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REVIEW

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Marine natural products

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Covering: January to the end of December 2024

This review covers the literature published in 2024 for marine natural products (MNPs), with 617 citations (578 for the period January to December 2024) referring to compounds isolated from marine microorganisms and phytoplankton, green, brown and red algae, sponges, cnidarians, bryozoans, molluscs, tunicates, echinoderms, the submerged parts of mangroves and other intertidal plants. The emphasis is on new compounds (1256 in 336 papers for 2024), together with the relevant biological activities, source organisms and country of origin. Pertinent reviews, biosynthetic studies, first syntheses, and syntheses that led to the revision of structures or stereochemistries, have been included. An analysis of the role of artificial intelligence in marine natural products research is discussed.

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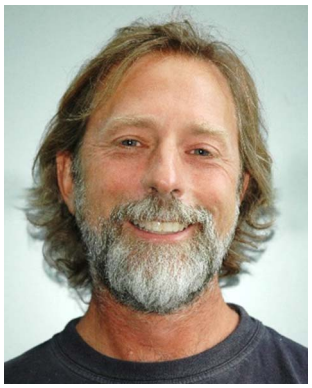
1 Introduction

This review is of the literature for 2024 and describes 1256 new compounds from 336 papers, compared to 1220 new compounds in 340 papers reported for 2023.[1] In addition, 30 known NPs were reported from a marine source for the first time, three artefacts were identified, and 48 known MNPs had their structures revised. Only new MNP structures or previously reported compounds where there has been a structural revision, or a newly established stereochemistry are shown in this review. The review also covers previously reported MNPs with significant new bioactivities or ones that have been synthesised for the first time, but their structures are generally not shown. A † symbol on the identifying diagram number is used to distinguish structures where the absolute configuration has been determined for all stereogenic centres, axes and/or planes in a compound. Reports of new MNPs that were identified based solely on a combination of biosynthetic gene cluster (BGC) information, MS/MS data and/or Global Natural Products Social (GNPS)-based molecular networking, with compounds not isolated and no NMR data recorded, are excluded from the review. Only a selection of highlighted structures (54) is shown. Compound numbers for structures not highlighted in the review are *italicised*, and all structures are available for viewing, along with their names, taxonomic origins, collection locations, and biological activities, in an associated SI document. Over the past 20 years we have discussed a variety of topics relevant to

MNP research in the conclusion section. Given that these treatises introduce new data derived from analysis of historical trends, we feel that it is more appropriate to include this discussion in a stand-alone section rather than the conclusion section. Therefore, this year we introduce a new "Perspective" section to the review, leaving the conclusion section to sum-up the overall insights gained from documenting the years MNP research outputs. Access to the curated MNP data held in the Marinlit database[2] provides all the structural and literature data used to prepare this review.

A pioneer of research on MNPs from Red Sea organisms, Professor Yoel Kashman from Tel Aviv University, passed away in mid-2025. Over his long career, Prof Kashman made significant discoveries, mostly associated with sponges, ascidians and soft corals. He will be sadly missed.

Trends in the number of new MNPs reported annually over the decade show that numbers of new sponge MNPs dropped again to a new low, while numbers of mollusc, (2) tunicate (6) and red algae MNPs (3) also dropped to decadal lows and numbers of reported cnidarian MNPs dropped to a near decadal low. No new bryozoan NPs were reported for the second year in a row and the number of new brown algae MNPs was lower than



Anthony R. Carroll

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.

Brent R. Copp

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.

Tanja Grkovic

Tanja Grkovic received her MSc and PhD degrees from the University of Auckland under the supervision of Professor Brent Copp. She then carried out postdoctoral research at the National Cancer Institute with Kirk Gustafson, and Griffith University with Professor Ron Quinn. She is currently a Staff Scientist at the Natural Products Branch and the Molecular Targets Program at the National Cancer Institute where her research is focused on the generation of prefractionated natural product libraries as well as the isolation and structure elucidation of natural products sourced from marine, plant, and microbial biota.

Robert A. Keyzers

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 an Associate Professor.

Michèle R. Prinsep

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 a Professor.

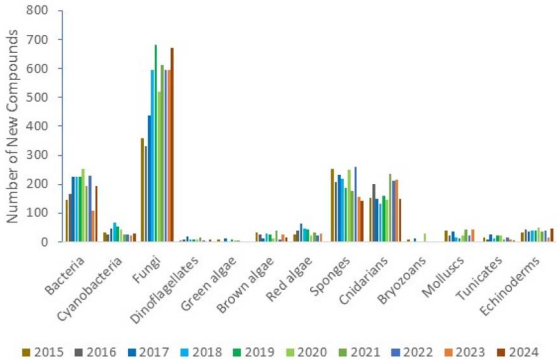


Fig. 1 Trends in new MNPs. The bars represent the total number of new MNPs reported each year over the last 10 years. Fungi MNPs only includes those isolated from submerged sources.

the decadal average. In contrast, numbers of MNPs isolated from fungi continue to rise and based on our changed reporting criteria, that now only includes fungi isolated from submerged environments, it is likely that 2024 had the highest number of new fungal MNPs reported over the 42-year history of this review. New MNPs reported from bacteria resurged to a decadal average level (Fig. 1).

2 Marine microorganisms and phytoplankton

2.1 Marine-sourced bacteria

Actinobacteria continue to be the most prolific source of MNPs reported from bacteria (with 165 new structures), even though they represent approximately 10% of total marine bacterial biodiversity. A sediment-derived *Actinomadura* sp. yielded five phenyl polyenes, maduraflavacins A–E ***1–5***.[3] Two rare genera of bacteria yielded MNPs for the first time in 2024, linear peptides cellulamides A ***6*** and B ***7*** were reported from *Cellulosimicrobium funkei*,[4] and glycoglycerolipids testacosides A–D ***8–11*** were the first MNPs reported from *Microbacterium testaceum*.[5] A sponge-derived *Microbispora* sp. strain yielded okichromanone, a chromanone NP isolated as a mixture of two interconverting epimers ***12*** and ***13*** due to hemiketal ring opening and recyclisation.[6]

The genus *Micromonospora* yielded two anthracyclinones ***14*** and ***15*** and one anthraquinone ***16***,[7] three nona-2,7-dienoic acid derivatives apocimycins A–C ***17–19***,[8] as well as three spiroketal-containing macrolides ***20–22***, and a diketopiperazine ***23***.[9] X-ray diffraction (XRD) analysis was used to define the absolute configuration of the co-occurring known MNP IB96212 ***24*** for the first time.[9] A large scale (96 L) fermentation of *Nocardiopsis maritima* yielded two cyclic hexapeptides, maritiamides A ***25*** and B ***26***, which showed moderate activity against *S. epidermidis* and weak activity against *E. coli* and *P. fluorescens*.[10] Two α-pyrone polyketides, nocardiopyrones D ***27*** and E ***28***, as well as an alkaloid, nocarterphenyl I ***29***, were reported from a sediment-derived *Nocardiopsis* sp.[11] Nocarterphenyl I ***29*** possesses a rare 2,2′-bithiazole-*p*-terphenyl scaffold, the structure of which was confirmed *via* XRD, and showed weak to moderate activity against four bacterial strains with MIC values ranging from 0.8 to 1.6 μM. Nocarterphenyl I showed no cytotoxicity against methicillin-resistant *S. aureus* (MRSA) or four human tumour cell lines (HTCLs).

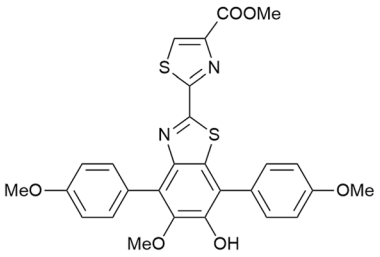
The genus *Saccharomonospora* yielded four pyridine-, thiazole-, and chloroquinoline-containing alkaloids lodopyridones D–G ***30–33***,[12] and two prenylated indole alkaloids, penipaline D ***34*** and *N*-acetyl-6-dimethylallyl-L-tryptophan ***35***.[13] Three sulfur-bridged angucycline dimers, spongisulfins A–C ***36***, ***37*** and ***38***, and one monomer, rubiginone A3 ***39***, were reported from a sponge-derived *Spongiactinospora rosea*, with the structures of ***37*** and ***39*** confirmed *via* XRD.[14]

As for previous years, the genus *Streptomyces* was the most studied, with 125 new MNPs reported. A discovery campaign targeting the identification of terminal oxazole-bearing NPs using PCR screening for oxazole cyclase encoding genes coupled with NMR-based screening measuring $^{1}J_{CH}$ one-bond coupling constants specific to oxazole rings, identified two polyketide MNPs, methyl-oxazolomycins A ***40*** and B ***41***.[15] The two compounds showed moderate cytotoxicity against two estrogen receptor (ER) positive breast cancer cell lines MCF7 and T-47D, but no activity against ER-negative cell line MDA-MB-231.

Two linear polyketides, alpiniamides H ***42*** and I ***43*** were isolated from a sediment-derived collection of *Streptomyces* sp.,[16] and a coral-derived *Streptomyces* sp. yielded polyketide chromone ***44***, polyketide ***45*** and fatty acids ***46*** and ***47***, and ***48*** and ***49***, each as racemates but purified to separate each enantiomeric pair.[17]

A series of methylene-bridged, dimeric, 4-hydroxy-2*H*-pyran-2-one (HPO)-containing pyrones, the phaeochromycins M–U ***50–59*** were produced by a large-scale culture of sediment-derived *Streptomyces* sp.[18] The authors showed that simple HPO-containing molecules can react with formaldehyde to generate methylene-bridged dimers, proposing a chemical defence mechanism where HPO-producing bacteria can sequester, and effectively neutralise formaldehyde from the environment for competitive gain. Cloning and heterologous expression of a type II polyketide BGC *spi*1 sourced from a marine sediment-derived *Streptomyces* sp. into a *S. coelicolor* host, led to the isolation and identification of six angucyclines ***60***, ***61–65***, including spirocyclinone A ***60*** with a rare oxaspiro [5,5]undecane motif.[19] Madeirone ***66***, a new mixed terpene- and polyketide-derived MNP, was reported from a sediment-derived *Streptomyces aculeoletus*.[20]

Glycosylated angucyclines reported from *Streptomyces* sp. included seven *C*-glycosides, chrysomycins F–J ***67–71***,[21] and grincamycins V ***72*** and U ***73***.[22] A mangrove rhizosphere-derived *Streptomyces* sp. yielded three glycosylated polycyclic xanthones, kebanmycins A–C ***74***, ***75*** and ***76***, and the aglycone kebanmycin D ***77***.[23] Kebanmycin A showed potent activity against *S. aureus*, including four MRSA isolates, with MIC values ranging from 0.125 to 0.5 μg mL^{-1}, but did not inhibit *B. subtilis* growth. Based on the annotations from the genome mining tool anti-SMASH, the candidate BGC *keb* was proposed to be responsible for assembly of the compounds.



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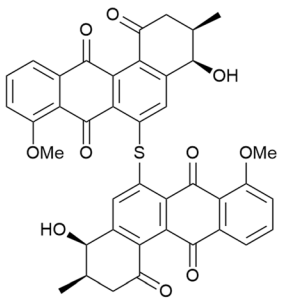

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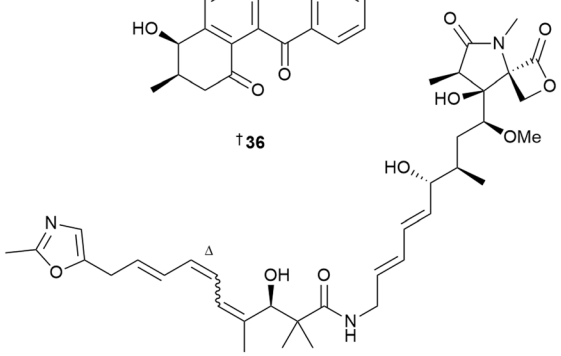
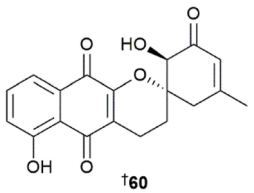


†40 *Z*
†41 *E*

Sediment-derived collections of *Streptomyces* sp. yielded four ansamycin-type macrolides ***78–81***,[24] five tetramate-containing macrolactams, aburatubolactams D–E ***82–84***,[25] as well as hydroxycapsimycin ***85*** and brokamycin ***86***.[26] Two 6,6-spiroketal-containing MNPs, streptospirodienoic acids D ***87*** and E ***88*** were reported from a coral-derived *S. cavourensis* together with two known compounds bafilomycins P ***89***, and Q ***90*** whose absolute configurations were revised based on nOe correlation analysis, DP4+ calculations, and XRD data.[27] Compounds ***89*** and ***90*** showed moderate activity against the A-549 and HCT-116 HTCLs. Five oligomycins ***91–95*** were reported from a sediment-derived *Streptomyces* sp.,[28] four polyene macrolides ***96–99***, were isolated from a mangrove-derived *S. hiroshimensis*,[29] and a sponge-derived *Streptomyces* sp. yielded a tricyclic polyene macrolactam, weddellamycin ***100***.[30]

Albusamides A–G, ***101–107***, a series of new long-chain hydroxylated acetamide MNPs, were reported from *S. albus*,[31] and a sediment-derived collection of *Streptomyces* sp. yielded additional acetamide-containing MNPs, streptothiomycin F ***108*** and *N*-(5-nitropentyl)acetamide ***109***, the latter reported as a first time MNP.[32] Albusamide C ***103*** displayed moderate cytotoxicity against a panel of fourteen HTCLs, but with no selectivity margin against the normal B lymphocyte cell line RPMI-1788. A genome-based mining strategy targeting the presence of the carpatamide BCG *ctd*, identified five acylated arylamines, carpatamides I–M ***110–114*** from *S. parvus*.[33]



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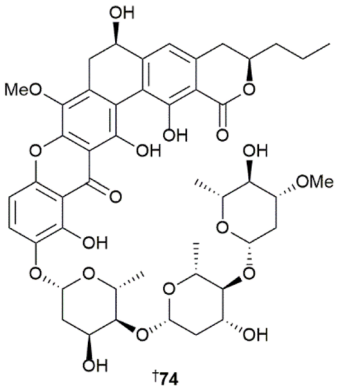

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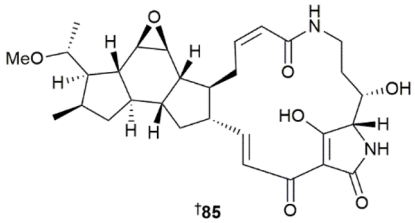

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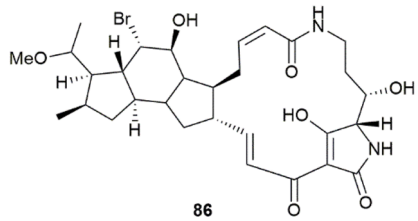

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A series of shorter chain aryl amines ***115–119***, a phenylpropanoid ***120***, unsaturated fatty acids ***121***, ***122***, and two thiazinone-containing MNPs ***123***, ***124*** were reported from a sediment-derived *Streptomyces* sp., with ***119*** and ***121–124*** representing new MNPs and others, known compounds reported from the marine environment for the first time.[34] A mudflat-derived *Streptomyces* sp. yielded a phenylthiazole anithiactin D ***125***,[35] and heterologous expression of an NRPS BGC *grsc*, sourced from a marine sediment-derived *Streptomyces* sp. into three different *Streptomyces* surrogate hosts, yielded eight new thiazole-containing MNPs, grisechelins E ***126***, F ***127***, and I–N ***128–133***, as well as two quinoline derivatives, grisechelins G ***134*** and H ***135***.[36] Notably, grisechelin E ***127*** showed moderate antimycobacterial activity, with an MIC of 8 μg mL^{-1} against *M. tuberculosis*.

A pyrrolo-pyrido-benzoquinone, streptoquinoneazaindole A ***136***, a urea-containing pyrrole alkaloid, streptoureipyrrole A ***137*** and 6-hydroxybenzothiazole ***138***, reported as a MNP for the first time, were isolated from a deep-sea sediment-derived *Streptomyces* sp.[37] A thioether-linked quinoline-quinazoline, quinosumycin ***139*** and a chromone, chromycone ***140***, were reported from a sponge-derived *S. diastaticus*.[38] A dithiolopyrrolone, ***141***

was isolated from a tunicate-derived *Streptomyces* sp.[39] and a staurosporine analogue, streptomholyrine A ***142*** was reported from a sediment-derived *Streptomyces* sp.[40]

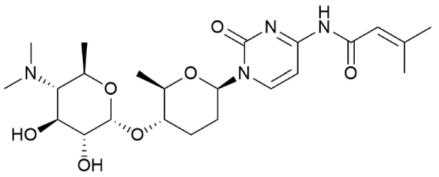
Pattern-based genome mining, combining mass spectrometry (MS) isotopic signature and biosynthetic sequence data, led to the identification of two chlorinated pyrroloketoindanes, indanopyrroles A ***143*** and B ***144***, with their candidate BGC *idp* identified and biosynthetic assembly proposed.[41] The trichlorinated indanopyrrole A showed moderate to potent activity against several Gram-positive and -negative bacteria, while the dichlorinated analogue indanopyrrole A was inactive.

In a high-throughput screening (HTS) discovery campaign against *Mycobacterium avium* complex (MAC) pathogens, seven disaccharide pyrimidine nucleosides, mavintramycins A–G ***145***, ***146–151*** were identified from a sediment-derived *Streptomyces* sp.[42] Mavintramycin A ***145*** showed potent activity against two different MAC strains, with only moderate activity against two TB-associated mycobacterial strains and no cytotoxicity against other bacterial and fungal pathogens and one mammalian cell line. Moreover, ***145*** was found to be active against 40 clinically isolated *M. avium* strains, including those that were multidrug-resistant. Mechanism of action studies found that mavintramycin A binds to 23S ribosomal DNA and inhibits protein synthesis. The structure of ***145*** was later unequivocally confirmed *via* total synthesis.[43]

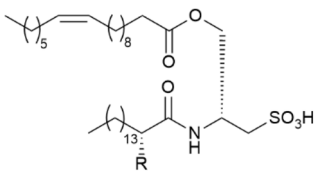
A Korean sediment-derived *Streptomyces* sp. yielded six linear depsipeptides, homiamides A–C ***152–154***,[44] and cavomycins A–C ***155–157***,[45] all of which contained a core amino acid sequence of α-hydroxyisovaleric acid (Hiv)-valine (Val)-lactic acid (Lac)-valine (Val) residues like the valinomycin family of cyclic peptides. Three linear lipopeptides, albubactins A–C ***158–160*** were isolated from a tunicate-derived *S. albidoflavus*,[46] with the structure of ***158*** confirmed *via* total synthesis. A sediment-derived *Streptomyces* sp. yielded *N*-acylated cyclic octapeptides, acyl-surugamides A1–A4 ***161–164***,[47] with the structure of acyl-surugamide A2 ***162*** also reported, but not fully characterised, from a tunicate-derived *S. albidoflavus*.[48] A sponge-derived *Streptomyces* sp. yielded four glycosylated cyclopeptides, pyridapeptides F–I ***165–168***,[49] and three cyclic lipopeptides olenamidonins A–C ***169–171*** were reported from a deep-sea derived *S. olivaceus*.[50] Six terpenoid structures reported from *Streptomyces* spp. included the iridoid lucknolide A ***172***, reported as a MNP for the first time,[51] and five labdanes, chlorolabdans A–C ***173–175***, and epoxylabdans A ***176*** and B ***177***.[52]

Only one MNP from the phylum Bacteroidota was reported in 2024; an alga-derived *Algoriphagus* sp. strain yielded a new carotenoid, 2-hydroxyflexixanthin ***178***.[53] Five MNPs were identified from the phylum Fimicutes, which included three glycosylated, 24-membered polyene macrolides, amylomacrolactines A–C ***179–181*** sourced from a sediment-derived *Bacillus amyloliquefaciens*,[54] and a new 25-membered polyene macrolide, macrolactin XY ***182*** and a new fatty alcohol ***183*** from a sponge-derived *B. subtilis*.[55]

Fourteen MNPs were reported from the phylum Proteobacteria in 2024. A deep-sea sediment-derived *Alcanivorax dieselolei* strain yielded two *p*-aminoacetophenonic acid analogues, mohangic acids E ***184*** and F ***185***,[56] and four lipopeptides, bokeelamides A–D ***186–189*** were isolated from snail egg mass-derived *Ectopseudomonas khazarica*.[57] Two ureido-containing linear peptides, alteropeptilides A ***190*** and B ***191*** were reported from an arthropod-derived *Pseudoalteromonas flavipulchra*.[58] Structures of sulfur-containing amino lipids cysteinolides A ***192*** and B ***193*** were reported from bacterial species *Ruegeria pomeroyi* and *R. meonggei* and represent the first members of the sulfonolipid structural class of MNPs that have been fully characterised by NMR spectroscopy and their structures confirmed by total synthesis.[59] A seawater-derived *Vibrio ruber* yielded a new pyrrolidine, vibripyrrolidine A ***194***, a piperazine vibripiperazine A ***195***, and two diazinanes, vibridiazinanes A ***196*** and B ***197***, with ***196*** showing potent activity against *S. aureus* with a MIC value of 0.98 μg mL^{-1} and no inhibitory activity against *E. coli*, *K. pneumoniae*, *P aeruginosa*, or a panel of three HTCLs.[60]



†**145**



†**192** R = H
†**193** R = OH

As in previous years, some MNPs reported from marine bacteria did not have adequate spectrometric or NMR spectroscopic data in full support of the proposed structures and were omitted from this review.[61–64] Total synthesis of the reported structure of marinoaziridine B[65] revealed significant inconsistencies in the ^{1}H and ^{13}C NMR spectroscopic data, suggesting the structure of this MNP should be revised. Total syntheses of other bacteria-sourced MNPs reported in 2024 included bacillimidazole B and discolins A, B, and E,[66] bacilotetrin C,[67] dassonmycins A and B,[68] caerulomycin K,[69] cihunamide B,[70] cyclomarin,[71] discoipyrroles A–C,[72] (+)-dixiamycin,[73] homoseongomycin,[74] ieodomycins A and B,[75] incarnatapeptins A and B,[76] iturin A,[77] lorneic acids C and D,[78] marformycins A and D,[79] mavintramycin A,[43] moiramide B,[80] octalactins A and B,[81] rufomycins 1, 2, 4, 11, 22, and 24,[82] salimabromide,[83] and sealutomicin C.[84]

Reviews focused on bacterial MNPs published during 2024 included a summary of metagenomic strategies for the discovery of MNPs,[85] and actinomycete promoter engineering approaches.[86] The bacterium *B. subtilis* was reviewed as a versatile host for heterologous expression of NP BGCs,[87] as was the regulation of acyl homoserine lactone-based quorum

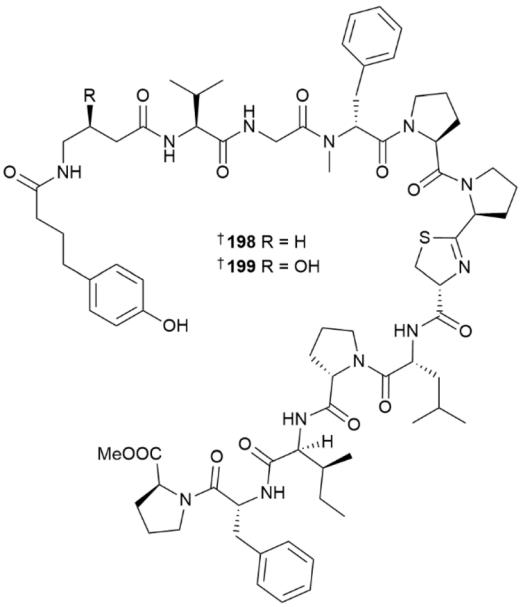
sensing effects on the biosynthesis of bioactive molecules in bacteria.[88]

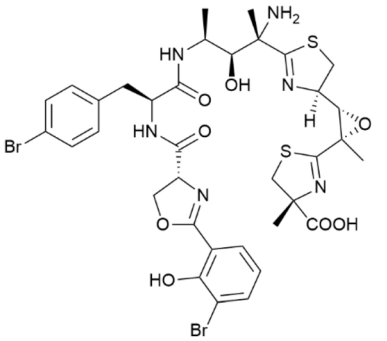
Structural classes of bacterial NPs reviewed included polyacetylenes,[89] alkaloids,[90] and γ-pyrones.[91] Groups of marine bacteria reviewed for biosynthetic potential and the diversity and activity of MNPs produced included the MAR4 clade of the genus *Streptomyces*,[92] and marine-sourced *Pseudomonas* sp.[93] Biological activity studies of previously reported bacteria-sourced MNP structures included *in vitro* and *in vivo* anti-*Toxoplasma gondii* activity assessment of a series of marinoquinolines,[94] mechanism of action (MoA) studies of marinopyrrole, pentachloropseudilin, and pentabromopseudilin,[95] tartrolon D,[96] and tunicamycins,[97] and structure–activity relationship (SAR) reports of thiomarinol A,[98] and sunshinamide.[99]

2.2 Cyanobacteria

Two linear lipopeptides, pemuchiamides A ***198*** and B ***199*** were isolated from a relatively large-scale (3 kg) Japanese collection of the filamentous cyanobacterium *Hormoscilla* sp.[100] The compounds contain 13 amino acid residues including four prolines and several unusual ones such as 4-aminobutanoic acid, 4-(4-hydroxyphenyl)butanoic acid and *N*-methyl-D-phenylalanine. Pemuchiamide A showed moderate activity against *Tryponosoma brucei rhodesiense* (IC_{50} = 0.63 μM), while the hydroxylated analogue pemuchiamide B was an order of magnitude less active. This indicated that the 4-aminobutanoic acid moiety may be an important pharmacophore feature involved in the antitrypanosomal activity for this peptide series. Laboratory cultures (some up to six months in length) of three geographically dispersed collections of *Lepthotoe* strains yielded three new PKS/NRPS hybrid linear peptide metallophones, leptochelins A–C ***200***, ***201*** and ***202***.[101] The leptochelin structures were challenging to elucidate since they contain modified amino acids and isolated stereogenic centres. Their structures were deduced using a combination of tandem mass spectrometry, NMR spectroscopy, and bioinformatic analyses of the putative leptochelin BGC *lec*. The leptochelins bind to zinc, copper, iron, and cobalt and were moderately cytotoxic against four HTCLs. They are also the first reported MNPs from the genus *Lepthotoe*.

Studies on *Moorena* spp. yielded seven MNPs. A glycosylated macrolide, moorenaside ***203*** was reported from a Florida collection of *Moorena* sp.[102] and a Guam-sourced collection yielded two polyketides ***204*** and ***205***.[103] A co-culture of *M. producens* with *Candida albicans* led to significant upregulation of the production of a linear depsipeptide hectoramide B ***206***. Its putative BGC *hca* was identified and its biosynthetic assembly annotated.[104] Even though the presence of a fungal competitor was crucial to produce hectoramide in liquid culture, when tested against two *Candida* strains, ***206*** did not show any antifungal activity. *M. bouillonii* collected in India, yielded a linear lipodepsipeptide, kavaratamide A ***207***,[105] and a cyclic depsipeptide, wajeepeptin ***208*** was isolated from a Japanese collection of *Moorena* sp.[106] A benthic collection of *Moorena* sp. (annotated as *Lyngbya* sp.) in Indonesia yielded a cyclic peptide PM170453 ***209***, the structure and absolute configuration of





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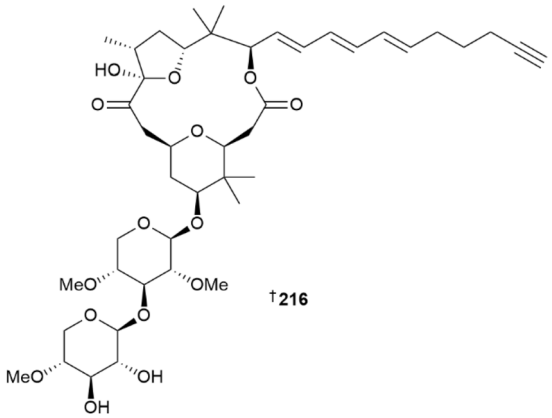
which was unequivocally confirmed *via* total synthesis.[107] Six lipopeptides, noducyclamides A1–A4 ***210–213*** and noducyclamides B1 ***214*** and B2 ***215***, were reported from a laboratory culture of *Nodularia* sp.[108] The dodecapeptide noducyclamide B1 was weakly cytotoxic against the MCF7 breast cancer cell line.

The genus *Okeania* yielded six new MNPs. A glycosylated, 16-membered macrolide, polycavernoside E ***216***, was reported from a coastal Okinawan collection of *Okeania* sp.[109] Related analogues of this MNP, the polycavernosides A–C were originally reported from the red alga *Polycavernosa tsudaii*[110–112] and were implicated in human illness and death from consumption of this edible seaweed in Guam. An additional congener, polycavernoside D, was later identified from a Caribbean collection of the cyanobacterium *Okeania* sp.,[113] and this latest report of ***216*** provides increasing evidence that polycavernosides are produced by cyanobacteria rather than red algae. Polycavernoside D did not show significant *in vitro* cytotoxicity against normal human fibroblast WI-38 cells, was weakly active against *T. b. rhodesiense*,[109] but demonstrated potent *in vivo* toxicity in mice with an ip. lethal dose of 0.81 mg kg^{-1}.[114] Additional *Okeania* sp. collections yielded a lipopeptide, *N*-

desmethylmajusculamide B ***217***,[115] cyclic peptide okeaniazole A ***218***,[116] and a linear lipopeptide, amantamide C ***219***.[117] Terpenoids continue to be reported from cyanobacterial sources, with kagimminols A ***220*** and B ***221*** representing the first examples of cembrene diterpenoids isolated from the genus *Okeania*.[118]



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An unidentified cyanobacterium with morphological resemblance to *Symploca* sp. collected in Guam, yielded three enyne-containing chlorinated fatty acid amides, taveuniamides L–N ***222–224*** and the absolute configuration of the C-8 stereogenic centre was reported for this MNP class for the first time.[119] Using genome-mining and metabolomic strain prioritization strategies, two dialkylresorcinol polyketides, 29-dechloro-29-hydroxybartoloside A ***225*** and 17-dechloro-17-hydroxybartoloside A ***226***, were isolated from *Synechocystis salina*,[120] with hydroxylation along the aliphatic side-chain in both compounds representing a novel structural modification of the bartoloside family of polyketides. Finally, a methyl-branched fatty acid, jobosic acid ***227*** was reported from an unidentified bacterial biomass collected in Puerto Rico.[121] Cyanobacteria remain one of the few marine taxa where reports of new MNPs from mixed assemblages or incomplete taxonomic identifications continue to be accepted for publication.

The total synthesis of the cyclic depsipeptide lagunamide C was achieved,[122] resulting in the revision of the proposed structure from a 27-membered macrocycle originally published[123] to the 26-membered odoamide,[124] a known cyanobacterial MNP. Other cyanobacterial MNPs with reported total syntheses included dragocins A–C,[125] irijimaside A,[126] jamaicamide B,[127] majusculamide D,[128] rivularin A,[129] serinolamides A and B,[130] and trichophycin-I.[131]

Reviews included summaries of anti-infective,[132] anti-protozoal,[133] and cancer-related[134] activities of cyanobacterial MNPs, as well as a comprehensive review of the diversity of NP structures and biological activities reported from the genus *Oscillatoria*.[135] Notable work on the biosynthesis of cyanobacterial MNPs included the identification and characterisation of the putative BGC for the assembly of dolastatin, reported from *Caldora penicillata*,[136] and SAR studies of known cyanobacterial compounds biselyngbyolide B,[137] carmaphycin B[138] iezoside,[139] and majusculamide D.[140] Environmental reports of cyanobacterial MNPs with broad implications to human health included a review on impacts of cyanobacterial blooms on fish farming,[141] and the first account of bioaccumulation of lyngbyatoxin A in edible shellfish along the coast of New Zealand.[142] This, together with a report of acute toxicity of a new polycavernoside analogue ***216*** in mice, accentuates the importance of accurate annotation, full structural characterisation, and monitoring of the occurrence of cyanobacterial MNPs that can impact marine ecosystems and human health.

2.3 Marine-sourced fungi

Co-culture of an *Acremonium* species with heat-killed *Pseudomonas aeruginosa*, resulted in isolation of acremosides A–G ***228–234***, linear sesquiterpenoids with a 2-phenyl acetoxy substituent conjugated to a sugar alcohol.[143] Other *Acremonium* strains were the source of the meroterpenoids, acremochlorins A ***235*** and B ***236***, pyridine alkaloid acremopyridone A ***237*** and cyclopentenone derivative acremoketene A ***238***,[144] sorbicillin-like compound ***239*** and acremonilactone ***240***,[145] and ascochlorin glycoside ***241***.[146]

Fermentation of *Albifimbria verrucaria* yielded the nonenes, verrucanonenes A–C,[147] ***242–244*** and tetrahydrofuran-containing polyketide derivatives[148] ***245–248***, the butanolide derivative, alterbutenolide ***249*** was obtained from *Alternaria alternata*,[149] and *Arthrinium arundinis* cultures, respectively derived from a sea anemone and a sponge, yielded arthrinoic acid ***250***, hexylaconitic anhydride methyl ester ***251***, nonanoic acid derivative ***252***, arthripenoids G ***253*** and H ***254***,[150] and alternapyrone derivatives, alternapyrones G ***255*** and H ***256***. The BGC for alternapyrone biosynthesis was identified and heterologous expression led to isolation of an additional alternapyrone, alterpyranone I ***257***.[151]

As has been the case in many previous years, the genus *Aspergillus* was the most common source of fungal metabolites. Co-culture of deep-sea-derived *Aspergillus aculeatinus* with a mangrove-derived *Penicillium* strain yielded the paraherquamides, aculeaquamides A ***258*** and B ***259***,[152] while addition of sodium bromide to growth media of *A. alliaceus* resulted in production of brominated anthraquinone monomer ***260*** and dimers ***261–263***.[153] *p*-Terphenyl ***264***, ***265*** and diphenyl ***266***, ***267*** ether derivatives were obtained from *A. candidus*,[154] whilst the benzophenone derivatives, carneusones A–F ***268–273*** were isolated from a culture of *A. carneus*.[155] *A. chevalieri* and *A. fischeri* respectively yielded prenylated indole diketopiperazine ***274*** and prenylated indole derivative ***275***,[156,157] ascidian-derived *A. clavatus* was the source of 12*S*-deoxynortryptoquivaline ***276***,[158] co-culture of *A. insulacola* with a mangrove-derived *Alternaria* strain led to isolation of cyclic tetrapeptides, violaceotides B–E, ***277–280***,[159] miniaturised cultivation profiling enabled isolation of 2,6-diketopiperazines, noonazines A–C ***281–283***, and azaphilone, noonaphilone A ***284***,[160] and a nitrobenzoyl sesquiterpenoid ***285*** was obtained from culture of *A ochraceopetaliformis*.[161]

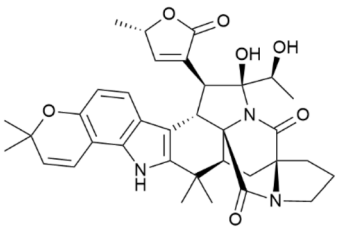
Stephaochratidin A ***286***, an unusual stephacidin-asperochratide hybrid, was isolated from culture of deep-sea-derived *A. ochraceus*.[162] Several deep-sea sediment-derived *Aspergillus* strains yielded a range of metabolites. The sesquiterpenoid malfilanol C ***287*** was isolated from a culture of *A.*

puniceus, along with known terrestrial but new MNP, malifilanol B ***288***,[163] phenol derivatives ***289–298***, (the last a known synthetic compound but new NP), were obtained from *A. subversicolor*,[164] and separate cultures of *A. sydowii* yielded sesquiterpenoid ***299*** and sydowimide A ***300***,[165,166] the latter by epigenetic modification. Another culture of *A. sydowii* was the source of xanthone-alkaloids sydoxanthones F–M ***301–311***,[167] and a prenylated indole alkaloid, aspertaichamide A ***312*** was obtained from *A. taichungensis*.[168]

Cultures of *A. terreus* were the source of a range of metabolites including caryophyllene-type sesquiterpenoids, asperporonins A ***313*** and B ***314***,[169] ergone derivatives ***315–324***,[170] (all deep-sea-derived), sulfur containing metabolites asperteretals L ***325*** and M ***326*** and terreins A ***327*** and B ***328***,[171] brominated aromatic butenolides ***329–342***,[172] meroterpenoids ***343–348***,[173] butenolide derivatives ***349*** and ***350*** (*via* epigenetic manipulation and the latter a known synthetic compound but new NP)[174] and a suite of azaphilones, ***351–361***, which were produced through activation of a cryptic BGC.[175]

Some polyketides ***362–365***, and a nucleoside ***366*** were isolated from a culture of *A. versicolor*,[176] while a range of metabolites were obtained from *Aspergillus* strains from a variety of sources. Sponge-derived *Aspergillus* strains yielded ergostane-type steroid ***367***,[177] terminal olefin-containing unsaturated fatty acid ***368***, 2-carboxy-2′-methyl-azobenzene ***369*** and 1,3,9-trimethyluric acid ***370*** (the last two known synthetic compounds but new NPs).[178] Sediment-derived *Aspergillus* strains yielded aspergilalkaloid A ***371***,[179] asperindopiperazines A–C ***372–374***, 5-methoxy-8,9-dihydroxy-8,9-deoxyaspyrone ***375***, and 12*S*-aspertetranone D ***376***,[180] emericelactones F ***377*** and G ***378***, preshamixanthones ***379*** and ***380***, 25-*O*-methylarugosin A enantiomers ***381*** and ***382***, furanones, 9-hydroxymicroperfuranone ***383*** and microperfuranone ***384***, (these last four known terrestrial but new MNPs), phthalimidinic acids ***385*** and ***386***, aspergilol G ***387***, anthraquinone ***388***, and propionic acid lactone derivative ***389***,[181] (the last a known synthetic compound but new NP). A seawater strain of *Aspergillus* yielded ceramide ***390*** while a coral-derived *Aspergillus* strain yielded megastigmanones A–C ***391–393*** and prenylterphenyllin H ***394*** and a starfish-derived strain yielded brominated isocoumarin ***395***.[182–184] The production of ***395*** resulted from addition of bromide salts to the culture medium.[184] *Aspergillus* species from mangrove sediments have yielded a nor-diterpenoid acid, asperbrunneo acid ***396***,[185] indole alkaloids asperdinones A–H ***397–404***,[186] isoprenyl phenol ***405***,[187] and alkyl resorcinols ***406–408***.[188]

Culture of *Aureobasidium melanogenum* yielded aliphatic δ-lactones ***409–411*** and fatty acid methyl esters ***412–414***,[189] sterol ***415*** was isolated from a culture of *Beauveria* sp.[190] and triterpenoid ***416*** was obtained from shellfish-derived *Ceriporia lacerata*.[191] Sesterterpenoids with a 5/8/6/5 tetracyclic ring system, sesterchaetins A ***417*** and B ***418*** and diepoxide polyketides ***419*** and ***420***, were isolated from deep sea sediment-derived *Chaetomium globosum*,[192] while sponge-derived *C. globosum* yielded six new cyctochalasans, marcytoglobosins A ***421*** and B ***422*** and marchaetoglobins A–D ***423***, ***424–426***, of which ***423*** is the first furan-containing cytochalasin.[193,194] Asepterpenedol A ***427***, an indole sesquiterpenoid obtained from culture of a *Chloridium* strain, contains a seven-membered ether ring as part of a 7/6/5/5/6/6 hexacyclic framework.[195]



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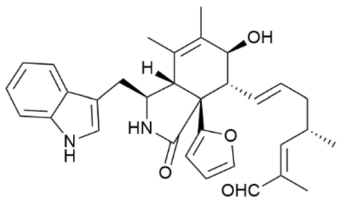

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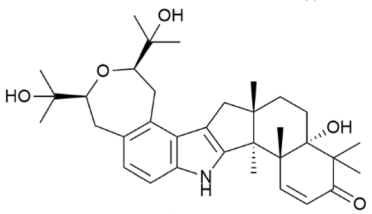

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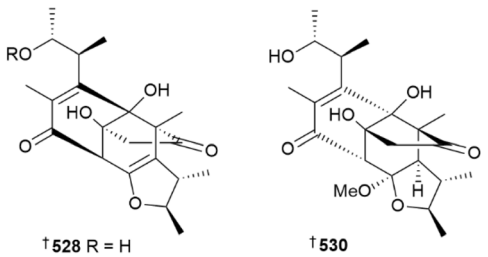
Cladosporium strains were the source of a range of new metabolites. Dodecanoic acid derivatives ***428–431*** (the last two known as semi-synthetic compounds) were isolated from a culture of *C. cladosporioides*,[196] *C. halotolerans* yielded pyrone derivatives ***432*** and ***433***,[197] drimane sesquiterpenoids ***434***, isocoumarin ***435***, coumarin ***436*** and known synthetic compound, 9-formylfluorene ***437*** as a new NP,[198] and a further *Cladosporium* strain was the source of isochromanone ***438***.[199] *Curvularia aeria* and *C. lunata* respectively yielded tricyclic aromatic polyketide ***439*** (as a racemic mixture) and resorcylic acid lactones ***440–443*** and isolation of curdepsidone A from a *Curvularia* strain led to revision of the structure to ***444***.[200–202]

A one strain many compound (OSMAC) approach to cultivation of *Emericellopsis maritima* resulted in isolation of eremophilane sesquiterpenoids ***445–447***,[203] 6/7/6 tricyclic diterpenoid eutyditerpenoid A ***448*** and cytochalasin ***449*** were obtained from a culture of *Eutypella scoparia*,[204] whilst another *Eutypella* strain yielded fifteen new sesquiterpenoids, eutypelides A–O ***450–464***.[205] Sesquiterpenoids, including eremophilane derivatives fureremophilanes A–D ***465–468*** and acorane analogues furacoranes A ***469*** and B ***470***, were isolated from culture of *Furcasterigmium furcatum*.[206] *Fusarium* strains were the source of fusarochromanone derivatives ***471*** and ***472***, hydroxyphenylacetic acid ***473–476*** and hydroxyphenylethanol derivatives ***477*** and polyketide ***478.***[207–209] The cyclic pentapeptides, avellanins D–O ***479–490*** were obtained from culture of *Hamigera ingelheimenensis*.[210] An *Irpex* strain produced the heptaketides irpetones A ***491*** and B ***492***,[211] catechol derivatives, meirols A–C ***493–495*** were obtained by fermentation of a *Meira* strain,[212] whilst a *Microascus* strain yielded eight decahydrofluorene alkaloids, ***496–503***.[213] Azaphilones ***504–506*** and a dihydroisocoumarin ***507*** were isolated from a strain of

Neopestalotiopsis,[214] mangrove sediment-derived *Nigrospora oryzae* contained four aniline derivatives ***508–511***,[215] spiro trione compound ***512*** and asparvenone derivatives ***513–516*** were obtained from a culture of *Paraconiothyrium sporulosum*,[216] and a culture of *Parengyodontium album* was the source of aromatic polyketide alternaphenol B2 ***517***.[217]

The *Penicillium* genus has proven once again to be a prolific source of new metabolites. A phenyl 6,7-dihydroxygeranyl ether derivative ***518*** was obtained from a *P. arabicum* culture,[218] *P. brasilianum* yielded diketopiperazine alkaloids ***519*** and ***520***,[219] soft coral-derived strains of *P. chrysogenum* were the source of aromatic heterocycles ***521–525***, and a sorbicillinoid ***526***,[220] and of anthranilic acid-peptide derivative ***527***.[221] Dicitrinols A–C ***528–530***, citrinin derivatives with a 6/5/7/5 core, were obtained from *P. citrinum* derived from hydrothermal vent sediment,[222] a culture of *P. corylophilum* yielded enantiomeric hydroxyphenylacetic acid derivatives ***531–534*** and α-pyrone analogue ***535***,[223] and co-culture of deep-sea sediment-derived *P. crustosum* with mangrove-derived *P. citrinum* resulted in production of the dihydropyrones, rhytismatones C ***536***, and D ***537***.[224]



†528 R = H
†529 R = CHO
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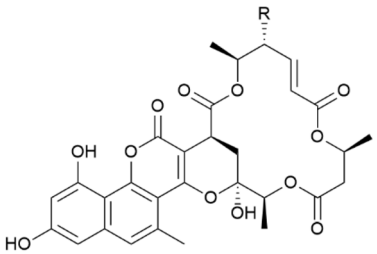
The relative and absolute configuration of the known indolone, notoamide X ***538*** was assigned for the first time. It was obtained from the mangrove sediment-derived *P. janthinellum* along with three other indolones with proposed new structures. However, the structures of these additional compounds were only proposed based on MS data and so are not included here.[225] Culture of *P. oxalicum* derived from a red alga yielded prenylated indole alkaloids ***539*** and ***540*** and polyoxygenated steroid ***541***,[226,227] sediment-derived strains, *P. pancosmium* and *P. rubens* respectively yielded meroterpenoids ***542*** and ***543*** (*P. pancosmium*)[228] and polyketide derivatives ***544*** and ***545*** and amino-bis-tetrahydrofuran derivatives ***546*** and ***547*** (*P. rubens*),[229] while a coral-derived *P. sclerotium* culture was the source of azaphilone pigments ***548–559***.[230] Highly oxygenated meroterpenoids, penisimplinoids A–K ***560–570***, were isolated from a *P. simplicissimum* culture,[231] steroid ***571*** and polyketides ***572*** and ***573*** were obtained from *P. variabile*[232] and seagrass-derived cultures of *P. velutinum* and *P. yezoense* respectively yielded piperazine derivative ***574***,[233] and decalin polyketides ***575–584***.[234] Meroterpenoids, including ***585–591*** and penicianstinoids F–K ***592–597***, indole diterpenoids, including ***598–604*** and penpaxilloids F ***605*** and G ***606*** and diketopiperazine alkaloid ***607*** (the last as a racemate), were isolated from cultures of various *Penicillium* strains.[235–239] Sediment-derived strains were the source of azaphilones ***608–612*** and clavatol derivative ***613***,[240,241] and *Penicillium* strains derived from seawater yielded racemic polyketides ***614*** and ***615*** and enantiomeric hydroxyphenylacetic acid derivatives ***616*** and ***617***,[242,243] which were separated by chiral chromatography. Cultures of various *Penicillium* strains also resulted in production of racemic phenalenone ***618***,[244] quinolone-citrinin hybrids ***619*** and ***620*** and speradines I ***621*** and J ***622***.[245,246]

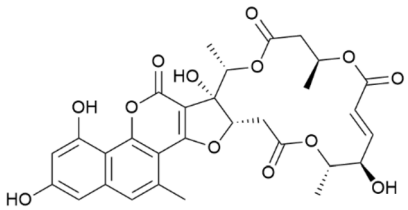
Mangrove sediment or root-derived *Penicillium* spp. have also yielded pyridyl derivatives ***623*** and ***624***, indole ***625***,[247] isobenzofuranone derivative ***626***, α-pyrone ***627***, quinone ***628***, phenol derivative ***629***,[248] steroids ***630*** and ***631***, benzopyran ***632***,[249] diterpenoid indole alkaloid derivatives ***633*** and ***634***,[250] sesquiterpenoids eupenicisirenins C–O ***635–647***,[251] aryl and decalin polyketide acids ***648–669***,[252] and alkyl resorcinols ***670–673***.[253]

Peniotrinins A–F, comprising cytochalasin derivatives ***674*** and ***675***, citrinin derivatives ***676–678*** and tetramic acid derivative ***679***, were isolated from a culture of a *Peniophora* strain[254] and merocytochalasans, perochalasin A ***680***, and inseparable C20 epimeric pairs ***681*** and ***682*** were obtained from a *Peroneutypa* species. The BGC responsible for cytochalasin biosynthesis was identified through full genome sequencing and the biosynthesis elucidated through feeding experiments utilising ^{15}N and deuterium labelled hydroxylamine.[255] *Pestalotiopsis* strains were the source of rearranged pimarane diterpenoid ***683*** and known terrestrial but new MNPs, nodulisporenones A ***684*** and B ***685***,[256] and polyketide derivatives, pestalotiopols E–J ***686–691*** (the last two as a racemic mixture).[257] Cultures of deep-sea sediment-derived *Phomopsis* strains yielded benzoic acid derivative ***692*** and 3-carboxy-indole derivatives ***693–695***.[258,259] Co-culture of two *Phomopsis* strains obtained from the roots of the Chinese mangrove *Rhizophora mangle* yielded pyrazine ***696***, steroid ***697*** and α-pyrone ***698*** (from *P. asparagi*)[260] and bis-chromones phomoxanthones L–N ***699–701*** (from *Phomopsis* sp.),[261] none of which were produced in monoculture of the individual strains.

Sediment-derived fungal strains have yielded the sesquiterpenoids ***702–711*** (from *Pseudallescheria boydii*)[262] naphthopyrone-macrolide hybrids, gymnoasins A–C ***712–714*** and glucolipid ***715*** (from *Pseudogymnoascus* strains) and sesquiterpenoids ***716*** and ***717*** (from *Pyrrhoderma noxium*).[263–265] Isolated from the Antarctic, the gymnoasins A–C ***712–714*** are the first naturally occurring compounds of their type and a biomimetic synthesis of ***712*** was achieved.[263] Deep-sea sediment-derived fungal strains were the source of bisabolene sesquiterpenoid ***718*** (from *Retroconis fusiformis*),[266] sesquiterpenoid-hydroquinone hybrids, saromacrophorins A–C ***719–721*** (from *Sarocladium terricola*)[267] and 4-hydroxyphenyl derivative ***722*** (from *Scedosporium apiospermum*), which displayed moderate to potent activity against a panel of *Candida auris* strains,[268] while mangrove sediment-derived *Roussoella* sp. contained two sesquiterpenoids ***723*** and ***724*** and a shikimate ***725***.[269]



†712 R = OH
713 R = H



†714

Dihydroisocoumarins, exserolides L *726* and M *727* were obtained from culture of a *Setosphaeria* species,[270] while various *Simplicillium* strains yielded a range of metabolites including nortriterpenoids *728–730*,[271] peptaibiotics *731–741*,[272] dipeptide *742* and acylated valine derivative, siamysin *743* (the last discovered *via* heterologous expression of a cryptic BGC).[273,274] Eleven brominated depsidones *744–754* were isolated from a culture of *Spiromastix* sp.,[275] *Stachybotrys* strains yielded atranones V–Z *755–759* and dolabellane diterpenoids *760–762*,[276] and phenylspirodrimanes, stachybotrins K *763* and L, *764*,[277] and cyclic tetrapeptides, endolides E *765* and F *766* were obtained from a *Stachylidium* strain. The endolides contain the unusual amino acid, *N*-methyl-3-(3-furyl)-alanine which was used in fragment pattern searches of tandem mass spectrometry data, in conjunction with molecular networking, to assist both isolation and structural elucidation of these metabolites.[278]

Culture of *Talaromyces* strains isolated from sediment have yielded a diverse array of metabolites, including oligophenalenone dimers *767–775*,[279] phenylhydrazone alkaloids *776–779*,[280] highly oxygenated *seco*-terpenoids *780–785*,[281] macrolactin *786*,[282] highly oxygenated phenol derivatives *787–789*, isocoumarin glycosides *790* and *791* and naphthalene glycoside *792*.[283] Culture of *T. purpureogenus* yielded six prenylated indole diketopiperazine alkaloids, talaromyines A–F *793–798*,[284] cyclic heptapeptides *799* and *800*, nor-diterpenoids *801* and *802*, diterpenoid acids *803* and *804* and triterpenoid *805* were obtained from cultivation of *T. scorteus*,[285] *T. siglerae* yielded cyclic heptapeptides, talaromides A–C *806–808* and *T. variabilis* was the source of hybrid pentaketide sesquiterpenoids *809–811*.[286,287] Seawater-derived *Talaromyces* strains yielded meroterpenoids *812–814* and benzoquinone *815* and furopyridinone *816* derivatives,[288,289] a strain derived from sediment was the source of ring-opened azaphilone derivative *817*,[290] thirteen new polyketide derivatives *818–830*, (6-hydroxy-4-methoxycoumarin *829* a known synthetic compound but new NP) were obtained from a deep-sea mussel-derived strain,[291] and duclauxin analogues, taladuxins A–N *831–844* were isolated from a culture of a coral-derived *Talaromyces* strain from which verruculosin B was also isolated and the configuration revised to 1*S* as in *845*.[292] Investigations of mangrove sediment-derived *Talaromyces* spp. have resulted in isolation of γ-lactams, talaroilactams A–D *846–849*,[293] sesquiterpenoids, talaroterpenoids A–D *850–853*,[294] *p*-terphenyl derivatives, talaroterphenyls A–D *854–857*,[295] diketopiperazines, *858–860*, a cyclopentenone *861*, an α-pyrone *862*,[296] and linear polyketides *863–868*.[297] Two of the talaroterphenyls *855* and *856* potently inhibited the enzyme phosphodiesterase-4 (PDE4) linked to inflammation.

Tetramic acid alkaloids, tolypyridones I *869* and J *870* were isolated from a culture of *Tolypocladium cylindrosporum*,[298] *Trichoderma* strains were the source of sesquiterpenoid derivatives *871–874*,[299] bergamotene-derived sesquiterpenoids *875–882*, (through heterologous expression of a cryptic BGC),[300] cyclonerane sesquiterpenoid *883*,[301] γ-butyrolactones *884–889* and monomeric sorbicillinoids *890–893*.[302,303] Steroid derivatives *894* and *895* and linear polyketide *896* have been isolated from mangrove sediment-derived *Trichoderma* spp.[304,305] and culture of *Xylaria acuta* yielded a range of metabolites; lactones *897*, *898*, *899*, glucopyranosides *900* and *901* and mannopyranoside *902*, epoxycytochalasins *903* and *904* and butylitaconic acid *905*.[306]

Total synthesis of acremolides A and B has been achieved and the absolute configurations established as *906* and *907*,[307] while asymmetric syntheses of aspilactonol F and aspiketolactonol were achieved in eight and ten steps respectively and their absolute configurations determined as *908* and *909*.[308] (±)-Notoamide N was prepared in a biomimetic approach *via* synthesis of (±)-stephacidin A,[309] and several prenylated indole alkaloids of the asperversiamide family, namely asperversiamides B, D, E, G and J and dihydrocarneamide A, were prepared in biogenetically patterned total syntheses. The structure originally proposed for asperversiamide A was shown to be incorrect with a revised structure proposed but not yet proven.[310] A unified approach was utilised to prepare cyclic tetrapeptide JM-47,[311] cyclocondensation of an electrophilic homoserine lactone was employed in the total syntheses of polonimides A and C[312] and synthesis of bipenicilisorin was achieved through initial preparation of its monomer penicilisorin and the absolute configuration was established as *910*.[313] Penostatin D was prepared in an asymmetric synthesis from L-ascorbic acid,[314] 12β-hydroxy conidiogenone C was also obtained in an asymmetric total synthesis[315] and total synthesis of the cyclic peptide, talarolide A showed that it (and by inference, talarolides C and D), occur naturally as atropisomers.[316] Racemic total synthesis of phomopsol B has been reported[317] and syntheses of asperisocoumarin A and fusarimatin C were achieved from commercially available starting materials.[318]

Alternariol monomethyl ether was shown to relieve food allergy symptoms in mice and reduce histamine release into blood serum,[319] the verticillin analogue, leptosin A showed potent inhibition of the parasite *Toxoplasma gondii*,[320] brevianamide F exhibited antithrombotic activity in a zebrafish model[321] and agonodepside B showed potential as a photoprotection agent.[322] Fumetremorgin derivatives 12*R*,13*S*-dihydroxyfumitremorgin C and tryprostatin A displayed antihypercholesterolemia activity in an *in vivo* model,[323] terphenyl

HN-001 was shown to attenuate obesity in mice through induction of thermogenesis in adipose tissue[324] and to suppress lipotoxicity from metabolic-associated fatty liver disease through inhibition of phospholipase A_2 (PLA2)[325] and linoleic acid ameliorated metabolic dysfunction-associated steatotic liver disease in mice by targeting fatty acid binding protein 4.[326] The mechanism underlying the antithrombotic activity of the peptide isaridin E was shown to be associated with inhibition of von Willebrand Factor secretion from activated platelets and this leads to protection against sepsis,[327] butyrolactone-I alleviated intestinal heat stress *in vivo* through inhibition of two key enzymes which are upregulated in heat stress[328] and pannorin was shown to be a selective, if relatively weak (IC_{50} 1.73 μM) monoamine oxidase inhibitor.[329]

The BGC responsible for the biosynthesis of the quinolone alkaloids, the asperalins in *Aspergillus alabamensis*, was identified and the biosynthesis elucidated through heterologous expression.[330]

'Omics'-based methods for isolation and cultivation of marine fungi have been reviewed[331] as have genome mining and biosynthetic pathways of biologically active marine fungal metabolites.[332]

2.4 Dinoflagellates

The number of new compounds reported from dinoflagellates and other microalgae is slightly lower than the decadal average.[1] *Prorocentrum lima* was the source of polyketides prorocentin-5 ***911*** and three okadaic acid derivatives ***912–914***, although surprisingly, the latter compounds were not assessed for bioactivity.[333,334] An indole methyl ether ***915*** and a biaryl ester ***916*** were reported from a Vietnamese *Aurantiochytrium* strain, although ***915*** is a likely artefact of isolation.[335]

Ectoine plays an important role in controlling osmotic responses in marine planktonic species. Using a hydrophilic interaction liquid chromatography (HILIC) based molecular networking approach, new analogue 2-homoectoine ***917*** was detected in multiple harmful algal bloom genera including *Alexandrium*, *Karenia*, and *Prorocentrum*. 2-Homoectoine plays a similar osmoadaptive role as archetype ectoine, for the producing strains. The synthesis of ***917*** was also achieved.[336]

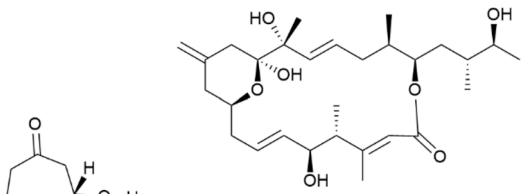
Other structures were suggested from MS and transcriptomics data but are not shown here.[337,338]

Although only isolated in 2023, the total synthesis of diatom sex pheromone peptide SIP^+ has been accomplished,[339] as has the production of *seco*-steroid gibbosterol A.[340] The original structure of iriomoteolide-1a was disproven by synthesis in 2010, but efforts have not revealed the correct constitution of the molecule until now. The corrected structures of iriomoteolides-1a ***918*** and 1b ***919*** have now been determined following an extensive integration of DFT calculations, NMR analysis, and total syntheses.[341]

A summary of the biomedical application of *Alexandrium* spp. NPs has been published.[342] Compound specific reviews focused on amphidinolides,[343] azaspiracids,[344] and ciguatera poisoning, especially related to ciguatoxins.[345] The structure of ciguatoxin-5 ***920***, originally proposed based solely upon MS data, has been confirmed by NMR spectroscopic analysis of only 40 μg of isolated material.[346] The ambiguity between synthetic and naturally occurring portimine B has been resolved, in part by use of the new i-HMBC NMR experiment that can differentiate $^2J_{C,H}$ *vs.* $^3J_{C,H}$ correlations. Differences in the NMR spectra observed were attributed to the formation of a transient hemiaminal hydrate, facilitated by acid modifiers added to aid HPLC separation. The authors advise researchers to be careful when adding acid or base to their chromatography solvents, especially when comparing spectroscopic data to literature values.[347]

The structure of 44-methylgambierone ***921*** was revised following extensive NMR analysis. The compound was also found to be unstable in DMSO-d_6 used for spectroscopic analysis hence a microstudy was undertaken to determine its stability towards various solvents over the course of eight months. The effects of the solvent chosen for NMR analysis, and potential storage, should be considered before committing an entire sample.[348] A study of the accumulation of domoic acid (DA), the causative agent of amnesic shellfish poisoning, in five species of invertebrate including commercially valuable shellfish, has shown that DA concentrates in scallops (*Pecten maximus*) at levels 20-fold higher than in clams, slippersnails and sea squirts. The *Pectenid* species showed a distinctive ability to accumulate, transform and distribute DA within their body tissue.[349] Transcriptomics analysis of saxitoxin and non-

†917



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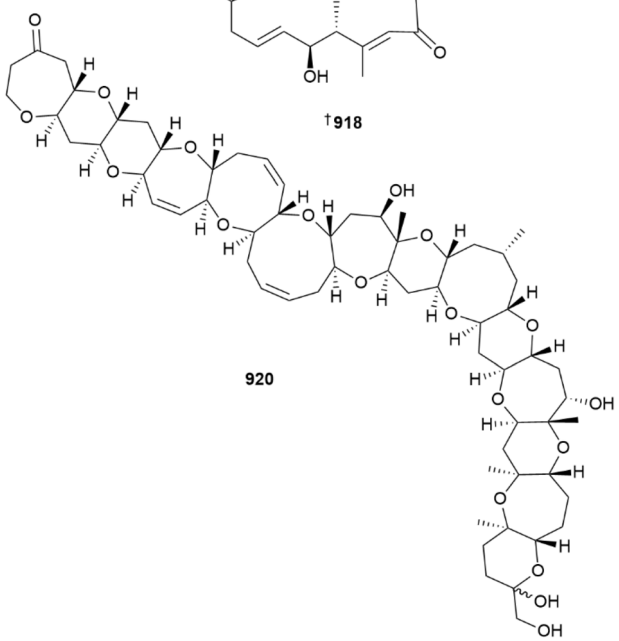


920

saxitoxin producing species of the prymnesins, super-carbon chain ladder polyethers produced by *Prymnesium parvus*, are some of the largest known natural products yet their biosynthetic origins have remained elusive until now. The megadalton PKZILLA-1 and -2 polyketide BGC's encode for 140 and 99 enzyme domains, respectively, that match the predicted "pre-prymnesin" linear backbone. A smaller PKZILLA-B1 encodes for a smaller analogue, prymnesin-B1. This works expands the state of knowledge of large biosynthetic genes and enzymatic pathways to important biomolecules.[350]

3 Green algae

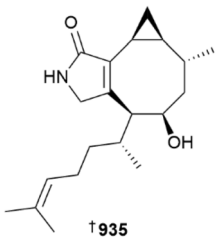
A Chinese *Caulerpa racemosa* var. *turbinata* yielded caulespiros A ***922*** and B ***923***, with neither being active against various bacterial strains.[351] Mebamamide C ***924*** was discovered from a Japanese specimen of *Bryopsis*, but also from the algal gastropod predator *Elysia marginata*.[352]

Two notable reviews of green algal metabolites were published in 2024. One focused on the allelopathic compounds from *Ulva prolifera*, the algal species that creates "green tides",[353] while the other summarised the metabolites and bioactivities of compounds from *Chlorella vulgaris*.[354]

4 Brown algae

Glyceroether ***925*** and polyunsaturated glycolipid ***926*** metabolites were reported from *Sargassum* sp. (China) and *Hizikia fujiformis* (South Korea), respectively.[355,356] Macrocarquinoid J ***927*** and sargasilols J–N ***928–931*** are meroterpenoid chromanes from *Sargassum* spp., although all are weak or inactive as anti-inflammatory agents.[357,358] An Indian *Stoechospermum marginatum* was the source of three new spatane diterpenoids ***932***, ***933***, ***934*** that showed weak cytotoxicity against four HTCLs.[359]

Coriaceumins A–D ***935***, ***936–938*** are rare examples of nitrogenous xenicane diterpenoids. Two diterpenoid lactams, dictyolactams C ***939*** and D ***940*** were also isolated from the same algal species, *Dictyota coriacea*, collected at Nanji Island, Zhejiang Province, China, although the lactams are artefacts of isolation as proven by extraction using different alcoholic solvents.[360]



†935

The total syntheses of ecklonialactones C and D, and of eiseniachloride A, have been achieved for the first time.[361] Notable reviews include a summary of the phytochemicals produced by *Halopteris scoparia*,[362] and those from the Sargassaceae between 1975 and August 2023.[363] Marine carotenoid fucoxanthin has been shown to reduce the effects of autism spectrum disorder induced by valproate treatment in a mouse model in a dose dependent manner. This reduction is induced by alterations to AKT/GSK-3β signalling.[364]

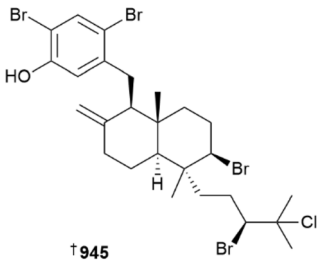
5 Red algae

An Okinawan *Portieria* sample yielded a new halogenated monoterpenoid ***941***, which also necessitated the reassignment of related compound ***942***. Both metabolites exhibited anti-fouling activity at 0.16 μmol cm^{-2}.[365] The only other report of new red algal metabolites was of diastereomeric sesquiterpenoids laurenolides A ***943*** and B ***944*** from a Vietnamese sample of *Palisada intermedia*.[366]

The same group has reported the total synthesis of pigments borolithochromes A, D, G, H1, H2, I1 and I2, metabolites of fossilised putative red alga *Solenopora jurassica*, *via* two separate publications.[367,368]

A review of the metabolites of *Gelidium corneum* including mycosporine-like amino acids, carotenoids and polyphenols, along with their associated bioactivities, has been published.[369] The current status of Japanese *Laurencia* complex focusing on chemical diversity and morphological characters for identification has been reviewed.[370] A comprehensive review of the metabolites of *Asparagopsis* and their use for chemotaxonomic analysis of the genus has also been released.[371]

A study has analysed the metabolic profile of different races of *Laurencia nipponica* considering their cytotoxicity, genotoxicity and their antifungal properties as it all relates to chemotaxonomy. Ultimately, with the aid of multivariate statistics, three different races can be differentiated using four different compounds, laurencin, laurallene, prepacifenol and laureatin.[372] An innovative approach to establishing the absolute configuration of metabolites with vicinal bromo-chloro substituents using chiroptical methods has been published. By applying van't Hoff's principle of optical superposition, and with the aid of chiroptical measurements of suitably well characterised model compounds for comparison, the contributions of different chromophores within a compound to the overall molar rotations can be mathematically disentangled to provide the absolute configuration of the metabolites in question. Use of this method has necessitated the revision of the structures of the halogenated meroditerpenoids callyphycols A ***945*** and B ***946*** and suggested that the structures of other metabolites within the class should be reinvestigated.[373]



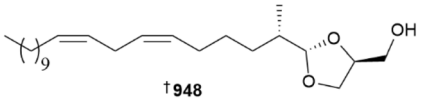
†945

6 Sponges

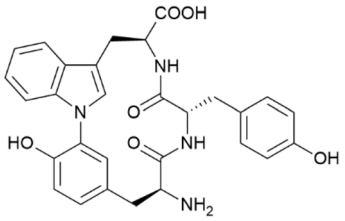
Aurantoside L ***947*** is an anti-leishmanial tetramic acid glycoside from *Siliquariaspongia japonica*.[374] A *Leucetta* sponge from Bohol Province, the Philippines, was the source of five dioxolane ether lipids ***948***, ***949–952***. To probe the relative and absolute configurations of their dioxolane cores, model compounds were prepared and their NMR data compared with that of the isolated metabolites. Even then, some compounds were indistinguishable, so the authors utilised europium-based lanthanide chiral shift reagents to ultimately discriminate all the compounds. None of the metabolites exhibited antibacterial activity.[375]

Endoperoxides, plakortides V ***953*** and W ***954***, and related gracilioether M ***955***, 11,12-dihydrogracilioether M ***956***, and 9,10-dihydrogracilioether G ***957***, were reported from *Plakinastrella* and *Plakortis* sponges, respectively.[376,377]

Somewhat surprisingly, only two new peptides from sponges were reported. Halichondamide A ***958*** was isolated from a USA-sourced *Halichondria bowerbanki*, with molecular dynamics simulation used to help delineate the biosynthetic formation of its disulfide bonds; note the name halichondamide should not be confused with that of the much more commonly found halichondramide macrolide family.[378] Neopetromin ***959*** is a cyclic tripeptide that contains a rare C–N biaryl link. Although inactive against two HTCLs, various enzyme targets, and as an antimicrobial, it does cause vacuole fragmentation in an actin-independent manner in tobacco BY-2 cells, like other actin polymerisation inhibitors but without disrupting cellular actin-dynamics. Studies of the impact of this unique bioactivity upon plant physiology are underway.[379]



†948



†959

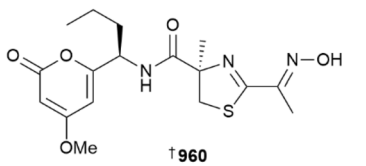
PM742 ***960*** is the first example of a polyketide α-pyrone coupled to an oxime-containing, non-ribosomal peptide. Isolated from a *Discodermia* sponge, PM742 is potently cytotoxic against eight cancer cell lines. Mode of action studies indicate it binds at the colchicine site, causing mitotic arrest *via* inhibition of microtubule dynamics at the G2/M checkpoint.[380] A synthetic SAR study identified an analogue, PM534, that covers 80% of the centres within the pharmacophore model of the colchicine binding site. PM534 has demonstrated a potent ability to reduce tumour growth in mouse xenograft models and has entered phase 1 clinical trials for the treatment of non-small cell lung cancer.[381]

A new mycothiazole congener ***961*** was reported from a sample of *Cacospongia mycofijiensis* collected in Vanuatu. Although a potent cytotoxin, the new analogue is still 400- to 700-fold less active than mycothiazole itself which is cytotoxic in the pM-range. Nematodes (*Caenorhabditis elegans*) treated with sub-lethal doses of ***961*** exhibited an extended mitochondrial lifespan. SAR analysis shows that *Z*-Δ^5 alkene geometry is critical for pM levels of cytotoxicity but does not impact the mitochondrial life extension bioactivity.[382]

Polybrominated ***962*** and lipidated ***963*** phenols have been reported from *Lendenfeldia* and *Hemimycale* sponges, respectively.[383,384] Several lactam- (***964–967***) and pyridine-based (***968*** and ***969***) alkaloids have been reported from a Chinese (Xisha Is.) *Pseudospongosorites suberitoides*. All are likely artefacts of extraction with methanol and are inactive as antivirals.[385] A pair of enantiomeric indolinone alkaloids, ***970*** and ***971***, were sourced from a *Tedania* sponge.[386] Neopetrotaurines A–C ***972–974*** are taurine-bridged isoquinoline dimer alkaloids that are weak suppressors of oncogenic fusion protein PAX3-FOXO-1 driven transcription. Neopetrotaurine C is a likely artefact of isolation.[387]

A new bromopyrrole glycoside has been reported from a deep sea *Lissodendoryx papillosa* specimen. Named in honour of the major contributions of Prof. Valentin Stonik to the field of MNPs, stonikacidin A ***975*** contains the unusual sugar, L-idonic acid, at its core. Although inactive against numerous mammalian cell lines, ***975*** has some antibacterial activity and it inhibits biofilm formation in *S. aureus*.[388]

Pyrroloiminoquinone alkaloids are commonly encountered in Latrunculid sponges. A dredged *Latrunculia* specimen yielded 6-chlorodamirone A ***976*** and its brominated analogue ***977***; over time the latter converts to the former when exposed to chloride in seawater in a non-enzymatic reaction. The total synthesis of both compounds was also achieved.[389]



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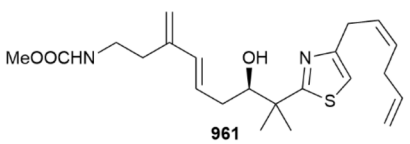
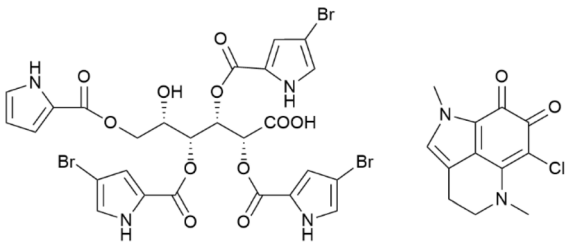


961



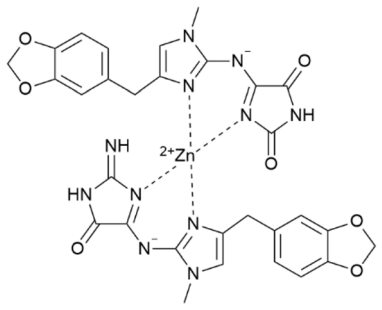
†975 976

The PIP-HSQCMBC-IPAP NMR pulse sequence was instrumental in helping identify a series of discorhabdin D congeners ***978–982*** reported from New Zealand *Latrunculia* spp.[390]

Multiple pyrrololactam alkaloids and their related metabolites (***983–1007***) have been reported from *Phakellia*, *Stylissa*, and

Pseudospongosorites sponges collected around Xisha Is. in the South China Sea.[391–393] A New Zealand *Dictyodendrilla* c.f. *dendyi* yielded five new sulfated denigrin metabolites ***1008–1012*** with one rearranging spontaneously upon short term storage in an NMR tube.[394]

A diverse series of metabolites, including multiple 2-aminoimidazolone derivatives, were isolated from calcareous sponge *Pericharax heteroraphis* collected from a lagoon in Wallis and Futuna Is.; a new Zn^{2+}-containing, heterodimeric clathridine A – clathridimine complex ***1013*** was one of the isolates. Although inactive as a stimulant of endochondrial ossification, the structure of the compound was confirmed by total synthesis as were those of several other congeners.[395]



1013

New bromotyrosine derivatives were isolated from *Suberea* ***1014–1017***, *Ircinia* ***1018–1021***, and *Pseudoceratina* ***1022*** sponges collected in Australia, the Philippines, and Okinawa, respectively.[396–398] Merosesquiterpenoids have been reported from *Pseudoceratina purpurea* (***1023***, ***1024***, and ***1025–1036***) and *Dysidea arenaria* (***1037–1042***), although all the isolates were only weakly active or inactive altogether in a variety of bioassays.[399,400] Further meroterpenoids were isolated from *Hyrtios* (***1043*** and ***1044***) and *Hippospongia* (***1045–1052***) sponges,[401,402] while a number of norditerpenoid cyclic peroxides ***1053–1062*** were reported from a Bohol Is. (the Philippines) specimen of *Diacarnus spinipoculum*.[403] A *Hemimycale* sponge was the source of *seco*-diterpenoid ***1063***, which was not assessed for bioactivity.[404] Isolation of ircinialactams ***1064–1066*** from a Mexican *Ircinia felix* also provided an opportunity to revise the structure of related variabilin metabolite ***1067***; the total synthesis of ***1064*** was also achieved.[405] An Antarctic *Suberites* sponge yielded suberitenone sesterterpenoids ***1068–1072*** although none showed activity as antivirals.[406] Given how common scalarane sesterterpenoids are in marine sponges, surprisingly only one, lendenfeldarane V ***1073***, was reported in 2024,[407] while two norsterol epoxide saponins ***1074*** and ***1075*** were obtained from a *Petrosia* sponge collected in the Philippines.[408] *Seco*-Sterols were reported from *Cliona* (***1076***), *Hippospongia* (***1077***, ***1078***) and *Spongia* (***1079***) sponges, respectively.[409–411] A series of 12 nor-terpenoid peroxides, ***1080–1091***, all derived from oxidative cleavage of larger metabolites, were isolated from a *Diacarnus* sponge and all with varying levels of antibacterial and cytotoxic activities.[412] Other structures have been claimed but are inconsistent with the spectroscopic data provided, or were only detected by GNPS-based molecular networking, and hence are not shown.[384,404,413]

Numerous first total syntheses were reported and these included halisphingosine A ***1092***, which necessitated a revision of structure,[414] cyclic peroxide ethyl plakortide Z,[415] peptide neopetromin,[416] and macrolide salarin C, which also validated a biosynthetic oxidation – Wasserman rearrangement to give salarin A in a biomimetic approach.[417] A multiplexed strategy using 96-well plates to optimise conditions for reactions between fatty dialdehydes, a nitrogen source and acrolein has been used to probe and verify the Whitehead – Baldwin biosynthetic proposal for the manzamines.[418] Indole alkaloids dragmacidins G and H, and tulongicin A, have been prepared for the first time,[419,420] as has aminophenol erebusinone, leading to its revised structure ***1093***.[421] The first total synthesis of spirodactylone has been accompanied by the synthesis of several related denigrin metabolites that has indicated the published structure of denigrin C is incorrect, although no alternative was proposed.[422] Guanidine alkaloids (−)-monanchoradin A and (−)-crambescin A2 393 have been prepared for the first time,[423] as have makaluvamines H, L, M and N.[424,425] A series of BF_2-complexes of ageladine A have been prepared as BODIPY mimics, with their fluorescence being heavily dependent on both pH and halogenation of the imidazole ring.[426] Psammaplysins M, O and Q, along with ceratinamide A, have been synthesised as racemates,[427] as have sesquiterpenoids halichonadins A, B and C.[428]

A review of sponge-derived MNPs with activity against lung cancer has been published.[429] Taxa-based reviews have focused on compounds from the *Spongia* and *Neopetrosia* genera, respectively,[430,431] while compound specific reviews have summarised the literature pertaining to the convoluted structure elucidation, total synthesis and biological evaluation of PUFA-derivative (−)-mucosin,[432] and also of the immunomodulatory effects of halichondrin and its derivatives.[433] Other compound specific reviews have concentrated on aaptamine and its derivatives,[434] the anti-cancer potential of marine bis-indoles such the dragmacidins and topsentins,[435] and of bromotyrosine-derivatives, mainly from Verongiid sponges.[436] A compendium of 413 sponge-derived alkaloids published between 2000–2023 has been released,[437] with a similar summary of diterpenoids isolated from sponges in 2009–2022 covering 228 structures.[438]

The inseparable endoperoxide pair, plakortinic acids C and D, isolated from the mutualistic symbiotic sponge pair *Plakortis symbiotica* and *Xestospongia deweerdtae*, have antiplasmodial activity without causing haemolysis. They exhibit promising absorption, distribution, metabolism, excretion, and toxicity (ADMET) properties making them potential antimalarial leads.[439] Chemoproteomic analysis has indicated that ubiquitin carboxyl-terminal lyase 5 (USP5) is the molecular target of endoperoxide gracilioether A (*Plakinastrella mamillaris*). USP5 is heavily involved in chromatin maintenance, catalyses degradation of abnormal proteins, and is over-expressed in multiple cancer types. Inhibitors of USP5 therefore have promise as anticancer agents.[440]

Peptide stylissatin A and its analogues inhibit membranous neuraminidase-1 functioning by modulating the chaperone

activity of cathepsin A, thereby having application in preventing the formation of adipose tissue.[441] Chemoproteomics has also been used to detect agents that protect against inflammatory acute lung injury. Naamidine J interacts with His745 and Phe903 in chromosome segregation 1-like protein (CSE1L) which inhibits nuclear translocation and transcriptional activity of SP1, suppressing inflammation. This study also provided more naturally occurring material which facilitated the reassignment of naamidines A, G and J ***1094–1096*** through X-ray crystallographic analyses.[442] Although only weak inhibitors of *M. tuberculosis* under standard culturing conditions, axinellamines A and B have moderate to potent activity against the pathogen when grown with limited protein supplementation, mimicking *in vivo* conditions. Both compounds are inactive against the RAW264.7 cell line as a model host.[443] Several bromotyrosine metabolites sourced from Verongiid sponges were found to be weak to moderate inhibitors of prion PrP^{SC} protein, which in turn reduces ER stress and also the spread of the pathogen in eukaryotic cells. This work provides a lead series of structures for development against a currently untreatable, and often fatal, disease.[444]

A nematocidal screen of over ten thousand fractions from approximately two thousand extracts held in the NatureBank repository highlighted seven extracts for further investigation. Deeper study of two chemotypes with activity against the sheep nematode *Haemonchus contortus* led to halaminol A and several agelasines as the dominant bioactive metabolites, but only agelasine B showed weak activity against the model roundworm *Caenorhabditis elegans*, suggesting either a minor component is responsible for the broader activity profile, or there is some instability in the isolated compounds.[445] Sterol sokotrasterol sulfate (*Topsentia* sp.) was found to inhibit IFN-γ-induced expression of programmed cell death – ligand 1 (PD-L1) protein *via* targeting Janus kinase with a downstream activation of signal transducer and activator of transcription (STAT) signalling, making it a new immunomodulatory lead.[446]

Assignment of the absolute configuration of antifungal depsipeptide cyclolithistide A ***1097*** has been completed by the synthesis of all four stereoisomers of the rare amino acid, 4-chloroisoleucine, and then use of Marfey's method to establish the configuration of the natural residue within the metabolite.[447]

An important cautionary observation regarding isolation of artefacts has been made. Pyrrole-containing MNPs including makaluvamines, discorhabdins, tambjamines and brominated tryptamines, were found to be trideuteromethylated when exposed to DMSO-d_6 NMR solvent in the presence of water, methanol, TFA and trace amounts of iron. Mechanistic investigation indicates that the reaction is caused by the presence of trideuteromethyl radical species generated by Fenton-type chemistry catalysed by the presence of iron or TFA.[448]

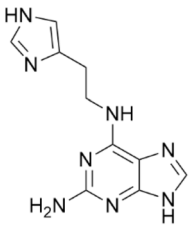
Finally, a study has explored the state of knowledge of the currently underexplored terpenoid biosynthesis in Dictyoceratid sponges, which, for example, are well known producers of spongian diterpenoids. While bioinformatic analysis detected analogues of known terrestrial prenyltransferases for one sesterterpenoid and multiple diterpenoid, steroid and carotenoid biosyntheses across various species, a lack of relatives to known terpenoid cyclase enzymes suggests the existence of divergent and unknown biosynthetic pathways in sponges. This study provides a start-point for exploring these currently cryptic biosynthetic genes and enzymes.[449]

7 Cnidarians

Tubastrine, sourced from the hard coral *Tubastraea tagusensis*, acts as an antioxidant to reduce levels of ROS, which is able to, in turn prevent neuron-like cell death caused by the oligomeric amyloid-beta peptide oAβ42.[450] While acroamine A ***1098***, a 2-amino adenine alkaloid reported from the soft coral *Acrozoanthus australiae*, was essentially inactive as an inhibitor of cAMP-dependent protein kinase A, several semi-synthetic analogues including di- and tri-bromo derivatives were active.[451] One of the more common family of alkaloids reported from cnidaria are the zoanthamines, isolated from *Zoanthus* sp. A new, efficient synthetic route to norzoanthamine has been reported, notable for including three photochemical reactions used at key steps.[452] Of a set of thirteen prostanoids isolated from *Clavularia* sp., two (***1099***, ***1100***) were new, with the former demonstrating weak *in vitro* cytotoxicity towards the Ca9-22 oral cancer cell line.[453] A new stereoselective approach to the synthesis of prostaglandin A_2 epimers (15*S*)-PGA_2 and (15*R*)-PGA_2 has been reported that makes use of only catalytic amounts of heavy metals.[454] The study also revealed that (15*R*)-PGA_2 is a weak inhibitor of $GABA_A$ receptors in *Xenopus laevis* oocytes, while the (15*S*)-epimer was inactive.

While the structures of sesquiterpenoids ***1101–1103***, isolated from *Lemnalia* sp., were established using a combination of NMR and MS techniques,[455] the structures of six of seven new sesquiterpenoids, bellissinanes A–G ***1104–1110*** (*Stereonephthya bellissima*) and each of lemneolemnanes A–D ***1111–1114*** (*Lemnalia* sp.) were secured using XRD.[456,457] Two new examples of isoledene-type sesquiterpenoids, ***1115*** and ***1116*** were isolated from the soft coral *Heteroxenia* sp., with both MNPs being inactive against SAR-CoV-2 and *Leishmania major*.[458] A deep-sea collection of *Anthothela grandiflora* afforded unusual dimethylamino substituted cadinene-type sesquiterpenoids, anthoteibinenes A–E ***1117***, ***1118***, ***1119–1121***.[459] Anthoteibinene B exhibited weak activity towards respiratory syncytial virus *in vitro*. Asymmetric syntheses of the sesquiterpenoids (−)-lemnalemnane A and (+)-lemnardosinane A, with both routes starting from (*S*)-carvone,[460,461] and a racemic synthesis of paralemnolin A have been reported.[462] Using a synthetic route starting from (−)-β-caryophyllene oxide, the structure of the chlorinated norsesquiterpenoid rumphellatin B has been confirmed, while those of rumphellatins A and C have been revised to ***1122*** and ***1123*** respectively.[463] During the study, it was observed that reaction of rumphelloilide C with HCl in dioxane afforded rumphellatin B, with the authors speculating that the simple transformation may occur in soft coral species.

The structures of meroterpenoids sinudenoids F ***1124*** and G ***1125***, isolated from *Sinularia densa*, are unusual due to the presence of a methylbenzoate core[464] while more classically functionalized meroterpenoids bearing hydroquinol or naphthoquinone cores ***1126–1129*** were isolated from *Litophyton brassicum*.[465] An asymmetric total synthesis of the pentacyclic benzosesquiterpenoid (+)-verrubenzospirolactone ***1130*** made use of a late-stage, spontaneous, intramolecular Diels–Alder reaction.[466]



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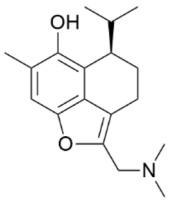

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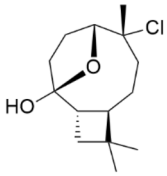

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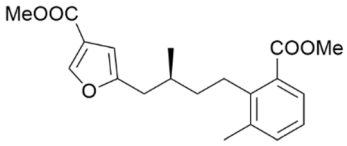

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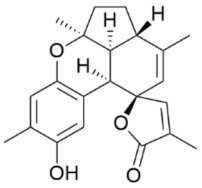


†1130

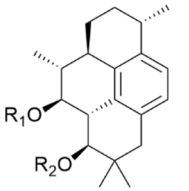
Four examples of diterpenoids embodying three different skeletons, herpetopanone B ***1131*** bellissimain A ***1132*** elisabethadienol B ***1133*** and elisabethadienol C ***1134*** were reported from extracts of *Stereonephthya bellissima*, with none showing activity in a pro-angiogenesis assay in zebrafish.[456] Further examples of biflorane-type diterpenoids, biofloranates E–I ***1135–1139***, in addition to two glycosides lemnabour sides H ***1140*** and I ***1141***, were reported from a South China Sea collection of *Lemnalia bournei*.[467] Of the set, only lemnabourside H exhibited antibacterial activity, weakly inhibiting the growth of *E. coli*. The structures and relative configurations of *seco*-diterpenoids kallopterolides A–I ***1142–1150***, isolated from the sea plume *Antillogorgia kallos*, were secured by combinations of NMR analysis and DFT DU8ML calculations.[468,469] Structures were assigned to polyoxygenated casbane-type diterpenoids sinueracasbanones E–O ***1151–1161***, isolated from *Sinularia erecta*, by extensive use of NMR data in conjunction with DFT DP4+ analysis, while absolute configurations were assigned by the use of Mosher's method and TDDFT-ECD calculations.[470] Sinueracasbanones I and O exhibited weak anti-inflammatory activity. The capnosane-type diterpenoid ***1162***, isolated from *Lobophytum pauciflorum*, was inactive in a zebrafish model of inflammation.[471] Three cycloamphilectane-type diterpenoids, sinucycloamtins A–C ***1163***, ***1164*** and ***1165*** were isolated from a South China Sea collection of *Sinularia brassica*.[472] Purification of sinucycloamtins B and C was hampered by the apparent ability of the two compounds to undergo intramolecular ester exchange. Subsequent saponification afforded a diol product, the structure and absolute configuration of which were established by XRD. Pseudopteroxazole and pseudopterosin G are antibacterial diterpenoids previously reported from the Caribbean soft coral *Antillogorgia* (formerly *Pseudopterogorgia*) *elisabethae*. Using an unbiased proteomics approach, the antibacterial mechanism of action of both MNPs appears to be associated with disruption of membrane-associated steps involved in cell wall biosynthesis.[473]

With over sixty new examples, cembranoids remain the dominant sub-structural class of diterpenoids reported from soft corals. Of a set of eleven cembranoids isolated from a South China Sea collection of *Litophyton amentaceum*, litoamentenes A–K ***1166–1176***, only litoamentene I exhibited cytotoxicity, being weakly active against the NCI-H446 cell line.[474] In addition to sterols (see later), an extract of *Lobophytum catalai* afforded the cembrane-type diterpenoids lobocalines A–E ***1177–1181***.[475] While XRD analysis provided the structure and absolute configuration of lobocaline A, interpretation of NMR data, including DP4+ probability analysis, and TDDFT-ECD calculations, secured the structures and configuration of the remaining examples. The structure of co-metabolite lobophytrol B, previously reported as a bicyclic terpenoid, was revised on the basis of XRD analysis to ***1182***.

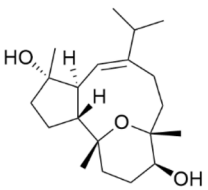
The 3D structure of 7*S*,8*R*-dihydrodeepoxysarcophytoxide ***1183*** has been secured by XRD analysis.[476] The findings call into doubt the structure and/or stereochemistry previously proposed for (+)-7β,8β-dihydroxydeepoxysarcophytoxide. Tetrahydropyrane ring-containing cembranoid ***1184*** and a 4-hydroperoxy derivative of sarcophine ***1185*** were reported from an extract of *Sarcophyton glaucum*, collected off the coast of Egypt.[477] Four sarcophine analogues, sarcoeleganolides H–K ***1186–1189*** were isolated from a South China Sea collection of *Sarcophyton elegans*,[478] while *S. crassocaule* was the source of meijicrassolins A–E ***1190–1194***.[479]

A series of capnosane-type cembranoids, sarcocinerenoids A–H ***1195–1202*** and two unusual variants, sarcocinerenoids I ***1203*** and J ***1204***, were reported from extracts of a Xisha Is., South China Sea collection of *Sarcophyton cinereum*.[480] XRD analysis was used to secure the structure and absolute configuration of sarcocinerenoids A–C, F, G and I. In a separate publication, details of the characterisation of a further nine cembranoid-type diterpenoids, sarcocinerenolides A–I ***1205–1213*** from the same *S. cinereum* biomass were reported.[481] Amongst these structures, sarcocinerenolide A is unusual in being a ring-opened variant, while the remaining congeners are more typical of cembranoids, displaying variation in the location of ether linkages. A structurally-related sarcophine derivative bearing a C-1 to C-12 peroxy linkage, sarcacutumolid A ***1214*** was reported from the extract of a Red Sea collection of *Sarcophyton acutum*.[482]

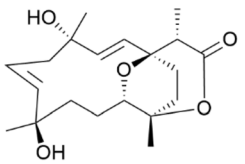
A Ximao Is., South China Sea collection of *Sinularia pedunculata* afforded a structurally-diverse set of cembranoid diterpenoids including sinupedunolides A–E ***1215–1219*** and sinupedunols A ***1220*** and B ***1221***.[483,484] The structures of sinupedunolide A and sinupedunols A and B were secured by XRD analysis. Of the five cembrane diterpenoids, sinudenoids H–L ***1222***, ***1223–1226*** isolated from *Sinularia densa*, sinudenoid H is an unusual example of a bisnorfuranocembrenolide skeleton.[464] The tricyclic structures of 4α-hydroxy-chatancin ***1227*** and sarcotoroid ***1228***, isolated from *Sarcophyton tortuosum*, were proposed to be derived from cembranoid precursors that had undergone transannular Diels–Alder cycloaddition reactions.[485]



†**1164** R_1 = H, R_2 = Ac
†**1165** R_1 = Ac, R_2 = H



†**1182**



†**1203**

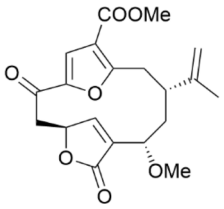
The possible role of photochemistry in the biosynthesis of the cyclobutane ring of the cembranoid diterpenoid (+)-providencin ***1229*** has been supported by its semisynthesis from (−)-bipinnatin E ***1230***, which was isolated from a Floridian specimen of *Antillogorgia kallos*.[486] The Norrish-Yang cyclisation step also afforded a C-14 regioisomeric product, which is likely an anticipated NP. Absolute configuration was assigned to all three compounds based upon XRD analysis of (−)-bipinnatin E. Two accounts of the synthesis of the revised structure of scabrolide B were reported in 2024. In the first publication, (−)-scabrolide B was synthesised in 19 steps (longest linear sequence), and in a simple set of reactions was converted into (−)-sinuscalide C ***1231***, (+)-ineleganolide and (+)-horiolide ***1232***.[487] A second route to (−)-scabrolide B was reported by another research group, with simple conversions again affording related diterpenoids (−)-sinuscalide C and (+)-ineleganolide, as well as a third MNP, (+)-fragilolide A ***1233***.[488] A full account of the synthesis of (−)-scabrolide F has been reported, with weak anti-fouling activity observed for the NP and synthetic intermediates.[489]

New biological activities were reported for cnidarian-derived cembranoids including the finding that 8-*epi*-sarcophinone and *ent*-sarcophine exhibit anti-fouling activity when included in marine paint preparations,[490] sinulariolide exhibits anti-inflammatory activity in a rheumatoid arthritis model,[491] 7,8-epoxy-11-sinulariolide acetate also exhibits anti-inflammatory as well as inhibition of osteoclastogenesis,[492] sinularin induces apoptosis in prostate cancer cells *via* a mechanism related to up-regulation of FOXO3 forkhead transcription factor,[493] and 4-carbomethoxyl-10-*epi*-gyrosanolide E inhibits oral cancer cell migration and invasion by inhibition of the MAPL pathway.[494] Sarcotrocheliol exhibits weak cytotoxicity, antimicrobial and larvicidal activities.[495] An aquaculture specimen of *Briareum stechei* afforded the epoxybriarane briastecholide O ***1234***, while wild-type specimens of the same organism yielded the related analogue briastecholide P ***1235***.[496]

XRD analysis was used to secure the structure and absolute configuration of not only briastecholide P, but also of the known co-metabolite briaexcavatin X ***1236***. In two separate accounts, a large array of new and known polyoxygenated briaranes were isolated from a collection of *Junceella fragilis* collected off the coast of Southern Taiwan. In addition to two new chlorine-containing congeners, ***1237*** and ***1238***, two previously reported analogues gemmacolide X ***1239*** and frajunolide I ***1240*** were re-isolated.[497] Absolute configurations of the latter two diterpenoids were determined for the first time. In the second publication, one new briarane example was reported, fragilide Z ***1241***, while the structures and absolute configurations of known co-metabolites 12-*epi*-fragilide G ***1242***, fragilide P ***1243***, junceellolide D ***1244***, junceellonoid A ***1245*** and juncin ZI ***1246*** were secured by XRD analysis.[498] Excavatolide B and two semisynthetic analogues, one being an ester linked piperidine salt and the second being an oxime-linked terminal alkyne, inhibit inflammation and angiogenesis in models of atopic dermatitis.[499]

As part of a SAR study of transient receptor potential melastatin 7 (TRPM7) ion channel inhibitors, six new xenicane diterpenoids 7*S*,8*S*-epoxywaixenicin A ***1247***, 7*S*,8*S*-epoxywaixenicin B ***1248***, 12-deacetylwaixenicin A ***1249***, waixenicin E ***1250***, waixenicin F ***1251*** and 20-acetoxyxeniafaraunol B ***1252*** were isolated from a Hawaiian collection of *Sarcothelia edmondsoni*.[500] The authors suggested that the epoxide-containing analogues 7*S*,8*S*-epoxywaixenicin A and 7*S*,8*S*-epoxywaixenicin B may be artefactual. Preliminary elements of an SAR were proposed, indicating the requirement of the 12-acetoxy group in combination with a dihydropyran ring for activity, and that bioactive compounds may act as latent electrophiles. Three examples of xenicane diterpenoids, miolenol ***1253***, epoxymiolenol ***1254*** and epoxycoraxeniolide A ***1255***, were reported from a deep-sea collection of the soft coral *Paragorgia arborea*.[501]

The first asymmetric total synthesis of the xenicane isoxeniolide A ***1256*** has been reported, confirming the structure and defining the absolute configuration.[502] Ten new sterols were reported from cnidarians in 2024, including lobocaloids A–D ***1257–1260*** from *Lobophytum catalai*,[475] cholesterols ***1261*** and ***1262*** from *Litophyton mollis*,[503] capnesterones A ***1263*** and B ***1264*** from *Capnella imbricata*,[504] and cholestene-7-ones ***1265*** and ***1266*** from *Lobophytum durum* and *Capnella imbricata*, respectively.[505,506] The study of *Lobophytum durum* also led to the re-isolation of the known ergosterol derivative ***1267***, the relative configuration of which was determined by XRD analysis, providing clarification on the configuration of the 24-methyl group.[505]



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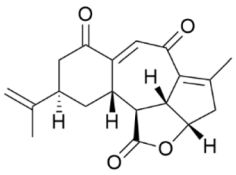
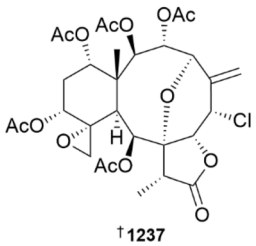


†1231



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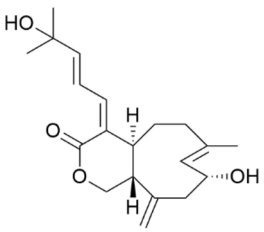


†1256

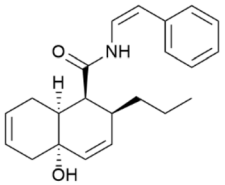
A secogorgostenone steroid, originally sourced from *Sinularia leptoclados*, exerts anti-inflammatory activity and brain-protective effects by attenuating matrix metalloproteinase-mediated events.[507] The MNP chemistry of cnidarians belonging to the genus *Capnella*, covering the period 1974 to May 2024, has been reviewed.[508] The background history of the palytoxins, originally isolated from the zoanthid *Palythoa* sp., as well as their biological activities and mechanism of action, has also been reviewed.[509] The chemistry and biological activities of ketosteroids, a number of which are isolated from soft corals, have been reviewed.[510] A recombinant variant of Tst2, a 38-residue peptide identified by transcriptomic and proteomic analysis of the Australian sea anemone *Telmatactis stephensoni*, is an inhibitor of the transient receptor potential subfamily V member 1 (TRPV1) ion channel.[511] HCIQ2c1, a Kunitz-peptide identified in cDNA analysis of the sea anemone *Heteractis crispa*, binds to the transient potential ankyrin 1 (TRPA1) calcium-permeable channel, appearing to stabilise the channel in the open conformation, and reduces pain and inflammation *in vivo*.[512] Bioassay-directed fractionation of toxins from a Japanese collection of the sea anemone *Heteractis aurora* led to the identification of one novel class of toxin, denoted Hau I, with as-yet undetermined target(s), two new examples of toxins from the boundless β-hairpin family (Hau II and III) and a new example of a sea anemone type 1 sodium channel toxin (Hau IV).[513]

8 Bryozoans

Brominated indole alkaloids, alternatamides B and D have been synthesised from 3,4-dibromoaniline *via* the intermediate 5,6-dibromotryptamine[514] and bryostatin 4 has been shown to inhibit the transforming growth factor (TGF)-β signalling pathway, responsible for disease progression in acute erthroleukaemia.[515]

9 Molluscs

Compared to previous years, there was a significant drop in new MNPs reported from molluscs in 2024, with the two examples being bursatamide A ***1268*** and bursatellin B ***1269*** isolated from the sea hare *Bursatella leachii*.[516] A general review covering the nutraceutical and medical potential of marine molluscs has been published.[517] The potential of dolastatin MNPs and synthetic analogues as anticancer agents has been reviewed.[518] Exploration of the SAR of (−)-doliculide A led to the identification of some analogues with similar levels of cytotoxicity as the MNP towards the HCT-116 cell line.[519] Furodysinin, isolated from the nudibranch *Hypselodoris tryoni*, exhibited weak *in vitro* activity against wild-type *Plasmodium berghei*, but was considerable less active against an atovaquone-resistant strain.[520]



†1268

Tambjamine analogues show interesting results as anti-leishmanials, with potent intracellular activity against *Leishmania mexicana* and *L. donovani*, and with one analogue demonstrating partial protection in a rodent *in vivo* model.[521] The structures, synthetic approaches and SARs of the lamellarin alkaloids have been reviewed.[522–524]

The global occurrence, toxicity and risk assessment of cyclic imine shellfish toxins have been reviewed.[525] Analysis of scallops *Argopecten irradians* exposed to paralytic shellfish toxin producing dinoflagellate *Gymnodinium catenatum* identified that the toxins accumulate in the adductor muscle, leading to levels exceeding regulatory limits.[526] Diatoms of the genus *Pseudo-nitzschia* produce the neurotoxin (DA), which in the case of king scallop contamination, can take many months to clear from the mollusc tissue. DA immunodetection has been used to examine the time course of subcellular location of the toxin, finding preliminary evidence of accumulation in autophagosomes in the digestive gland, which may go some way to explain the toxin's persistence.[527] Two fatty acid desaturase genes were identified in the dwarf surf clam *Mulinia lateralis*, the expression levels of which varied during embryonic development.[528]

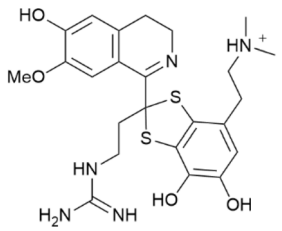
Investigation of the transcriptome of the carnivorous snail *Raphitoma purpurea* has identified putative conotoxin-like peptides in both salivary gland and venom duct tissue, proving further implication for venom evolution in molluscs of the family Conidae.[529] The predatory and defensive venoms associated with 16 different cone snail species have been reviewed,[530] while another review has focused more on the effects of κ-conotoxins on potassium channels.[531] A cyclic octapeptide Am931, identified from analysis of the venom duct transcriptome of *Conus amadis*, appears to act as a catalyst promoting formation of the natively folded disulfide form of α-conotoxin ImI.[532] Replacement of L-lysine or L-arginine residues in α-conotoxin RgIA with their corresponding D-enantiomers has afforded weakly antifungal analogues.[533]

Six synthetic α-conotoxins, sequences of which were derived from transcriptome sequencing of venom of *C. quercinus*,

exhibited potent cytotoxicity towards fall army worm cells as well as anti-larval toxicity.[534] In two separate studies by the same research group, α-*O*-conotoxin GeXIVA[1,2] was shown to both reduce cancer chemotherapy induced neuropathic pain in a mouse model and to exhibit *in vivo* tumour growth suppression of triple-negative breast cancer.[535,536] A disulfide bond deleted and truncated conotoxin analogue, based on the sequences of α-*O*-conotoxins GeXIVA and GeXXVIIA, demonstrated retained potency of human α9α10 nAChR inhibitory activity, inhibited $Ca_V2.2$ channels and alleviated pain in a mouse model.[537] Conotoxin kM-RIIIJ has been used to explore the physiological functions of hetereodimeric $K_V1.2$ channels, revealing that the receptors are highly expressed in proprioceptive (sensory motor) neurons in the dorsal root ganglion.[538]

10 Tunicates (ascidians)

Further investigation of an extract of an unidentified Didemnidae ascidian collected in Palau, afforded guanidine alkaloids 1-carboxydopargimine ***1270***, mellpaladines D–F ***1271***, ***1272*** and ***1273***, serodopalgimine ***1274*** and a 4,4′-dimer of serotonin ***1275***.[539] Sulfoxide-containing mellpaladines D and E were isolated in equal amounts, with the authors suggesting they may be products of autoxidation. Mellpaladines D–F were evaluated for effects on behavioural activities in mice, with mellpaladine F being found to induce whole body convulsion.



1273

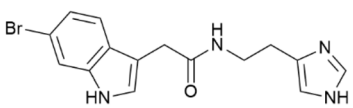
The original proposed disulfide, homocoupled, cyclic hexapeptide structures of antatollamides A and B have been revised by total synthesis to be simpler monomeric cyclic peptides ***1276*** and ***1277***, respectively.[540] The first enantioselective synthesis of (+)-cylindricine B has been reported.[541] The synthesis and multidrug resistance reversing activities of the pyrrole-containing ningalin MNPs have been reviewed,[542] as have the cytotoxic properties of cephalostatin and ritterazine dimeric steroidal pyrazine alkaloids.[543] The human triggering receptor expressed on myeloid cells 2 (TREM2) is an important regulator of immunological responses. *N*-Deacetylshermilamine B, as well as a partially characterised mixture of glucosylceramides, isolated from the ascidian *Cystodytes dellechiajei*, bind to TREM2, with the pyridoacridine alkaloid also exhibiting weak *in vitro* cytotoxicity towards two multiple myeloma cell lines.[544]

Phosphoeleganin and one semisynthetic analogue decrease interleukin 6 secretion, suggestive of this compound class having potential in the management of diabetes.[545] A further SAR study of phosphoeleganin has identified the importance of the phosphate and glycine groups, in combination with backbone carbinol stereochemistry, for inhibition of h-15-LOX-1 enzyme.[546] Of a library of synthetic analogues of rubrolide-related γ-butenolides, a non-halogenated example exhibited weak to moderate cytotoxicity towards a panel of HTCLs, induced ROS production leading to apoptosis, arrested cell cycle at G_2/M and exhibited *in vivo* activity in a HeLa xenograft assay.[547]

A structurally simplified analogue of diazonamide A exhibited potent antiproliferative activity towards a panel of HTCLs which appeared to be related to inhibition of tubulin assembly, and the simplified analogue was less toxic to noncancerous cell lines.[548] Screening a library of marine bacterium *Tistrella* strains revealed the majority were capable of producing didemnin B.[549] The introduction of a second copy of the didemnin B BGC afforded a modified strain with excellent production capability (75 mg L^{-1}), enabling semisynthesis of a series of new analogues. A cytotoxic photoaffinity probe successfully enriched a large number of potential protein targets, including the previously noted depalmitoylating enzyme PPT1. Before being discontinued due to a lack of COVID-19 related hospitalisations, a Phase III trial of plitidepsin showed it to be generally well tolerated and suggested a two-day improvement in the median time to withdrawal of supplementary patient oxygen support.[550] Screening a library of marine-derived compounds identified polycarpine as a potent inhibitor of the main protease M^{pro} of coronaviruses, also inhibiting SARS-CoV-2 and PEDV (porcine epidemic diarrhoea virus) viral replication in Vero-E6 cells.[551] The uses of the tetrahydroquinoline alkaloid trabectedin and its related analogue lurbinectedin in oncology, mechanisms of action, and uses in combination therapies have been reviewed.[552]

11 Echinoderms

Extracts of the arms of the starfish *Thromidia catalai* afforded three indole derivatives, catalindoles A–C ***1278***, ***1279*** and ***1280***, the structures of which were confirmed by synthesis.[553] When collected, the starfish were found feeding on the sponge *Theonella swinohei* – LCMS analysis of extracts of the starfish digestive gland identified the presence of *Theonella*-sourced NPs while none were located in the arms, suggesting that the starfish does not accumulate sponge metabolites.



1278

Steroidal disulfates ***1281–1284*** were isolated from a Far Eastern collection of the slime sea-star *Pteraster marsippus*.[554] The biological activities of saponins isolated from sea-stars has been reviewed.[555] Several examples of polyhydroxylated steroidal saccharides were also reported from starfish, including pectiniferosides A–J ***1285–1294*** from *Patiria* (= *Asterina*) *pectinifera*,[556] spiculiferosides A–D ***1295–1298*** from *Henricia leviuscula spiculifera*,[557] and ceramasterosides A, B, D and E ***1299–1302***, from *Ceramaster patagonicus*.[558] Of these MNPs, only the latter, ceramasteroside D, exhibited a notable

biological activity, of moderate ability to inhibit the release of nitrite from LPS stimulated microglial cells.

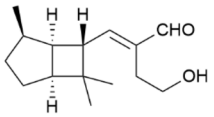
Ten examples of triterpenoid glycosides were reported from sea cucumber species, including pacificusosides L–Q ***1303–1308*** from *Solaster pacificus*,[559] peronioside A ***1309*** from *Psolus peronii*,[560] conicospermiumosides A_3-1, A_3-2, A_3-3, A_7-1 and A_7-2 ***1310–1314*** from *Cucumaria conicospermium*,[561] and ***1315–1318*** from *Apostichopus japonicus*.[562,563] Investigation of extracts of the sea urchin *Clypeaster humilis* afforded sulfate ***1319***, methane sulfonate ***1320*** and boldine ***1321***, the latter two compounds being reported from a marine organism for the first time,[564] while bibenzochromenones phanogracilins A–C ***1322–1324*** were isolated from the crinoid *Phanogenia gracilis*.[565] Additional biological properties have been reported for echinochrome A, including anti-asthmatic activity[566] and the ability to modulate migration of non-small cell lung cancer cells.[567] 2-Butoxytetrahydrofuran, previously reported from *Holothuria scabra*, attenuates the aggregation and oxidative properties of α-synuclein, lessening its toxicity in a *C. elegans* model of Parkinson's disease.[568]

The effect of the saponin frondoside A on inflammation associated with tooth replantation has been investigated.[569] Holothurin B and related saponins are able to prevent mast cell degranulation, as measured by the release of β-hexosaminidase, and to alleviate the action of a number of pro-inflammatory cytokines.[570] The chemistry and biological activities of MNPs reported from the sea cucumber *Apostichopus japonicus* (2000 to late 2023) have been reviewed.[571] Holotoxin A_1, isolated from *A. japonicus*, inhibits candidiasis by a wide range of mechanisms including increased intracellular accumulation of ROS, mitochondrial membrane depolarisation, and inhibition of mitochondrial complex I.[572] Further examination of the triterpenoid glycoside cucumarioside A_2-2 has identified it to be active against prostate cancer cell lines, while cucumarioside A_0-1 and djakonovioside A are active against triple-negative breast cancer cell lines.[573,574]

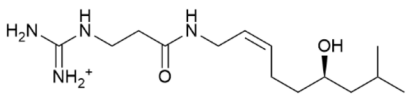
12 Miscellaneous

The structure of the bicycloheptane derivative (−)-raikovenal ***1325***, originally isolated from the marine ciliate *Euplotes raikovi*, has been confirmed by total synthesis, with the study also assigning absolute configuration.[575] The potential of seagrass meadows as a source of bioactive molecules has been reviewed,[576] while species specific reviews of *Halophila stipulacea*[577] and *Halodule uninervis*[578] covered the phytochemistry and pharmacological activities of metabolites. Further investigation of the diarylheptanoid heterodimer zosterabisphenone B determined it can induce apoptosis in HCT116 cells and reduce the growth of human colon tumour xenografts in mice.[579]

Five pyridyl and dihydroisoquinoline alkaloids, ***1326–1330***, were reported from the nemertean worm *Amphiporus angulatus*.[580] Collections of the intertidal sipunculid worm *Phascolosoma granulatum* afforded phascolosomines A–F ***1331–1336***, characterised as linear guanidine amides.[581] A SAR study of the antimicrobial peptide capitellacin, originally isolated from the annelid worm *Capitella teleta*, identified the importance of one of the disulfide linkages for activity, that the linear peptide was devoid of activity, the D-enantiomer had reduced activity and that most analogues only exhibited weak, at best, activity towards *E. coli*.[582] Bioinformatic searching of the transcriptome of the lugworm *Arenicola marina* identified a new BRICHOS-related antimicrobial peptide, AmBRI-aaa, that was prepared using heterologous expression in *E. coli*.[583] The 44 residue peptide exhibited weak to moderate activity towards some Gram-positive bacterial strains and whole genome sequencing of a resistant mutant identified targets associated with cell wall biosynthesis.



†1325



†1331

The ribbon worm *Cephalothrix simula*, known to contain tetrodotoxin, may be a source of toxin in British farmed Pacific oysters *Magallana gigas*.[584] A truncated variant of a peptide, bolespleenin,[334–347] derived from a gene product of the mudskipper fish *Boleophthalmus pectinirostris*, exhibited broad-spectrum antibacterial activity, inhibited biofilm formation and was active in a mouse model of skin infection.[585] Further investigation of the peptide epinecidin-1, originally isolated from the orange-spotted grouper *Epinephelus coioides*, has determined its ability to suppress lipoteichoic acid-enhanced proliferation of non-small cell lung cancer cells and induce necrotic cell death *via* a mechanism involving mitochondrial damage and elevated levels of ROS.[586] The 22-residue peptide piscidin-1, derived from mast cells of the hybrid striped bass *Morone saxatilis* × *M. chrysops*, induces apoptosis in oral squamous cell carcinoma cells, and also increases ROS levels leading to mitochondrial dysfunction.[587]

GC/electron capture negative ion-MS analysis of seventeen species of fish from the Seychelles in the Western Indian Ocean identified the predominant polyhalogenated compounds present were halogenated NPs of the methoxylated diphenyl ether class, rather than PCB or DDT anthropogenic persistent pollutants.[588]

13 Perspective – the emerging role of artificial intelligence in MNP research

The rapid uptake of artificial intelligence (AI) in commerce, health, transportation and communications is stunning. In the sciences, AI is equally transformative. AI is used to analyse large amounts of different kinds of data, to find patterns and relationships, and use those to make predictions, decisions or generate "new" content. Machine learning (ML) is the most

widely used approach, and it identifies patterns in data without relying on rules-based programming, allowing tasks to be performed automatically. When dealing with more complex, high-dimensional, unstructured data, a specialized subset of ML called deep-learning (DL) employs multilayered neural networks to identify intricate patterns.

Given that computer assisted analysis have been used for decades in MNP research, how much has the introduction of AI improved productivity in MNP research and increased the discovery of novel and/or potently active MNPs in the last decade?

Two main applications of AI are generative (GAI) and predictive (PAI). GAI is based on large language models (LLN) mostly trained on online data and analysed using ML or DL algorithms to produce content copied from existing data but stitched together in different ways to construct a "new" amalgam as text, image or video that mimics the training data. GAI can aid researchers to write code for software packages such as Python and R, produce images for graphical abstracts or presentations, help construct grammatically correct text, and provide suggestions for areas to investigate when initiating research on an unfamiliar topic. But GAI needs to be used with caution because it has a propensity to generate wildly inaccurate results. For instance, creating images of chemical structures using GAI is particularly unreliable (Fig. 2).[589]

PAI by contrast can analyse large amounts of orthogonal data to identify patterns that are often hidden or difficult to discern and predict new findings based on this pattern recognition.

The application of AI in the molecular sciences is mostly predictive rather than generative and this approach has resulted in important breakthroughs. The development of AlphaFold, an AI model to predict 3D protein structures based on amino acid sequence, for example, is transformative. Using a transformer neural network architecture trained on ~180 000 experimentally determined 3D protein structures, AlphaFold can now predict the 3D structures of over 200 million proteins.[590] These predictions are now being incorporated into the drug discovery process.

Computational methods such as TDDFT calculations to assign absolute configurations to molecules, spectroscopic simulations, computer-assisted structure elucidation using rules-based algorithms, compound similarity and substructure searches, dereplication using spectral database matching tools and virtual screening of compound libraries through *in silico* protein docking are all computer assisted (but not AI) methods developed since the 1990s. Many of these methods are still widely used in NP research and most involve complex calculations that require supercomputers to do time-consuming analyses. The arrival of ML has created opportunities to massively reduce the time required to generate similar results without using supercomputers.[591–594]

ML is now used as an aid in MS-based dereplication by predicting MS fragmentation patterns for NPs found in databases to augment the annotation of molecules in molecular networks that were previously only identified through matching experimental spectrometric data.[595] Augmenting lower level DFT calculations with ML generated parametric corrections has resulted in more accurate predictions of NMR data in minutes rather than days.[596,597] ML assisted genome mining has resulted in the discovery of BGCs and the prediction of their products.[598,599]

MNP researchers still face many bottlenecks that impede productivity. These include access to biota, accurate taxonomic identification of biota, identification of "interesting" NPs especially those present in complex mixtures, separation and structure identification of NPs, identifying a NP's bioactivity and understanding its mode of action, identifying the genes and proteins responsible for NP biosynthesis and synthesis of the NP for sustainable production or to generate analogues.

We analysed 13 582 papers and reviews published in the field of MNP (obtained from MarinLit) over the last 10 years (January 2015–December 2024) to help understand how AI has contributed to solving these MNP research bottlenecks. Keyword searches (Table S2) of all abstracts allowed a connectivity map (using VOSviewer)[600] to be generated by creating a thesaurus of keywords that we grouped into ontologies based on relatedness of the words. The limitation of this approach is that some abstracts do not necessarily include information regarding the use of AI, but the keyword connectivity maps can help to infer AI usage based on a combination of ontologies. A network from genome mining to bacteria, BGC, molecular network, predict, MS, bioinformatic, model and database for example infers AI is being used in microbial MNP research even though there is no explicit association between the use of AI terms and genome mining in the abstracts (Fig. 3).

We found that very few studies have used AI to aid in the discovery of new MNPs or bioactivities over the last decade. Overall, the rate of discovery of new MNPs (Fig. 1) and potently active MNPs has remained static over the decade.[601]

AI has only been explicitly referred to in 82 MNP papers over this period (Fig. 4 and 5) and is most highly associated with the prediction of bioactivity through either *in silico* docking of MNPs to proteins or inferred bioactivity based on structural similarities. ML augmentation of DFT calculations for more accurate NMR chemical shift prediction (DU8ML),[596] pattern recognition within 2D NMR spectra to predict structures (SMART2.0/DeepSAT),[602,603] pattern recognition within structures to predict NP structure classes (NPclassifier),[604] prediction of peptide quaternary structures (conotoxins),[605] and genome

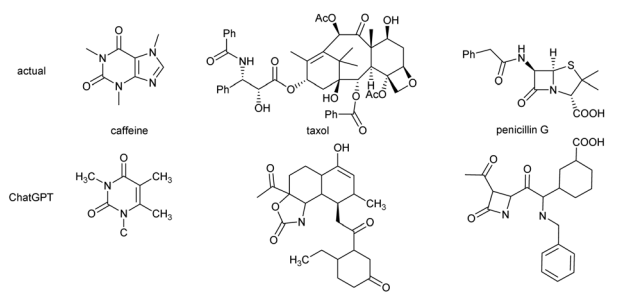


Fig. 2 Structures of three ubiquitous NPs (caffeine, taxol and penicillin G) and their "structures" generated by ChatGPT. The "structures" generated by Gemini and DeepSeek are presented in the SI (Fig S1).

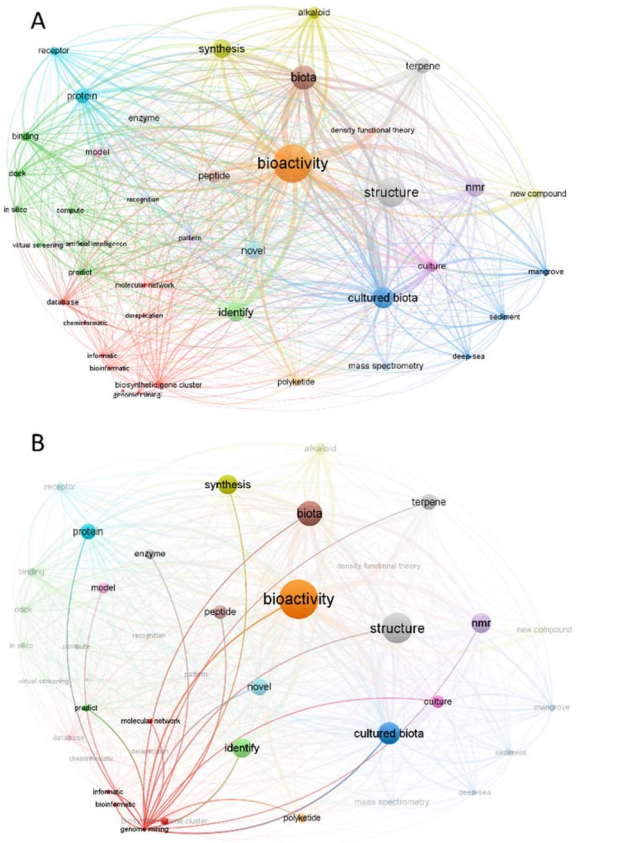


Fig. 3 Connectivity map (created in VOSviewer)[600] of network linkages between keywords extracted from MNP paper abstracts. The size of a keyword node is proportional to the number of papers that contain that ontology and the width of the linkage to another node is proportional to the number of instances that the linkage occurs. Each of the top 21 nodes has a individual colour. (A) Total network; (B) highlighted network from "genome mining".

mining to generate hypothetical virtual libraries of potential structures of undiscovered molecules for MS molecular network matching (HypoRiPPAtlas)[599] are examples of other uses. To date, none of these methods have been widely adopted.

In silico screening methods (233 papers) have steadily increased over the decade. There have been several studies that have used databases to predict MNPs active against specific targets (such as the SARS-COV 19 virus) through virtual screening. This approach previously used the knowledge of the protein structure (obtained mostly by XRD) and *in silico* docking techniques to identify molecules that interacted with the protein. However, AI methods now tend to use the knowledge gained from molecules that have been shown experimentally (or virtually through *in silico* methods) to interact with a specific target protein (or a structurally related protein) and these interactions are used to train a model to identify other compounds that will also interact. This pattern recognition approach is potentially quicker to implement and can be used to analyse large datasets. Unfortunately, where this approach has been adopted using MNPs, the verification of "hits" obtained mostly relies on virtual screening of the "hit" molecules

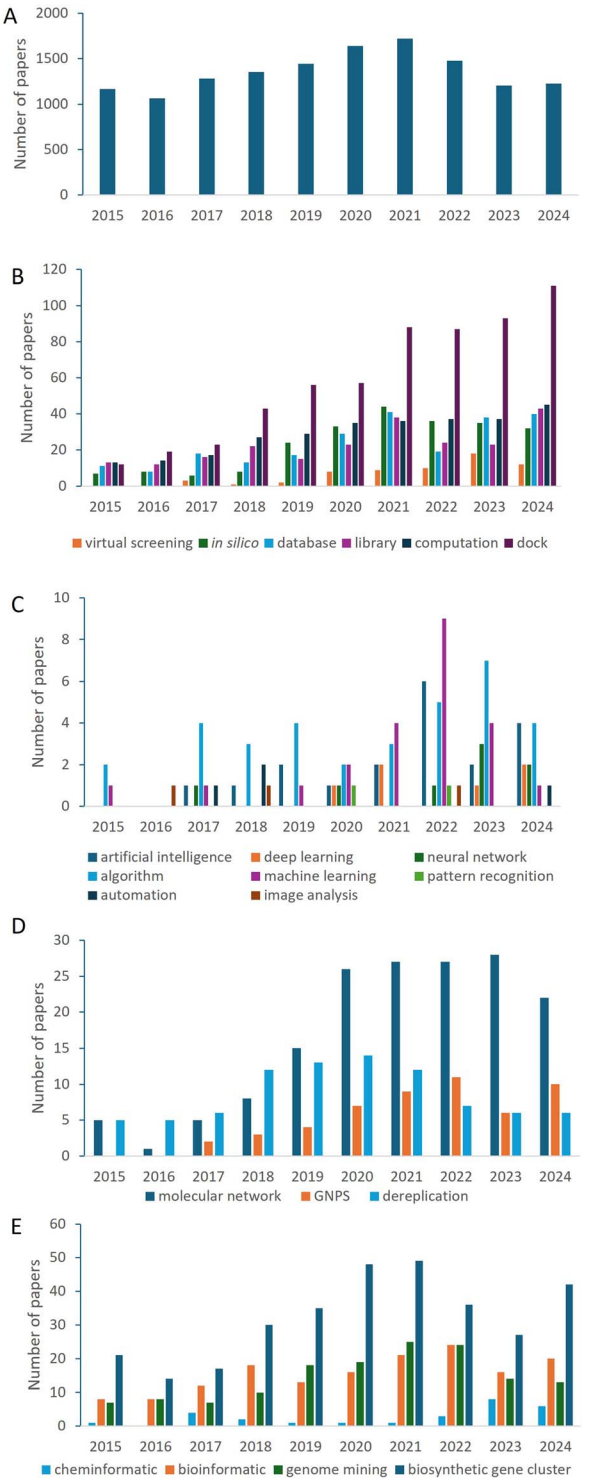


Fig. 4 Trends in number of papers using keywords in their abstracts from January 2015 to December 2024. (A) Total MNP papers, (B) papers discussing virtual screening and interactions of MNPs with proteins; (C) papers discussing AI; (D) papers discussing MS molecular networking; (E) bioinformatics application in MNP research. The bars represent the total number papers.

through molecular docking experiments. This seems to be a circular approach given that in some cases, the training data was originally obtained through virtual screening. There has

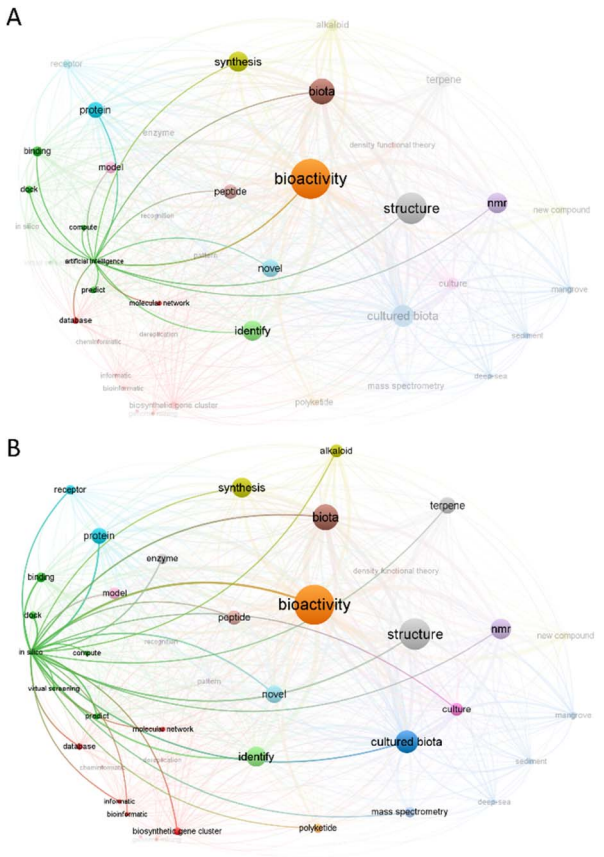


Fig. 5 Connectivity map (created in VOSviewer)[600] of network linkages between keywords extracted from MNP paper abstracts. (A) Highlighted network from "artificial intelligence" ontology; (B) highlighted network from *"in silico"* ontology.

been limited examples where the virtual "hit" compounds have been tested in real laboratory assays.

One of the key advantages of studying marine biodiversity is the propensity to discover unprecedented chemistry. PAI learns from previous discoveries to identify known molecules and their close unknown analogues and to predict new bioactivities for these compounds based on their structural similarity to other molecules with demonstrated bioactivity against a known target. This has the potential to "de-orphan" known MNPs lacking any bioactivity data, but to date this has not occurred.

Linking biosynthetic genes with known NPs or predicting new NPs from bioinformatics associated with BGCs are also effective outcomes from PAI assisted research (BGC is mentioned 561 times over the decade). MNP research increasingly uses ML augmented molecular networking of LCMS data as an aid in the selection of biota to investigate or to link molecular network clusters to BGC predictions. Molecular networking has been discussed in 294 papers over the last 10 years, but invariably the selection of clusters has resulted in the isolation of closely related analogues of known compounds. So, based on this, one might expect that greater effort would now be focused on the unknowns with AI doing the heavy lifting in dereplication by sifting through the large volumes of "known" chemistry to identify the uniquely unknown metabolites that can then become the focus of new research. There is little evidence that this approach is being used yet but hopefully, the future use of this powerful tool can be tailored to highlight the unknowns rather than the knowns.

There is no evidence of an increase in new MNP novelty (*i.e.* structures possessing new ring systems or features not reported before; information from previous MNP reviews). The term "novel" has been used 2255 times and "unprecedented" 344 times, but virtually never in relation to either truly novel scaffolds or bioactivities. The lack of increased structural novelty might be related to diminishing returns that are anticipated when specific biota resources have been extensively studied.[606] However, it could also reflect the sustained use of standard purification, biota selection, culture methods, or bioassay methods that unintentionally exclude certain structures. An example of this bias can be seen when normal-phase silica gel separation methods which were almost exclusively used until the early 1990s started to be supplemented with reversed-phase separation methods, thus expanding the latent chemical diversity accessible within well studied species (Fig. 6). This resulted in a significant increase in average polar surface area of MNPs after the early 1990s that also reflected a difference in structural novelty. If a ML approach were adopted, it could potentially identify specific purification methods that have a higher chance of yielding novel chemistry.

There are still very significant pockets of marine biodiversity that remain unstudied. Small animals and plants (species with individuals occupying 1 cm^3 or less) are rarely sampled but potentially account for most biodiversity in an area.[607,608] They likely occupy different niches where environmental pressures could drive differences in MNP expression. ML from ecological data could prove insightful. The propensity to isolate and culture microorganisms sourced from marine environments using media suited to fast growing, terrestrial soil-derived microbes, does not help to exploit truly marine biodiversity and their potentially unique chemical diversity. The choice of isolation media and culture media used to access truly marine microbes might benefit from an AI approach. Combining data from microbial ecology, biogeochemistry, aquaculture, food

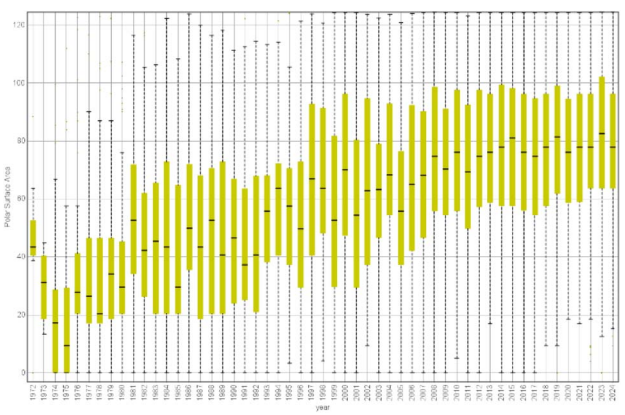


Fig. 6 Box plot of calculated polar surface area (PSA) for all new MNPs published each year between 1972 and 2024. Black horizontal lines represent median PSA.

sciences, ecological studies and metagenomics for ML might help to uncover specific nutrient requirements that could enhance the isolation and cultivability of truly marine species.

MNP discovery continues to rely on serendipity. There is no evidence so far that AI has been applied to the biota selection process. At its most basic, MNP researchers have used prior knowledge to target specific species for study (soft bodied marine invertebrates, algae, fungi, bacteria, cyanobacteria). AI could aid in this process. However, we know from experience that the random collection of an invertebrate that was collected simply because it looked different or was found in an unusual place, has led to some of the most unusual molecules we have discovered. Perhaps an AI assistant on a SCUBA dive could help find that elusive organism.

Genome mining helps to identify the vast potential of BGCs (most of which are silent) in microorganisms, but what about the NPs that are not made by enzymes encoded by clustered genes? Pertinent examples are many types of alkaloids which represent a substantial proportion of MNPs and drugs derived from NPs. ML methods using Alphafold could provide insights to predict proteins from genomic data for non-clustered biosynthesis.

The structure determination of NPs has always been challenging, and continued reports of MNP structure revisions show that improved methods to aid in structure determination are always welcomed. Computationally demanding DFT methods to predict NMR chemical shifts have been augmented with parametric rules obtained through ML approaches using NMR data obtained from large libraries of published structures. This has resulted in much faster predictions and resulted in two MNP structure revisions.[596] The tool can also be used to establish if a proposed structure is correct prior to publication. SMART2.0 is an image-based AI tool that predicts the structure of a compound based on the similarity of its HSQC image to those generated from a training set of over 53 000 experimental HSQC images or HSQC images generated through predictions of H and C chemical shifts for compounds lacking experimental data.[602] The four MNP studies that have used SMART2.0 to guide prioritisation all resulted in the isolation of either close analogues of known compounds or a chimeric molecule containing features from two known molecules. Overall, these ML NMR methods can improve the structure determination workflow, but do not necessarily result in greater novelty of structures being published. Hopefully, it will reduce the number of incorrect structures appearing in the literature (if used judiciously at the time of peer review prior to publication).

Open Molecules 2025 (OMol25), released in mid-2025 by Meta and the US Department of Energy's Lawrence Berkeley National Laboratory, contains 100 million DFT calculations covering a huge variety of molecular systems that can now be used in ML applications.[609] This has the potential to massively reduce the time required to predict NMR and ECD data with estimates suggesting a 10-thousand-fold reduction in cpu time to generate equivalent quality predictions to those generated by DFT calculations. This resource has the potential, through ML algorithms, to predict NMR and ECD data for all known MNPs with comparable accuracy to quantum mechanical calculations virtually instantaneously on a laptop or smart phone. Linking this potential capability with other AI platforms such as 2D NMR image recognition, MS^2 data recognition and NP structure prediction from genomic data and NP bioactivity predictions could provide a paradigm shift in NP discovery efforts.

Since ML methods are trained on known data (such as molecules, NMR spectra, MS^2 data, genes) there is an obvious bias to match unknowns that mimic knowns. "Rare" structures might not feature in a training set, and undiscovered novel molecules clearly cannot be there at all. Therefore, important potential matches might be missing in the model. This is analogous to the inherent bias used by Amazon to recruit IT staff where female applicants were culled early in the automated selection process because the data used to train the algorithm did not include applications from women.[610] Training models to highlight and prioritise the unknowns will help to counter these biases.

There is a danger when AI is used as a "black box" method by non-experts who accept the results as gospel without any understanding or questioning because of a lack informed knowledge. If these unverified AI results are subsequently used to train new models, a misinformation loop is exacerbated, rendering future models even less accurate than their predecessors. There is, therefore, an ongoing need for orthogonal testing of results obtained from AI tools in MNP research. In structure determination, this means that XRD and total synthesis will continue to be important verification tools. Laboratory-based bioassays need to be used to confirm or reject an AI computational result and MS^2 fragmentation matching for structure annotation will still rely on real MS data from standards.

There have been highly innovative AI methods developed, and some have been widely adopted while others have not. It is somewhat baffling to know why this is the case. For instance, molecular networking is now widely adopted with platforms such as GNPS and applications such as MZmine providing a simple computational engine for upload of raw data and download of clustered and annotated results.[611,612] The barrier to adopting this technology is low and labs using these platforms benefit from their simplicity to access. Equally as innovative is the SMART2.0/DeepSAT platform for interpretation of 2D NMR data to match known compounds and to predict structure classes for unknown compounds. However, the adoption of these methods by the MNP community is low (only four papers refer to SMART2.0 in their abstract). One reason may be that many MNP research groups have NMR "gurus" who can identify a known structure from limited experimental data extremely quickly and do not need to rely on computational methods. However, the most likely reason for slow uptake is that these methods still do not work well with NMR of mixtures (apart from matching major components). The time involved to purify an extract and then obtain HSQC data on individual molecules compared to injecting a minute quantity of an extract into an HPLC column and generating MS^2 data for thousands of compounds in minutes is likely to be a serious impediment.

Negative results are a major gap in published knowledge. For example, many MNP groups have large inventories of marine

species collected or cultured randomly. These collections invariably contain individual samples that have been studied but either produce no MNPs or only known MNPs or the isolated MNPs showed low or no bioactivity in a range of bioassays. These results do not generally make it into research papers but are a gold mine of information that could be used in AI analysis. To make the most of all research outputs therefore requires an acceptable mechanism to capture open access, validated negative research results as well as positive ones.

Linking datasets will be key to seeing the most productive uptake and application of AI in MNP research. The World Register of Marine Species (WoRMs) contains up-to-date taxonomic classifications, collection records and distributions for over 500 000 species,[613] the Ocean Biodiversity Information System contains 167 million geolocation records for over 200 000 marine species and 27 million gene sequences,[614] citizen science initiatives such as iNaturalist have images of species linked to taxonomic data and geolocation with close to 280 million observations linked to 536 000 species (including 4041 observations from 1280 marine invertebrate species),[615] the Lotus initiative provides taxonomic links to over 750 000 NP structure/organism pairs,[616] the newly released Minimum Information about a Tailoring Enzyme (MITE) links biosynthetic enzymes with reaction type and precursor and product structures,[617] SMART2.0 uses a database of over 50 000 HSQC images to link with structures, GNPS and other MS based platforms link MS^2 data to structures. Linking these disparate datasets could provide opportunities to guide new discoveries and to unlock novelty in plain sight.

This perspective has highlighted that MNP researchers are only beginning to embrace the power of AI and it will be fascinating to see where MNPs research in the AI era evolves over the coming decade.

14 Conclusion

New MNP discoveries in 2024 have shown an increasing prevalence of MNPs compounds derived from marine fungi and a decreasing trend in macro-organism studies. Although close to half of all new MNPs were reported from marine fungi in 2024, the majority are minor variations on already well represented structure classes. Actinomycetes are a minor contributor to overall marine procaryote biodiversity, but they continue to be the major source of new bacterial MNPs.

BCGs associated with MNPs obtained from bacteria, cyanobacteria and to a lesser extent fungi continue to be characterised, and genome mining is increasingly being used to find new bacterial MNPs. In some cases, these genomic approaches have resulted in proposed new MNP structures without comprehensive and convincing spectroscopic evidence and from our perspective these compounds have not been structurally verified. Rigorous spectroscopic characterisation including NMR spectroscopy and or XRD analysis should complement MS-based evidence in future studies.

The bioactivities reported for select MNPs continue to be a highlight, with the clinical development of a synthetic derivative of the Indonesian sponge-derived alkaloid PM742 holding promise for lung cancer patients. Synthetic studies continue to be an important technique to support or refute MNP structures and in 2024, over 120 first total syntheses of MNPs were accomplished, leading to several structure revisions and over 10 absolute configurational assignments being established.

New sponge-derived MNPs had fewer than expected examples of peptides and alkaloids based on previous trends. Cnidarian studies were predominantly associated with organisms collected from Chinese, Vietnamese and Taiwanese waters in the South China Sea pointing to reduced emphasis on cnidarian biodiversity hotspots. Molluscs, ascidians, bryozoans, red, green and brown algae were, in total, only represented by a handful of studies, while echinoderms were mainly studied from Russian collections. These observations point to diminished geospatial and phylogenetic diversity of MNP discoveries. If these biodiversity trends continue, the overall relevance of MNP research will be diminished. That notwithstanding, the contributions of new MNPs to the structural diversity of organic chemistry remains high and they represent a valuable resource for biodiscovery.

15 Conflicts of interest

There are no conflicts to declare.

16 Data availability

Supplementary information (SI): all structures along with their names, taxonomic origins, collection locations, and biological activities. See DOI: https://doi.org/10.1039/d5np00080g.

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