines with IC_{50} values ranging from 0.1 to 1.4 μM. Furthermore, ***14*** blocked the cell cycle in the G_0/G_1 phase and induced apoptosis. Important



tumour metabolic enzymes, HK2, PFKFB3, PKM2, LLS and FASN were downregulated by ***14***.[23]

A bioassay-guided investigation of a fermentation culture of *Pseudonocardia carboxydivorans* M-227, which was collected from water at a depth of 3000 m in the Aviles Canyon (Cantabrian Sea, Biscay Bay), yielded two antibiotics, branimycins B **16** and C **17** (ref. 24) that displayed moderate to significant antibacterial activities against 28 strains (18 Gram-positive; 10 Gram-negative) with MICs ranging from 1 to >160 μg mL^{-1}. A recent X-ray and NMR study resulted in the structure revision of branimycin, the first congener in this series.[25]

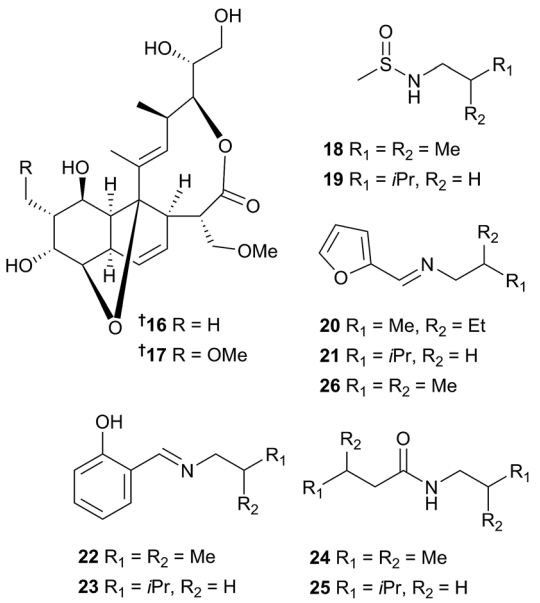
Chemical investigations of nitrogen-containing volatiles from *Salinispora pacifica* and three different strains belonging to the *Roseobacter* group of bacteria resulted in the identification of 20 compounds, several of which are new NPs (**18–25**) or have been reported from a marine organism for the first time (**26**).[26] The first examples of naturally occurring sulfonamides, *N*-isobutylmethanesulfinamide **18** and *N*-isopentylmethanesulfinamide **19** were identified during these studies. New imines such as (*E*)-1-(furan-2-yl)-*N*-(2-methylbutyl) methanimine **20**, (*E*)-1-(furan-2-yl)-*N*-isopentylmethanimine **21** and (*E*)-2-((isobutylimino)methyl)phenol **22** were also identified together with several other acetamides, and formamides. The authors propose that these nitrogenous volatiles originate from biogenic amines derived from isoleucine, valine and leucine.[26]

The known bromotyrosine-derived alkaloids fistularin-3, 11-hydroxyaerothionin, verongidoic acid, aerothionin, homopurpuroceratic acid B, purealidin L and aplysinamisine II were identified from cultures of *Pseudovibrio denitrificans* Ab134 isolated from the tissue of the Brazilian sponge, *Arenosclera brasiliensis*.[27] This report unambiguously demonstrated for the first time that this unique alkaloid structural class, the majority of which have previously only been isolated from Verongida sponges, can be biosynthesised by a marine bacterium.[27] Attempts to identify the bacterial gene clusters responsible for the biosynthesis of these alkaloids are currently underway.

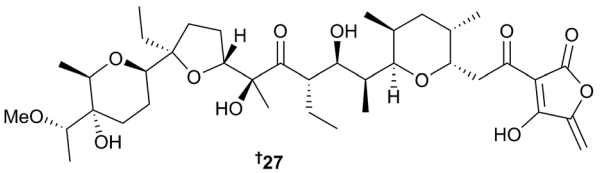
The challenging goal of using biosynthetic machinery to selectively transform inert C–H bonds to other functional groups is slowly being addressed. As this field of study matures, the impact on the chemical industry will be significant, with new chemical entities with commercial impact being generated. An unprecedented two-step biosynthetic conversion of the natural product thiotetromycin to thiotetroamide C involving the tandem oxidation and amidation of an unreactive alkyl group has been reported.[28] This is the first report of an oxidation–amidation enzymatic cascade reaction leading to the selective formation of a primary amide group from a chemically inert alkyl group.[28] Furthermore, this methodology was applied to the generation of several unnatural thiotetroamide C

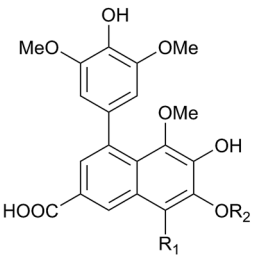
analogues, one of which potentiated bioactivity compared to the parent NP.

The search for new antibacterial agents continues unabated, primarily due to the worrying trend of the emergence of new multi-drug resistant strains. The first SAR study of the potent antibiotic merochlorin A has been undertaken, with 16 analogues generated using a biosynthetic blueprint.[29] Lead compounds were identified against several drug-resistant Gram-positive bacteria and minimal toxicity towards human cell lines translated to promising selectivity indices. Encouragingly, one derivative showed activity towards *Bacillus*-infected cells with similar potency to rifampicin. The biodata for this derivative was superior to that of the NP and this highlighted the potential of the merochlorin chemotype as a new class of antibiotics.[29] Mechanism of action (MoA) studies have been initiated. Ecteinamycin **27**, the *Actinomadura*-derived polyether antibiotic, is the first antibacterial agent where chemical genomics has been used to study its MoA against *Escherichia coli*. Drug sensitive strains of *E. coli* were shown to be potassium cation transport and homeostasis barcode deletion mutants, and this supported an ionophoric mechanism for ecteinamycin induced bacteria cell death.[30] Ecteinamycin **27** has now been isolated from both marine- and soil-derived *Actinomadura* strains, with the latter studies (also reported in 2017)[31] securing the absolute configuration *via* X-ray diffraction studies.



Algal–bacterial symbioses have been the source of numerous new secondary metabolites. The examination of the secondary metabolome of *Phaeobacter inhibens* in response to the algal NP, sinapic acid led to the production of known roseobacticide and the new phenylpropanoids, roseochelins A **28** and B **29**.[32] Roseochelin B **29** was shown to bind to iron and is algacidal against the algal host *Emiliania huxleyi*. A proposed biosynthesis of roseochelins A and B occurs *via* a combination of non-enzymatic and enzymatic transformations.[32]





Additional new *Streptomyces* derived metabolites were ***30–151***.[33–76] New compounds ***152–200*** were reported from the Actinobacteria genera *Nocardiopsis*, *Micromonospora*, *Saccharomonospora*, *Rubrobacter*, *Nesterenkonia*, *Microbacterium*, *Actinoalloteichus* and *Saccharopolyspora*.[77–97] New compounds from the Firmicutes genera *Bacillus*, *Geobacillus* and *Thermoactinomyces* include ***201–216***,[98–106] while new NPs from the Proteobacteria genera *Pseudoalteromonas*, *Pseudomonas*, *Pseudovibrio*, *Vibrio*, *Thalassiosira*, *Enhygromyxa* and *Labrenzia* include ***217–241***.[107–116]

Closer inspection of the spectroscopic and spectrometric data (or lack thereof) presented in several articles reporting new marine bacteria NPs, suggest that the structure assignment of several compounds (***202–205***,[99] ***210***, ***211***,[102] and ***215*** (ref. 105)) remains questionable.

The first total syntheses of the marine-derived bacteria compounds neomaclafungin A **242**,[117] usabamycins A and C,[118] (−)-marinisporolide C,[119] seoaracenes A and B,[120] actinoranone **243**,[121] enhygrolide A,[122] discoipyrroles C and D,[123,124] and metagenetriindole A[125] has been achieved.

The enantioselective total synthesis of neomaclafungin A **242** resulted in the relative and absolute configurations of this potent antifungal agent being revised.[117] These data should, in the future, assist in the complete structures of other neomaclafungins and the closely related metabolite, maclafungin being revised.

The synthesis of actinoranone **243**, along with three stereoisomers, led to a revision of the originally proposed relative configuration, as well as the definitive assignment of the absolute configuration for this cytotoxic meroterpenoid.[121]

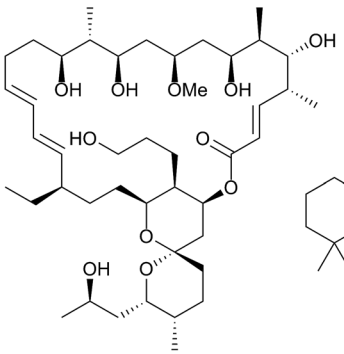
A total synthesis of the proposed structure of baulamycin A along with two diastereomers has been described in the literature.[126,127] Spectroscopic data discrepancies between the synthetic material and the NP indicate that the reported structure for baulamycin A needs to be revised. It is proposed that the actual structure of the MNP is most likely a different diastereomer.[126]

Madurastatin C1 **244** was isolated for the first time from a marine organism (*Actinomadura* sp., deep sea sediment,

Canary Basin, Atlantic Ocean).[128] Through a combination of NMR and partial synthesis studies by two independent research groups,[128,129] the structure of **244** was revised from the originally postulated and reported aziridine containing pentapeptide to an *N*-terminal 2(2-hydroxyphenyl-oxazoline) derivative. The absolute configuration of **244** was determined *via* Harada's advanced Marfey's method.[128] Subsequently, the structures of other madurastatin class NPs, including the terrestrial-derived madurastatins A1, B1, and MBJ-0035, were revised. This research provides a framework that may facilitate future structure revisions for other "aziridine" containing NPs.[128,129]

the commonly used therapeutic drug pentamidine (IC_{50} 4.7 nM).[131] Furthermore, significant selectivity (>25 000 fold) towards the trypanosome parasite was identified, since no *in vitro* cytotoxicity (IC_{50} > 25 μM) towards the human foetal lung fibroblast cell line (MRC-5) was observed.[131]

NMR and MS guided isolation of an extract derived from a Red Sea collection of *Okeania* sp. led to the isolation of serinolamides C ***247*** and D ***248***, and lyngbyabellins O ***249*** and P ***250***.[132] All compounds were evaluated for MCF-7 cytotoxicity and anti-fouling activity (*vs. Amphibalanus amphitrite*). Lyngbyabellins O ***249*** and P ***250*** exhibited strong anti-fouling activity



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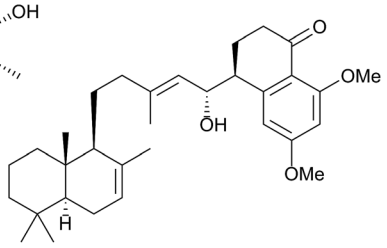

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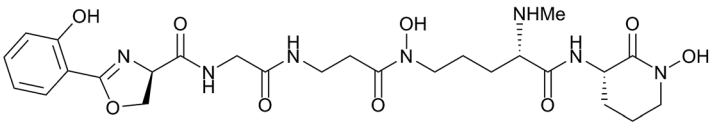
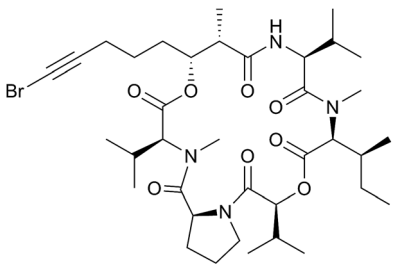


†244

3.2 Cyanobacteria

There has been a significant increase (71%) in the number of new NPs reported from cyanobacteria since 2016. The 48 new compounds for 2017, which have been isolated from nine genera, are the most ever reported for this phylum since the inception of this annual review.

As in previous years, the majority of new metabolites (29 of 48) contain one or more amide functional groups, with 11 cyclic and five linear peptides reported in 2017. Some of the more potent and biologically interesting metabolites are described below. Odobromoamide **245** was identified following bioassay-guided fractionation of an extract from an Okinawan *Okeania* specimen.[130] This cyclodepsipeptide contains a unique C8 alkynoate that incorporates a terminal alkynyl bromide; a moiety rarely observed in nature. Furthermore, initial cytotoxicity testing of **245** towards HeLa S3 cells showed promising activity (IC_{50} of 310 nM), resulting in it being evaluated against a panel of 39 HTCLs; the average GI_{50} for **245** across all cell lines was 29 nM. Secondary assays showed that odobromoamide-induced toxicity was dependent on the caspase family of proteins.[130] Hoshinolactam **246** (*Oscillatoria* sp., Hoshino, Japan), a cyclopropane-lactam, showed potent antitrypanosomal activity towards *Trypanosoma brucei brucei* GUT at 3.1 (IC_{50} 3.9 nM), which was comparable to with EC_{50} values of 0.38 μM and 0.73 μM, respectively, while showing minimal or no cytotoxicity. Grassystatins D–F ***251–253***, new modified linear peptides from a Guam cyanobacterial strain (related to the poorly resolved, polyphyletic genera *Leptolyngbya* and *Phormidium*), were evaluated for aspartic protease



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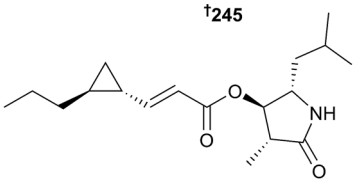
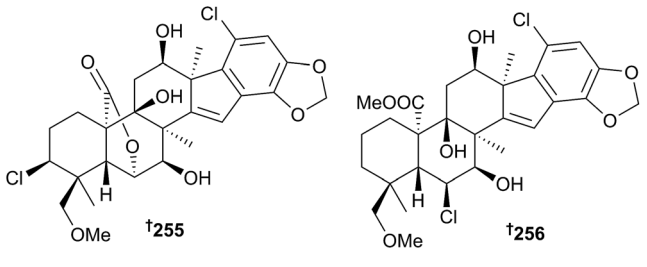


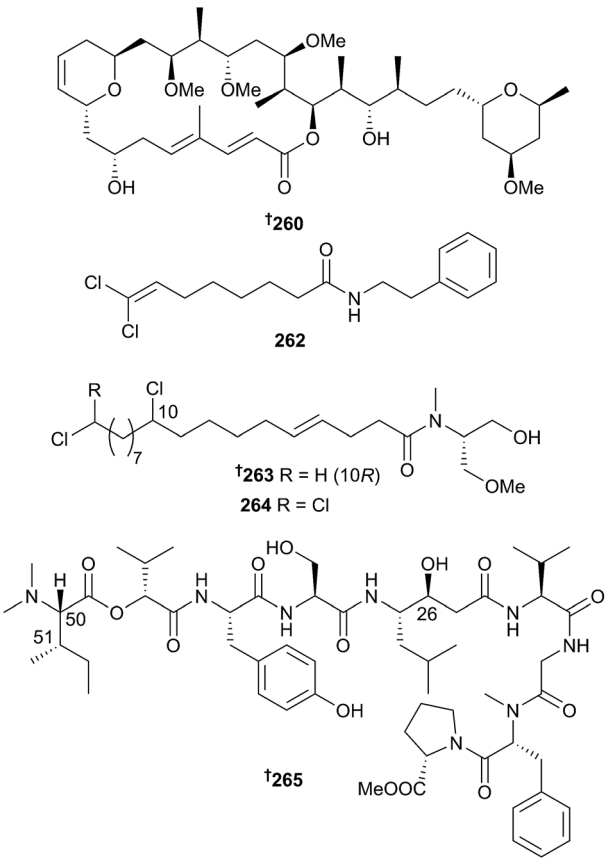
†246

inhibitory activity.[133] While all congeners were active towards cathepsins D and E, grassystatin F was the most potent analogue with IC_{50} values of 50 and 0.5 nM, respectively. Additionally, grassystatin F inhibited the cleavage of cystatin C and PAI-1, and interrupted migration of highly aggressive, triple negative breast cancer cells.[133] Integrating LC-MS/MS molecular networking and bioassay-guided fractionation using a cytotoxicity assay facilitated the targeted isolation of the octapeptide, samoamide ***254***, from a *Symploca* species collected from Samoan waters. It was shown to be most active towards H460 cells with an IC_{50} of 1.1 μM.[134] A genome-mining strategy identified a cyanobacterial-derived (*Scytonema* sp., Bermuda) biosynthetic gene cluster for highly modified and cytotoxic meroterpenoids. Subsequent studies enabled the functional characterisation of the terpene cyclase MstE and showed that it produces an *ent*-sterol type skeleton that is fused to an aryl group; in addition to the identification of merosterols A **255** and B **256**.[135] These molecules were shown to display weak cytotoxicity towards HeLa cells, with IC_{50} values of 1.8 and 11.6 μM. This is the first report on the identification of biosynthetic meroterpenoid-specific enzymes from marine organisms. This strategy holds great promise as a tool for the chemoenzymatic preparation of synthetically challenging chemical scaffolds. A strategy for detecting nitrogen rich and unique compounds from cyanobacterial cultures, which relies on a "MALDI isotopic" technique, has been presented. This method involves the incorporation of ^{15}N (using the feed reagent ^{15}N nitrate), and enables the expressed metabolome of single cyanobacterial filaments to be analysed by MALDI mass spectrometry. Application of this process to a cultured *Moorea producens* strain led to the identification of the non-ribosomal peptide/polyketide hybrid metabolite, cryptomaldamide **257**.[136]



The first total syntheses of seven cyanobacterial compounds have been achieved. Impressively, several of these "first synthesis" papers included the initial isolation and structure elucidation of new NPs, followed by the total synthesis of one of the new compounds. Leptolyngbyolides A–D ***258***, ***259***, **260**, ***261***, were isolated from an extract derived from a *Leptolyngbya* species (Okinawa, Japan).[137] NMR studies enabled the planar structures of these macrolactones to be determined, while the absolute configuration for leptolyngbyolide C **260** was assigned following the asymmetric total synthesis. Furthermore, detailed biological evaluations showed that the leptolyngbyolides depolymerise filamentous F-actin.[137] A dichlorovinylidene – phenethylamide containing NP, caracolamide A **262**, was identified and synthesised using a three-step process.[138] Caracolamide A **262** showed *in vitro* calcium influx and calcium-channel oscillation modulatory activity against murine cortical neurons at concentrations as low as 10 pM. Two chlorinated fatty acid amide derivatives, columbamides D **263** and E **264**, were isolated from *Moorea bouilloni*, collected near Mantanani Island, Sabah, Malaysia.[139] Total synthesis of all four stereosiomers of **263** enabled its absolute configuration to be assigned.[139] The remaining first total syntheses were for 2,2′,5,5′-tetrabromo-3,3′-bi-1*H*-indole,[140] biselyngbyaside,[141] and wewakazole B.[142,143]

Only one structure revision was reported for this phylum in 2017. Symplocin A **265**, a sub-nanomolar cathepsin E inhibitor, had its absolute configuration corrected from 26*R*, 50*R*, 51*R* to 26*S*, 50*S*, 51*S*, following the first total synthesis of the natural linear peptide, along with two non-natural diastereomers.[144]



A total synthesis of the proposed structure of nhatrangin A has been described in the literature.[145] Discrepancies in the spectroscopic data between synthetic and natural nhatrangin A led to the synthesis of six diastereomers of the proposed structure. While this is not the first synthesis of nhatrangin A, it is the first to suggest that its proposed structure was mistakenly reported in the original isolation publication. Current data

suggests that the original stereochemical assignment is probably incorrect.[145]

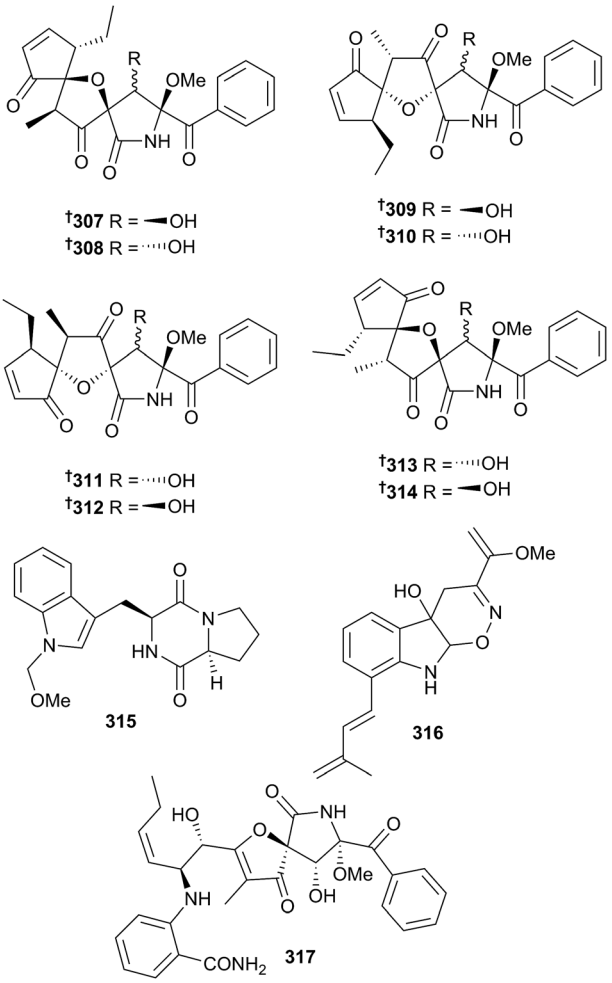
Other new cyanobacterial metabolites were obtained from the genera *Trichodesmium* ***266***,[146] ***267***,[147] and ***268–270***,[148] *Moorea* ***271***, ***272***,[149] ***273–276***,[150] ***277–279***,[151] and ***280–284***,[152] *Okeania* ***285–287***,[153] and ***288–290***,[154] *Oscillatoria* ***291***,[155] *Caldora* ***292*** (ref. 156) and *Leptolyngbya* ***293***.[157]

3.3 Marine-sourced fungi (excluding from mangroves)

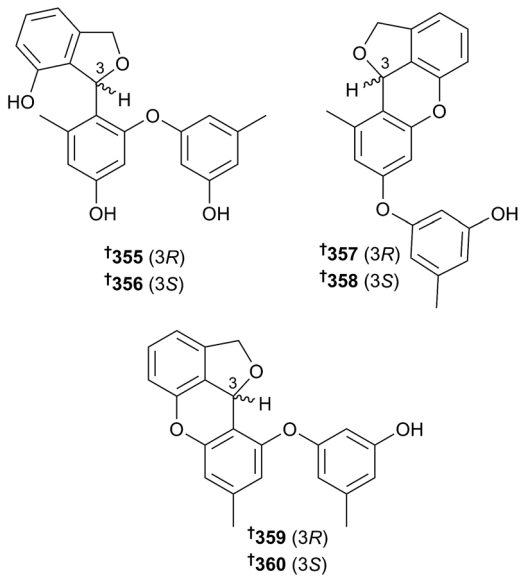
Studies of fungi continue to significantly increase with 448 new compounds reported in 2017 compared to 369 in 2015 and 328 in 2016. The majority of the fungi yielding new MNPs were isolated from sediments (34%), sponges (19%), cnidarians (16%), other invertebrates (12%) or algae and sea grass (10%). A number of new metabolites have been obtained from the genera *Alternaria* (sesterterpenes ***294–298***,[158] perylenequinones ***299***, ***300***, alternaric acid ***301***, butyrolactone derivative ***302*** and cerebroside ***303*** (ref. 159)) and *Arthrinium* (C-8 diastereoisomeric meroterpenoid alcohols ***304***, ***305*** (ref. 160) and phenethyl 5-hydroxy-4-oxohexanoate ***306*** (ref. 161)), the last of which was previously obtained *via* synthetic biology but was isolated here as a new NP.

The genus *Aspergillus* has again been well studied and has yielded many new metabolites. A strain of *Aspergillus fumigatus* isolated from a fish yielded a series of compounds with spiroheterocyclic γ-lactam cores; cephalimysins E–L **307–314**, which contain six chiral centres. Treatment of these compounds with acidic methanol yielded their enantiomers. Molecular orbital calculations were used to propose a plausible biosynthetic route to the cephalimysins *via* an intramolecular annulation.[162] Separate co-cultures of marine sediment derived *A. fumigatus* with each of two terrestrial bacterial strains led to dual induction of metabolites in both organisms. In axenic culture, *A. fumigatus* produced the diketopiperazine brevianamide X **315** but in co-culture with one strain of bacteria, production of the indole alkaloid luteoride D **316** and heterospirocycle pseurotin G **317** was induced whilst the bacterium was induced to produce a peptide. In co-culture with a different terrestrial bacterial strain, *A. fumigatus* did not produce any of these new metabolites but induced the bacterium to produce metabolites not obtained in axenic culture which may have suppressed production of the fungal compounds.[163]

Other metabolites produced by *Aspergillus* species included flavones ***318***, ***319***,[164] phenyl ether derivative ***320***,[165] furan ***321*** (ref. 166) and an indole alkaloid ***322***.[167] Co-culture of an *Aspergillus* strain with *Penicillium citrinum* induced the *Aspergillus* species to produce furanone derivatives ***323–325*** (the last two isolated as individual C-8 epimers which immediately epimerised), oxadiazin derivative ***326***, pyrrole derivative ***327*** and neoaspergillic acid complexes, aluminiumneohydroxyaspergillin ***328*** and ferrineohydroxyaspergillin ***329***.[168] *Aspergillus* strains were also the sources of diphenyl ether ***330***,[169] sesquiterpene glycoside ***331***,[169] phenolic polyketide ***332***,[170] sydonic acid derivative ***333***,[171] candidusin derivative ***334***,[172] napthoquinones ***335***, ***336***,[172] quinazoline derivatives ***337–339***,[173] oxepin alkaloid ***340***,[173] cyclopenin derivatives ***341–344***,[173]



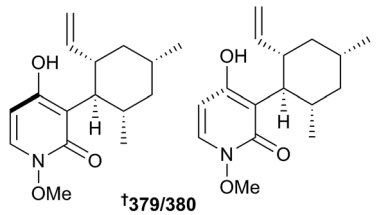
anthraquinones ***345–348*** (ref. 174) (the last as a first time marine isolate), diphenyl ether ***349***,[174] benzaldehyde derivative ***350*** (ref. 174) and cyclopeptides ***351–353***,[175] ***354***.[176] Versiorcinols A–C **355–360**, were isolated from a sponge-associated *Aspergillus versicolor* as racemates and although the structures of the



enantiomers could not be determined unambiguously at room temperature, a combination of variable temperature NMR experiments, DP4 probability analysis, DU8 proton–proton spin–spin coupling constant calculations and ECD calculations enabled assignment of the absolute configurations.[177]

Further metabolites obtained from the *Aspergillus* genus included cyclic peptide ***361*** and linear peptides ***362–364***,[178] diastereoisomeric liptopeptidyl benzophenones ***365*** and ***366***,[179] benzaldehyde derivatives ***367–369***,[180] dioxopiperazine alkaloids ***370***, ***371***,[180] polycyclic aspochalasin ***372***,[181] dioxomorpholine derivatives ***373–377*** (ref. 182) and diphenyl ether glycoside ***378***,[183] the last of which was obtained *via* chemical epigenetic manipulation of the culture.

An endophytic *Chaunopycnis* sp., isolated from a pulmonate false limpet, has previously yielded a number of NPs including pyridoxatin, a potent siderophore and antifungal compound which occurs as atropisomers. Cultivation of this strain with a co-occurring endophyte, *Trichoderma hamatum*, resulted in complementary chemical-ecology responses that were elucidated with time course studies. In monoculture and co-culture, the *Chaunopycnis* strain produced pyridoxatin but in co-culture, *Trichoderma hamatum* quantitatively biotransformed pyridoxatin to methyl-pyridoxatin atropisomers **379**, **380**, which had no Fe(III) binding affinity and were not cytotoxic. It then appeared that the *Chaunopycnis* sp. activated a silent PKS to produce **381**, a new example of the rare 2-alkenyl-tetrahydropyrans. Although **381** was not antifungal, prior studies have established that glycinyl esters of this structure class are extremely potent antifungal agents so it was speculated that either such esters are produced at levels below detection or that the bacterial gene cassette (BGC) in *Chaunopycnis* sp. may be corrupted and unable to incorporate the glycinyl residue.[184]



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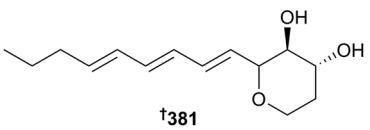
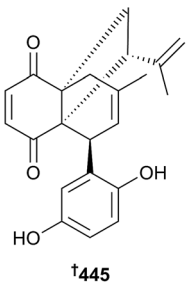


†381

New metabolites were obtained from the genera *Chondrostereum* (sesquiterpenes ***382–389*** (ref. 185)), *Cladosporium* (cladosporol derivatives ***390–394*** (ref. 186)), *Dichotomomyces* (phenolic ketones ***395***, ***396***,[187] amides ***397–400***,[188] polyketide ***401*** (ref. 188) and diketopiperazines ***402***, ***403*** (ref. 188)), *Engyodontium* (benzoic acid derivative ***404*** (ref. 189)) and *Epicoccum* (azaphilonoid ***405*** (ref. 190)). ^{13}C labelling studies indicated that the skeleton of this last compound is biosynthesised from two polyketide chains.[190] *Eurotium* strains yielded steroid ***406***,[191] benzaldehyde derivatives ***407***, ***408***,[192] and indolediketopiperazine alkaloids ***409–412***.[193] The absolute configuration of rubrumline M was determined as ***413***.[193] Norsesquiterpenoid ***414***, monoterpene ***415*** and 2-(2-hydroxy-4-methylcyclohex-3-enyl)propanoic acid ***416*** (the latter a known synthetic compound but obtained here as a new NP) were isolated from a *Eutypella* strain,[194] as were diterpenes ***417*** and ***418***,[195] hexahydrobenzopyran derivative ***419*** (ref. 196) and thiodiketopiperazine alkaloids ***420–432***,[197] ***433–438***.[198] A *Fusarium* species was the source of pyripyropenes ***439–441***, (the last a known terrestrial fungal metabolite) but isolated from the marine environment for the first time[199] and a *Gaeumannomyces* strain yielded glycosylated dialkylresorcinol derivatives ***442***, ***443*** and anthraquinone derivative ***444***.[200]

Gliomastix sp. yielded hydroquinone derivatives **445**, **446**, ***447–449***, **450**, **451**, ***452***. Gliomastin A **445** possesses a novel skeleton which could be biosynthetically derived from a Diels–Alder reaction between the known hydroquinone derivatives acremonin A and F-11334A, both of which were co-isolated, whilst **446** is a rare sulphur containing alkaloid derived from F-11334A.[201] The absolute configurations of **445** and aglycones **450**, **451** were determined *via* Time-Dependent Density Functional Theory-Electronic Circular Dichroism (TDDFT-ECD) and optical rotation (OR) calculations.[201]



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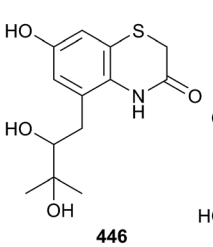

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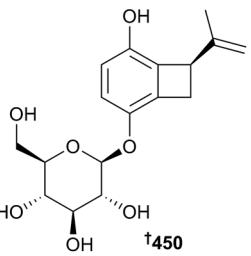

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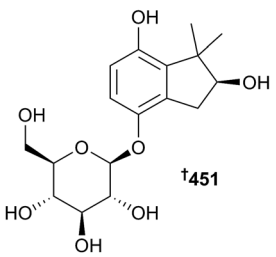
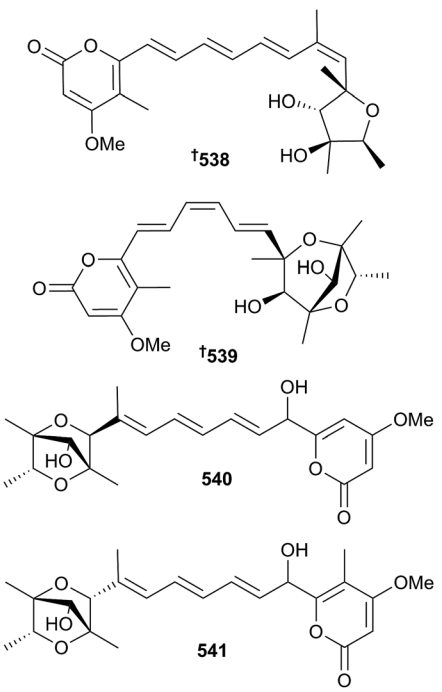


†451

A *Graphostroma* species yielded a number of sesquiterpenes ***453–461***, including xylariterpenoids A and B. The stereochemistry of C-10 in each was revised to that shown in xylariterpeneoids A ***463*** and B ***462*** respectively and both were also obtained as first time MNPs.[202] Other genera to yield new NPs were *Hansfordia* (resorcinol derivatives ***464–473*** (ref. 203)), *Hypoxylon* (butyrolactone derivatives ***474***, ***475*** (ref. 204)) and *Isaria* (polyketides ***476***, ***477*** (ref. 205)). *Leptosphaeria* sp. was the source of isobenzofuranones ***478–481*** and isochromenones ***482***, ***483***, in addition to the known isobenzofuranones clearanols E and D (reported from a marine source for the first time and absolute configurations determined as ***484*** and ***485*** respectively[206]). Cyclic heptapeptides ***486–489*** were obtained from a *Mortierella* sp.,[207] dihydrochromone dimer ***490*** was isolated from a *Neosartorya* strain[208] and 3,4-dihydroisocoumarin derivatives ***491***, ***492*** were reported from a *Paraconiothyrium* species.[209] As always, the genus

Penicillium has been a prolific source of new metabolites, including alkaloids ***493***, ***494*** (ref. 210) and isobenzofuranones ***495***, ***496***,[210] ***497–500***.[211] A deep-sea derived strain yielded a diverse range of metabolites including dimeric isocoumarin ***501***, merosesquiterpenoid ***502***, citrinin dimer ***503***, alkaloid ***504*** and lactone ***505***.[212] Other metabolites obtained from the *Penicillium* genus include polyketide ***506***,[213] cladosporin derivative ***507***,[214] spiro tetracyclic diterpene ***508***,[215] secalonic acid derivatives ***509*** and ***510***,[216] azaphilone derivative ***511***,[217] quinolinone ***512*** (ref. 218) and acetylenic aromatic ether ***513*** which has the same planar structure as the antibiotic WA but the opposite optical rotation.[219] Polyketides ***514–518***,[220] ***519–530***,[221] lactones ***531–534*** (ref. 222) and sesquiterpene ***535***, guaiadiol A ***536*** and 4,10,11-trihydroxyguaiane ***537*** were also isolated from *Penicillium* species, the last two as first time MNPs.[223]

Metal-stress experiments with a *Penicillium* strain cultured separately in the presence of each of six heavy metals, elicited production of cryptic metabolites. Culture in the presence of cobalt induced production of antibiotic polyenes **538–541**.[224] Other *Penicillium* strains produced indole diterpenoids ***542–544***,[225] amino acid conjugated anthraquinones ***545–552***,[226] chlorinated polyketide ***553***,[227] polyketides ***554*** (ref. 228) and ***555***,[229] sesquiterpene methylcyclopentenedione ***556***,[230] meroterpenoids ***557–561***,[231] napthalene derivative ***562***, ketone ***563*** and chromone ***564***,[232] xanthone ***565*** and chromone ***566***,[233] austin derivative ***567*** and isochromones ***568–571*** (ref. 234) and meroterpenoids ***572–577***.[235] The absolute configurations of the meroterpenoids chrodrimanin F and A were determined as ***578*** and ***579*** respectively.[235]

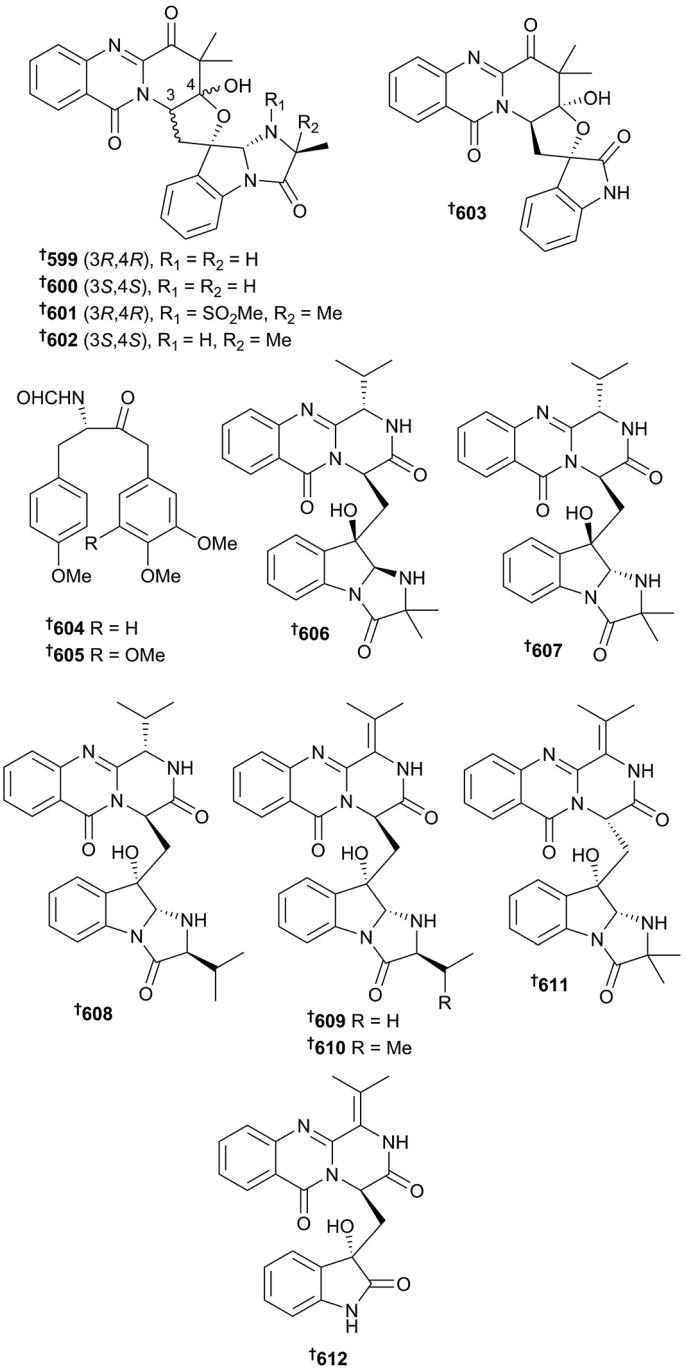


The genus *Pestalotiopsis* yielded isocoumarins ***580***, ***581***,[236] degraded sesquiterpene ***582***,[236] furan derivative ***583*** (ref. 236) and polyketide derivatives ***584–595***,[237] while a *Phoma* species was the source of diphenyl ethers ***596***, ***597*** and ***598***.[238] Culture of *Scedosporium apiospermum* on media supplemented with various amino acids induced production of a range of alkaloids including scedapins A–G (**599–605**) and scequinadolines A–G (**606–612**). Of these, scedapin C **601** and scequinadoline D **609** displayed significant activity against hepatitis C virus (HCV).[239]

Other genera of fungi to yield new metabolites included *Scopulariopsis* (triterpenoids ***613***, ***614*** and naphthoquinone ***615*** (ref. 240)), *Stachybotrys* (sesquiterpenes ***616–619***,[241] meroterpenoids ***620–622***,[242] ***623–628***,[243] atranones ***629–635*** (ref. 244) and isoindolinone derivatives ***636–639***, (these isoindolinone derivatives were produced *via* supplementation of the culture with amino compounds[245])), *Stilbella* (diterpenoids ***640–642*** and polyketide-peptides ***643***, ***644*** (ref. 246)) and *Talaromyces* (hydroanthraquinones ***645–649***,[247] lactones ***650–663***,[248] ergosterol analogue ***664***,[249] bis-anthraquinone ***665*** (ref. 249) and cyclic hexapeptide hydroxamate ***666*** (ref. 250)). Mixed culture of a deep-sea derived *Talaromyces* strain and a mangrove derived *Penicillium* strain produced four new polyketides ***667–670*** but which of the two fungi was the producing species was not resolved.[251] Depsides ***671–685*** were produced by a *Thielavia* sp., of which ***682–685*** were obtained for the first time from a marine source.[252] A *Tolypocladium* strain yielded acyl tetramate ***686*** and a number of known metabolites, efrapeptin D ***687***, terricolin ***688***, malletenin B ***689***, malletenin E ***690*** and tolypocladenols A1/A2 ***691***, which were isolated from the marine environment for the first time.[253] *Trichoderma* strains were the source of diterpenes ***692–694***,[254] and cyclopentenone ***695*** (ref. 255) and a *Truncatella* strain yielded α-pyrone analogues ***696–701***, of which the known synthetic ***701*** was isolated from a natural source for the first time.[256] The *Westerdykella* genus yielded tetrahydropyran derivatives ***702–707*** and lanomycinol ***708***, the last of which was obtained as a first time marine isolate,[257] cytochalasans ***709–714***,[258] tyrosine derived alkaloid ***715***,[258] phomacin B ***716*** (ref. 258) and triticone D ***717***,[258] the last two of which were also first time marine isolates. *Xylaria* strains yielded cytochalasin ***718*** (ref. 259) and hydroxylated fatty acid ***719***.[260]

Other metabolites reported from the marine environment for the first time include 4″-dehydroxycandidusin A ***720***,[261] aspochalasin K ***721***,[262] fuscin ***722***, dihydrofuscin ***723***, dihydrosecofuscin ***724*** and secofuscin ***725***,[263] brevicompanine B ***726***,[264] 4-hydroxy-3,6-dimethyl-2-pyrone ***727***, 4-methoxyisoquinolin-1(2*H*)-one ***728*** and *N*,*N*-diethyl-3-methylbenzamide ***729***,[265] isotorquatone ***730***, chartabomone ***731*** and dichoromone ***732***.[266] Chartabomone and the racemate of isotorquatone were growth inhibitory towards the alga *Chlorella fusca* while the (*S*)-enantiomer of isotorquatone was much less active.[266]

The structure of the meroterpenoid acremine P, originally isolated from a sponge-derived *Acremonium persicinum*, was revised to ***733***, through re-evaluation of NMR chemical shift values and NOESY data followed by DFT calcultaions.[267] Synthesis of two possible diastereoisomers of gliomasolide E, a 14-membered macrolide isolated from a sponge-derived *Gliomastix* sp., enabled determination of the absolute configuration of the natural product as ***734*** (ref. 268) while the absolute configuration of gliomasolide D was determined as ***735*** *via*



synthesis of the C-17 epimers.[269] Total synthesis of the bicyclic polyketide lactone, protulactone A, originally obtained from a sediment-derived *Aspergillus* sp., has been achieved and resulted in determination of the absolute configuration as ***736*** (ref. 270) and asymmetric total synthesis of the azaphilone derivative felinone A (*Beauveria felina*) resulted in revision of the absolute configuration to ***737***.[271]

Asymmetric total synthesis of aspergillide D, a 16-membered macrolide originally obtained from a gorgonian-derived *Aspergillus* sp., was achieved using a convergent strategy,[272] while synthesis of the tetramic acid derivative cladosin C (*Cladosporium sphaerospermum*) was achieved starting with inexpensive, commercially available compounds.[273] The dimeric *trans*-epoxyamide, chrysamide B (*Penicillium chrysogenum*) was synthesised utilising a convergent approach[274] and a parallel synthetic approach was used to achieve synthesis of the 1,4-benzoquinones anserinones A and B (*Penicillium* spp.).[275] Lumazine peptides penilumamides B–D (*Aspergillus* sp., *Penicillium* sp.) were prepared *via* a straightforward method from 1,3-dimethyllumazine-6-carboxylic acid[276] and penicinoline E

(*Penicillium* sp.) was prepared in high yield.[277] Syntheses of both naturally occurring enantiomers of pericosine E (*Periconia byssoides*) were completed, with synthesis of the minor naturally occurring enantiomer, (+)-pericosine E, being a first synthesis.[278]

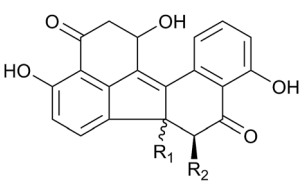
A number of new activities were reported for known fungal metabolites. Penicillenols A1, A2, B1, B2, C1 and C2 inhibited *Candida albicans* biofilm formation and eradicated exisiting biofilm effectively.[279] The cyclic peptide unguisin A was shown to be a receptor for a range of anions, with highest affinity for phosphate and pyrophosphate.[280] The polyketide pallidopenillines were found to be growth stimulatory to a variety of grain crop seedlings[281] and ustusol A exhibited moderate activity against three species of plant pathogenic fungi.[282]

Studies of the biosynthetic pathway to the indole diketopiperazine alkaloids echinulin and neoechinulin (originally isolated from various *Aspergillus* species), revealed that two prenyltransferases control a prenylation cascade in the biosynthesis and the second of these is able to accept its own polyprenylated derivatives as substrates and to catalyse prenylations at different sites.[283] The terretonins are a family of meroterpenoids obtained from various marine and terrestrial fungi. Biochemical and crystallographic analyses have revealed that one multifunctional enzyme is involved in unusual skeletal reconstruction in terretonin biosynthesis, catalysing both a ring expansion and a hydrolysis reaction.[284]

3.4 Fungi from mangroves

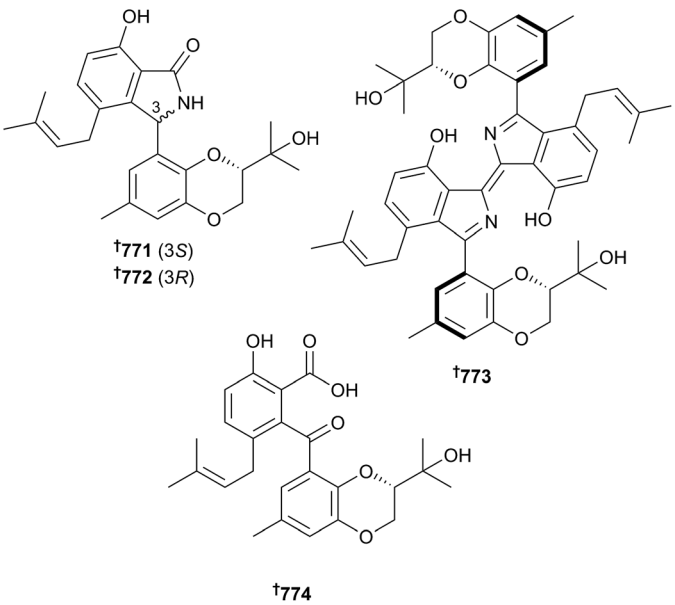
In recent years there has been a continued increase in the number of new metabolites reported from mangrove-associated fungi (103 in 2014, 133 in 2015 and 149 in 2016), but in 2017 there was a considerable drop in numbers with 101 new compounds reported, mostly from endophytic species. According to the recently proposed concensus definition, for mangrove-associated fungi to be considered to be marine the fungi must be obtained from the rhizosphere, roots and sediment of mangroves. Only 30% of the current studies fit into this category.[285] Interestingly, a number of the leaf and branch derived fungal endophytes were grown on saline media, even though they would not be exposed to these conditions in their natural environment.

An *Annulohypoxylon* strain was the source of the benzo[*j*] fluoranthenes, daldinones H **738** and J **739**. The previously proposed biosynthetic precursor to the daldinones, 1,8-dihydroxynaphthalene (DHN) was fed to the culture and resulted in accumulation of **738** and daldinone B, providing support for the hypothesis that the biosynthesis of these compounds begins with oxidative coupling of two DHN units.[286]



738 R_1 = ····OH, R_2 = H
739 R_1 = ▬H, R_2 = OH

An *Ascomycota* sp. yielded enantiomeric polyketide dimers ***740*** and ***741***,[287] whilst *Aspergillus* strains were the source of coumarin derivatives ***742***, ***743***,[288] chromone ***744***,[288] sterone ***745***,[288] anhydride derivative ***746***,[289] lipid amide ***747***,[289] xanthonoids ***748–751*** and derivative ***752***,[290] meroterpenoid ***753*** (ref. 291) and chromanone ***754***.[292] Sesquiterpenes ***755***, ***756*** (ref. 293) and furan derivatives ***757–764***,[294] were obtained from *Coriolopsis* strains, of which ***763*** and ***764*** were obtained for the first time as NPs. *Diaporthe* species were the source of a number of alkaloids including ***765–768***,[295] isoindolinones ***769***, ***770***,[295] diaporisoindoles A **771**, B **772**, C **773** (an unusual diisoprenylisoindole dimer) and tenellone C **774**.[296] Although diaporisoindoles A **771** and B **772** are C-8 epimers, diaporisoindole A **771** exhibited significant inhibition of *Mycobacterium tuberculosis* protein tyrosine phosphatase B (MptpB) while diaporisoindole B **772** was inactive at the concentrations tested.



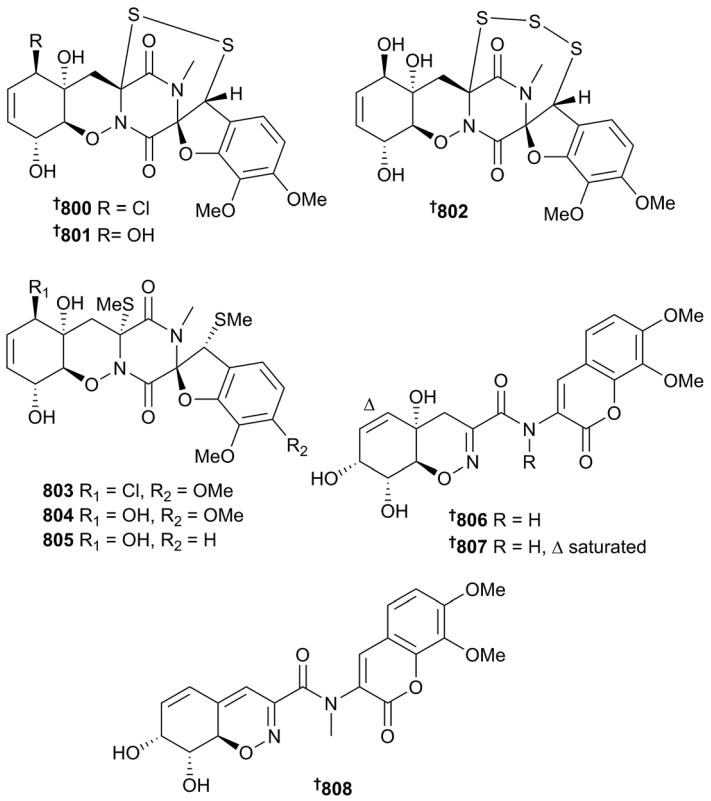
†**771** (3*S*)
†**772** (3*R*)

†**773**

†**774**

A *Eurotium* strain yielded anthraquinone ***775***, prenylated indole 3-carbaldehyde derivatives ***776***, ***777***, anthranilic acid derivative ***778*** and isochromone derivative ***779***,[297] whilst a *Eutypella* species was the source of sesquiterpene lactones ***780–783***.[298] Macrocyclic lactones ***784***,[299] ***785***, ***786*** (ref. 300) were isolated from *Lasiodiplodia* species and pyranonaphthazarin ***787*** and 2-naphthoic acid derivative ***788*** were isolated from a *Leptosphaerulina* strain.[301] A number of new metabolites were obtained from *Penicillium* species. These include chromone ***789***,[302] chaetoglobosin ***790***,[303] chlorinated xanthone ***791*** and anthraquinone ***792***,[304] azaphilones ***793–796***,[305] chlorinated alkaloid ***797*** (ref. 306) and xanthone derivatives ***798***, ***799***.[307] A *Penicillium janthinellum* strain was the source of two different families of complex NPs. Penicisulfuranols A–F **800–805** are epipolythiodioxopiperazine alkaloids with an additional spirofuran ring[308] and trichodermamides D–F **806–808**, heterocyclic dipeptides with a 1,2-oxazadecaline core.[309] Other genera to

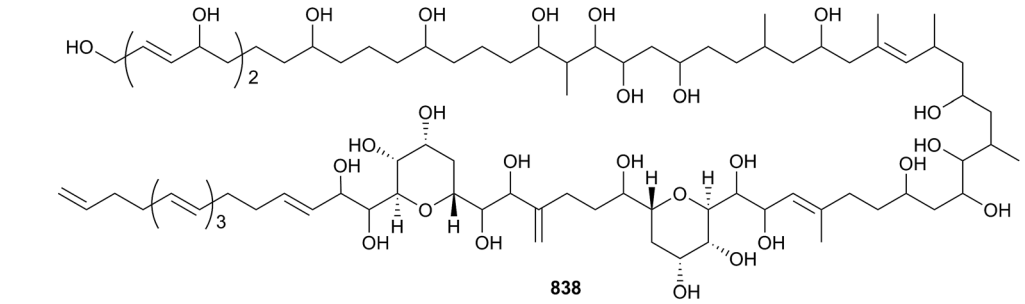
yield new metabolites were *Pestalotiopsis* (salicyloid derivatives ***809–818*** (ref. 310)), *Phoma* (anthraquinone ***819*** (ref. 311)), *Phomopsis* (biphenyl derivative ***820***,[312] α-pyrone ***821*** (ref. 313) and polyketides ***822–824***),[314] *Rhytidhysteron* (spiro-bisnaphthalenes ***825***, ***826*** (ref. 315)) and *Talaromyces* (depsidones ***827***, ***828*** (ref. 316) and alkaloid ***829*** (ref. 317)). Co-cultivation of a *Trichoderma* sp. with a pathogenic bacterium induced production of sesquiterpenes ***830***, ***831*** and lasiodiplodins ***832–834***, the last of which is a known synthetic compound but was isolated here as a new NP,[318] whilst co-culture of two unidentified fungi yielded a nonadride derivative, ***835***.[319]



Synthesis of the proposed structure of an alkaloid obtained from endophytic *Hypocrea virens* was carried out but inconsistencies in the data between the synthetic and NP led to structural revision of the NP to gliotoxin derivative ***836*** which was then also synthesised.[320] Synthesis of the structure proposed for polyketide-macrolactone phomolide H (*Phomopsis* sp.) also led to structural revision of the NP to ***837***, the diatereoisomer of the originally proposed structure.[321] The 14-membered ring macrolides, pestalotioprolide C[322] and pestalotioprolide G[323] have been synthesised and mismatch of the spectroscopic data of the synthesised proposed structure of pestalotioprolide H and that of the isolated metabolite indicated that structural revision of the NP is required.[323] Fused tetrahydrofuran lactone 2106 A (unidentified endophytic fungus, *Avicennia marina*) has been synthesised *via* an approach utilising radical addition to activated olefins.[324] A study of the effect of monochromatic red, blue and green light on polyketide production in an endophytic *Halorosellinia* sp. revealed that all monochromatic light enhanced octaketide production and inhibited heptaketide production but green light had the most dramatic effect.[325]

3.5 Dinoflagellates

The number of new metabolites reported from dinoflagellates has risen somewhat, with 20 compounds reported in 2017 compared with 15 in each of 2014 and 2015 and 11 in 2016. Culture of a strain of *Amphidinium carterae* collected in South Korea has yielded the polyketides amphidinol 20 **838** and 21 **839**, with **839** possessing the longest linear structure reported in all isolated amphidinols to date.[326] An Italian strain of *A. carterae* was the source of two further amphidinols ***840***, ***841*** and investigations into the biosynthesis of ***840*** utilising ^{13}C labelled acetate and glycolate proved that glycolate is the starter unit for polyketide chain assembly in amphidinols.[327] A further strain of *Amphidinium* yielded ***842***, ***843***, 15- and 19-membered macrolides respectively.[328] Karmitoxin **844**, isolated from a culture of *Karlodinium armiger*, is a polyhydroxy-polyene that is structurally related to both amphidinols and karlotoxins but contains a primary amino group. It exhibited potent cytotoxic activity in a rainbow trout gill cell-based assay and caused mortality to the copepod *Acartia tonsa* which grazes on the dinoflagellate.[329] Two polyenes; a trioxilin ***845*** and a sulphoquinovosyl diacylglycerol ***846*** were obtained from *Oxyrrhis marina*[330] while *Prorocentrum lima* yielded the polyhydroxy compound limaol ***847*** (ref. 331) and a number of diol and sulfated diesters of okadaic acid and



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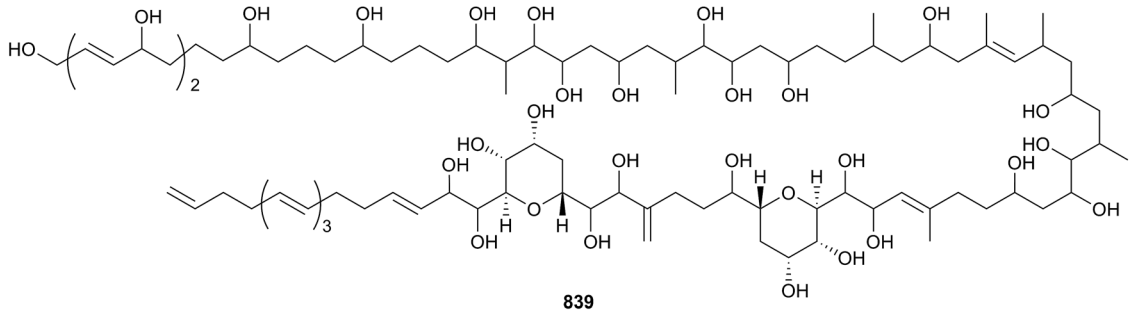

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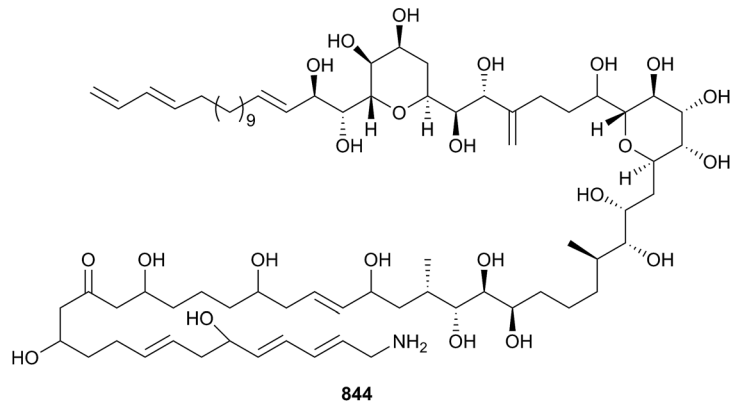


844

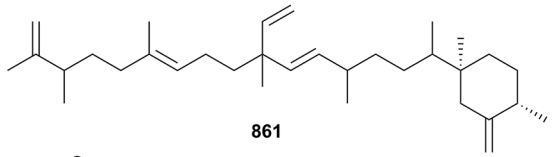
dinophysistoxin-1 (DTX-1), ***848–855***.[332] The presence of these compounds confirms that okadaic acid and DTX-1 are initially formed in dinoflagellates as non-toxic sulphated esters.[332] Two tetrodotoxin-like compounds ***856***, ***857***, were identified in *Prorocentrum minimum* but characterised by mass spectrometry only.[333]

Comparison of transcriptome profiles of *Alexandrium catenella* cells at different toxin biosynthetic stages within the cell cycle identified 138 homologues of 15 toxin genes and indicated that toxin biosynthesis was not regulated at a transcriptional level but may be regulated either translationally or post-translationally.[334] Sequencing of transcriptomes of two ciguatoxin producing *Gambierdiscus* strains revealed a great diversity of PKS genes, many of which may be associated with toxin production. There was also evident distinction between the genes responsible for polyketide and fatty acid synthesis.[335] A study of *Karenia brevis* pooled RNA sequence libraries to generate a large combined library, enabling identification of transcripts encoding both single and multidomain PKS genes.[336] Bioluminescence in dinoflagellates is considered to be an anti-grazing strategy. Strains of *Lingulodinium polyedra* and *Alexandrium tamarense* were shown to be capable of increased bioluminescence capacity in response to chemical cues from grazer copepods.[337]

4 Green algae

Thirteen new MNPs were isolated from five species of green algae in 2017. Three new curcuphenol sesquiterpenes ***858–860*** were isolated from *Udotea orientalis*.[338] Curcuphenol type sesquiterpenes have not previously been reported from green algae. Three new cyclic C33 botryococcene terpenes **861–*863*** and one new trimethylsqualene isomer ***864*** were isolated from the green micro-alga *Botryococcus braunii*.[339] One of the compounds, **861** contains an unusual methylenecyclohexane

ring attached to the linear part of the structure. The new oxidised steroids ***865–867*** were isolated from *Ulva australis*.[340]



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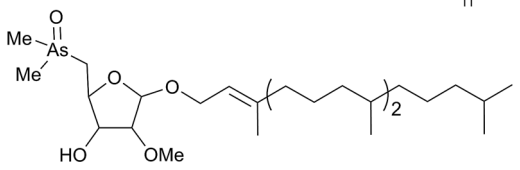
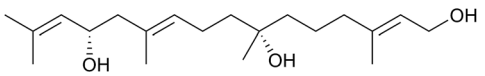


868

The first 2-*O*-methylriboside **868** discovered outside of RNA and containing arsenic was reported from the unicellular alga *Dunaliella tertiolecta*.[341] Two new sulfated diglycerides ***869***, ***870*** were isolated from *Codium dwarkense*.[342] 3-Hydroxy-4,7-megastigmadien-9-one ***871***, isolated from *Ulva pertusa*, has been shown to attenuate TLR-9 mediated inflammatory responses in murine bone marrow-derived dendritic cells.[343] The structure of the brominated monoterpene, 1-(*R*)-bromo-*ent*-maaliol isolated from *Neomeris annulata*,[344] has been revised to its C-4 epimer ***872*** based upon comparison of experimental and DFT calculated ^{13}C chemical shifts.[345] Research related to the diverse bioactivity of astaxanthin continues to appear in the literature and in the latest study, astaxanthin has been reported to have promising chemo-photothermal toxicity towards drug resistant tumours.[346]

5 Brown algae

Thirteen new compounds were reported from brown algae in 2017. A new triterpene carboxylic acid, padinolic acid ***873*** was isolated from *Padina boergesenii* from Oman.[347] Two new oleanene triterpenes ***874*** and ***875*** were isolated from *Sargassum wightii* and shown to be moderate inhibitors of PTP1B, an enzyme implicated with diabetes.[348] Two labdane diterpenes ***876*** and ***877*** were also isolated from *Sargassum wightii*.[348] Bifurcatriol **877** is a new linear antiplasmodial trihydroxyditerpene isolated from *Bifurcaria bifurcata*. The absolute configuration of **878** was determined from analysis of experimental and calculated Vibrational Circular Dichroism (VCD) spectra and calculation of ^{13}C-NMR shielding constants, the first time this combined approach has been used to determine the absolute configuration of a long chain linear structure.[349]



†**878**

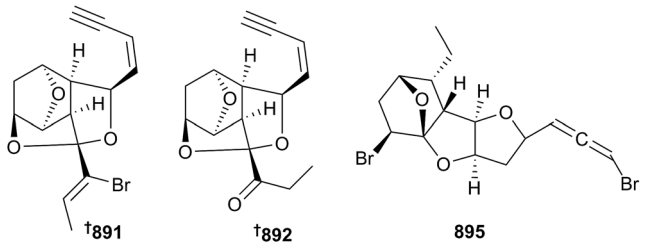
Two dolastane diterpenes ***879***, ***880*** were isolated from the Jamaican alga *Canistrocarpus cervicornis*.[350] A 3*R*/3*S* diastereomeric mixture of the meroditerpene, tetraprenyltoluquinone ***881***, ***882*** was isolated from *Cystoseira baccata*,[351] and a carotenoid metabolite, an isololiolide derivative, schiffnerilolide ***883*** has been reported from a Tunisian collection of *Cystoseira schiffneri*.[352] An Indian collection of *Sargassum wightii* was claimed to contain two unusual polyketide lactones, ***884***, ***885***.[353] Unfortunately the evidence provided both in the paper and in the ESI raises serious doubts about the true identity of these compounds.

Various known brown algal metabolites have been reported to have anticancer activities. The carotenoid pigment, fucoxanthin shows antiproliferative effects on non small cell lung cancer,[354] and inhibits the growth of colon cancer cells,[355] fucoxanthinol inhibits human colorectal cancer tumorigenicity,[356] tuberatolide B inhibits the growth of breast, lung, colon, prostate and cervical cancers.[357] Fucoxanthin also weakly inhibits monoamine oxidase A and B, prevents H_2O_2-induced neuronal apoptosis[358] and is antioxidant and antihypertensive.[359] Molecular modelling docking studies have helped to identify the binding mode of three very weakly active thrombin inhibitors pachydictyol A, isopachydictyol A and dichotomanol.[360] Indole-2-carboxaldehyde and indole-5-carboxaldehyde isolated from *Sargassum thunbergii* have been shown to inhibit adipogenesis.[361] The phlorotannins, eckol and dieckol moderately inhibit human monoamine oxidase A and B.[362] Dieckol also possesses chemopreventive activity in *N*-nitrosodiethylamine (NDEA)-induced hepatocarcinogenesis in rats.[363] Pretreatment of peripheral blood cells and macrophages with dolabelladienetriol led to the inhibition of HIV-1 replication.[364] The monoacylglyceride, monoolein and the carotenoid metabolite apo-9′-fucoxanthinone have been shown to possess anti-inflammatory activity.[365,366] The polyphenol, 6,6′-bieckol, shows angiotensin I-converting enzyme (ACE) inhibition leading to antihypertensive activity in rats.[367] Sargachromenol D shows potent vasodilatory and antihypertensive activity[368] The meroditerpenes sargahydroquinoic acid, sargachromenol and sargaquinoic acid show moderate inhibition of Protein Tyrosine Phosphatase 1B (PTP1B),[369] acetylcholine esterase (AChE), butyrylcholine esterase (BChE) and beta-secretase 1 (BACE1).[370] Sargahydroquinoic acid also reduced the production of Aβ1-42 in CHO-751 cells and therefore has potential as an inhibitor of neurotoxic Aβ peptide production.[371] Diphlorethohydroxycarmalol suppresses osteoclastogenesis by downregulating the RANK-NF-κB signaling pathway.[372] Saringosterol exhibits an anti-obesity effect by inhibiting the expression of adipogenic transcription factors and marker genes.[373]

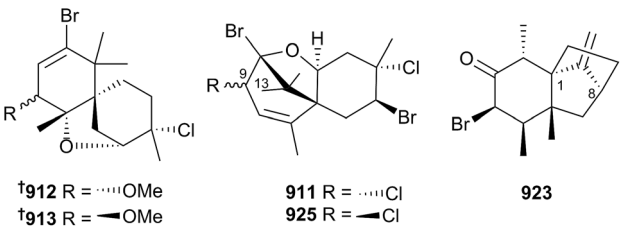
6 Red algae

There was an upsurge in the discovery of new NPs from red algae reported in 2017 with 63 new compounds reported in 14 papers. In addition, the structures of 19 previously reported red algae NPs were revised and one compound was reported from a marine source for the first time. A new linear pentachloromonoterpene ***886*** was isolated from *Plocamium cartilagineum*.[374] Although the paper claimed the compound to be a diastereomer of a compound also previously isolated from *P. cartilagineum,* it would appear this it is actually the (−)-enantiomer of this compound.[375] An unusual cyclic ether ***887*** has been reported from *Kappaphycus alvarezii*, however the NMR

and MS spectra reported for this compound do not support the proposed structure.[376] Two brominated C15-acetogenins, isolaurenidificin ***888*** and bromolaurenidificin ***889*** have been isolated from *Laurencia obtusa*.[377] Using an inhouse ^{13}C NMR database to dereplicate known marine derived mono-, sesqui- and diterpenes, sterols, C15-acetogenins and fatty acids, a new tetrahydropyran C15-acetogenin sagonenyne ***890*** was identified from a Corsican collection of *Laurencia obtusa*.[378] Three new tetracyclic C15-acetogenins, jeddahenynes A **891** and B **892**, both possessing a novel ring system and 12-debromo-12-methoxy isomaneonene A ***893***, were reported from a Red Sea collection of *Laurencia obtusa*.[379] A Japanese *Laurencia* sp. yielded two new allene containing C-15 acetogenins, hachijojimallenes A and B ***894***, **895**. Both compounds showed potent inhibition of barnacle larval settlemental at concentration >20 fold lower than they were toxic.[380]



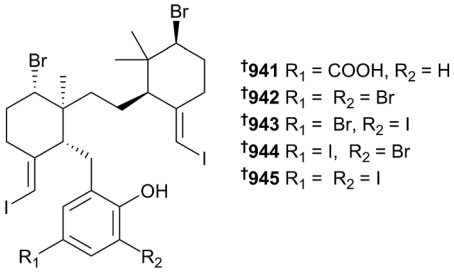
New brominated/chlorinated sesquiterpenes continue to be reported from red algae and include the cuparane, 8-deoxyalgoane ***896*** (*Laurencia natalensis*),[381] laurane dimers ***897–899*** (*Laurencia okamurai*)[382] and chamigranes, compositacins A–L ***900–911*** (*Laurencia composita*).[383] The structures of an additional two chamigranes, cycloelatanene A and B **912**, **913** have been revised through application of the crystalline sponge method.[384] Only 5 μg of **912** was required to determine its absolute configuration. Kutateladze and Reddy have developed a computational method to validate the structures of halogenated NPs by comparing parametric corrected DFT calculated ^{13}C NMR chemical shifts and spin–spin coupling constants with experimental data.[345] The method was used to validate the structures of 85 of 100 published halogenated MNPs and their derivatives. The method also permitted proposal of the full relative configuration for a further five red algae compounds ***914–918*** for which relative configurations were previously unassigned. Alternative structures for the 15 invalidated structures were proposed. The majority of the revised structures either have: a change in the relative configuration at one or two stereogenic centres (***914***, ***919–925***); the position of bromine and chlorine atoms within a molecule swapped (***926***); replaced a hydroxy with a hydroperoxy group ***927***; substituted a 3-iodo-1-hydroxyphenyl group with a 6-chloro-1-hydroxyphenyl group (***928***); or substituted a chlorine with a bromine atom (***929***). Although comparison of the experimental and calculated ^{13}C NMR data for the revised structures of compositacin L, **925** and 4-bromo-2,5,6-trimethyl-l 1-methylenetricyclo[6.2.1.0]undecan-3-one **923** had lower deviations to those calculated for the original structures, an intense ROESY correlation between H-9/H-13 in the ESI for the original study of compositacin L **911** and a NOE between H-2 and H-15 reported previously for 4-bromo-2,5,6-trimethyl-l 1-methylenetricyclo[6.2.1.0]undecan-3-one clearly support the original configurational assignments at C-9 in **911** and C-1/C-8 in 4-bromo-2,5,6-trimethyl-l 1-methylenetricyclo[6.2.1.0]undecan-3-one.[383,385] This new methodology clearly has its place for validating structures but MS and/or NMR correlated evidence should not be ignored, particularly when the root mean squared deviations between experimental and calculated data are relatively similar between original and revised structures.



The first total synthesis of the (+)-6*R* and (−)-6*S*-diastereomers of a 7*S*,9*S*,10*R* 6-chlorotetrahydrofuran acetogenin has allowed the relative and absolute configuration of the NP ***930*** isolated from *Laurencia glandulifera* to be determined. The NMR data for the (+)-6*R* isomer was identical to that of the NP while its specific rotation was opposite that of the NP indicating that the NP is the 6*S*,7*R*,9*R*,10*S* isomer.[386] Other new non-halogenated sesquiterpenes reported were the laurane ***931*** (*Laurencia* sp.),[380] and the monocyclofarnesols ***932***, ***933*** (*Laurencia snackeyi*),[387] while the relative configuration of the stereogenic centres in the known red algae metabolite palisadin A ***934*** also isolated from *Laurencia snackeyi* have now been defined.[387] A study to investigate the enzymes involved in the biosynthesis of sesquiterpenes in red algae has identified, through transcriptomics, three sesquiterpene synthases from *L. pacifica*. One of these enzymes (LphTPS-A) was expressed in yeast and this led to the production of the known sponge and liverwort sesquiterpene, prespatane ***935*** (also detected in *L. pacifica*). Application of the crystalline sponge X-ray method (on 5 μg) in combination with microscale NMR studies, resulted in the revision of the relative configuration at C-8 and determination of the absolute configuration as ***935***.[388]

New diterpenes were omaezol ***936*** and 11,12-dihydro-3-hydroxyretinol ***937*** (*Laurencia* sp.),[380] 13-acetyl pinnatol A ***938*** isoconcinndiol 13-acetate ***939***, and concinndiol 13-acetate ***940*** (ref. 389) (*Laurencia alfredensis*). Iodocallophycoic acid A **941**, and iodocallophycols A–D **942–945**, from *Calophysus* sp. are unique meroditerpenes since they contain both iodine and bromine.[390] The compounds contain two polysubstituted cyclohexyl groups bridged by a flexible ethyl linker and because of this, the determination of their relative configuration proved challenging. The problem was solved through the development of a DFT model to predict inter proton distances and these were then compared to distances calculated from NOEs. The absolute configurations of the compounds were then assigned through comparison of experimental and DFT computed ECD

spectra. The alga also contained bromophycoic acid F ***946*** and bromophycoic acid A methyl ester ***947***.[390]



†**941** R_1 = COOH, R_2 = H
†**942** R_1 = R_2 = Br
†**943** R_1 = Br, R_2 = I
†**944** R_1 = I, R_2 = Br
†**945** R_1 = R_2 = I

New polyether triterpenes intricatriol ***948*** (*Laurencia* sp.)[380] and alfredensinols A–C ***949–951*** (*L. alfredensis*)[389] were reported. This alga also contained three new oxygenated steroids, alfredensterol ***952***, 14α-hydroxy alfredensterol ***953*** and 3-deacetoxy alfredensterol ***954***.[389]

Six new citric acid derivatives aconitates A–F ***955–960*** were isolated from a Chinese collection of *Symphyocladia latiuscula*.[391] The alga also contained 12 bromophenol citric acid adducts, ten of which were new symphyoclidins H–Q ***961–970***. All were isolated as racemates and symphyoclidins H/I and J/K were isolated as inseparable *Z*/*E* mixtures.[391] Two additional bromophenols odonthalol ***971*** and odonthadione ***972*** were reported from *Odonthalia corymbifera*.[392]

The first total syntheses of avrainvilleol,[393] oxidised levuglandin D2,[394] similisines A and B[395] and (+)-intricenyne[396] have been reported. The first asymmetric total synthesis of laurenidificin has deduced that it possesses 6*R*,7*R*,9*R*,10*R*,12*R*,13*S* configuration.[397] An asymmetric total synthesis of isodehydrothyrsiferol has revealed that the majority of its chiral centres are enantiomeric with those of other squalene-derived thyrsiferol triterpenoids.[398]

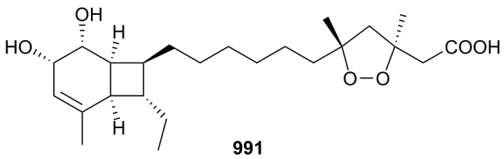
Y-Maze experiments have demonstrated that (+)-elatol isolated from *Laurencia dendroidea* is an attractant for the sea hare *Aplysia brasiliana*.[399]

7 Sponges

In 2017, 231 new or new to the marine environment compounds were reported from sponges, a slight increase over the number in 2016 (224) but still far below the rolling average over the past decade (277).[1] There is no question that the importance of microbiological samples in MNP investigations is growing every year, to the detriment of sponge-derived compounds; perhaps the heyday of phylum Porifera as the premier source of new MNPs is formally over?

A small number of glyceride ***973***,[400] ceramide ***974*** (ref. 401) and fatty acid lipids ***975–983*** (ref. 402) were reported from sponges. Given their normal abundance, new acetylenic compounds were less common than in previous years ***984–987***,[403,404] as were methylated lactones ***988–990***.[405,406] The synthesis of ***990*** was also achieved.[406]

Plakortinic acids A **991** and B **992** are inseparable endoperoxide-containing polyketides from the symbiotic two-sponge association between *Plakortis halichondrioides* and *Xestospongia deweerdtae* collected in Puerto Rico. These compounds are among the first examples of bicyclo[4.2.0]octene-containing NPs (with the only others also reported from sponges in 2017; see below) and given the relative configuration within this motif, they are likely to be formed chemically from disrotatory electrocyclic ring closure of a cyclo-octatriene precursor, itself likely formed from a octatetraene that could be formed from oxidation of the triene co-isolated with the new metabolites. The optical rotation of the mixture appears to be set by the remote chiral 1,2-dioxolane unit. The mixture also showed potent activity against two HTCLs.[407]



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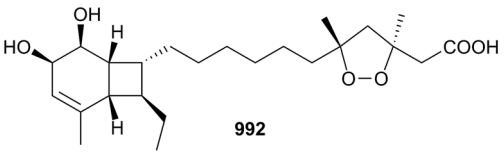
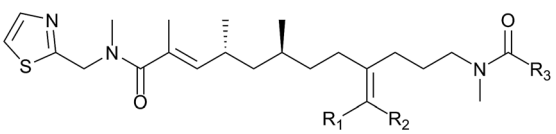
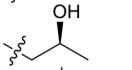


992

An Indonesian specimen of *Petrosaspongia* yielded biakamides A–D **993–996**, chlorinated thiazole-containing polyketide metabolites, isolated as two pairs of inseparable geometrical isomers. The relative configuration of the pendant methyl units was solved by spectroscopic means while the configurations of the secondary alcohols in **993** and **994** were determined using Mosher's method. The compounds' relative and absolute configurations were determined following total synthesis of all four compounds. The anti-proliferative activity of the four compounds against the PANC-1 pancreatic cancer cell line was assessed under normal and glucose-deficient conditions, indicating that the mode of action of these compounds involves the inhibition of complex I in the mitochondrial electron transport chain, with biakamides C **995** and D **996** being more potent than the other two congeners.[408]

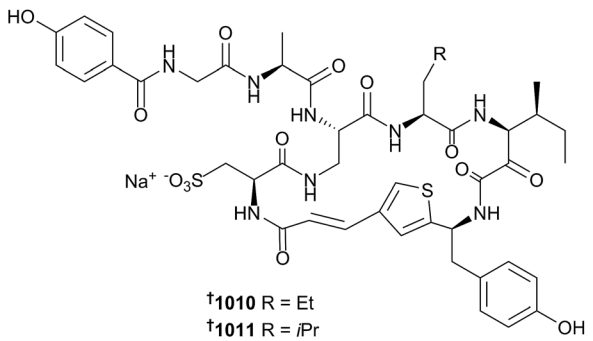


†**993** R_1 = Cl, R_2 = H, R_3 = 
†**994** R_1 = H, R_2 = Cl, R_3 = 
†**995** R_1 = Cl, R_2 = H, R_3 = 
†**996** R_1 = H, R_2 = Cl, R_3 = 

A small number of ethyl branched polyketides ***997–999*** (ref. 409 and 410) were reported in 2017, as were naphthoquinones ***1000***, ***1001*** (ref. 411) and a bisquinone ***1002***.[412] Somewhat surprisingly, only three reports of new sponge-derived peptidic

compounds were made in 2017, as sponges are normally a lucrative reservoir of such compounds. Peptides were isolated from *Daedalopelta* ***1003*** (ref. 413) and *Clathria* ***1004–1009*** (ref. 414) sponges.

A specimen of *Theonella swinhoei* collected in Madagascar (Dos de la Balieme), was the source of cyclotheonellazoles A–C **1010–1012**, with the isolation directed by the promising antiplasmodial activity noted for the extract, although this activity was finally ascribed to the co-isolated and commonly found swinholide A. The three new metabolites, however, all exhibited potent inhibition of chymotrypsin (IC_{50} = 0.62–2.8 nM) and extremely potent inhibition of elastase (IC_{50} = 34–100 pM). The elastase inhibitory activity was suggested to be due to complexation of the cyclotheonellazoles with Ser195 of elastase *via* their unusual 3-amino-4-methyl-2-oxohexanoic acid moiety.[415]

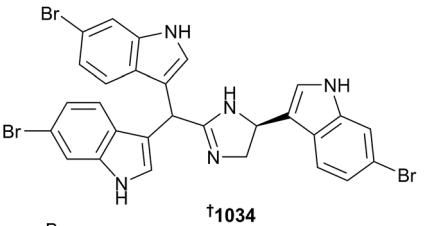


†**1010** R = Et
†**1011** R = *i*Pr
†**1012** R = Me

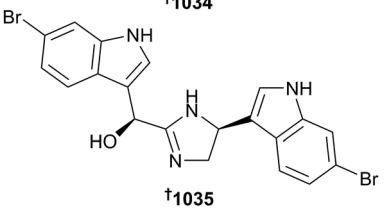
Equally surprising as the lack of new peptides, only one report of new macrolides ***1013***, ***1014*** was published in 2017.[416] Four new amino acid compounds ***1015–1018*** were reported from the sponge genera *Hymeniacidon*, *Lendenfeldia* and *Oscarella*; the synthesis of the last compound was also achieved.[417–419]

Pyrazole NPs ***1019–1021*** were reported from *Cinachyrella* sp.,[403] while a *Haliclona* yielded three new 3-alkylpyridinium alkaloids ***1022–1024***.[420] New manzamine-type alkaloids appeared in three separate reports ***1025–1032*** from *Acanthostrongylophora*, *Amphimedon* and *Lissodendoryx* sponges.[421–423] An Indonesian *Acanthostrongylophora* sponge was also the source of a new β-carboline alkaloid ***1033***.[424]

The sponge *Topsentia* sp., collected using a manned submersible at 140 m depth in Ulong Channel, Palau, yielded a series of new indole alkaloids. In particular, tulongicin A **1034** and dihydrospongotine C **1035** are tris- and bis-bromoindole alkaloids possessing strong antibiotic activity against Gram-positive, but no activity against Gram-negative bacteria. Additionally, both compounds show moderate inhibition of HIV infectivity yet were essentially inactive against two mammalian cell lines, giving a useful therapeutic index. The absolute configuration of both compounds was determined using comparison of calculated and experimental ECD spectra. Tulongicin A is the first compound containing a bis(6-bromo-1*H*-indol-3-yl) moiety linked to an imidazole core.[425]



†**1034**

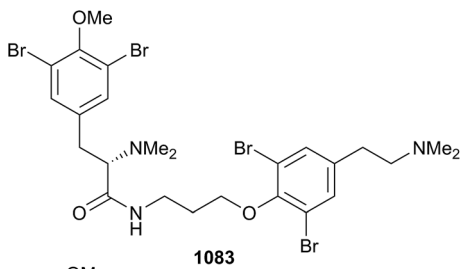


†**1035**

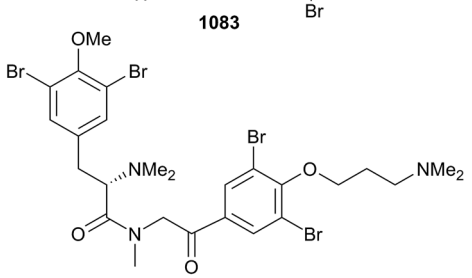
Additionally, a new imidazole ***1036*** was reported from *Dercitus japonensis*.[426] As always, guanidine and pyrrole motifs were common features of sponge derived compounds reported in 2017. These included cyano-containing metabolites ***1037***, ***1038***,[427] pyrrolo-amides ***1039–1041***,[428] enantiomeric pairs of pyrrolic amides ***1042–1053***,[409,429] and oroidin-type alkaloids ***1054***, ***1055***.[430] Sceptrin- ***1056–1058*** (ref. 431) and phakelin-type ***1059–1061*** (ref. 431–433) alkaloids were isolated from three collections of *Agelas* sponges, along with betaine ***1062***.[433] Reports of pentacyclic guanidine-type metabolites were made from *Monanchora pulchra* (Bering Sea; ***1063***, ***1064***)[434] and *M. unguiculata* (Madagascar; ***1065–1069***).[435]

Amphimedon sp. ***1070***, ***1071***,[436] *Iotrochota iota* ***1072–1078*** (ref. 437) and *Pseudoceratina verrucosa* ***1079–1082*** (ref. 438) were the sources of new bromotyrosines alkaloids.

In addition, anomoian B **1083** and aplyzanzine B **1084** are related bromotyrosine compounds isolated from *Hexadella* sp. and a two sponge association between *Jaspis* sp. and *Bubaris* sp., respectively; the sponge samples were collected from different locations in Indonesia. Anomoian B and aplyzanzine B were both found to induce apoptosis, which is a common antiproliferative activity for bromotyrosine alkaloids, however neither compound generated ROS nor inhibited histone deacetylase, two mechanisms of action associated with other bromotyrosine alkaloids. The absolute configuration of **1084**



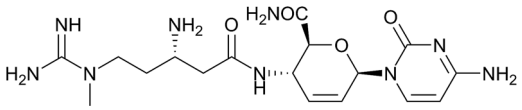
1083



†**1084**

was established by a combination of chemical degradation into known compounds along with optical rotation.[439]

A small number of purines were reported from *Haliclona cymaeformis* ***1085–1088*** (ref. 440) and *Clathria strepsitoxa* ***1089***, ***1090***.[441] A campaign to find new broad spectrum antibiotics highlighted a bioactive extract from the chemically rich sponge *Theonella swinhoei* (Anguar Island, Palau). Bioassay-guided isolation yielded compound P10 **1091**, an analogue of the known and commerically available antibiotic blasticidin S (also called blasticidine S) from *Streptomyces griseochromogenes*. The semi-synthesis of P10 from blasticidin S was also reported. A detailed investigation using genome sequencing of resistant *Staphylococcus aureus* and genome-wide assessment of barcoded *Escherichia coli* showed that P10 is 16-times more potent than the parent blasticidin S, and most surprisingly, that resistance to the drug was due to inactivation of the drug efflux pump NorA in *S. aureus*. This finding suggests that NorA can facilitate entry of peptidyl nucleosides into the cell, as well as its better characterised role in the efflux of common antibiotics such as the fluoroquinolones.[442]

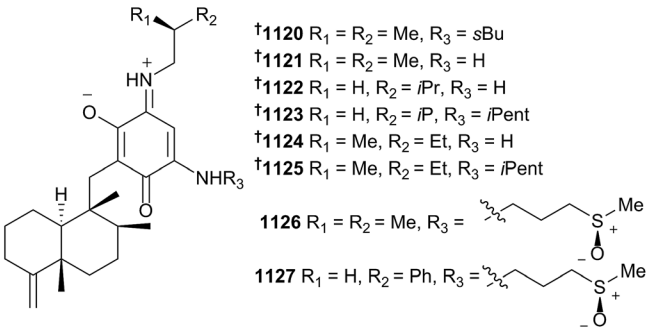


†**1091**

As always, new isoprenoid compounds were the dominant structural class of sponge metabolites, accounting for approximately half of the newly reported sponge compounds. Merosesquiterpenoids were isolated from the genera *Dactylospongia* ***1092***, ***1093***,[443] *Hyrtios* ***1094–1097***,[444,445] *Smenospongia* ***1098–1103*** (ref. 446 and 447) and *Spongia* ***1104–1107***,[448] ***1108–1119***.[449]

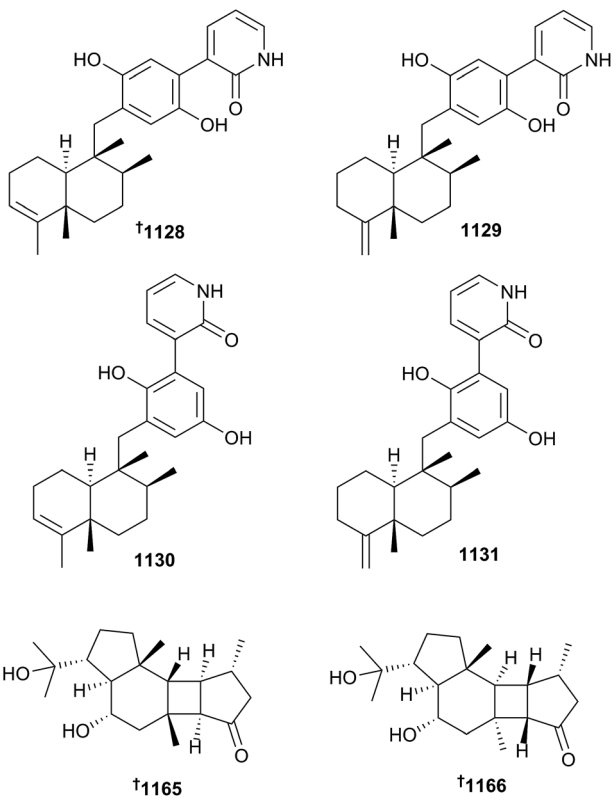
Naturally occurring blue pigments are rare and hence are desirable for the food and beverage industry. The blue hemisynthetic zwitterionic quinonoid derivative of ilimaquinone, a common sponge derived sesquiterpenoid quinone, was used as a MS probe to discover natural quinonoid-containing terpenoids. Screening for quinonoid terpenoids was achieved using molecular network matching of tandem MS fragmentation patterns obtained from LCMS analysis of sponge extracts using the online Global Natural Product Social Molecular Networking platform (GNPS).[450] This analysis highlighted that French Polynesian (Fakarava and Rangiroa islands) collections of *Dactylospongia metachromia*, a rich source of merosesquiterpenoids, contained several possible quinonoid candidates leading to the isolation of dactylocyanines A–H **1120–1127**. These compounds are the first naturally occurring blue, zwitterionic biscyanine pigments and all show a strong solvatochromic sensitivity. Hence their colour can be tuned according to the desired hue required by altering solvent polarity.[451]

Dysivillosins A–D **1128–1131** are anti-allergic avarol-type compounds from a South China Sea (Yongxing Island) *Dysidea villosa*. None of the compounds exhibit cytotoxicity to rat basophilic leukaemia cells, but all potently inhibit release of β-hexosaminidase, a marker of degranulation. All four compounds also down-regulate the production of various cytokines and pro-inflammatory biomarkers in stimulated RBL-2H3



mast cells. Further characterisation indicated that **1128** supresses Syk/PLCγ1 signalling to effect its potent anti-allergic activity. These compounds are the first terpene/polyketide/pyridine hybrids known.[452]

Adenine-containing meroditerpenoids ***1132–1134*** were reported from two species of *Agelas* collected in the South China sea,[409,429] while *Axinyssa variabilis* ***1135–1138*** (ref. 453) and *Lamellodysidea herbacea* ***1139–1143*** (ref. 454) yielded new sesquiterpenoids. Dolabellane ***1144***, ***1145***,[455] tricyclic ***1146***,[456] spongian ***1147–1158***,[457–459] and rearranged spongian diterpenoids ***1159–1164*** (ref. 460 and 461) were all reported in 2017. Investigation of a South China Sea (Xisha Islands) *Hippospongia lachne* yielded two stereoisomeric diterpenoids hipposponlachnin A **1165** and B **1166**. The relative configurations between these two unprecedented tetracyclo-tetradecane compounds suggest they are formed from a photochemical [2 + 2] cycloaddition reaction of a dolabellane skeleton. The absolute configuration of both compounds was solved by X-ray crystallography. Neither compound showed significant cytotoxicity but both suppressed



†**1128** **1129**

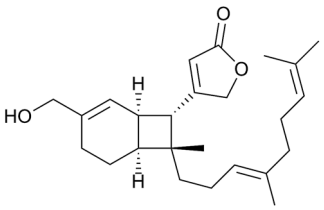
1130 **1131**

†**1165** †**1166**

β-hexosaminidase and hence are anti-allergic compounds, similar to the dysvillosins described above.[462]

Linear furanosesterterpenoids are commonly reported from marine sponges. New variants were isolated from *Ircinia* ***1167–1169***,[463,464] *Luffariella* ***1170***, ***1171*** (ref. 465) and *Psammocinia* ***1172–1174*** (ref. 466) sponges while surprisingly, scalarane-type sesterterpenoids were largely under represented with only one new phyllactone ***1175*** (*Phyllospongia papyracea*) reported.[467]

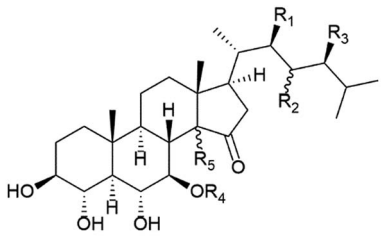
Hippolide J was isolated as a pair of enantiomers **1176**, **1177** in a non-racemic ratio from the sponge *Hippospongia lachne* (Yongxing Island, South China Sea). The enantiomers were separable by chiral HPLC with the dextrorotary form (**1176**) being the more potent antifungal compound against eight strains of hospital-acquired fungi, although both enantiomers exhibit MIC values in the sub μg mL^{-1} range. These compounds possess the bicyclo[4.2.0]octene skeleton that was unprecedented prior to 2017, although the plakortinic acids mentioned above also contain this motif. It is likely the hippolide J skeleton is formed from a photochemical [2 + 2] cycloaddition of a linear polyene structure. The absolute configuration of these compounds was established by comparison of calculated and experimental ECD spectra.[468]



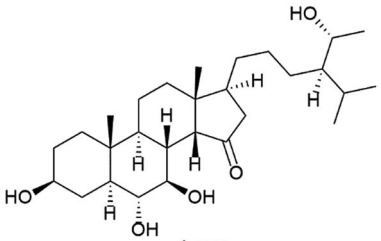
†**1176**
†**1177** *ent*

A number of steroid ***1178–1187***,[469,470] steroidal saponin ***1188–1194*** (ref. 471) and ring-contracted norsterones ***1195***, ***1196*** (ref. 472) were isolated from sponges of the genera *Crella*, *Dysidea*, *Petrosia* and *Poecillastra*. A Korean specimen of *Clathria gombawuiensis* was the source of gombasterol A–F **1197–1202**, heavily oxygenated sterols with four congeners having the unusual C/D *cis* ring fusion, yielding new members of the H-14β family of marine sterols. None of the compounds exhibited cytotoxicity against two HTCLs, nor did they show any antibacterial or antifungal activity. However, the compounds did enhance 2-deoxy-2-[(7-nitro-2,1,3-benzoxadiazol-4-yl)amino]-D-glucose uptake in differentiated adipocytes, which coupled with the lack of mammalian cytotoxicity, suggest these compounds are promising antidiabetic agents. The gombasterols also enhanced phosphorylation of AMP-activated protein kinase and acetyl-CoA carboxylase. The absolute configuration of the gombasterols was established using an exhaustive combination of NOE and J-based analyses, Mosher's method and X-ray crystallography. Additionally, the full absolute configuration of the co-isolated clathriol A **1203**,[473] including the previously undefined side-chain, was established using similar metholodology.[474]

The only reports of triterpenoid saponins were made from *Erylus goffrilleri* ***1204–1210*** (ref. 475) and an unidentified sponge from the Solomon Islands ***1211***.[476]



†**1197** R_1 = OH, R_2 = ◄OH, R_3 = Me, R_4 = H, R_5 = ◄H
†**1198** R_1 = OH, R_2 = ◄OH, R_3 = R_4 = H, R_5 = ◄H
1199 R_1 = OH, R_2 = ◄OH, R_3 = Me, R_4 = SO_3Na, R_5 = ◄H
†**1200** R_1 = H, R_2 = ····OH, R_3 = R_4 = H, R_5 = ◄H
†**1201** R_1 = OH, R_2 = ◄OH, R_3 = Me, R_4 = H, R_5 = ····H
†**1202** R_1 = H, R_2 = ◄OH, R_3 = Me, R_4 = H, R_5 = ····H



†**1203**

New bioactivities continue to be ascribed to old compounds. The pharmacokinetics of jaspine B (pachastrissamine) have been investigated and indicated that oral bioavailability is enhanced when complexed with bile acids.[477] X-ray crystallographic analysis has shown the binding of discodermolide to the taxane site of tubulin at 1.9 Å resolution, giving insight to the compound's mode of action.[478] A new assay designed to avoid detecting inherently fluorescent "nuisance" compounds has identified that halenaquinol sulfate inhibits human tyrosyl phosphodiesterase I (TDPI) with μM affinity for the enzyme. TDPI has been implicated in enhancing resistance to anticancer camptothecin derivatives and may hold promise in the clinic when used in conjunction with topoisomerase inhibitors.[479] A suggestion for the MoA of the arthropod moult inhibitor erebusinone *via* inhibition of the cytochrome p450 CYP315a1 has been determined using computational approaches,[480] while aaptane alkaloids have been shown to stimulate seedling growth in important agricultural crops.[481] A joint HR-ESI-MS study in conjunction with data mining of published bioactivity data has allowed a comprehensive analysis of the SAR related to the discorhabdin alkaloid family to predict the most important components of the pharmacophore;[482] additionally several discorhabdin congeners have been shown to be reversible inhibitors of various cholinesterases and hence may have potential as treatments for Alzheimer's disease.[483] The treatment of pain and epilepsy are potential outcomes from the discovery that oroidin-type alkaloids are modulators of the K_v1 subfamily of voltage gated potassium ion channels.[484] The merosesquiterpenoid metachromin A has been found to have nanomolar antiviral activity against hepatitis B *via* impairment of viral promoter activity,[485] while several furanoditerpenoids are new protein tyrosine phosphatase 1B inhibitors.[486]

The number of first total syntheses of sponge metabolites is similar in 2017 (51) to that reported in 2016 (53). Compounds

that have been synthesised include ancorinoside A,[487] diacarnoxide C,[488] and plakinidone C.[406] The structures of gracilioether I and mucosin have been disproven by synthesis but no alternatives have been proposed.[489,490] Jaspisin, isojaspisin and (*Z*)-narain have been synthesised,[491] as have smenothiazoles A[406,492] and B.[492] The peptides ciliatamides A and D (revised to ***1212*** and ***1213*** respectively),[493] euryjanicin E,[494] reniochalistatin E,[495] and theonellapeptolide Id[496] and the diaryl ether tedarene A have all been synthesised.[497] The absolute configurations of purpuroine A ***1214*** and nakirodin A ***1215*** have been revised following their synthesis,[498] while spiroleucettadine[499] and both madangamines C and E[500] have been made. Similarly, the signs of the specific rotation for natural hyrtioreticulins C and D have been reversed following their synthesis,[501] while the structure of topsentin C has also been revised to ***1216*** some 37 years after its initial report.[502] A large number of indole-containing alkaloids have been synthesised including makaluvamine O and batzelline D,[503] dictyodendrin G,[504] leucettamine C,[505] isonaamidine E,[506] and cylindradine B,[507] while both enantiomers of the bromotyrosine itampolin A have been produced.[508] Smenospongine A,[509] smenoqualone,[509,510] dehydrocyclospongiaquinone-1,[511] haterumadienone (done in a protecting group-free manner),[512] cheloviolenes A, B and dendrillolide C,[513] astakolactin (revised to ***1217***),[514] phorbaketal A,[515,516] alotaketals B, C and D,[516] and ansellones A, B and phorbadione[517] are all terpenoids that had syntheses published in 2017. One notable synthetic campaign was published by Kishi's group. The amazingly efficient and highly scalable zirconium/nickel mediated coupling of two halves of the halichondrin family has allowed for 1.07 g of halichondrin B to be prepared from only 1 g of D-galactal. In addition, the X-ray structure of halichondrin C was solved during this epic process.[518]

A chemo-ecological study explored chemical defence strategies within the "symbiotic" two sponge association between *Xestospongia deweerdtae* and *Plakortis simplex*. The feeding deterrent activity of extracts of each sponge was measured against three common coral reef predator fish species. Correlating the deterrent activity of associated and non-associated sponge specimens indicated that both members of the symbiosis benefit from the presence of the other through shared metabolic defensive chemistry, although it is suggested that *Plakortis* is more chemically defended than *Xestospongia* and only minimal translocation of metabolites is likely.[519]

The production of discodermolide in aquaculture studies of *Discodermia dissoluta* at two sites in the Colombian Caribbean using fixed or suspended culturing showed that production of the target compound was independent of both location and the conditions used, although a negative correlation with temperature was noted. Production of this important bioactive compound ranged from 20–270 μg g^{-1} of dry sponge.[520] In a similar vein, comparison of production of polycyclic guanidine compounds in wild and farmed *Crambe crambe* in the north western Mediterranean Sea found no difference between the populations, nor did other abiotic factors seem to influence metabolite levels. Conversely, seasonal effects were apparent.[521]

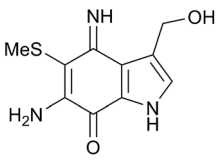
An independent detailed investigation of the absolute configuration of aurantosides G and J using computational approaches has validated the results suggested by total synthesis.[522] A metagenomic analysis of the microbial community associated with the chemically prolific sponge *Theonella swinhoei* has identified a new pathway to the keramamides, sponge-derived peptides containing the rare halohydroxytryptophan motif. In addition, a new flavin-dependent halogenase enzyme with potential biocatalytic applications was also identified as a key component of the biosynthetic pathway.[523] A further wide ranging study of a large number of Dysideidae holobionts using unbiased metagenome-mining and heterologous expression has shown that endosymbiotic cyanobacteria are responsible for the production of brominated biphenyl ethers in these Dictyoceratid sponges, one of the most prolific natural sources of such compounds in the environment.[524]

A LCMS-based metabolomics study of 28 sponge specimens collected from shallow water hydrothermal vents (25 m depth around Arnatnesstrýtur, Eyjafjordur, Iceland) was used to delineate chemical differences in taxonomically related sponges. In particular, three specimens were all identified as *Haliclona rosea* yet all three had different metabolic distributions. 3-Alkylpyridinium alkaloids were found to be the main drivers of differences in observed bioactivity in the sponge extracts. A new congener of the cyclostellettamine family was proposed based upon ion mobility MS data acquired in the time aligned parallel (TAP) fragmentation mode.[525]

NMR data for spongia-16-one,[526,527] dendrillol-1 and -2,[528–530] and aplyroseol-6,[531] have been reassigned using a 800 MHz spectrometer after inconsistencies between the legacy spectroscopic data for these common spongian diterpenoids was noted.[532]

8 Cnidarians

The 149 new compounds reported from cnidarians in 2017 represents an approximately 30% drop below the previous decadal average. While the structures of the majority of NPs isolated from cnidarians are derived from terpene biosynthesis, a handful of nitrogenous metabolites were reported in 2017, including the cytotoxic ceramide ***1218*** (*Cespitularia stolonifera*),[533] the cytotoxic iminoquinone macrophilone A **1219** (hydroid *Macrorhynchia philippina*),[534] and the 2-aminoimidazole alkaloids terrazoanthines A–C ***1220–1222*** from the zoanthid coral *Terrazoanthus onoi*.[535] Of note was macrophilone A **1219** which inhibited the small-ubiquitin-like modifier (SUMO) conjugation cascade, *via* an oxidative mechanism. The structure of **1219** was confirmed by synthesis.

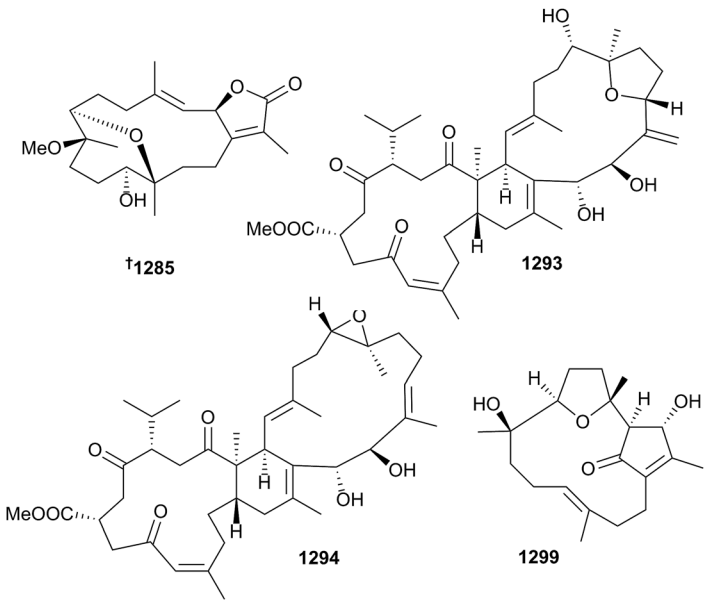


1219

Only a limited number of sesquiterpene-derived metabolites were reported from cnidarians in 2017, including ***1223***, ***1224*** (*Sinularia nanolobata*),[536] clavuridins A and B ***1225***, ***1226***

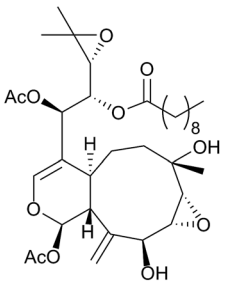
(*Clavularia viridis*),[537] parathyroidins E–G ***1227–1229*** (*Paralemnalia thyrsoides*),[538] a di-sesquiterpenoid rumphellolide J ***1230*** (*Rumphella antipathies*),[539] and some unusual guaiazulene alkaloids ***1231–1235*** and bis-sesquiterpenoids ***1236***, ***1237*** from *Muriceides collaris*.[540] The structure and absolute configuration of ***1226*** was secured by single crystal X-ray diffraction – the study also determined an error in the structure of a previously reported un-named variant, identifying it to be a stereoisomer of ***1226***. The six structures ***1232–1237*** were isolated as racemic mixtures, and were separated by chiral HPLC. Their absolute configurations were determined by comparison of calculated with experimental ECD spectra. Investigation of Madagascan soft corals afforded a range of metabolites including a cembrane diterpenoid ***1238*** (*Sarcophyton stellatum*), lipid ethyl esters ***1239***, ***1240*** and guaiane sesquiterpene ***1241*** (*Capnella fungiformis*).[541]

Of 12 assorted nor-diterpenes, and diterpenes, fragilolide A ***1242*** (*Junceella fragilis*),[542] chabrolin A ***1243*** (*Nephthea chabroli*),[538] xishacorenes A–C ***1244–1246*** (*Sinularia polydactyla*),[543] euplexaurenes A–C ***1247–1249*** (*Euplexaura* sp.),[544] and lobovarols A–E ***1250–1254*** (*Lobophytum varium*),[545] the xishacorenes promoted concanavalin A-induced T-lymphocyte proliferation, and the euplexaurenes exhibited moderate cytotoxicity. The latter study of the lobovarols also led to determination of the absolute configuration of the secondary alcohol in ***1255***, a metabolite previously reported from Indian Ocean specimens of *Lobophytum* sp. Just over 50 cembrane-related metabolites were reported from cnidarians in 2017. Amongst these were the mildly cytotoxic klyflaccicembranols A–I ***1256–1264*** (*Klyxum flaccidum*),[546] sarcophytrols G–U ***1265–1279*** (*Sarcophyton trocheliophorum*),[547,548] lobophylins F–H ***1280–1282*** and the anti-inflammatory lobophyolides A ***1283*** and B ***1284*** (both series from *Lobophytum crassum*),[549,550] and mildly cytotoxic sarcoehrenbergilids A–C **1285**, ***1286***, ***1287*** (*Sarcophyton ehrenbergi*).[551] Single crystal X-ray diffraction analysis was used to solve the structure and absolute configuration of sarcoehrenbergilid A **1285**. Further examples of cembranoids included sarelengans C–G ***1288–1292*** and bis-cembranoids sarelengans A **1293** and B **1294** (*Sarcophyton elegans*),[552] and cembranoids ***1295–1298*** (*Sarcophyton glaucum*).[553] The structures of **1293**, ***1289–1291*** were each secured by single crystal X-ray diffraction, while the latter study also led to revision of the structure of lobocrasol (*Lobophytum crassum*) to that shown (**1299**).

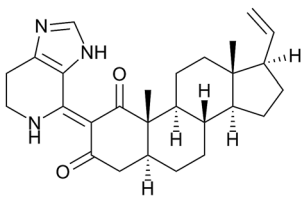


Twenty-one new briarane diterpenes were reported in 2017, including briarenols B–E ***1300–1303*** (*Briareum excavatum*),[554,555] and fragilolides B–Q ***1304–1319*** (*Junceella fragilis*).[542] The structure of fragilolide J ***1312*** was established by single crystal X-ray diffraction, with the same technique being used to establish absolute configuration of known co-metabolite frajunolide N (*J. fragilis*) ***1320***, which in turn secured the absolute configuration of ***1304***. Of five new chlorinated briaranes ***1321–1325*** (*Junceella fragilis*), ***1321*** and 2-deacetylpraelolide represent deacetyl derivatives of known cnidarian metabolite praelolide (*Menella praelonga*).[556] Acetylation of ***1321*** and known co-metabolite 2-deacetylpraelolide (fragilide J) afforded praelolide, the structure of which was confirmed by single crystal X-ray diffraction. The same study also led to the isolation of a mixture of 2-deacetyl- and 3-deacetyljunceelin, which were found to interchange during variable temperature NMR experiments. Cooling the mixture afforded crystals of 3-dacetyljunceelin ***1326***, X-ray diffraction analysis of which confirmed its structure and absolute configuration and by analogy that for 2-deacetyljunceelin, previously reported from *Junceella fragilis*. The remaining three examples of diterpenes, all possessing a xenicane skeleton, comprised cytotoxic fatty acid esters protoxenicins A **1327** and B ***1328*** (*Protodendron repens*)[557] and antibacterial xeniumbellal ***1329*** (*Xenia umbellata*).[558]

Cnidarians yielded a variety of sterols including polyhydroxysterol ***1330*** (*Xenia umbellata*),[558] pregnane ***1331*** (*Carijoa riisei*),[559] ergostane ***1332*** and pregnane glycoside ***1333*** (*Sinularia brassica*),[560] spinaceamine–pregnane hybrid scleronine **1334** (*Scleronephthya* sp.),[561] seco-sterols pinnisterols D–J ***1335–1341*** (*Pinnigorgia* sp.)[562] and ameristerenols A and B and ameristerol A ***1342–1344*** (*Pseudopterogorgia americana*),[563] seco-sterol ***1345*** and ergostane ***1346*** (*Pinnigorgia* sp.),[564] ergostane ***1347*** (*Palythoa caribaeorum*) and rearranged ergostane ***1348*** (*P. variabilis*),[565] and klyflaccisteroids K–M ***1349–1351*** (*Klyxum flaccidum*).[566] The structure of scleronine **1334**, which inhibited tumour cell migration, was confirmed and its absolute configuration determined by X-ray diffraction.[561]



1327



†**1334**

Further examples included cytotoxic sterols ***1352–1356*** from Vietnamese specimens of *Sinularia conferta*,[567] leptosteroid ***1357*** and an epoxy-gorgosterol ***1358*** (*Sinularia leptoclados*),[568] 16-deacetylhalicrasterol B ***1359*** (*Sarcophyton glaucum*),[553] columnaristerols B ***1360*** and C ***1361*** (*Nephthea columnaris*),[569] sinubrasones A–D ***1362–1365*** from cultured specimens of *Sinularia brassica*,[570] cytotoxic cholestane ***1366*** (*Lobophytum crassum*),[571] 4α-methylergosterol ***1367*** (*Nephthea columnaris*),[572] and withanolides sinubrasolides H–L ***1368–1372*** (*Sinularia brassica*).[573]

The structure of pubinernoid A (*Pinnigorgia* sp.) has been revised to that of the previously reported fused furanone (+)-loliolide ***1373***,[564] while the structure of an epoxyergostane, also reported from *Pinnigorgia* sp. has been corrected to a C-7 diastereomer ***1374***.[574]

A 35-amino-acid peptide containing two disulfide bridges Ms 9a-1, isolated from the venom of the sea anemone *Metridium senile*, produced significant potentiating effect on the transient receptor potential ankyrin-repeat 1 (TRPA1) implying utility as an analgesic or anti-inflammatory agent.[575] Crassicorin-I and putative homologue crassicorin-II were isolated from pharynx extracts of the anemone *Urticina crassicornis*.[576] Recombinant crassicorin-I was an antimicrobial peptide (AMP) exhibiting activity towards both Gram-positive and -negative bacteria, with its transcript being upregulated by immune challenge, implying a defensive role. There was sequence similarity with β-defensin fold neurotoxins, and crassicorin-I was indeed found to exhibit paralytic activity towards a crustacean. Other anemone neurotoxins were also found to act as AMPs.

APETx4, isolated from *Anthopleura elegantissima*, is a new inhibitor of the oncogenic ether-à-go-go voltage-gated potassium channel Kv 10.1, inducing cytotoxic and proapoptotic effects on various cell lines.[577] The membrane interaction exhibited by the pore-forming toxin sticholysin II (*Stichodactyla helianthus*) depends upon lipid head group properties.[578] A combination of proteomic analysis, of milked venom, and tentacle transcriptomics of *S. haddoni* has enabled positive identification of 23 families of putative toxins, 12 of which were new.[579] Further studies of recombinant HCGS-peptides from *Heteractis crispa* have identified them as being able to decrease cytoplasm Ca^{2+} levels in macrophages stimulated by histamine.[580]

First syntheses were reported for paralemnolide A (*Paralemnalia thyrsoides*),[581] (−)-pavidolide B (*Sinularia pavida*),[582] an un-named furanosesquiterpene (*Sinularia* sp.),[583] 7-acetylsinumaximol B (cultured *Sinularia sandensis*),[584] caribenols A and B (*Pseudopterogorgia* sp.),[585] and *rac*-verrubenzospirolactone (*Sinularia verruca*) and its proposed biosynthetic precursor capillobenzopyranol (*Sinularia capillosa*) have been achieved.[586] A biosynthetically-inspired conversion of capillobenzopyranol to verrubenzospirolactone supported this proposed biosynthetic relationship.[587] A new more efficient synthesis of stolonidiol (*Clavularia* sp.) facilitated investigation of the mechanism of potentiation of choline acetyltransferase reported for the NP, identifying that potentiation derives from stolonidiol binding to PKC.[588] An isopropyl/isopropylene truncated variant of the cubitane calyculone H maintains cytotoxicity towards a panel of HTCLs.[589]

Further biological studies have identified that sinulariolide suppresses human bladder cancer cell migration and invasion[590] and also inhibits LPS-induced murine bone marrow-derived dendritic cell maturation,[591] whilst sinularin induces DNA damage, phase arrest and apoptosis in hepatocarcinoma cells,[592] and 5-*epi*-sinuleptolide disrupts the actin cytoskeleton.[593] Leptolide improves insulin sensitivity in obese mice,[594] pseudopterosin inhibits NF-KB signaling,[595] a capnellene-diol derivative impairs vascular development in zebrafish,[596] a cholestane-monoacetate derivative inhibits human small cell lung cancer growth *in vitro* and *in vivo*.[597] Cembranes exhibited antifouling activity against bryozoan and barnacles,[598] the antiproliferative effects of cnidarian isoprenoids towards MCF-7 cells were noted and *ent*-deoxysarcophine was identified as being able to potentiate the activity of doxorubicin.[599] Pachycladin A deactivates EGFR signaling in tumour cells,[600] and lobocrassin B induces apoptosis and inhibits human lung cancer cell growth *in vivo*.[601]

Excavatolide B induces apoptosis in nonsmall cell lung cancer cells through ROS generation, inhibition of catalase and SOD activity and inhibition of Akt and NF-KB expression.[602] The same diterpene also inhibits osteoclastogenesis[603] and can modulate the electrophysiology and calcium ion homeostasis of rabbit atrial myocytes.[604] The cytotoxicity of *Sarcophyton* sp. extracts towards androgen-dependent and androgen-independent tumour cell lines does not correlate with diterpene levels as measured by NMR metabolomics methods.[605]

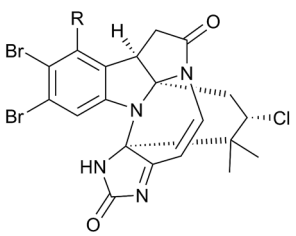
A new source of palytoxin and related congeners has been identified as the Atlantic zoanthid coral *Palythoa canariensis*.[606] Exposure of *Sarcophyton glaucum* to prostaglandin E1 and methyl jasmonate elicited increased production of campestene-triol and cembrane metabolites, while exposure to arachidonic acid or geranylgeranyl pyrophosphate or physical wounding failed to

alter the metabolomics profile.[607] The soft coral *Lobophytum pauciflorum* was less responsive to elicitation. Exposure of juvenile and adult coral *Acropora millepora* to hyposaline conditions led to increased dimethylsulfoniopropionate (DMSP) levels and upregulation of candidate genes associated with production of DMSP *via* an alga-like pathway.[608] The genes associated with biosynthesis of the blue pigment biliverdin IXα present in the blue coral *Heliopora coerulea* have been identified,[609] and furanosesquiterpenes from the Mediterranean soft coral *Maasella edwardsi* can act simultaneously as toxins, inducers of avoidance-learning and as aposematic odorant cues.[610]

9 Bryozoans

Few compounds are usually reported from this understudied phylum and in fact, in 2016, no new metabolites were reported but in 2017 there was a remarkable upswing, with 16 new compounds isolated. The indole alkaloid, 2,6-dibromo-*N*-methylgramine ***1375*** was obtained from *Amathia verticillata*,[611] while sterol ***1376*** and ceramides ***1377***, ***1378*** were isolated from *Cryptosula pallasiana*.[612] *Schizomavella mamillata* was the source of 5-alkylresorcinol derivatives, schizols A–F ***1379–1384***, with ***1379*** and ***1380*** possessing high radical scavenging activity. Schizol A ***1379*** was synthesised from 3,5-dimethoxybenzaldehyde.[613] The Arctic species *Securiflustra securifrons* yielded a number of halogenated alkaloids including securidine A ***1385*** (ref. 614) and securamines H **1386** and I **1387**. The latter two are hexacyclic indole-imidazole alkaloids that contain both bromine and chlorine. An analogue, securamine J was also isolated but shown to be an artefact formed *via* methanol addition to a NP, the exact structure of which is yet to be determined. When securamines H **1386**, I **1387** and co-isolated securamines C and E were dissolved in methanol, they were converted to adducts but the NPs were reformed when these artefacts were dissolved in a non-nucleophilic solvent.[615]

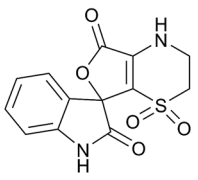
Terminoflustrindoles B ***1388*** and C ***1389*** were obtained from *Terminoflustra membranaceatruncata* along with the known alkaloid, terminoflustrindole A. Terminoflustrindole A was cytotoxic to both tumour and normal mammalian cell lines. LC-MS analyses of *T. membranaceatruncata* colonies indicated that these alkaloids are most highly concentrated at the base of the colonies, implying that their biological role is not to protect embryos, a potential role postulated for alkaloids in other species.[616]



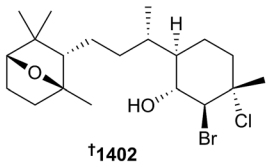
1386 R = Br
1387 R = H

10 Molluscs

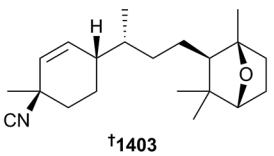
The number of new metabolites reported in 2017 from molluscs (38) is an increase on the average number reported per year over the past decade. Four new odd-chain fatty acids ***1390–1393*** were isolated from the ovaries of the limpet *Cellana toreuma*,[617] with the structures of ***1390*** and ***1392*** being confirmed by synthesis.[618] Australian samples of the keyhole or slit limpet *Scutus antipodes* afforded scutinin A ***1394*** a hexaester of D-sorbitol, and scutinin B as a 3 : 2 mixture of epimers ***1395*** and ***1396***.[619] The structures of scutinins A and B were confirmed by synthesis and antibacterial and antifungal activities reported. Bivalve molluscs were the sources of racemic spiro-indolothiazine orbicularisine **1397** (*Codakia orbicularis*)[620] and sterols ***1398*** and ***1399*** (*Paphia malabarica*).[621] Two tetrahydropyrans monodontins A ***1400*** and B ***1401***, the latter of which exhibited weak cytotoxicity, were reported from the Vietnamese top snail *Monodonta labio*.[622] The venerable opisthobranch *Dolabella auricularia* was the source of a bromo-chloro-diterpenoid dolabellol A **1402**, the structure and absolute configuration of which was secured by X-ray diffraction analysis.[623] Three new isonitriles, pustulosaisonitriles-1 **1403**, -2 ***1404*** and -3 ***1405*** were isolated from 14 specimens of the nudibranch *Phyllidiella pustulosa*.[624] Absolute configuration was assigned to the cyclohexene subunit of **1403** by NOESY in combination with TDDFT-ECD calculations of structurally-simpler model compounds, while stereocontrolled synthesis established the absolute configuration of the complete molecule. The isolated NP **1403**, synthesised NP and a stereoisomer all exhibited similarly moderate levels of antimalarial activity *in vitro*.



1397



†**1402**



†**1403**

A Bay of Naples collection of the opisthobranch *Spurilla neapolitana* afforded the cyclohexenyl terpenoid spurillin A ***1406*** while a Patagonian collection of *Spurilla* sp. gave the farnesol derivative ***1407***, previously known as a synthetic product, and diterpene spurillin B ***1408***.[625] Both mollusc samples contained the known laevorotatory alkaloid bursatellin. None of the four metabolites were detected in extracts of sea anemones *Aiptasia diaphana* or *Parabunodactis imperfecta* that *S. neapolitana* or *Spurilla* sp., respectively, were associated with in the wild. Secogorgosterol ***1409***, methylsarcoate analogue ***1410***, 2*R*-isosarcophine ***1411***, bisepoxide ***1412*** and isobisglaucumlides B ***1413*** and C ***1414*** were isolated from extracts of an Australian collection of the nudibranch *Phyllodesmium longicirrum*.[626] Of the new MNPs, ***1412*** exhibited feeding deterrent activity

towards the tropical puffer fish *Canthigaster solandri*. Diacylguanidine actinofide ***1415*** was reported from skin tissue of the dorid nudibranch *Actinocyclus papillatus*.[627] A straight forward synthesis of ***1415*** was expanded to include the preparation of a range of analogues. These compounds were evaluated against a panel of six HTCLs, and actinofide and the known related diacylguanidine dotofide (*Doto pinnatifida*) were moderately antiproliferative. Three new ulapualide congeners, C–E ***1416–1418*** were isolated from egg masses of the nudibranch *Hexabranchus sanguineus* collected in Oahu, Hawaii.[628] The structures represent dihydro or desmethyl analogues of ulapualides A and B, with close similarity in ^{13}C chemical shifts suggesting they share common stereochemical configuration. Ulapualide C was typically 2–4-fold less active than ulapualides A and B towards a panel of 4 HTCLs. Investigation of the chemistry of a single specimen of the nudibranch *Goniobranchus collingwoodi* has revealed that the new spongian-16-one analogues ***1419–1424*** are located in only the mantle tissue.[629] Bioinformatic analysis identified a new family of linear cationic AMPs, the myticalins, present in mussels of the genus *Mytilus*.[630] Transcriptomic analysis identified preferential expression in gill tissue, with six of seven solid-phase synthesised peptides exhibiting moderate to strong broad spectrum antimicrobial properties. Interestingly, no activity was observed for these peptides against *Vibrio anguillarum*. This lack of activity was hypothesised to be associated with either the elevated NaCl concentration needed to grow the bacterium that is known to reduce binding of AMPs to bacteria, or possibly from an intrinsic resistance mechanism as *Vibrio* sp. are typically hosted by mussels.

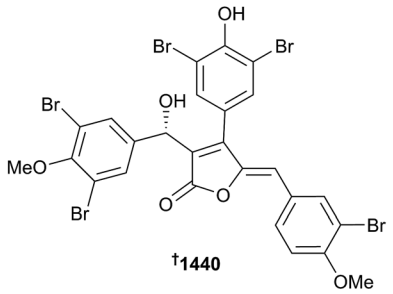
UVA wavelength-dependent photochemical conversion of dactylone into the brominated sesquiterpene aplydactone has been studied more closely.[631] The study also reported the conversion of another *Aplysia dactylomela* metabolite 10-*epi*-dactylone into a single product, 8-*epi*-isoaplydactone which should now be considered an anticipated NP. Total synthesis of dolastatin 16 has been reported, and its potent antifouling activity towards barnacle cyprids confirmed.[632] Three studies reported on new biologically active analogues of the dolastatin 10 peptide scaffold.[633–635] Novel tambjamine analogues have been shown to activate the p38 MAPK pathway leading to *in vitro* and *in vivo* activity towards lung cancer cell lines.[636] An extract derived from the hypobranchial gland of *Dicathais orbita*, and 6-bromoisatin and analogues found in this gland, exhibit anti-inflammatory activity in *in vitro* and *in vivo* models.[637,638] A series of halogenated *N*-methylpyrroles were synthesised and used as standards to quantify their presence in European Atlantic, North Sea and Baltic Sea specimens of *Mytilus* sp.[639] Exposure to okadaic acid has been found to reduce the antioxidant and non-specific immune responses of bay scallops *Argopecten irradians*.[640] Feeding of a dinophysitoxin-producing microalga (*Prorocentrum foraminosum*) to Gray's mussel (*Crenomytilus grayanus*) led to accumulation of dinophysitoxin-1 in the digestive gland and gills while dinophysitoxin-3 levels were highest in the digestive gland.[641] Feeding two strains of the dinoflagellate *Alexandrium tamarense* to uncontaminated mussels *Mytilus galloprovincialis* led to detection of known and new paralytic shellfish toxins related to saxitoxin.[642] The roles and substrate selectivity of *Octopus vulgaris* proteins Scd and Evol4 as fatty acid desaturase and elongase enzymes, respectively, have been studied.[643]

The cone snail *Conus pulicarius* was the source of the moderately cytotoxic steroidal glycosides ***1425–1427***.[644] The synthesis, 3D structure and antiproliferative effects of conotoxin Φ-MiXXVIIA (*C. miles*), a member of the new G2 toxin superfamily have been reported.[645] Fifteen-residue C-amidated conorfamide-Sr3 (*C. spurius*) blocks activity of *Shaker* subtype voltage-gated potassium channels.[646] Two hormone-like peptides related to bee brain prohormone-4 and invertebrate neuropeptide elevenin were isolated from *C. victoriae*,[647] and two 17-residue peptides including anti-mollusc active pn4c were purified from the venom of *C. pennaceus*.[648] *Conus betulinus* venom was the source of N-terminus pyroglutamamide-derivatised contryphan-Bt, which exhibited 'stiff-tail' syndrome in mice,[649] while transcriptomics of the venom identified six toxin sequences that were subsequently synthesised and found to exhibit insecticidal activity towards the mealworm.[650] Anti-parallel dimeric *N*-terminal domain peptides related to α_D-conotoxin GeXXA (*C. generalis*) are antagonists of nAChR, acting by a 'lid-covering' mechanism, which is distinct from other neurotoxins that bind to the interface between extracellular domains or are pore blockers.[651] Venom of *C. generalis* was also the source of O-conotoxin GeXXVIIA which in its native form is a disulfide-linked homodimer.[652] The linear peptide of the toxin exhibited potent inhibition of human nAChR. Turripeptide ubi3a, a 17-residue peptide isolated from the venom of the Turridae gastropod *Unedogemmula bisaya* belongs to the M-superfamily of conotoxins and incorporates unusual disulphide connectivity in its cysteine framework III scaffold.[653] Weak inhibition of nAChR was observed. Molecular dynamic computational studies suggest that examples of conotoxins could act as antagonists by binding to the extracellular region of the lysophosphatidic acid receptor 6 (LPAR6).[654] Recombinant *Conus* protein disulphide isomerase catalyses disulphide bond formation in linear lt14a, a 13-residue peptide that includes four cysteines, affording a mixture of four single disulphide bond-containing peptide products.[655]

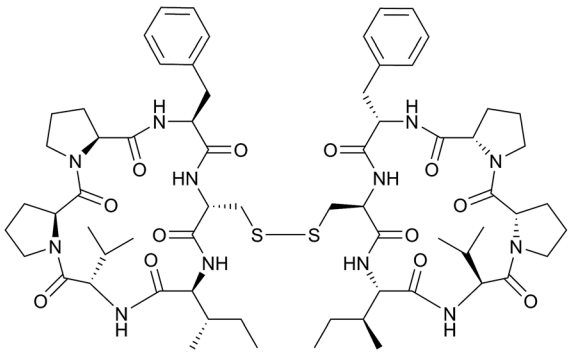
11 Tunicates (ascidians)

The 27 new tunicate-derived NPs presented in this review is about 15% lower than the average number reported per annum over the last decade.

The metabolites reported in 2017 included carbamates ***1428–1431***, ureas ***1432–1435*** and isoquinoline ***1436*** (*Didemnum molle*),[656] isoquinoline-quinones ***1437–1439*** (*Ascidia virginea*),[657] antibacterial cadiolides J–M **1440**, ***1441–1443*** (*Pseudodistoma antinboja*),[658] new congeners in the mandelalide series ***1444–1450*** (*Lissoclinum mandelai*),[659] cytotoxic bistratamides M and N ***1451***, ***1452*** (*L. bistratum*),[660] and cyclic hexapeptide dimers antatollamides A **1453** and B ***1454*** (*D. molle*).[661] The absolute configuration of the constituent amino acids of antatollamide A were secured by making use of a new tryptophan-based chiral derivatisation reagent.



†1440

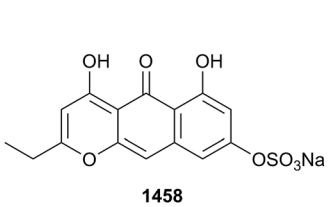


†1453

Strong Gram-positive antibacterial activity associated with the cadiolide family of butenolides isolated from *Pseudodistoma antinboja* has been reported.[658] As with a number of other rubrolide/cadiolide-type MNPs, cadiolide J was characterised as a Δ^4 mixture of *Z* (major):*E* (minor) isomers. The authors also reported X-ray crystallographic analysis of co-metabolite cadiolide H (desbromo cadiolide J) which, with supporting ECD data and Mosher's derivatisation, established absolute configuration at the secondary alcohol as 6*S*. Also of note was the continued investigation of biological activity of mandelalide macrolides, prompted by the isolation and characterisation of six new family members.[659] Complemented with synthesis, these studies revealed preliminary aspects of a SAR and that the class can act as inhibitors of mitochondrial ATP synthase. The finding that antiproliferative potency observed in biological assays depends upon the oxidative phenotype of the cell line in question, and that this phenotype can vary with cell density, provides an explanation for the previously reported disparities noted for synthetic-sourced mandelalide A. Antatollamides A **1453** and B **1454** are unusual dimeric cyanobactins, apparently the first such examples that embody intermolecular dimerisation through cysteine.[661] The two structures differ in the *S-trans*/*S-cis* configurations of the proline residues.

Two groups reported asymmetric syntheses of diyne-containing 1,2-amino alcohol lipid distaminolyne A (*Pseudodistoma opacum*) – one group suggested revision of absolute configuration to 2*R* was required,[662] while the second group supported the original 2*S* assignment.[663] Unfortunately both groups relied upon comparison of low magnitude optical rotation values to make their respective conclusions, rather than preparation, and ECD analysis, of the corresponding *N*,*O*-dibenzoyl derivatives. Further work is required to clarify these findings. The structures of a number of NPs have been confirmed by total synthesis including halocyamine A,[664] biselides A[665] and E,[666] ningalin G,[667] (+)-arborescidine C,[668] and (−)-pseudodistomin E.[669] Absolute configuration was assigned to the phosphorylated polyketide phospoeleganin (*Sidnyum elegans*) by synthesis of model fragment alicyclic compounds and their cyclic derivatives, and comparison of ^{1}H and ^{13}C NMR chemical shifts.[670] Improved semisyntheses of ecteinascidin 743 and (−)-jorumycin from safracin B have been reported.[671] The absolute configuration of (+)-lepadiformine C (*Clavelina moluccensis*), established by enantioselective synthesis of both enantiomers, has an enantiomeric core to co-metabolites lepadiformines A and B, suggesting that the ascidian is capable of enantiodivergent biosynthesis.[672] Cytotoxic activities were also reported for the NP and related analogues. Rubrolides and analogues were found to inhibit NO production by LPS-stimulated macrophages.[673] 6-Azaindole analogues related to meridianin G inhibit the kinase Dyrk1a and increased nucleus residence time for nuclear factor of activated T-cells, with implications for the treatment of muscular dystrophy.[674] Virtual screening has identified the meridianins as potential templates for the development of treatments for Alzheimer's disease.[675] Voltammetry and computational studies suggest biologically active thiazinoquinones, such as thiaplidiaquinones and conicaquinones, undergo single electron reduction to semiquinone radical species which can be either reduced or oxidised depending upon the protonation state.[676] SAR studies exploring the anti-*Plasmodium falciparum* and farnesyltransferase inhibiting properties of thiaplidiaquinones A and B and analogues have been reported.[677] The lissoclimides, cytotoxic succinimide-containing labdane diterpenoids, act as protein synthesis inhibitors.[678] The molecular basis of this inhibition was revealed by X-ray analysis of a co-crystal between chlorolissoclimide and the eukaryotic 80S ribosome and was further explored by biological evaluation of a library of analogues and by computationally modelling their interactions with the ribosome. Benzotrithioles and their sulfoxides related to varacin demonstrate less potent cytotoxicity than the MNP but also act to inhibit colony formation of tumour cells exposed to EGF tumour promoter.[679] Lissoclibadins, polysulfur aromatic alkaloids, also related to varacin, exhibit moderate cytotoxicity to HTCLs including HCT-15, promoting apoptosis through a caspase-dependent pathway.[680] *In vivo* activity against HCT-15 cells in mice was observed for lissoclibadin 1. Intragastrically administered dosing of astaxanthin at 100 mg kg^{-1} led to significant inhibition of subcutaneous PC-3 cells in a murine *in vivo* model.[681] Under oxidative conditions, 1,2-dehydro-*N*-acetyldopamine, a structurally simplified model of tunichrome MNPs, forms adducts with *N*-acetylcysteine or oligomeric products.[682,683] The conformation of novel ascidiacyclamide analogues, containing substitutions replacing the oxazoline-subunit, have been studied by NMR, X-ray diffraction and ECD spectroscopy.[684] Proline- and piperidine-containing analogues exhibited cytotoxicity. A fluorescently-tagged patellamide cyclic peptide is uptaken passively by *Prochloron* sp. cells and forms Cu^{2+} complexes, as determined by confocal microscopy and flow cytometry.[685] The antimicrobial activity of clavanin A, a 23-residue antimicrobial peptide, is enhanced by the presence of Zn^{2+} over other cations. Swapping out certain

histidine residues in clavanin A abrogates its antimicrobial activity. Its antimicrobial activity is partially associated with depolarisation of bacterial lipid membranes, but the clavanin–Zn^{2+} complex has also been shown to cleave DNA.[686] Molecular dynamics has been used to investigate the binding of didemnin B to the domain I/III interface of translation elongation factor (eEF1A1), providing a rationalisation of the binding and also why A399V replacement in the protein leads to a loss of affinity and hence drug resistance.[687] Bacterial symbionts typically undergo genome reduction as they adapt to their new host. However, in the case of bacteria from the phylum *Verrucomicrobia* associated with the mandelalide-containing ascidian *Lissoclinum* sp. (*mandelai*), the symbiont maintains seven copies of the PKS pathway for the mandelalides accounting for 19% of the genome and 25.8% of the coding capacity.[688] The authors conclude the genes are "under strong purifying selection and are important to the symbiotic relationship". Trabectedin (ecteinascidin 743) exhibits *in vitro* and *in vivo* activity towards clear cell sarcoma cell lines,[689] while phase I trial data for plitidepsin (Aplidin®) in combination with sorafenib or gemcitabine shows them to be manageably safe with some objective responses observed.[690] Pharmacokinetic experiments with ^{14}C-plitidepsin show that 77.4% is excreted over 20 days, mainly in faeces, and that higher levels were detected in whole blood compared to plasma, indicating that red blood cells are the major distribution compartment.[691]



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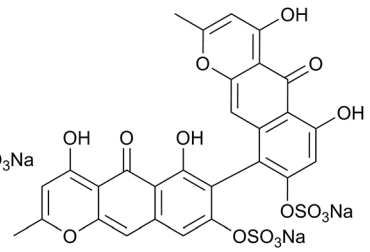


1459

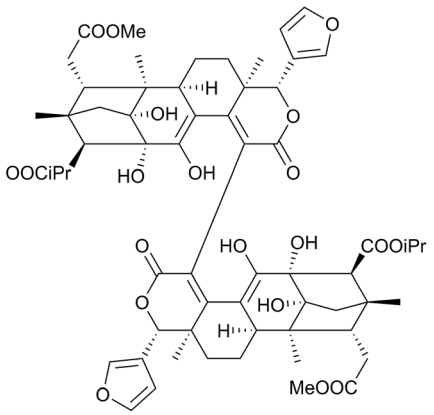
12 Echinoderms

The 36 new metabolites reported from echinoderms in this review is about average for the number reported per annum over the last decade. Chamigrene sesquiterpenes isoobtusadiene ***1455*** and its acetate ***1456*** were isolated from the brittle star *Ophionereis reticulata*.[692] This compound class is typically reported from red algae, suggesting a dietary source for the brittle star. Phenolic sulfate monomers ***1457***, **1458** and dimers **1459**, ***1460***, from the crinoid *Alloeocomatella polycladia* exhibited weak (former two) to moderate (latter two) inhibition of HCV NS3 helicase.[693] MNPs ***1458*** and ***1459*** were only weakly active in an anti-HCV assay, suggesting a lack of correlation between whole cell antiviral activity and helicase inhibition.

The remaining metabolites reported from echinoderms included steroidal glycosides pentacerosides A ***1461*** and B ***1462*** (*Pentaceraster gracilis*),[694] regulusosides S1 ***1463*** and S2 ***1464*** (*P. regulus*),[695] pentaregulosides A–G ***1465–1471*** (*P. regulus*),[696] and granulatoside C ***1472*** (*Choriaster granulatus*),[697] all isolated from starfish, as well as sea cucumber-sourced triterpene glycosides nobiliside D ***1473*** (*Holothuria nobilis*),[698] stichorrenosides A–D ***1474–1477*** (*Stichopus horrens*),[699] magnumosides A_1–A_4, B_1–B_4, C_1–C_4 ***1478–1489*** (*Neothyonidium* (=*Massinium*) *magnum*),[700,701] and fallaxosides B_1 and D_3 ***1490***, ***1491*** (*Cucumaria fallax*).[702] Nobiliside D ***1473*** showed strong cytotoxicity towards a panel of six HTCLs,[698] while magnumoside C_4 ***1489*** enhanced the antiproliferative effects of γ-radiation on DLD-1 human colorectal adenocarcinoma cells.[700] Biological evaluation of carbohydrate conjugates of the sea urchin quinonoid ethylmompain identified analogues with enhanced cytotoxicity and the ability to inhibit urchin spermatozoids.[703] The total synthesis of the triterpene glycoside echinoside A has been reported, using an adaptable synthetic route that allowed preparation of desulfated echinoside A and the closely related echinoside B.[704] Surface plasmon resonance spectroscopy was used to assess the ability of starfish-derived lanosterol analogues to interact with human and fungal 14α-demethylase (CYP51).[705] Two examples, henricioside H and levisculoside G, were confirmed as modest inhibitors in a purified enzyme assay. In other biological testing, frondoside A exhibits synergistic effects against acute leukaemia cell lines in the presence of conventional anticancer drugs,[706] semi-purified triterpene glycoside extracts exhibit antifouling activity in coated-plate sea trials,[707] intraperitoneal administration of cucmarioside A_2-2 leads to an increase in markers associated with spleen macrophage activation,[708] luzonicoside A was found to be more potent than luzonicoside D at inhibiting proliferation of human melanoma cells *in vitro* by cell cycle regulation and induction of apoptosis,[709] anthraquinones from *Comanthus* sp. exhibited cytotoxicity and were strong kinase inhibitors,[710] and (*Z*)-2,3-diphenylacrylonitrile (*Holothuria parva*) caused an increase in ROS generation and collapse of the mitochondrial membrane potential in hepatocellular carcinoma cells.[711] Purified spinochromes, naphthoquinones from urchins, exhibit wide ranging activities against bacteria, as antioxidants, as pro-inflammatory agents and as cytotoxins.[712] Mature eggs of the sand dollar *Scaphechinus mirabilis* contain a jelly coat bearing pigment cells which contain spinochromes D and E, and to a lesser degree, spinochrome A and echinochrome A.[713] Eight of the core triterpenoid biosynthetic enzymes were identified in cell wall tissue of the sea cucumber *Holothuria scabra*,[714] while Δ^5 and Δ^8 fatty acyl desaturases were identified in the sea urchin *Paracentrotus lividus*.[715] Metabolism of triterpenoids holothurin A and echinoside A, both by incubation with gut microflora (*in vitro*) or *in vivo* in rats, identified deglycosylation to be the dominant process.[716] The profiles of triterpenoid glycosides in five different body components (respiratory trees, body walls, gonad tubules, guts, and aquapharyngeal bulbs) of the sea cucumber *Eupentacta fraudatrix* were found to be qualitatively similar but with some variability in minor compounds.[717] Transcriptome sequence data for three brittle star species (*Ophionotus victoriae*, *Amphiura filiformis*, *Ophiopsila aranea*) identified a number of neuropeptide precursor proteins.[718]

13 Mangroves

Mangroves or their associates were the sources of benzaldehyde ***1492*** (*Rhizophora mangle*),[719] sesquiterpenes ***1493***, ***1494*** (*Rhizophora annamalayana*),[720] terpenoids ***1495***, ***1496*** (*Rhizophora mucronata*),[721] diterpenes ***1497–1499*** (*Ceriops tagal*),[722] ***1500*** (*Wedelia prostrata*),[723] triterpenes paracaseolins A–E ***1501–1505*** (*Sonneratia paracaseolaris*),[724] ***1506***, ***1507*** (*Rhizophora mucronata*),[725] limonoids granaxylocartin A ***1508***,[726] xylomexicanins I and J ***1509***, ***1510***,[727] sundarbanxylogranins A–E ***1511–1515***,[728] and thaixylogranins A–H ***1516–1523*** (all from seeds of *Xylocarpus granatum*)[729] and krishnolides A–D ***1524–1527*** (from seeds of *X. moluccensis*)[730] and krishnadimer A **1528**, an unusual C_2-symmetric limonoid dimer (also from seeds of *X. moluccensis*).[731] The structure of the latter is notable for being the first example of a dimeric limonoid. The structure and P-configuration chiral axis were secured by ECD analysis and X-ray crystallography.



†**1528**

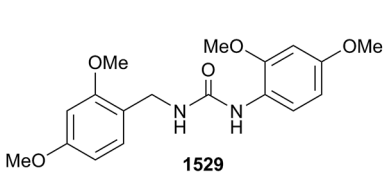
A two-step biomimetic synthesis of kishnadimer from co-metabolite moluccensin A was achieved. The synthesis also afforded the M-configured atropo-diastereomer intermediate, the structure of which was secured by X-ray diffraction. This M-configured dimer exhibited strong cytotoxicity to a panel of six HTCLs.

The reader is cautioned regarding the veracity of structures ***1493–1496***, ***1506***, ***1507*** as in each case sample purity is questionable as is the quality of NMR spectroscopic data used for their characterisation.[720,721,725] The ability of avicequinone C (*Avicennia marina*) and synthetic analogues to exhibit cytotoxicity and to inhibit steroid 5α-reductase has been studied.[732]

14 Miscellaneous

Ureas **1529**, ***1530*** and biaryls ***1531–1533***, the latter three reported as NPs for the first time, were isolated from the lichen *Lichina pygmaea*.[733] Moderate antioxidant activities were observed for ***1529***, ***1530*** and ***1533*** and the structures of ***1529*** and ***1530*** were confirmed by synthesis. A new flavone *O*-glycoside sulfate, thalassiolin D ***1534*** was isolated from extracts of the seagrass *Thalassia hemprichii*.[734] Moderate inhibition of HCV NS3-N54A protease was observed. The isolation and characterisation of ten new fatty acids (***1535–1544***) from amoeboid protist foraminiferan genera *Bathysiphon* and *Rhabdammina* prompts questions as to their dietary or biosynthetic origins and raises the possibility that some of them could be used as trophic markers in food web studies.[735]

Bioassay-directed fractionation of liver extracts of Hawaiian pufferfish *Arothron hispidus* that had suffered a mass mortality event led to the partial purification of polar molecules such as **1545** as being associated with the fish deaths.[736] Difficulty with purification, combined with only extremely small quantities of toxin (35 μg) and the presence of several related molecules, means the structure shown is somewhat speculative.



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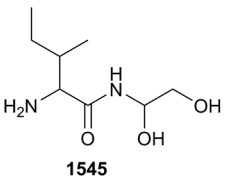

1545

Aberrant aggregation of α-synuclein is a characteristic of Parkinson's disease. Squalamine, a steroidal polyamine originally isolated from the dogfish shark *Squalus acanthias* inhibits α-synuclein aggregation in model vesicles and in neuroblastoma cells.[737] Fermentation of processing waste derived from the spiny head croaker fish (*Collichthys lucidus*) yielded a 22-residue antimicrobial peptide SBF-3-1p that was synthesised and found to be a strong antifungal agent.[738]

15 Conclusion

As in previous years, a small number of papers published in 2017 report structures of questionable identity. In general terms these papers had either poor or no supporting spectroscopic data, spectroscopic data that called into question the purity of the compound being reported, NMR assignments that were inconsistent with the functional groups that they were assigned to or 2D NMR correlations that were inconsistent with the molecular frameworks proposed. In an era where chem-informatics tools are increasingly used to aid in dereplication, structure determination and drug discovery it is important that a rigorous structure determination process is supported by high quality ESI.

Recent reviews discussing the temporal and geographic distribution of MNPs have highlighted the rise in MNP research in China and the substantial increase in discoveries of new MNPs from marine microorganisms over time.[2,3] In 2018, a review of the chemical diversity of MNPs at a phylum level concluded that targeting diverse collections of both macro and micro marine organisms would maximise the chances of finding new chemistry.[1] To expand on these findings, this conclusion section uses data obtained from MarinLit[18] to investigate if there are phylum specific differences in MNP discoveries geographically and if this has changed over time. It is now 68 years since the first report of a MNP, spongothymidine[739] There are now over 29 000 MNPs with approximately 41% discovered in the last 10 years. Three phyla (Ascomycota,

Cnidaria and Porifera) account for 63% of the total MNP inventory and another 12 phyla (Actinobacteria, Cyanobacteria, Proteobacteria, Dinophyta, Chlorophyta, Ochrophyta, Rhodophyta, Mollusca, Bryozoa, Echinodermata, Chordata and Tracheophyta) are sources for all but 1% of the remaining MNPs. Fig. 2 shows the locations of all 28 785 MNPs that have collection location data.

Mapping this data individually for each phylum reveals some clear differences in the location of MNP discoveries. Sponge (Porifera) derived MNPs, for example, have a cosmopolitan geographic distribution while marine fungi (Ascomycota) derived MNPs emanate from South East Asian and Chinese sources.

There are also clear differences in geographic distributions of MNPs from the other 13 major MNP producing marine phyla (see ESI‡ for their maps). Analysis of this data by climatic zones indicates that 49.5% of MNPs have come from tropical, 28.1% from sub-tropical, 21.6% from temperate and 0.7% from polar regions. There are differences in the proportion of MNPs discovered from different phyla and climatic zones (Fig. 3) with the majority of cyanobacteria (72%), cnidarian (70%) and mangrove (85%) (Tracheophyta) MNPs being isolated from tropical species while temperate Proteobacteria, Bryozoa, and brown algae (Ochrophyta) species account for the majority of the MNPs reported from these phyla. A more even distribution of MNPs reported from the remaining nine phyla occurs across species found in tropical, sub-tropical and temperate zones. There are substantially more MNPs discovered from the northern hemisphere (79.7%) compared to the southern hemisphere (20.3%). This is not surprising since the majority of

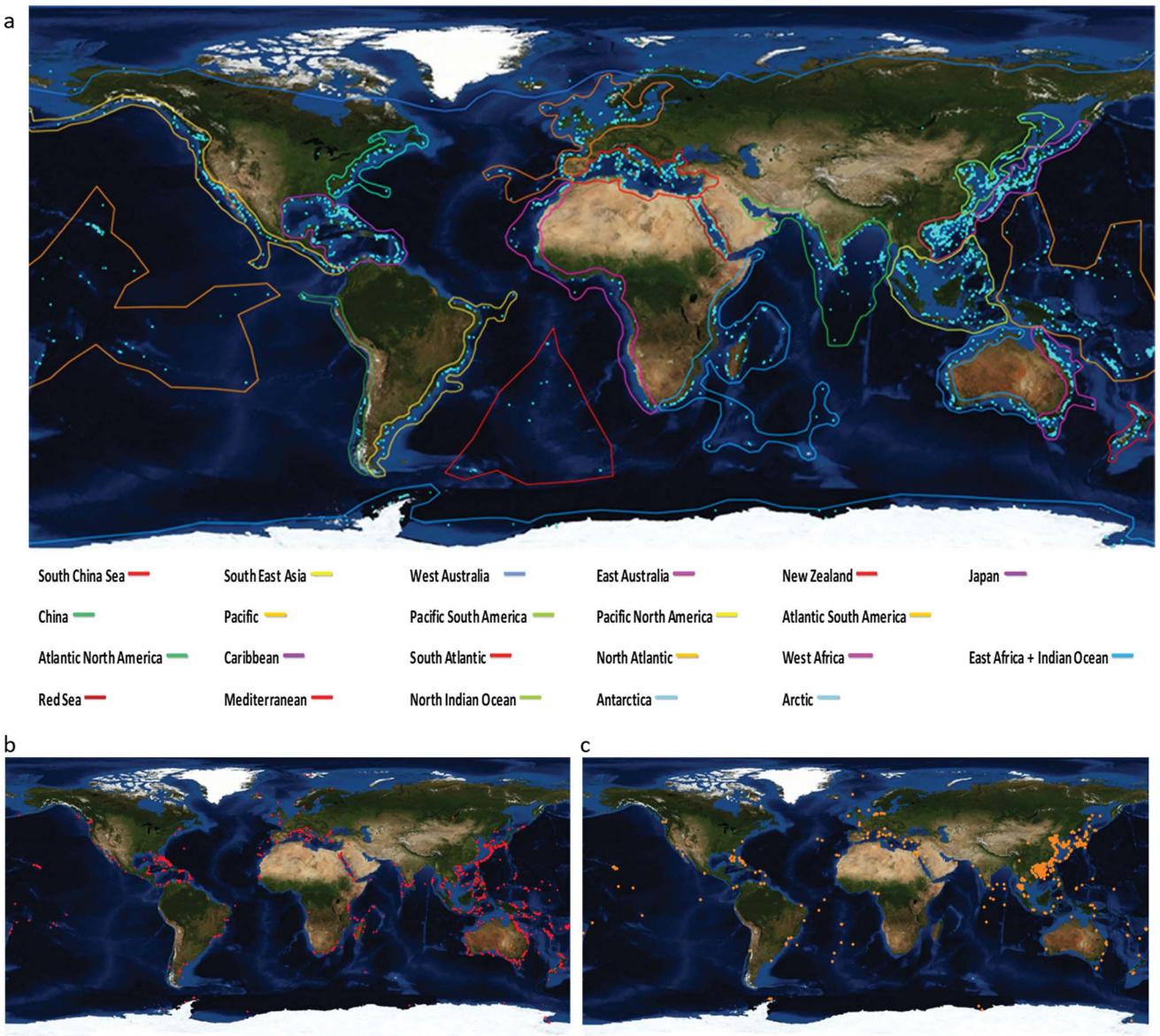

Fig. 2 Worldwide distribution of MNPs that have collection location details in MarinLit (28 785). (a) Total MNP geographic distribution 1957–2017, with geographic zones delineated. (b) Worldwide distribution of Porifera MNPs. (c) Worldwide distribution of Ascomycota MNPs.

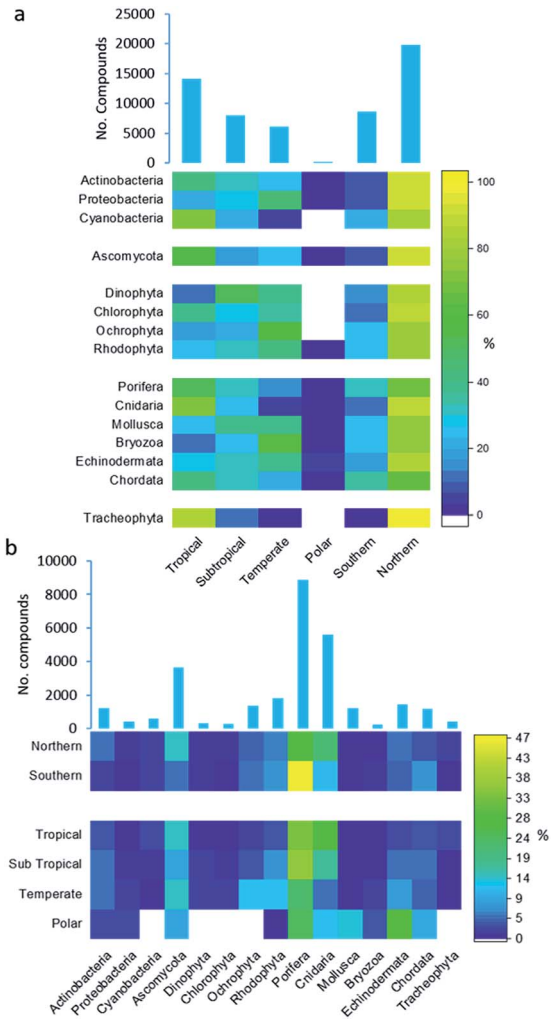

Fig. 3 Proportion of MNPs obtained from different climatic zones (tropical, subtropical, temperate and polar) and hemispheres (northern and southern). (a) Heatmap of proportions of MNP reported from each phylum *vs.* climatic zone and hemisphere. (b) Heatmap of proportions of MNPs reported from each climatic zone and hemisphere *vs.* phylum.

MNPs have been isolated from coastal species and considering that there is more coastline in the northern hemisphere (0.66 million km) compared to the southern hemisphere (0.18 million km).[740] However, if one compares the average number of MNPs discovered per km of coastline there is a difference between the northern and southern hemispheres with 11% more MNPs per km reported from the north compared to the south. Large stretches of coastline along the South American Pacific and African Atlantic coasts remain under investigated to any significant extent.

There are proportional differences in MNPs reported from each phylum in different climatic zone and hemisphere. Sponges (33%) and cnidarians (28%) are the leading sources of MNPs in the tropics, while sponges (22%), marine fungi (14%) and red and brown algae (12% each) are the main sources of MNPs in temperate areas. In the polar regions, 50% of MNPs have been reported from sponges and echinoderms while cnidarians, marine fungi, molluscs and tunicates each contribute ~10% to total polar-sourced MNPs.

Other trends appear through analysis of this data at a regional level (Fig. 2 and 4). Over 60% of all MNPs come from only five regions: South China Sea, Japan, China, Pacific reefs, islands and atolls and the Caribbean. Research in some of these regions has been highly focused on specific phyla. For instance, research in the South China Sea (mainly from Taiwan, and the Chinese island of Hainan) has yielded 5506 MNPs (20% of the worldwide total) yet only two phyla, Ascomycota (1514 MNPs and 41% of the total for this phylum), and Cnidaria (2317 MNPs and 41% of the total for this phylum) account for the majority (70%) of these outputs. In the Caribbean, 67% of all MNPs have

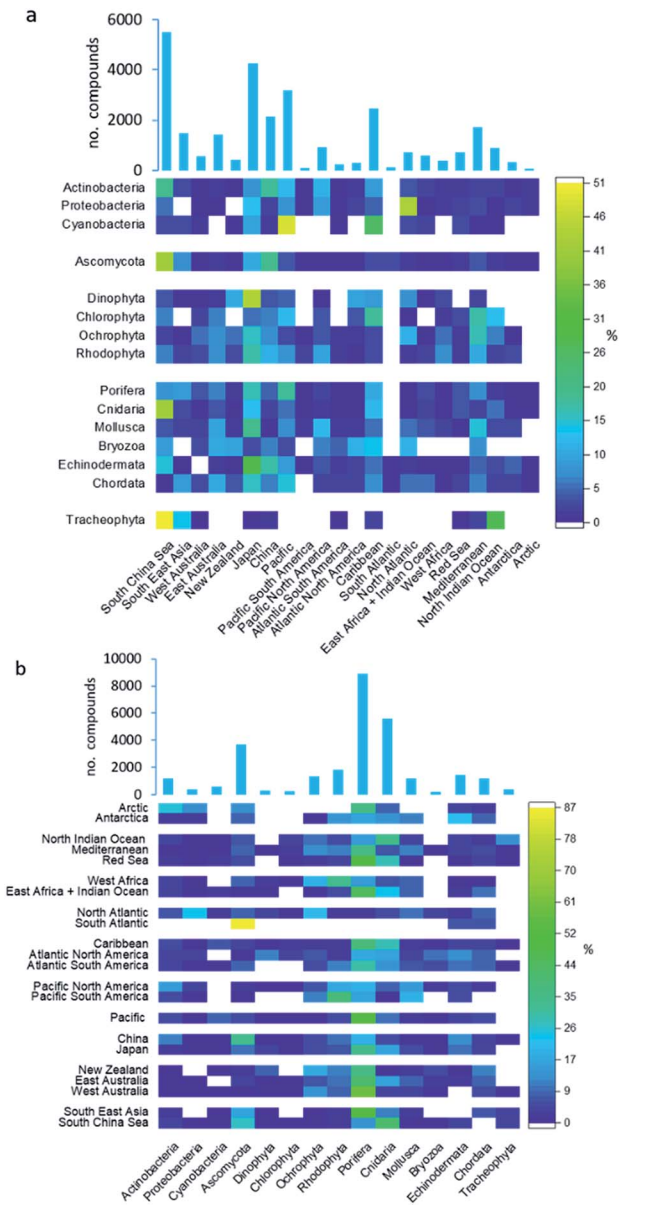

Fig. 4 Proportional distribution of MNPs across geographic regions. (a) Heatmap of proportions of MNP reported from each phylum *vs.* geographic region. (b) Heatmap of proportions of MNPs reported from each geographic region *vs.* phylum.

been isolated from sponges (39%) and Cnidarians (28%) while in the Pacific over half of the MNPs reported have come from sponges (52%) with cyanobacteria and cnidarians (10% each) being the next most prolific organisms to yield MNPs. Japan and China, with a more even spread of MNP discoveries across the 15 phyla, more closely match worldwide trends. Mapping the proportional distribution of MNPs by phylum across the regions shows clear gaps and thus opportunities for directed research. Firstly, Bangladesh, Myanmar, Sumatra, Irian Jaya, the Pacific coast of Central America, western Africa, western South America and north eastern South America are largely unexplored for MNPs and thus provide opportunities for discoveries across all kingdoms and phyla. Marine fungi, being the third most prolific producer of MNPs, have not been studied to any extent in regions outside of SE Asia, China and Japan, suggesting that there are great opportunities to find new chemistry from this kingdom in neglected areas.

Cyanobacterial studies have largely been restricted to Japan, the Pacific and the Caribbean. Considering that MNPs from these organisms have mainly come from tropical locations, opportunities exist for their further study in tropical Africa, South America, SE Asia and Australia. Actinobacterial MNPs mostly emanate from studies in the South China Sea, Japan, China, the Pacific, the Pacific coast of North America and the Caribbean. Hence, there are significant opportunities for research on this important microorganism group in other parts of the world. Mangrove-derived MNPs have only been substantially reported from the South China Sea, SE Asia and the Northern coast of the Indian ocean and considering that there are major stands of mangroves in New Guinea, Australia, East and West Africa, Central America and Northern South America, opportunities for research in these areas should be considered. Studies on echinoderm chemistry have largely been restricted to the South China Sea, China and South Korea, Japan and the North Eastern Pacific. Bryozoan investigations are lacking in the tropics, South America, Africa, Western Australia and Polar regions.

What has changed in the last ten years? Fig. 5 shows the proportion of total MNPs isolated from each phylum and each geographic region between 2008 and 2017. Actinobacteria, Ascomycota, Proteobacteria and Tracheophyta have all increasingly been sources of new MNPs (albeit many from a very low regional base) over the last ten years. Macro-organism phyla including most of the marine invertebrates and algae were already intensively studied up to 2007 in North America, Caribbean, Australia and New Zealand and so the pace of discoveries in these phyla and regions is relatively low by comparison. This is likely to be a reflection of shifting career paths of key researchers, a change in emphasis on biota sources (specifically now targeting microorganisms), funding opportunities and access.

Finally, this analysis suggests that access to highly biodiverse regions is not a limiting factor in relation to new MNP discoveries, since some of the most prolific MNP discoveries have occurred in regions of average biodiversity. This suggests that there is a bright future for new MNP discoveries with strategically targeted collection efforts. It also highlights key

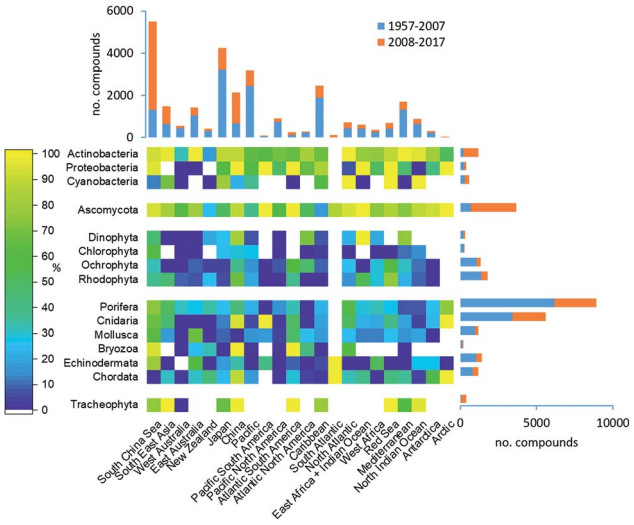

Fig. 5 Heatmap of the proportion of total MNPs discovered by phylum and geographic region between 2008 and 2017.

opportunities to use global information system (GIS) data to identify specific previously overlooked locations within relatively well studied regions and to target under represented phyla, both in these locations and in poorly studied locations.

16 Conflicts of interest

There are no conflicts to declare.

17 Acknowledgements

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