



## Marine natural products‡

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Covering: January to the end of December 2023

This review covers the literature published in 2023 for marine natural products (MNPs), with 582 citations (541 for the period January to December 2023) 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 (1220 in 340 papers for 2023), 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 progress in the study of prokaryote involvement in macro-invertebrate MNP production is discussed.

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## 1 Introduction

This review is of the literature for 2023 and describes 1220 new compounds from 340 papers, compared to 1417 new compounds in 384 papers reported for 2022.<sup>1</sup> In addition, 25 known NPs were reported from a marine source for the first time, 9 artefacts were identified and 45 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 gene cluster 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 (58) is shown in the review. 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 ESI document.‡ Access to the curated MNP data held in the Marinlit database<sup>2</sup> provides all the structural and literature data used to prepare this review.

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The section reporting MNPs from mangrove-associated fungi that appeared in previous installments of this review has now been amalgamated into a general marine-sourced fungi section.



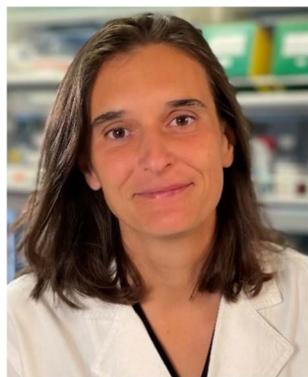
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*Anthony (Tony) Carroll initially studied the alkaloid and lignan chemistry of rainforest plants (BSc (Hons) and PhD, Prof Wal Taylor, Sydney University) but marine natural products became a major focus after postdoctoral fellowships at the University 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.*



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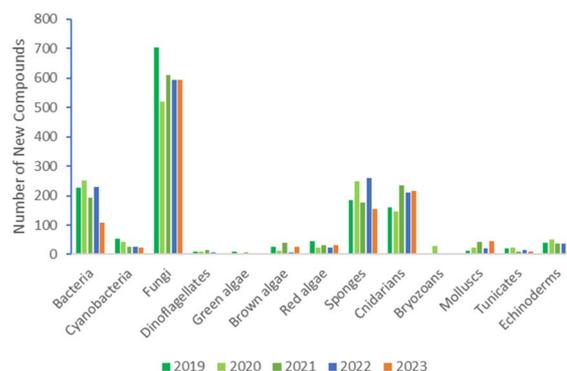
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**Fig. 1** Trends in new MNPs. The bars represent the total number of new MNPs reported each year over the last five years.

Trends in the number of new MNPs reported annually over the semi-decade show a substantial drop in new MNPs reported from bacteria in 2023. A decreasing trend in reporting of cyanobacterial MNPs continues. New sponge metabolites are at a decadal low. The number of new compounds reported from



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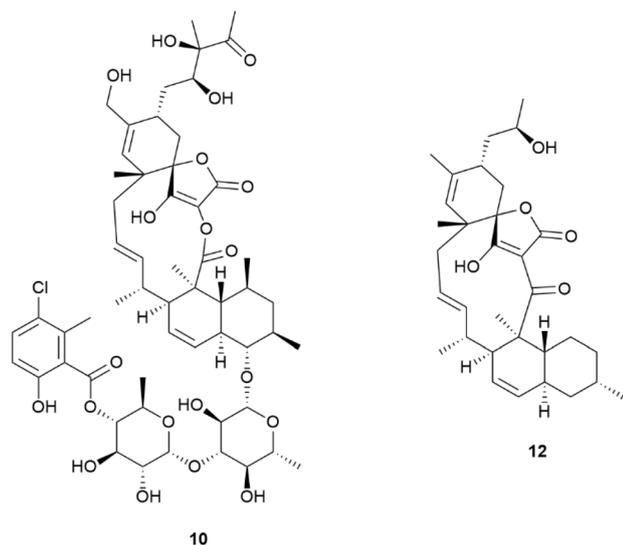
the brown algae rebounded after an anomalous low in the previous year (Fig. 1).

## 2 Marine microorganisms and phytoplankton

### 2.1 Marine-sourced bacteria

Actinobacteria were the most common source of bacterial MNPs with 94 new structures reported. A sponge-derived *Actinalloteichus cyanogriseus* yielded three new cyclolipopeptides, cyanogriptides A–C **1–3**.<sup>3</sup> Based on the annotations from the genome mining tool antiSMASH, the candidate biosynthetic gene cluster (BGC) *cgpV* was proposed to be responsible for the assembly of the compounds. A cyclic tetrapeptide, arthropeptide **4** was isolated from *Arthrobacter humicola* sourced from composted material of marine origin,<sup>4</sup> and a new diketopiperazine janibatide **5** was reported from a deep-sea sediment-derived *Janibacter* sp.<sup>5</sup> A new pyrroline, nocarpyrroline **6** was reported from a krill-derived *Nocardiopsis* sp.,<sup>6</sup> and two new furan derivatives, nicardifurans **7** and **8** were isolated from a sediment-sourced *Nocardiopsis* sp.<sup>7</sup>

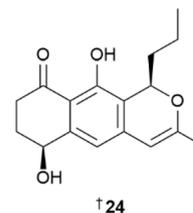
A coral-derived *Micromonospora* sp. yielded a new phenolic acid, 1-(6-methylsalicyloyl)glycerol **9**, the absolute configuration of which was confirmed *via* total synthesis.<sup>8</sup> Three new spirotronate polyketides, phocoenamycin **10** and **11** and maklamicin **12** were reported from a sediment-derived *Micromonospora endophytica*.<sup>9</sup> When tested against a panel of human pathogens, only the aglycone **12** showed weak activity against methicillin-resistant *S. aureus* (MRSA), and *M. tuberculosis*, and moderate activity against *E. faecium*.



Pyrrolizine alkaloids, phenopyrrolizins **13** and **14** were isolated as racemates from a sediment-derived *Micromonospora* sp., and their structures confirmed by X-ray diffraction analysis (XRD).<sup>10</sup> A series of new benzoxazole alkaloids, microechmycins **15–20** were reported to be the products of a BGC *mich* originally found in *Micromonospora* sp. but here, heterologously expressed in the host *Streptomyces albus*.<sup>11</sup> A large-scale, 70 L culture of *Salinispora arenicola* yielded three new rifamycin

analogues, salinisporamycins **21** and **22**, and salinifuran **23**.<sup>12</sup>

The genus *Streptomyces* continues to be the largest source of chemical novelty with 71 new MNPs reported in 2023. Pre-alnumycin **24** was isolated from a polychaete-derived *Streptomyces sundarbansensis*.<sup>13</sup> The putative BGC *als* was identified to be responsible for the biosynthesis of the compound and, when expressed in a heterologous host *Streptomyces coelicolor*, an additional new MNP phaeochromycin **25** was detected and isolated.



Five new aromatic polyketides, RM18 C–G **26–32**, were reported from a mangrove soil-derived *Streptomyces* sp.<sup>14</sup> Compounds RM-18 E and F were isolated as racemates and separation by chiral HPLC allowed the absolute configurations of the enantiomers **28/29** and **30/31** to be assigned. Strategies involving one strain many conditions (OSMAC) and addition of epigenetic modifiers yielded five new aromatic polyketides **33–37** from a cnidarian-derived *Streptomyces griseorubiginosus*,<sup>15</sup> and wailupemycins **38** and **39** from a green alga-derived *Streptomyces* sp.<sup>16</sup> A marine sediment-derived *Streptomyces massiliensis* yielded a new indanone derivative streptinone **40**,<sup>17</sup> and two new lactones **41**, **42** were reported from a sediment-derived *Streptomyces* sp.<sup>18</sup> Three new angucyclines, angumycins **43** and **44**, and kanglemycin **45** were reported from a *Streptomyces* sp. engineered to overexpress the native global regulator cyclic AMP-receptor protein (Crp).<sup>19</sup> A coral-derived *Streptomyces* sp. yielded naphthohydroquinones, iseoic acids **46** and **47** and a new naphthoquinone propanoic acid dimer bis-iseoate **48**.<sup>20</sup> Two new, ether-bridged C-glycosyl benz[*a*]anthracenes tandocyclinones **49** and **50** were isolated from a sediment-sourced *Streptomyces* sp.,<sup>21</sup> and a cold-seep-derived *Streptomyces olivaceus* yielded four new linear ansamycin analogues olimycins **51–54**.<sup>22</sup>

Four new enediyne-derived, cycloaromatised polyketides, jejucarbosides **55–58** were isolated from a sediment-sourced *Streptomyces* sp., with only **58** showing sub-micromolar cytotoxic activity against a panel of five human tumour cell lines (HTCLs), demonstrating that carbonate and methoxy functional groups are crucial for the activity of the series.<sup>23</sup> Two new *Streptomyces*-sourced antimycin analogues have been reported, antimycin **59**,<sup>24</sup> and antimycin **A2c 60**.<sup>25</sup> A hydrothermal vent sediment-derived *Streptomyces* sp. yielded two new linear polyketides kueishanamides **61** and **62** that exhibited weak antifungal activity against the pathogenic fungus *Cryptococcus neoformans* with no cytotoxicity against two HTCLs.<sup>26</sup>

Two new chlorinated, pyrrole-containing alkaloids, streptopyrroles **63** and **64** were reported from a sediment-derived

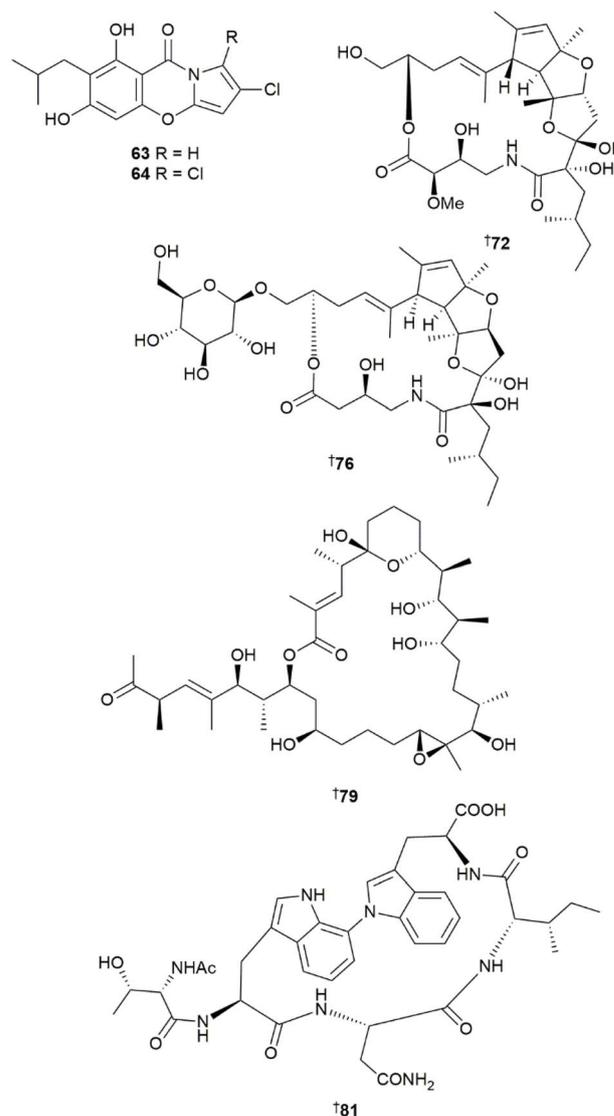


*Streptomyces zhaozhouensis*.<sup>27</sup> Both compounds showed moderate activities against Gram-positive bacteria but no activity against Gram-negative strains and only weak activity against a panel of six HTCLs. A new naphthyridine MNP, actinoquinazolinone **65** was isolated from a sediment-derived *Streptomyces* sp.,<sup>28</sup> and a new 3-hydroxybutanoic acid-containing quinazolinone streptonaphthyridine A **66** was reported from a *Streptomyces* sp. strain derived from a sediment sample collected from a submarine canyon.<sup>29</sup> Two new diphenazines, baraphenazine H **67** and izumiphenazine E **68** were reported from a sediment-derived *Streptomyces* sp.<sup>30</sup> Structural and stereochemical elucidation of these proton-deficient compounds was aided by computationally-predicted NMR and ECD spectra and the established methodology was used to revise absolute configurations of three co-occurring known dimeric phenazines, namely phenazinolin D **69**,<sup>31</sup> izumiphenazine A **70**,<sup>32</sup> and baraphenazine G **71**,<sup>33</sup> which were also reported as MNPs for the first time.

Structures and absolute configurations of four new polyketide-peptide hybrid macrolide lactams, somalactams A–D **72**, **73–75** isolated from an Arctic sponge-derived *Streptomyces somaliensis*, were unambiguously assigned by XRD.<sup>34</sup> Somalactams A **72** and B **73** possess a novel hexahydro-2*H*-cyclopenta [*b*]furo[2,3-*d*]furan tricyclic ring system. This structural motif has also been reported in argenteolide A **76** isolated together with a simpler analogue argenteolide B **77** from a deep-sea sediment-derived *Streptomyces argenteolus*.<sup>35</sup> Notably, while the planar structure of the macrocyclic ring is almost identical in **72** and **76**, the assigned absolute configurations at multiple stereogenic centres are different. An additional new glycosylated macrolide lactam, haneummycin **78** was isolated from a sediment-derived *Streptomyces* sp.<sup>36</sup> Two new 24- and 26-membered macrolactones, marinolides A **79** and B **80** were reported from a sponge-derived marine bacterium that was identified by partial 16S rDNA analysis to likely be a new genus within the Streptomycetaceae family.<sup>37</sup> Recognising the challenges of assigning the structures and absolute configurations of complex macrolactones, the authors identified a 97 kb BGC *mld* to be responsible for the assembly of the compounds and used bioinformatic analyses of ketoreductase and enoylreductase domains within the BGC to predict the structures and configuration of 13 out of 16 stereogenic centres in **79** and **80**. Full structural and stereochemical assignment of the two compounds was achieved through complete NMR analysis and XRD data and this matched the bioinformatic predictions.

Four new ribosomally synthesised and post-translationally modified peptides (RiPPs), cihunamides A–D **81**, **82–84**, were isolated from a volcanic island sediment-derived *Streptomyces* sp.<sup>38</sup> The compounds possess a rare C–N crosslink between the two tryptophan units formed through oxidative coupling catalysed by cytochrome P450. The authors proposed a new naming classification for this RiPP family, the “bitryptides”, defined by a single biaryl linkage between two tryptophan units and canonical atropisomerism. A cyclic undecapeptide, streptnatamide A **85** was reported from a sponge-derived *Streptomyces* sp.<sup>39</sup> Peptides such as **85** that contain non-canonical amino acids that can adopt different conformers in

solution represent significant structural elucidation challenges by NMR due to insolubility or line broadening. To facilitate fast structural elucidation, the authors developed a MS-based structure confirmation tool using isotopic fine structure (IFS) analysis and an in-house MS<sup>2</sup> analysis workflow. Other new peptide MNPs sourced from sediment-derived *Streptomyces* sp. included octadepsipeptides, quinomycins K **86** and L **87**,<sup>40</sup> and piperazic acid-containing decapaptides, lenziamides B1 **89** and D1 **88**.<sup>41</sup>



Four new sesquiterpenoids, pentalenomycins A–C **90–92**, and bolinane A **93** were reported from a sediment collection of *Streptomyces qinglanensis*,<sup>42</sup> and four new geosmin- and germacrane-type sesquiterpenoids, odoripenoids A–D **94–97** were isolated from a sponge-derived *Streptomyces* sp.<sup>43</sup>

Five new MNPs were identified from the phylum Firmicutes. An intertidal mudflat sediment-derived *Bacillus* sp. yielded two new glycosylated macrolactin analogues succinyl glyco-oxydifficidin **98** and succinyl macrolactin O **99**,<sup>44</sup> while a cold-seep sediment-derived *Bacillus* sp. collection yielded a further new macrolactin analogue 6'-O-succinyl methyl ester macrolactin O



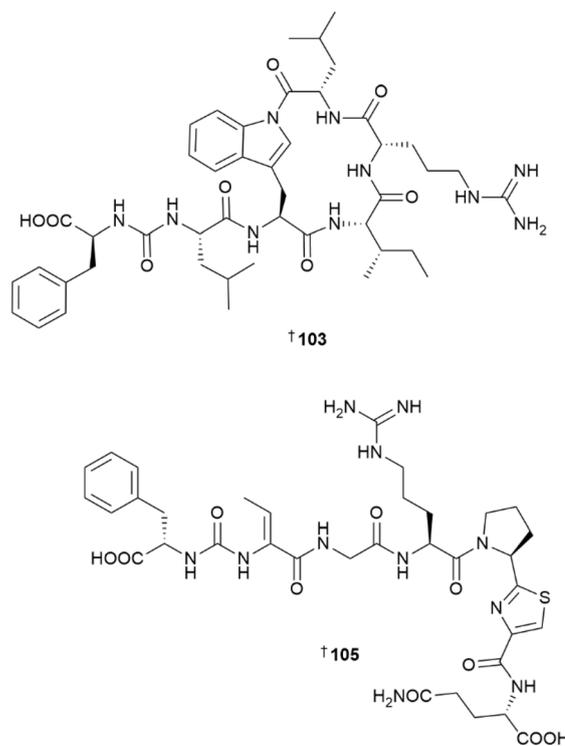
**100**, as well as two new hydroxy unsaturated fatty acids **101** and **102**.<sup>45</sup>

Eight new MNPs were reported from the phylum Pseudomonadota. A coral-derived *Microbulbifer* sp. yielded a new ureido-hexapeptide bulbiferamide **103**, with a rare *N*-aminoacylated indole linkage between tryptophan and leucine residues.<sup>46</sup> The same structure, referred to as bulbiferamide A, was also reported from a sponge-derived *Microbulbifer* sp., together with a related bulbiferamide analogue **104** where the terminal threonine unit was dehydrated to a dehydrobutyrine residue.<sup>47</sup> The latter study identified a putative BGC *bulb* to be responsible for the assembly of the bulbiferamides. Another *Microbulbifer* sp. strain derived from the sponge *Smenospongia aurea* yielded four related linear analogues, the pseudobulbiferamides A–C **105**, **106**, **107** and a truncated shunt metabolite **108**, and their BGC was identified and named *mbp*.<sup>48</sup> Interestingly, the BGCs for both pseudobulbiferamides and the bulbiferamides were present in this strain, with the pseudobulbiferamide BGC *mbp* found to be plasmid encoded, while the bulbiferamide BGC *bulb* was chromosomally encoded. The strain was shown to be capable of producing both families of ureido-peptides, and mass spectrometry imaging showed they occupy different physical spaces within the colony when grown on solid media. A genome-mining strategy targeting BGCs with siderophore related genes identified a marine-derived *Tistrella mobilis* as a possible producer of siderophores, from which two new *C*-diazoniumdiolate-containing MNPs, tistrellabactins A **109** and B **110** were identified.<sup>49</sup> Both compounds were found to coordinate Fe(III), but were also photoreactive upon exposure to UV light, releasing an equivalent of NO and H<sup>+</sup> from the bacterial cells in the process.

As with previous years, a small number of MNPs published in the literature from marine bacteria did not have adequate spectrometric and spectroscopic data to support the proposed structures.<sup>50–54</sup> Some MNPs reported in 2023 had structures proposed from mass spectrometric data, but without full NMR structural characterisation and were omitted from this review.<sup>47,48,55</sup> Total synthesis of the reported structure of cahuitamycin A revealed significant inconsistencies in the <sup>1</sup>H NMR spectroscopic data, putting the proposed structure in doubt.<sup>56</sup> Other total syntheses of bacterial NPs included *rac*-abyssomicin 2 and *rac*-neoabyssomicin B,<sup>57</sup> chejuenolides A–C,<sup>58</sup> *rac*-cyanogramide D,<sup>59</sup> dixiamycins A and B,<sup>60</sup> enhypprazinone A,<sup>61</sup> levesquamide,<sup>62</sup> lysiformine,<sup>63</sup> marinoquiquinoline A,<sup>64</sup> mindapyrroles A and B,<sup>65</sup> neaumycin B,<sup>66</sup> (–)-nenestatin A,<sup>67</sup> and sorangiolide A.<sup>68</sup>

Reviews focused on marine bacterial NPs published during 2023 included structures and biological activities of NPs reported from sponge-derived microorganisms,<sup>69,70</sup> deep-sea sourced actinobacteria,<sup>71</sup> phylum *Bacillota*,<sup>72</sup> and the genus *Pseudoalteromonas*.<sup>73</sup> Specific classes of bacteria-sourced MNPs reviewed included peptides with antimicrobial activity,<sup>74</sup> biosynthesis and biological activities of *Streptomyces*-sourced lipopeptides,<sup>75</sup> and structures and biological activities of pederin-type polyketides.<sup>76</sup> MNPs from marine-derived-bacteria with antibiotic and antibiofilm activities were reviewed,<sup>77,78</sup> as

were various strategies for the co-culture of marine-sourced bacteria.<sup>79</sup>



## 2.2 Cyanobacteria

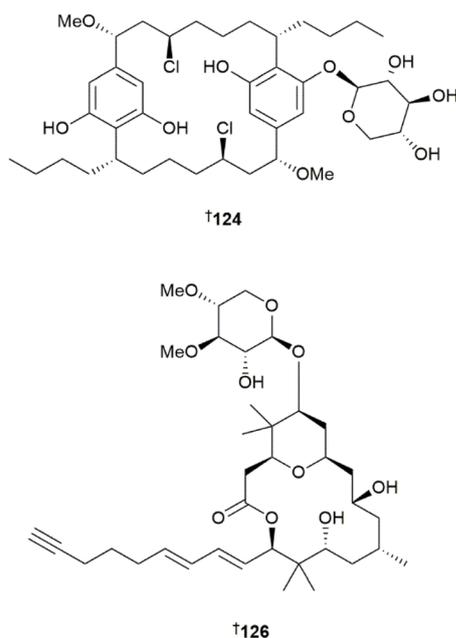
The lower number of MNPs reported from cyanobacteria in 2023 also coincided with a decrease in the chemical diversity of new structures. Another general observation is an increase in the isolation of new NPs from mixed assemblages of cyanobacteria. A mixed cyanobacterial collection of predominantly *Lyngbya* and *Dichothrix* spp. yielded a new peptide–polyketide hybrid NP, iezoside B **111**,<sup>80</sup> while a South China Sea collection of *Lyngbya* sp. yielded two new aplysiatoxin analogues, neo-debromoaplysiatoxin I **112**, and neo-debromoaplysiatoxin J **113**.<sup>81</sup> A new cyclic depsipeptide, alotamide B **114** was reported from a mixed assemblage comprised mostly of *Moorena* sp. (annotated as *Moorena* sp. in the manuscript)<sup>82</sup> and a new cyclopropane-containing fatty acid derivative, benderadiene **115** was reported from a bloom forming assemblage of *Lyngbya* sp.<sup>83</sup> Notably, there is still significant inconsistency in reporting of the correct naming of this genus with *Moorena* being the accepted genus name.

Four new peptide glycosides, suomilides B–D **116–119** were isolated from a laboratory cultivation of *Nostoc* sp.<sup>84</sup> Nostocyclophanes E–J **120–123**, **124**, **125**, were minor cyclophane metabolites reported from a 240 L culture of *Nostoc linckia* with the compounds showing weak to moderate cytotoxicity against the breast epithelial adenocarcinoma MDA-MB-231 cell line with GI<sub>50</sub> ranges from 0.72 to 8.2 μM.<sup>85</sup>

Akunolides A–D **126**, **127–129**, four new 16-membered macrolide glycosides bearing alkylated substitution at C-15, were isolated from the cyanobacterium *Okeania* sp.<sup>86</sup> Akunolide A **126** possesses a rare terminal alkyne structure in the alkylated



sidechain. The structure of 30-methylscillatoxin reported in 2019,<sup>87</sup> was revised to 7-*epi*-30-methylscillatoxin D **130** following a comparison of the NMR data with that of synthesised analogues.<sup>88</sup> Moreover, the taxonomic classification of the producing organism, initially assigned *via* morphological observations under light microscopy, was changed from *Moorea producens* to *Okeania hirsuta* based on 16S rRNA phylogenetic analysis. This taxonomic revision has implications for over 20 other new cyanobacterial MNPs in seven other articles reported from this collection (and obtained from a single extraction) over the last four years.<sup>87,89–94</sup>



Two new lipopeptides, okeaniamides A **131** and B **132** were isolated from a coastline collection of *Okeania* sp.<sup>95</sup> The compounds showed no cytotoxicity at 10  $\mu$ M but demonstrated an increase in adipocyte differentiation of 3T3-L1 pre-adipocyte cells in the presence of insulin when tested at concentrations of 5 and 10  $\mu$ M. Finally, a black band disease-forming, filamentous cyanobacterium *Roseofilum reptotaenium* collected from the massive starlet coral *Siderastrea siderea* yielded a new mixed polyketide/peptide 20-membered macrocycle, lookeyolide D **133**.<sup>96</sup>

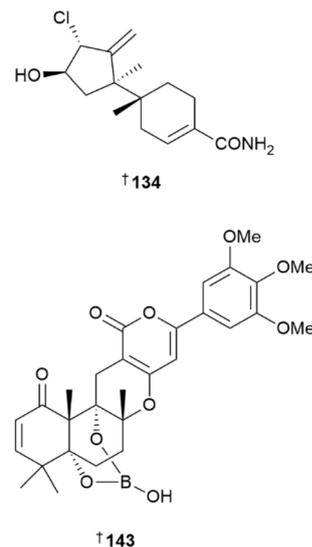
Reviews focused on marine cyanobacteria included summaries of various biological activities annotated for cyanobacterial MNPs including antifungal,<sup>97</sup> antiviral,<sup>98</sup> and cytotoxic activity against human cancer cell lines.<sup>99</sup> Notable work on the biosynthesis of cyanobacterial MNPs has included identification and characterisation of the putative BGC *lynB* for the assembly of lyngbyapeptin B,<sup>100</sup> a comprehensive review on the incorporation of fatty acids in the biosynthesis of cyanobacterial NPs,<sup>101</sup> and a review on NPs from symbiotic cyanobacteria and their biosynthesis.<sup>102</sup> Total syntheses of cyanobacterial MNPs included the polyketides (10*E/Z*)-trichotoxin A and dechlorotrictoxin A<sup>103</sup> as well as caldazole<sup>104</sup> and the peptides ikoamide,<sup>105</sup> odookeanynes A and B,<sup>106</sup> and a high yielding synthesis of gallinamide A which was achieved in nine steps of longest linear sequence and an overall yield of 32%.<sup>107</sup> The large-scale synthesis of complex polyketides has been

hampered by access to complex chiral building blocks. Access to the apratoxin A fragment, (2*R*,3*R*,5*R*,7*R*)-3,7-dihydroxy-2,5,8,8-tetramethylnonanoic acid, has been achieved through heterologous expression in the cyanobacterium *Anabaena* sp. PCC78120.<sup>108</sup>

### 2.3 Marine-sourced fungi

The sesquiterpenoid marinobazzanan **134** was isolated from an *Acremonium* species and shown to decrease cancer cell migration and invasion at nontoxic concentrations by downregulating transcription factors and modulating the expression level of other enzymes involved in cell motility and  $\beta$ -catenin expression. Additionally, **134** reduced the number of metastatic nodules in an intraperitoneal xenograft mouse model.<sup>109</sup> Myrchromanol analogues **135–142** were obtained from a culture of *Alfimbria verrucaria*<sup>110</sup> and an *Alternaria* species yielded territrem F **143**, a boronic ester of the co-isolated drimane meroterpenoid territrem B, both of which were weak synchronous Ca<sup>2+</sup> oscillation inhibitors.<sup>111</sup> Culture of *Amphicorda felina* resulted in isolation of meroterpenoids **144–152**.<sup>112,113</sup>

Three *Arthrinium* strains isolated from mangrove sediments contained an oxime **153** and pyridyl derivative **154**,<sup>114</sup> four sesquiterpenoids; arthroliferins A–D **155–158**,<sup>115</sup> and two tetrahydroisobenzofurans arthrinones A **159** and B **160**, respectively.<sup>116</sup> A further *Arthrinium* strain was the source of the pyridine alkaloids arthpyrones M–O **161–163**,<sup>117</sup> of which **161**, inhibited growth and metastasis of gastric cancer *in vivo* *via* targeting a signalling pathway.<sup>118</sup>



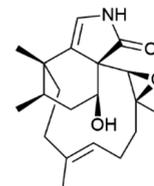
The *Aspergillus* genus was once again a source of many new metabolites including the dimeric tetrahydroxanthones, aculeaxanthones A–E **164–168**,<sup>119</sup> and benzoic-acid containing alkaloids, **169–176**.<sup>120</sup> Production of a further alkaloid **177**, by *Aspergillus aculeatus*, was induced *via* chemical epigenetic regulation with suberohydroxamic acid<sup>121</sup> and culture of *A. austwickii* yielded polyketides **178** and **179**, 2,3-dihydrobenzofuran derivative **180** and kojic acid derivative **181**.<sup>122</sup> Isocoumarin **182**,<sup>123</sup> quinazoline alkaloids felicarnezolines A–E **183–187**,<sup>124</sup> oxygenated chromene derivative **188**,<sup>124</sup>



anthraquinone derivative **189** and 2-aminoprop-2-enoic acid derivative **190**,<sup>125</sup> were all obtained from various *Aspergillus* cultures. *A. chevalieri* was the source of indole diketopiperazine alkaloids **191–195**,<sup>126</sup> and nonadride **196**,<sup>127</sup> cyclohexanone derivative **197** and drimane sesquiterpenoids **198** and **199** were obtained from a seagrass-derived strain.<sup>128</sup> Sediment-derived *Aspergillus* strains yielded indole alkaloid **200**,<sup>129</sup> thio-diketopiperazines **201–203**,<sup>130</sup> oxygen bridged phenolics **204** and **205** and dimeric isobenzofuran **206**,<sup>131</sup> whilst sponge-derived strains were the source of numerous indoloquinazoline alkaloids **207–227** and depsidone **228**.<sup>132,133</sup> Alkaloids were also obtained from several *Aspergillus* strains derived from various sources; sediment-derived *A. noonimiae* yielded indole diterpenoid glycosides noonindoles G–L **229–234**,<sup>134</sup> diketopiperazines **235–238** and **239–241** were obtained from coral-derived *A. puniceus*<sup>135</sup> and sponge-derived *A. sclerotiorum*<sup>136</sup> respectively. Culture of deep-sea-derived *Aspergillus* strains led to the isolation of cyclopentapeptides **242–248**,<sup>137</sup> bisabolane sesquiterpenoids **249** and **250–253** (the last four as racemates separated by chiral chromatography)<sup>138,139</sup> and *N*-acyl adenosine derivative **254**.<sup>140</sup> *A. terreus* strains were the source of chlorinated biphenyls **255–258**,<sup>141</sup> maleimides **259**, **260** and butenolides **261** and **262**,<sup>142</sup> sesquiterpenoid **263** and nitrobenzene derivatives **264** and **265** (the last two known synthetic compounds but new NPs).<sup>143</sup> *A. terreus* strains also produced terrein derivatives **266** and **267**, octahydrocoumarin derivative **268** and eurylene **269**, (the last a known terrestrial NP but new MNP).<sup>144</sup> Deep-sea-derived *A. versicolor* strains yielded pyrazinopyrimidine **270–273** and quinolinone **274**, **275** alkaloids (the last two known synthetics but new NPs),<sup>145</sup> diketopiperazine alkaloids **276–279**, (**276** and **278** not new but the absolute configuration as determined),<sup>146</sup> macrolactone **280**, quinazoline alkaloid **281** (ref. 147) and phenolic bisabolane sesquiterpenoids **282–292**,<sup>148</sup> while other strains of *A. versicolor* were the source of pyrroloindoline-containing cycloheptapeptide **293**, (also synthesised from the co-isolated asperversiamide A),<sup>149</sup> indole diketopiperazine alkaloids **294** and **295**,<sup>150</sup> dimeric citrinin derivatives **296–299**, isochromene derivative **300** and acetamide **301**.<sup>151</sup> Indole alkaloids, including dimeric diketopiperazines (**302–307**)<sup>152,153</sup> and indole diterpenoids **308–315**,<sup>154</sup> austalide derivative **316**,<sup>155</sup> ophiobolin sesterterpenoid **317** and drimane sesquiterpenoids **318–322**,<sup>156</sup> phenolic bisabolane **323**,<sup>157</sup> benzofuran derivative **324**,<sup>158</sup> glyoxylate-containing benzene derivative **325**,<sup>159</sup> unsaturated fatty acid **326**,<sup>160</sup> azaspiroenes **327–331**,<sup>161</sup> in addition to the *p*-terphenyls, asperterphenyls A–N **332–352**, were also obtained from various *Aspergillus* strains.<sup>162</sup>

*Asteromyces cruciatus* was the source of anthraquinone derivatives **353–355**,<sup>163</sup> an integrated genomics and metabolomics approach was utilised to isolate cyclopeptides **356–361** from *Beauveria felina*,<sup>164</sup> and a combination of metabolomics, chemometrics and traditional NP techniques resulted in isolation of phomactinine **362**, the first nitrogen containing phomactin, from a *Biatrispora* strain.<sup>165</sup> Chlorinated azaphilones **363–365** were isolated from strains of *Chaetomium globosum*<sup>166,167</sup> and diterpenoids **366** and **367**, sesquiterpenoids **368** and **369** and ecdysteroid **370** were all obtained from *Cladosporium oxysporum*.<sup>168</sup> Synthesis of all possible enantiomers of the indole alkaloids

colletotrichindoles A–E, **371–377**, was utilised in their structure determination. These, along with further indole alkaloids **378–382**, were obtained from a culture of *Colletotrichum gloeosporioides*,<sup>169</sup> while  $\alpha$ -pyrone derivatives **383** and **384** were isolated from a *Curvularia* strain<sup>170</sup> and 4a-*O*-methoxyarugosin H **385** was isolated from *Emericella nidulans* but may be an artefact resulting from the use of MeOH in the isolation procedure.<sup>171</sup>



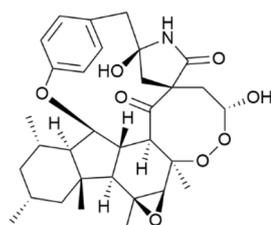
† 362

Various sediment-derived fungal strains have yielded a range of compounds. *Emericellopsis maritima* was the source of eremophilane sesquiterpenoids **386–389**,<sup>172</sup> a chlorogentisyl alcohol derivative **390** was isolated from *Epicoccum sorghinum*,<sup>173</sup> *Eutypella* strains yielded prenylated dihydroisocoumarin **391** and chromene amide derivative **392**,<sup>174</sup> pimarane diterpenoids **393–395** and cytosporin derivatives **396–402**,<sup>175,176</sup> epipolthiodioxopiperazines, graphiumins K–N **403–406** were obtained from *Exophiala mesophila*,<sup>177</sup> two anthraquinones **407** and **408** were obtained from a mangrove sediment-derived *Fusarium* sp.<sup>178</sup> and culture of *F. solani* led to isolation of the polyketides, fusarisolins F–K **409–414** and another polyketide **415**.<sup>179</sup> This last compound was named fusarin I but this name has already been used for another compound isolated from *F. solani* previously.<sup>180</sup> Culture of *Hamigera avellanea* strains yielded pentaketides **416–419** and *p*-hydroxyphenyl-2-pyridone derivative **420**,<sup>181</sup> and enantiomeric alkaloids **421** and **422** (resolved by chiral chromatography).<sup>182</sup> Steroidal lactone **423** was obtained from a deep-sea mussel-derived *Hypocrea* strain,<sup>183</sup> thio-diketopiperazines, lecanicilliums A–G **424–429** were reported from *Lecanicillium kalimantanense* isolated from mangrove sediment<sup>184</sup> and supplementation of growth media with amino acids led to isolation of alkaloids **430–432** and sterol **433**, (the last two are new MNPs but are a known synthetic product and plant metabolite, respectively).<sup>185</sup> A seawater-derived *Meira* strain was the source of thiolactones **434** and **435** and steroids **436** and **437**. Thiolactone **435** is a known terrestrial fungal metabolite but isolated here as a new MNP and the absolute configuration was revised from 3*R*,4*S* to 3*R*,4*R*.<sup>186</sup> A *Metarhizium* strain, also derived from seawater, yielded  $\alpha$ -pyrone glycosides **438–440** and phenolic glycoside **441**.<sup>187</sup> The chlorinated benzopyrone **442**, and two known terrestrial (but new to marine) NPs, a dichloropyrrol-2,5-dione **443** and meroterpenoid **444** were reported from mangrove sediment-derived *Mollisia* sp.<sup>188</sup> Fungal cultures derived from sediment were the source of a number of metabolites; cultures of *Paraconiothyrium sporulosum* yielded eremophilane **445–451** and santalane sesquiterpenoids **452–454**,<sup>189,190</sup> and isobenzofuranones **455** and **456**,<sup>190</sup> whilst *Paraphoma radicina* was the source of isobenzofuranone **457** and polyketide amino acid hybrid **458**.<sup>191</sup>

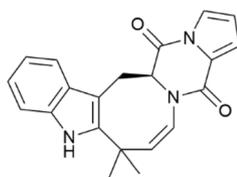
The *Penicillium* genus has always been extensively studied as a source of new metabolites but this year, there were even more



studies reported on this genus than on *Aspergillus*. Cultures of *Penicillium antarcticum* yielded  $\beta$ -resorcylic acid derivatives **459–463**,<sup>192</sup> cyclopiane diterpenoids **464** and **465** and pentaketide derivative **466**,<sup>193</sup> seawater-derived cultures of *P. chrysogenum* were the source of cerebroside A aglycone **467** and tyrosine derivative **468**,<sup>194,195</sup> sediment-derived *P. citrinum* strains yielded indole diterpenoid **469**,<sup>196</sup> polyketides **470–472** and three pairs of C-9 verrucosidin epimers **473–478**,<sup>197,198</sup> (the first two pairs revising C-6 configuration from 6*S* to 6*R*), while *P. citrinum* strains sourced from crustaceans were the source of alkaloid **479** and unusual hirsutellone analogues **480** and **481–486**.<sup>199,200</sup> Of these, perpyrrospirone A **480** consists of an unprecedented 6/5/6/8/5/13/6 oxahexacyclic scaffold with a peroxide-bridged 8,9-dioxa-2-azaspiro[4,7]dodecane core. Indole diketopiperazine alkaloid **487**, obtained from fermentation of *P. dimorphosporum*, resensitised drug-resistant prostate cancer cells to the anti-androgen drug enzalutamide through specific downregulation of the androgen receptor without associated toxicity.<sup>201</sup>



†480

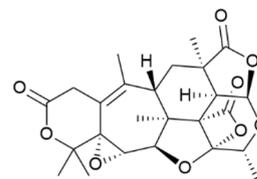


†487

Polycyclic alkaloid communesin M **488**, was isolated from a culture of *P. expansum* and synthesised from co-isolated communesin A.<sup>202</sup> Co-culture of *P. janthinellum* with *Paecilomyces formosus* (both collected from the same source) led to isolation of nine indole diterpenoids, janthinellumines A–I **489–497**,<sup>203</sup> meroterpenoid **498** was obtained from axenic culture of *Penicillium ochrochloron* and on co-cultivation with the bacterium *Bacillus subtilis*, the known synthetic compound but new NP, ochrocholic acid **499** was produced.<sup>204</sup> Sesterterpenoid **500** was obtained from *P. oxalicum*,<sup>205</sup> as was phenalenone derivative **501**.<sup>206</sup> Verrucosidin derivatives, poloncosidins G–K **502–506** were isolated from cold-seep sediment-derived *P. polonicum*,<sup>207</sup>  $\beta$ -carboline **507–509** and 2-quinolinone alkaloids **510** and **511** were obtained from *P. raistrichii*,<sup>208</sup> deep-sea coral-derived *P. rubens* was the source of polyketide **512**, sesquiterpenoid **513** and steroid **514**, the last of which exhibited potent activity against *E. coli* and *Vibrio parahaemolyticus*.<sup>209,210</sup> Co-culture of a natural complex/association of sea urchin-derived *P. sajarovii* and *Aspergillus protuberus* led to production of polyketides **515** and **516**,<sup>211</sup> seven meroterpenoids **517–523** and a range of azaphilones were also obtained from *P. sclerotiorum*.<sup>212,213</sup> Of these, **524–527** were

obtained from a red alga-derived strain by Taiwanese researchers, with **524** and **525** designated penicilazaphilones H and I, however, the name penicilazaphilone H had already been very recently designated to a brominated analogue by Chinese researchers and this same group isolated further azaphilones **528–534** from a sponge-derived strain, including **524** and **525** (here named penicilazaphilones L and K respectively).<sup>214</sup> Very close timing/overlap in the publication process of these reports has led to this confusion; **524** and **525** were reported first but should be renamed. Green algal-derived *P. stecki* cultures yielded tanzawaic acid **535–541** and benzene **542** derivatives,<sup>215</sup> in addition to fusarin derivatives **543–547**.<sup>216</sup> Deep-sea sediment-derived *Penicillium* strains yielded sulfated isonitrile, sulfoxanthicillin **548** and xanthenes **549** and **550**,<sup>217,218</sup> whilst other sediment-derived strains were the source of citrinin derivatives **551–559**,<sup>219</sup> polyketides **560** and **561**,<sup>220</sup> indole alkaloid **562**,<sup>221</sup> eremophilane sesquiterpenoid **563** and meroterpenoids **564** and **565**.<sup>222,223</sup> A soft coral-derived strain yielded the linear peptides, penicamides A **566** and B **567** and alkaloids **568** and **569** and butenolide **570** were obtained from a sponge-derived *Penicillium* strain.<sup>224,225</sup>

Two studies on a *Penicillium* species isolated from the roots of the Chinese mangrove *Lumnitzera litorea* furnished 15 rearranged merosesquiterpenoids, littoreanoids A–O **571–585**, one (**573**) possessing a rare oxetan-2-one ring<sup>226</sup> and nine related merosesquiterpenoids, peniciacetals A–I **586–594**.<sup>227</sup>



†573

Other MNPs isolated from mangrove-derived *Penicillium* spp. were meromonoterpenoids cyclohexenoneterpenes A–J **595–604**,<sup>228</sup> sesquiterpenoid **605**,<sup>229</sup> diterpenoids **606** and **607**, merosesquiterpenoid **608**, oxime **609**, carboxylic acid **610**, stilbene **611** (known, but absolute configuration now determined), phenol **612** and indoloditerpenoids **613–616**.<sup>230,231</sup>

Polyketides **617–623**, were reported from culture of a *Peroneutypa* strain, the latter being a known terrestrial NP but new MNP and with absolute configuration determined for the first time.<sup>232</sup> Another compound was claimed as new but the structure subsequently corrected to that of known NP daidzein.<sup>233</sup> Farnesyl hydroquinones **624** and **625** were obtained from culture of *Pestalotiopsis diploclisia*,<sup>234</sup> phaeosphaerins A–E **626–630** are isocoumarins isolated from a *Phaeosphaeriopsis* strain,<sup>235</sup> a *Phoma* strain was the source of polyketides and a sesquiterpenoid, **631–633**,<sup>236</sup> and a mangrove sediment-derived *Phomopsis* sp. contained isocoumarins **634–636**, and an  $\alpha$ -pyrone **637**.<sup>237</sup> Tetracyclic steroids **638–643** were obtained from a *Rhizopus* strain,<sup>238</sup> culture of *Samsoniella hepiali* led to isolation of aminated fusaric acid derivatives **644–646** and PKS-NRPS derived polyketide **647**,<sup>239</sup> a *Spiromastix* strain yielded chlorinated diphenyl ethers **648–650** and cyclopentanone **651**,<sup>240</sup> phenyl spirodrimanans **652–656** (with **655** being a known



semi-synthetic product but new NP) were isolated from culture of *Stachybotrys* strains<sup>241,242</sup> and cyclopropane derivatives **657**, **658** and  $\alpha$ -pyrones **659–665** were obtained from a *Stagonospora* strain.<sup>243</sup> In the course of the structural determination of the latter  $\alpha$ -pyrones, computational chemistry and NMR analyses suggested that the structures of the terrestrial plant metabolites, chenopodalans A–F should be revised from furofuran to  $\alpha$ -pyrones.<sup>243</sup> *Talaromyces* strains were the source of a number of polyketides, including azaphilone derivatives **666** and **667**, (C-8 epimers) **668–670**,<sup>244</sup> nonadride derivatives talarodrides G **671** and H **672** and depsidone derivative botryorhodine K, **673**,<sup>245</sup> and the spirocyclic talaromyacins A–C **674–676**.<sup>246</sup> Dibenzodioxepinones **677–680**, diphenyl ether **681**, benzopyran **682**, benzophenones **683** and **684**,<sup>247</sup> maleic anhydrides **685** and **686**, and three simple isoprenyl phenyl ethers **687–689**,<sup>248</sup> were obtained from mangrove sediment-derived *Talaromyces* species. The glucosidic polyketides, talaminosides A–C **690–692**, enantiomers **693** and **694**, (resolved by chiral chromatography) and azaphilones **695** and **696** were isolated from a culture of *T. minioluteus*.<sup>249</sup> *T. pinophilus* yielded a range of metabolites including known terrestrial fungal metabolites bacillisporins A **697** and B **698** as new MNPs, hybrid phenalenone dimer talaropinophilone **699**, azaphilone **700**, phthalide dimer **701**, steroid **702** and 1-deoxyrubralactone **703**, the configuration of which was revised to 11S.<sup>250</sup> The structure of talarolide A was revised to **704** after its isolation from a *Talaromyces* strain along with three analogues, talarolides B–D **705–707**. Talarolide B **705** was prepared *via* solid phase peptide synthesis.<sup>251</sup> Heterologous expression of a silent BGC from a *Talaromyces* strain in *Aspergillus nidulans* led to isolation of labdane diterpenoid derivatives, talarobins A–E **708–712**, with three P450 enzymes determined to catalyse multi-step reactions in the biosynthetic pathway.<sup>252</sup> Culture of a *Tolypocladium* strain resulted in the isolation of lipopeptaibols, tolypocaibols A **713** and B **714**,<sup>253</sup> whilst phomalone derivatives, tricholichenones A–D **715–718**, were obtained from deep-sea sediment-derived *Trichobotrys effusa*.<sup>254</sup> *Trichoderma* strains derived from red or brown algae were the source of numerous metabolites including bisabolene sesquiterpenoids **719–722**, cyclopentene **723** and cyclopentenone **724**, derivatives,<sup>255</sup>  $\gamma$ -lactone trichonafurin A **725**,<sup>256</sup> sorbicillinoid derivative **726**,<sup>257</sup> carotane sesquiterpenoid, trichocarotin N **727**,<sup>258</sup> harziane diterpenoid, harziaketol A **728** and sterol, trichosterol A **729**,<sup>259</sup> hydroxylated lipids, trichoderols B–G **730–735**,<sup>260</sup> and cyclopentenone **736** and wickerol **737** derivatives.<sup>261</sup> Further *Trichoderma* strains yielded sesquiterpenoid (**738–743**) and diterpenoid (**744** and **745**) aminoglycosides,<sup>262</sup> and the peptaibols, trichorzins A–G **746–752**.<sup>263</sup>

Computational methods were used to show that the structure assigned to janthinolide A obtained from coral-derived *Penicillium janthinellum* is incorrect and that the compound isolated was actually the known terrestrial NP, janthinolide C **753**.<sup>264</sup> The isolation was the first report of janthinolide C from the marine environment however.<sup>264</sup> The absolute configuration of vismione E, previously obtained from a sponge-derived *Aspergillus* strain, was established as **754**.<sup>265</sup>

Total synthesis of aflaquione I was achieved by two parallel strategies in nine and four steps respectively,<sup>266</sup> and a divergent

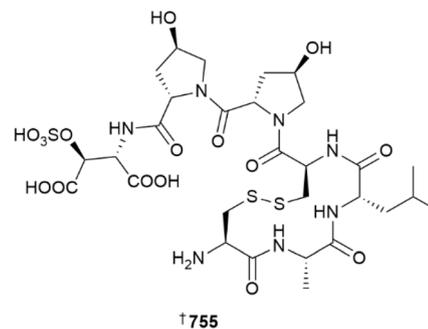
synthetic strategy was employed to synthesise conidiogenones E, F and 12 $\beta$ -hydroxyconidiogenone C.<sup>267</sup> Chemoenzymatic synthesis of 13-oxoveeruculogen was achieved in ten steps from commercially available materials,<sup>268</sup> gram scale preparation of a key intermediate facilitated the successful synthesis of tanzawaic acid B<sup>269</sup> and syntheses of marlines B and C were accomplished utilising a multicomponent reaction method.<sup>270</sup> Syntheses of colletopeptide A and colletotrichamide A were achieved *via* a cyclic tripeptide derivative as a key intermediate<sup>271</sup> and varioxiranol B and C were prepared by convergent strategies.<sup>272</sup>

Penicopeptide A was shown to promote osteoblast-related bone formation, indicating its potential in osteoporosis prevention,<sup>273</sup> pimarane diterpenoid scoparasin B was shown to inhibit angiogenesis, vascular mimicry and tumour growth,<sup>274</sup> naphtho- $\gamma$ -pyrone aurasperone F inhibited amyloid- $\beta$  (A $\beta$ ) aggregation and exhibited a protective effect against A $\beta$  toxicity so could be useful in treatment of Alzheimer's disease<sup>275</sup> and pretrichodermamide B inhibited the transcription activator STAT3 *in vivo*, also promoting cell cycle arrest and apoptosis.<sup>276</sup>

Gene deletion, heterologous expression, and biochemical characterisation were utilised to demonstrate that a unique fungal P450 enzyme, CtdY catalyses amide bond cleavage in the 2,5-diazabicyclo[2.2.2]octane system and subsequent decarboxylation to form the 6/5/5/6/6 pentacyclic ring system in (21R)-citrinadin A. Seven enzymes were implicated in subsequent post-translational modification to produce the metabolite.<sup>277</sup> The biosynthetic pathway to the dipeptide (+)-azonazine was reconstituted using four enzymes and the study revealed that the route to the benzofuranoindoline core occurs *via* an oxidative coupling reaction catalysed by the P450 enzyme AznC.<sup>278</sup> The BGC responsible for biosynthesis of (–)-protubonine B was identified *via* heterologous expression, gene deletion experiments and isolation of subsequently accumulated products.<sup>279</sup> Techniques utilised for activation of silent BGCs in fungi such as epigenetic regulation, co-culture, precursor feeding, heterologous expression and altering fermentation conditions were reviewed.<sup>280</sup>

## 2.4 Dinoflagellates

The number of reported dinoflagellate and diatom derived MNPs continues to decline.<sup>1</sup> A novel peptide **755** from *Seminavis robusta*, obtained from a culture collection, is the first sex-inducing pheromone of diatoms known. Compound **755** induces production of a diproline diketopiperazine dimer as an attraction pheromone at doses estimated to be in the low fM range. Assuming the peptide is chemically stable, each *S. robusta* cell



produces ~150–400 amol of 755 within five days, leading to nanomolar concentrations at a cell density of 20 k cm<sup>-2</sup>. Stable isotope incorporation (<sup>13</sup>C/<sup>15</sup>N) during culturing was crucial to providing material suitable for NMR-based structural studies.<sup>281</sup>

A new ergosterol derivative 756 has been obtained from a Vietnamese *Thraustochytrium pachydermum*, although it was inactive against seven bacterial strains.<sup>282</sup> As is commonplace, new azaspiracid and gambierone congeners were proposed based solely on mass spectrometric studies and so are not definitively assigned, therefore, these structures are not shown here.<sup>283,284</sup> The first total synthesis of amphidinolide S has been achieved using allyl alcohol as a key acrolein equivalent. The same study also completed the syntheses of amphidinolides J and R in half the number of steps previously needed.<sup>285</sup> A “two-phase” synthetic approach has resulted in the total synthesis of portimine B 757, resulting in its structural revision, and has also provided portimine A in amounts suitable to probe its molecular target using photoaffinity labelling studies; portimine A targets the 60S ribosomal exporter NMD3.<sup>286</sup> The reported structure of prorocentoin, and its subsequent revised form 758, have been synthesised for the first time.<sup>287</sup> The relative configuration of the C-61 to C-83 segment of the super carbon chain compound symbiodinolide 759 has been established by synthesis.<sup>288</sup>

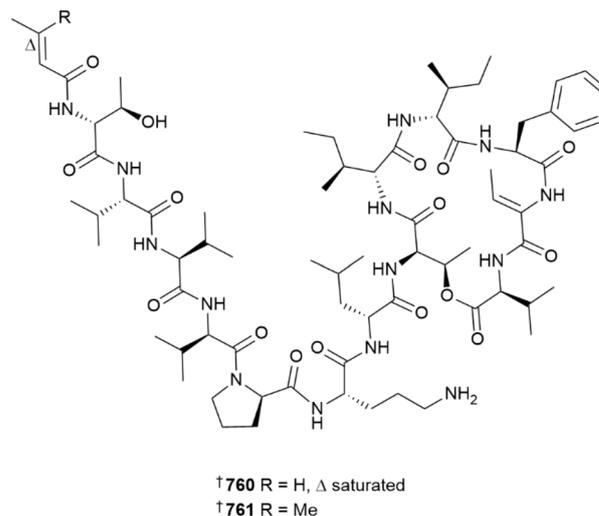
A review of the low molecular weight carbohydrate-derived MNPs of microalgae has been published,<sup>289</sup> as has a summary of the synthesis and mechanisms of action of the amphidinolides.<sup>290</sup> The genus *Amphidinium* is one of the most prolific producers of toxic metabolites known. Rather than focusing on their biotoxins, the antimicrobial, anticancer and antifungal properties of *Amphidinium*-derived metabolites have been examined.<sup>291</sup> The role microalgal compounds may play as biopesticides has also been reviewed.<sup>292</sup>

The mechanism of action of okadaic acid (OA) continues to intrigue researchers. OA downregulates the metabolism of xenobiotics in the liver by activating NF-κB signalling that stimulates the release of various interleukins, with downstream activation of JAK (Janus Kinase)-signalling.<sup>293</sup> Domoic acid (DA) is a potent neurotoxin produced by diatoms of the genus *Pseudonitzschia*. A recent study has shown that even trace amounts of DA can alter the make-up and biodiversity of marine protists, altering the ecosystems they inhabit at a functional level, largely driven by alteration of phototroph composition and subsequent downstream effects.<sup>294</sup> In a different study, *Desulfovibrio* and *Clostridiales* bacteria have been shown to metabolise DA via a novel reductive biotransformation pathway.<sup>295</sup>

A new method to perform metabolite fingerprinting using magic angle spinning solid-state NMR spectroscopy has been applied to study biofuels and nutritional components from three microalgal species.<sup>296</sup> Biotoxins produced by harmful algal blooms (HABs) continue to plague communities around the world through contamination of shellfish food stocks. SoundToxins is a new research and monitoring partnership in Puget Sound, Washington State, established to help monitor HABs in the area. Over 30 partner organisations including indigenous peoples, local citizens and aquaculture fisheries, use real-time monitoring of their local areas to help provide critical information in a timely fashion to regulators and testing agencies.<sup>297</sup>

### 3 Green algae

A species of *Bryopsis* (Mie Province, Japan) was the source of new kahalalide congeners Z<sub>3</sub> 760 and Z<sub>4</sub> 761, both of which are weakly cytotoxic against murine fibroblasts. To support their assignment of absolute configuration using standard Marfey's approaches, the authors also analysed the metagenomic DNA from the *Bryopsis* specimen to determine a genetic reasoning for the differences in amino acid configuration between the new compounds and earlier members of the class. The presence of a key E-domain was found to control the configuration of the kahalalide products, based upon A-domain specificities. In addition, the authors identified a suitable BGC from symbiotic bacteria belonging to a new taxon *Candidatus* Endrobryopsis kahalalidefaciens. They related kahalalide biosynthesis to the presence of different strains. Notably, the lack of production of both kahalalide F and Z within a single strain implies that different strains have evolved independently without genetic crossing.<sup>298</sup> Other peptide-based MNPs were reported from a green alga but the structures were identified only using mass spectrometry and therefore are not shown here.<sup>299</sup>



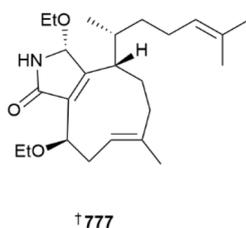
The synthesis of a weakly cytotoxic, 22-amino acid linear peptide from *B. plumosa* has been achieved,<sup>300</sup> as has a revised total synthesis of indolocarbazole racemosin B in two steps with over 50% yield.<sup>301</sup> *In vivo* testing of acetylated carotenoid siphonoin, isolated from the edible green algae *Caulerpa lentillifera* and *Codium fragile*, has shown that it is absorbed in the gut (mouse model) with minimal degradation whilst still exhibiting anti-inflammatory effects by inhibiting production of pro-inflammatory cytokines.<sup>302</sup> Whole genome sequencing of *Chrysothrix taylorii*, which has been linked to coastal algal blooms, has revealed that the alga has an extensive suite of BGCs alongside a small microbiome with limited biosynthetic potential.

### 4 Brown algae

It is surprising that all of the new compounds reported from brown algae were exclusively terpenoid in origin. There were two reports of macrocarquinoid meroditerpenoids from *Sargassum macrocarpum*, one from a Japanese specimen (762 and 763) and



the other sourced from Korea (764–767), respectively.<sup>303,304</sup> A new nor-meroterpenoid sargasilol A **768** and eight related compounds, sargasilols B–I **769–776** were reported from *S. siliquastrum*.<sup>305</sup> Five new xenicane meroditerpenoids, including rare lactams **777**, **778**, and **779**, a butanone **780** and a more standard structure **781**, were isolated from a Chinese *Dictyota coriacea*; all exhibited weak antioxidant activity with three as likely artefacts from extraction with EtOH.<sup>306</sup> A Chinese (Hainan Province) collection of *Sargassum polycystum* yielded an unusual spiro-cyclic sesquiterpenoid **782** of the spheciospongone series.<sup>307</sup> A series of new diterpenoids from different biosynthetic classes **783–790** with varying levels of ability to inhibit NO production were obtained from the invasive alga *Rugulopteryx okamurae* sourced from Punta Carnero, Spain.<sup>308</sup>



Further structures were claimed in another publication, but one of the authors of this review (RAK) has independently raised concerns about the spectroscopic data to the publishing journal.<sup>309,310</sup>

A review of the diterpenoid metabolites of *Dictyota* and *Canistrocarpus* algae sourced from Brazil has been published,<sup>311</sup> as has a summary of Ochrophyta compounds with potential to treat neurodegenerative diseases.<sup>312</sup> Two reviews focusing on *Sargassum* have been published; one is a review of antioxidant metabolites sourced from the genus,<sup>313</sup> while the other focuses specifically on the bioactivities and active principles of the edible alga *S. fusiforme*.<sup>314</sup> As noted above, *R. okamurae* is a member of an invasive genus of algae that has spread from native waters in Asia through to Europe. A review of *R. okamurae* and potential applications of it as an economic resource during efforts to control its spread has been published.<sup>315</sup> Bioinformatic analysis has suggested that a series of non-canonical PKS genes from the host alga are responsible for the biosynthesis of the antibiotic, macrocyclic halogenated ether, chrysopaentin A.<sup>316</sup>

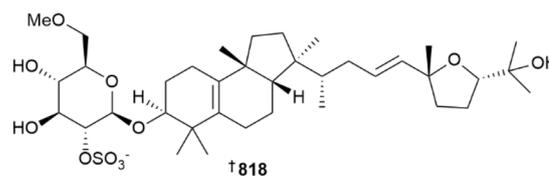
## 5 Red algae

New C15-acetogenins were reported from *Laurencia* species collected in Japan (**791**) and Egypt (**792–800**), respectively.<sup>317,318</sup> Chlorobenzoate solieriate **801** was isolated from a *Solieria* species from Zhangjiang City, Guangdong Province, China,<sup>319</sup> while a *Laurencia* species from the same geographical area yielded halogenated laurenhalogens A **802** and B **803**, respectively.<sup>320</sup> A tribrominated diphenyl methane **804** was obtained from *Symphocladia latiuscula*, although the metabolite was inactive as an antioxidant.<sup>321</sup> A series of new mycosporine-like amino acids (MAAs) were isolated from two intertidal species, *Bostrychia scorpioides* (**805–810**) and *Catenella caespitosa* (**811**, **812**), respectively, both collected from France (Brittany). Such MAAs help protect the producing alga from UV damage.<sup>322</sup>

The Rhodophyta are well known for their repertoire of poly-halogenated monoterpenoids. *Portieria hornemannii*, collected at the Penghu (Pescadore) Is., was the source of three halogenated linear monoterpenoids and while **813** was a weak inhibitor of TNF- $\alpha$  expression, congeners **814** and **815** were inactive, hinting at a potential SAR.<sup>323</sup> Two brominated aplysin derivatives **816** and **817** showed activity against settling of the mussel *Mytilus galloprovincialis* at 0.16  $\mu\text{mol cm}^{-2}$  so may be antifouling leads.<sup>324</sup>

Two separate collections of Fijian *Peysonnellia* spp. have resulted in the isolation of sulfated triterpenoid glycosides peyssobaricanosides A–C **818**, **819–820**. Genetically and otherwise chemically similar *Peysonnellia* samples from the Solomon Is., collected from similar geological locations, did not produce these metabolites, indicating population-level differences in metabolite profiles. Cryo-electron microscopy-based micro-crystal electron diffraction (microED) was used to establish the absolute configuration of **819**.<sup>325</sup>

Red algal metabolites that have been synthesised include a butyl dibromobenzoate,<sup>326</sup> dibromoindole glossobalol,<sup>327</sup> and histidine-derived alkaloid colensolide A.<sup>328</sup> A survey of the chemistry and bioactivity of metabolites from genus *Gelidium* has been published.<sup>329</sup> Both enantiomers of elatol have been assessed for activity against the primary amoebic meningoencephalitis-causing *Naegleria fowleri*; (+)-elatol showed weak to moderate activity against two strains of the amoeba while the (–)-enantiomer was inactive.<sup>330</sup> Laurequinone (*Laurencia johnstonii*) shows weak to moderate anti-leishmanicidal activity against the promastigote form of *Leishmania amazonensis* and seems to cause apoptosis of the parasite.<sup>331</sup> Analysis of seasonal variation of metabolites of *Jania rubens* (Israeli Mediterranean coast) coupled with bioactivity against non-small cell lung cancer has highlighted essential fatty acid eicosapentaenoic acid as the main driver of activity.<sup>332</sup> Polyhalogenated carbazoles are increasingly detected in the environment and are products of both human and natural sources. A study of the bromoperoxidase reaction has determined the regiospecificity of halogenation of carbazole and has subsequently detected the products in algal samples from the South China Sea.<sup>333</sup>



## 6 Sponges

A series of sixteen new phytoceramides **821–836** were obtained from *Monanchora clathrata* collected in Western Australia.<sup>334</sup>

Assimiloside A **837** is an unusual, branched glycolipid lactone isolated from a dredged (160 m) sample of *Hymeniacidon assimilis* collected from the Urup Is. The structure of **837** was determined using a combination of spectroscopic, computational and degradative studies, although the configuration at C-16 remains unresolved. Assimiloside A stimulated both ROS production and lysosomal activity in RAW 264.7 cells



at non-toxic concentrations between 0.01 to 10  $\mu\text{M}$ , making it a new immunomodulatory lead.<sup>335</sup>

A specimen of *Clathria faviformis* yielded a dihydropyridinium-containing lipid, favilipid A **838**. This lipid contains a chromophore and hence is UV-active, which is unusual for the class. The dihydropyridinium core exhibited slow protium-deuterium exchange of carbon-bound hydrogen nuclei when stored in  $\text{CD}_3\text{OD}$  NMR solvent. In addition, **838** is a weak inhibitor of five of 24 kinases, three of which are involved in immune system regulation.<sup>336</sup>

Two pairs of enantiomeric butenolide lipids **839/840** and **841/842** were obtained from a Chinese *Suberites* sponge.<sup>337</sup> Both the first isolation and total synthesis of adamantane-like arsenicin D **843** were achieved,<sup>338</sup> while a new valine-containing formamide **844**, baeramide, was obtained from a *Haliclona baeri*.<sup>339</sup> SAR analysis of two new onnamide congeners **845**, and **846**, which are weak to moderately cytotoxic to mammalian cell lines, revealed the importance of the sidechain alkene geometry for bioactivity.<sup>340</sup> The genus *Phakellia* remains a rich source of new peptides, with phakellisin A–E **847–851** being isolated from a Chinese sample; note the names given to these compounds are very similar to other *Phakellia*-derived metabolites (for example the pyrrole imidazole alkaloid phakellin) from different biosynthetic classes and readers should exercise care to not confuse them.<sup>341</sup>

An Australian (Coral Sea, Far North Queensland) *Theonella* species gave six new cyclotheonellazoles **852**, **853–857**. All incorporate non-proteogenic amino acids including the key protease transition state mimic, 3-amino-4-methyl-2-

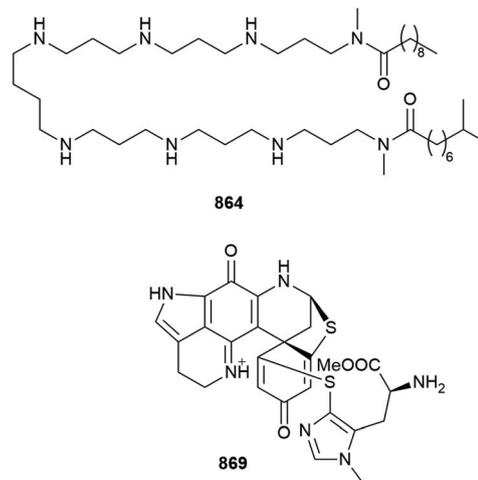
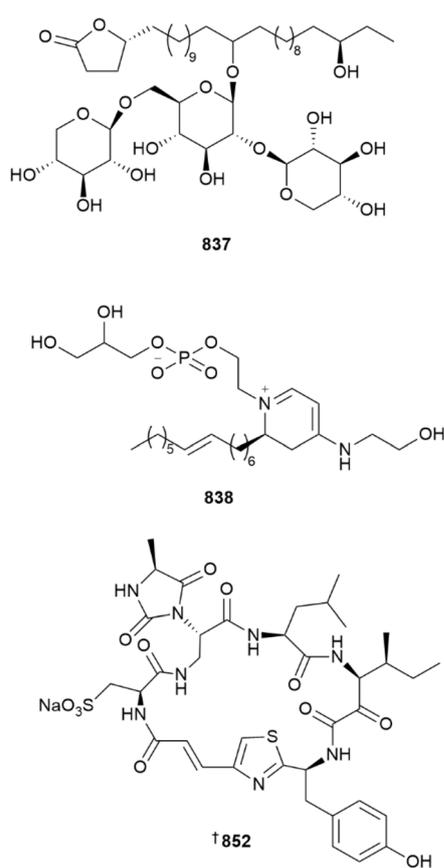
oxohexanoic acid. It is therefore logical that all six isolates are potent ( $\text{IC}_{50}$  16–61 nM) inhibitors of mammalian elastase but at non-toxic concentrations ( $\text{IC}_{50}$  > 100  $\mu\text{M}$  vs. 3 HTCLs).<sup>342</sup>

Somewhat surprisingly, there was only one report of new sponge-derived macrolides in 2023, that of four new enigmazole congeners **858–861** from a Papua New Guinean *Cinachyrella enigmatica*.<sup>343</sup> Two new brominated diphenyl ethers **862** and **863** came from a *Dysidea fragilis* from Mozambique.<sup>344</sup>

Aptolobamine A **864** is a polyamine from *Aaptos lobata*. Analysis of mass spectrometric fragmentation data was key to assigning the structure of this metabolite, and also highlighted the presence of other homologues in the sponge extract. The purified compound showed a broad range of activities against cancer cell lines, bacterial strains, and the mixture of homologues also inhibited  $\alpha$ -synuclein aggregation.<sup>345</sup>

The isolation and total synthesis of 2-piperidone alkaloid dysidone A **865** has been reported,<sup>346</sup> while two new naphthyridine isomers **866** and **867** came from an *Aaptos* sponge collected at the Xisha Is., China.<sup>347</sup> Geobarretin D **868** is a bromoindole alkaloid containing the rare herbipoline motif.<sup>348</sup>

A semisynthetic approach has been used to assess SAR within the discorhabdin class of aromatic alkaloids. Three new metabolites of the class (**869**, **870**, **871**) have been isolated although **869** is likely an artefact of methanolic extraction. The effects of structural modification upon the bioactivity within the discorhabdin B, C and L series against Merkel cell carcinoma have been assessed. The presence of sulfur on the E-ring, as well as a protonated imine B-ring, are important for potent activity.<sup>349</sup>



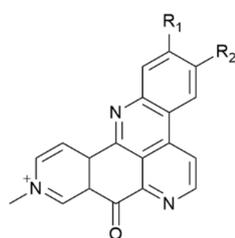
A partnership between the United States of America National Institute of Allergy and Infectious Disease and the National Cancer Institute (NCI) to screen the NCI's collection of over 326 000 extract fractions for antimicrobial activity has led to the isolation of two new amphimedine alkaloids from a Malaysian *Petrosia* sponge. Both 2-bromo- and 3-bromo-deoxy-amphimedine **872**, **873** are moderate to potent antimicrobial agents with activity against five of nine ESKAPE pathogens. The screening campaign resulted in  $\sim 3000$  leads from the pre-fractionated library, representing  $\sim 1\%$  hit-rate.<sup>350</sup>

A dredged (97 m) *Isabela* sponge from Zuytdorp, Western Australia, was the source of three new porphyrin metabolites

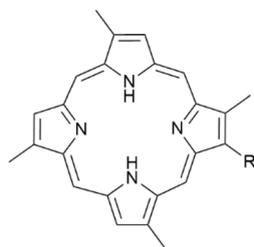


isabellins A **874** and B **875**, and Fe<sup>3+</sup>-containing isabellihemin A **876**. Heavily reduced porphyrin **874** is potently active against two HTCLs while the other isolates were inactive. Other related Fe-complexes were also detected by LCMS but were not isolated and fully characterised due to paramagnetic effects of the bound metal. NMR characterisation of the compounds required extensive analysis of NOESY correlations.<sup>351</sup>

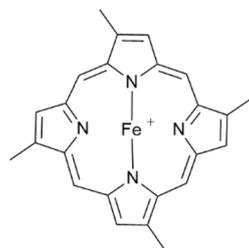
Terpenoid MNPs continue to dominate the new metabolites reported from Porifera. A seco-meroterpenoid, dysambiol **877**, is moderately potent as an anti-inflammatory agent but is non-toxic against RAW 264.7 cells at concentrations up to 20 μM.<sup>352</sup> Ten new merosesquiterpenoids, pseudoceranolids A–J **878–887** were isolated from a Xisha Is. (China) sample of *Pseudoceratina purpurea*,<sup>353</sup> while merosesquiterpenoid dimer thorectidiol A **888**, isolated as a racemate from a Papua New Guinean *Dactylospongia elegans*, is a moderate inhibitor of the SARS-CoV-2 spike protein receptor-binding domain interaction with the host ACE2 receptor.<sup>354</sup> Meroditerpenoid alkaloids **889–891**, along with the first time isolation of core adenine derivative **892**, were reported from a Taiwanese *Agelas nakamurai*.<sup>355</sup> Five stronglyphosphorine-class metabolites **893–897** came from a Solomon Is. collection of *Petrosia* sp. It is likely **893** and **894** are artefacts from **895** and **896**, respectively.<sup>356</sup> Linear (**898–904**) and cyclic (**905**) sesquiterpenoids have been reported from the *Aaptos* and *Cliona* genera, respectively.<sup>357,358</sup>



**872** R<sub>1</sub> = Br, R<sub>2</sub> = H  
**873** R<sub>1</sub> = H, R<sub>2</sub> = Br

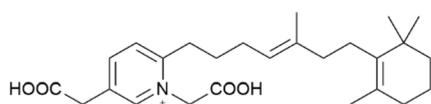


**874** R = H  
**875** R = CH<sub>2</sub>CH<sub>2</sub>COOH



**876**

The conscinoderines are a series of unusual pyridinium-containing terpenoid-based alkaloids. Conscinoderines A–J **906**, **907–915** were obtained from *Coscinoderma bakusi* collected at Fannuk Is., Chuuk (Federated States of Micronesia). The conscinoderines possess a rare 1,2,5-trisubstituted pyridinium motif. Alkaloids are very uncommon metabolites from this genus.<sup>359</sup>



**906**

Diterpenoids from sponges reported include kalihacyloxamides A–H **916–923** from *Acanthella cavernosa*,<sup>360</sup> and spongian-class metabolites **924** and **925** from *Spongia officinalis* and **926** and **927** from *Dendrilla* sp. respectively.<sup>361,362</sup> A series of di- and sesterterpenoids **928–935** were isolated from a Chinese *Sarcotragus* specimen collected from the South China Sea.<sup>363</sup> The suberitienones **936–944**, obtained from an Antarctic collection of *Suberites*, are sesterterpenoids with a new carbon skeleton.<sup>364</sup> A large number of scalarane-class metabolites were reported from *Lendenfeldia* (**945–948**), *Hyrtios* (**949–954**), and *Phyllospongia* (**955–974**) specimens, all of which were collected in the waters surrounding China and Taiwan.<sup>365–370</sup> Rhabdastrellosides A **975** and B **976** are new isomalabaricane triterpenoids from *Rhabdastrella globostellata*,<sup>371</sup> while a Papua New Guinean *Melophus sarasinorum* yielded seven new triterpenoids (**977–983**) although none were found to be bioactive.<sup>372</sup> Surprisingly, no steroids were reported from sponges in 2023.

The published structures of peptide solomonamide B and alkaloid 1-(1-*H*-indol-3-yloxy)propan-2-ol have been synthesised but are spectroscopically different from the NP suggesting that their structures should be revised.<sup>373,374</sup> Sponge NPs that have been synthesised for the first time are enigmazole B,<sup>375</sup> lissodendoric acid A,<sup>376</sup> which was also the subject of a review,<sup>377</sup> and njaoamine C which also established the absolute configuration **984** as shown.<sup>378</sup> Pyrroloiminoquinone NPs discorhabdin H, K and V and related compound aleutianamine have been synthesised by two independent groups.<sup>379–381</sup> Alkaloids (–)-chelolin A,<sup>382</sup> naamidine J,<sup>383</sup> nagelamide W,<sup>384</sup> longamide F, agelasines A and B, and nakamurine B,<sup>385</sup> respectively, have also been synthesised for the first time. Although the total synthesis of (–)-agelastatin A has already been achieved, a recent development of a flow-based photorearrangement to generate the central core has resulted in a scalable route to gram-level production of the compound with the use of only a single protecting group.<sup>386</sup> The total syntheses of merosesquiterpenoids dysiherbol B, D and E,<sup>387</sup> diterpenoids dysidealactams E and F, dysidealactone B,<sup>388</sup> mycaperoxide B, C, D, and G methyl ester,<sup>389</sup> and hamigerans C, I and debromo-I,<sup>390</sup> were also reported.

Notable sponge-related reviews include a summary of the development of the anti-proliferative polyketide plocabulin,<sup>391</sup> macrolide neopeltolide,<sup>392</sup> and the synthesis and bioactivity of faspaplysin and the aplysinopsins respectively.<sup>393,394</sup> Summaries of the cytotoxicity and anti-inflammatory activities of the nor-topsentins,<sup>395</sup> and of the general chemistry of dimeric pyrrole-imidazole alkaloids,<sup>396</sup> and of sponge sterol and triterpenoid glycosides,<sup>397</sup> have also been published. Taxa specific reviews include a focus on the sterols obtained from *Theonella* spp.,<sup>398</sup> the biosynthesis of compounds from the Theonellidae,<sup>399</sup> and of the MNPs obtained from *Acanthella* spp.<sup>400</sup> Case studies of the application of GNPS in studying Australian sponge chemistry have also been reviewed.<sup>401</sup>

Several reports of new biological activity for known sponge metabolites have been reported. Bis-indole dragmacidin D has been found to selectively induce apoptosis in aggressive triple



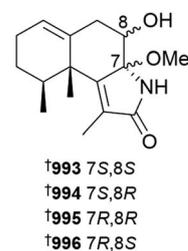
negative breast cancer spheroids, and can work synergistically with paclitaxel.<sup>402</sup> Curcuphenol, an aromatic marine sesquiterpenoid that is also commonly found in food spices, rescues immune recognition of metastatic cancers by restoring expression of antigen presentation machinery. This is achieved by eliciting histone deacetylase-enhancing activity, which causes changes resembling those caused by interferon- $\gamma$ , a cytokine that has an important role in regulating the innate and adaptive immune systems.<sup>403</sup> Fusion of a GFP-label to defensive steroid formoside has shown it distributes to the lips, tastebuds and olfactory epithelium in zebrafish as a model of fish predator-prey interactions, which helps to explain chemoreception in an ecological setting.<sup>404</sup>

A comparison of the metabolic and bioactivity profiles of two farmed and wild Mediterranean sponge species, *Agelas oroides* and *Sarcotragus foetidus*, has shown that both populations shared similar chemical profiles. The antibiotic activities of the sponge extracts were also generally similar, albeit slightly lower in the farmed sponges, while only *S. foetidus* extracts from both treatment groups were weakly cytotoxic.<sup>405</sup> A chemoecological study of Indonesian *Aaptosuberitoides* has explored the effects of the sponge microbiome upon the composition of aaptamine and other alkaloids. A wide variability of alkaloid concentrations across the sponges sampled showed no direct correlation with the presence of microbial symbionts, and no direct link for microbial biosynthesis of aaptamine could be found.<sup>406</sup>

An assessment of the combination of DFT-calculated NMR chemical shifts, as determined with several variants of the DP4+ algorithm combined with artificial neural network pattern recognition, along with a comparison of calculated and experimental chiroptical analyses, has confirmed the relative and absolute configurations of a marine endoperoxide as proposed using biogenic reasoning.<sup>407</sup> Several new poly-arsenic compounds, like arsenicins A–D, have been reported from sponges in the preceding years. However, DFT-methods, including study of the use of different functionals and basis sets for calculating their NMR chemical shifts for comparison with experimental data, has been lacking. A recent report describes a systematic examination of the use of two DFT methods, four functionals and five basis sets to establish the best approach for calculating <sup>1</sup>H and <sup>13</sup>C NMR chemical shifts and coupling constants within this growing class of metabolite, and validated by comparison with experimental data.<sup>408</sup> An evaluation of currently accessible, state of the art computational tools for assisting with structure elucidation has been carried out, in particular, focusing on their application by non-specialist users. The tools assessed included HOSE, CASCADE, DP4, DP4+ and ML-J-DP4. The study was exemplified by a computational examination of the compound dysiherbol A, the structure of which was recently reassigned following total synthesis, and where the erroneous structure proposed could have been flagged using the assistance of digital technologies prior to the synthetic campaign beginning. A pathway for structural confirmation prior to publication and embarking on a total synthesis was proposed.<sup>409</sup>

## 7 Cnidarians

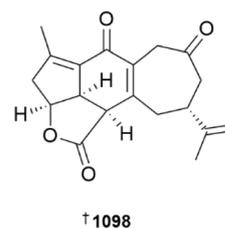
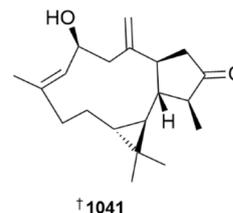
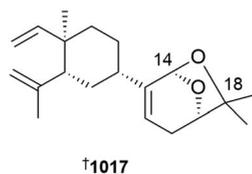
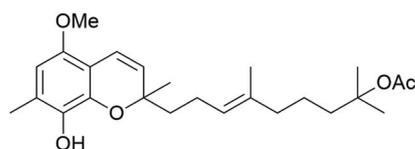
In addition to an anti-inflammatory cembranoid (discussed later), investigation of a Red Sea collection of *Sarcophyton glaucum* afforded  $\delta$ -lactone sarcoglaucanoate **985**.<sup>410</sup> As well as three known alkyl glycerol ethers, a new example 3-(*n*-henicosyloxy)propane-1,2-diol **986** was isolated from the soft coral *Nephthea mollis*, also collected in the Red Sea.<sup>411</sup> A large number of sesquiterpenoid and merosesquiterpenoid NPs were reported from soft corals. Two farnesane-type sesquiterpenoids, sinulalides A **987** and B **988** were isolated from extracts of the South China Sea soft coral *Sinularia scabra*.<sup>412</sup> The capnellene skeleton sesquiterpenoid **989** was isolated from an Orchid Is. Taiwan collection of *Capnella imbricata* and is somewhat unusual in possessing hydroxylation at C-15.<sup>413</sup> A guaiane sesquiterpenoid, litoarbolide A **990** was purified from a Red Sea collection of *Litophyton arboretum*<sup>414</sup> while a eudesmane sesquiterpenoid, cespilamide F **991** was isolated from a Taiwanese collection of *Cespitularia* sp.<sup>415</sup> – both MNPs were inactive when evaluated for anti-*P. falciparum* (the former), and cytotoxicity and anti-inflammatory (the latter) properties. Thirteen examples of nitrogen-containing nardosinane sesquiterpenoids, clavukoelloids A–M **992–1004** were isolated from a South China Sea collection of *Clavularia koellikeri*.<sup>416</sup> The stereochemical relationships between clavukoelloids B–E (**993–996**) were secured by CP3 analysis of calculated NMR chemical shifts, comparison of calculated and experimental ECD spectra and, in the cases of clavukoelloid B and E, by XRD analysis. The absolute configuration of clavukoelloid H was also secured by XRD analysis. In addition to several dolabellane diterpenoids (discussed later), a Taiwanese collection of *Clavularia* sp. afforded five eudensamane-type sesquiterpenoids, clasamanes A–E **1005–1009**, with clasamane E having a peroxide bridge.<sup>417</sup>



New congeners were added to the tuaimenal family of merosesquiterpenoids, with tuaimenals B–H **1010–1016** being isolated from deep-sea collections of the Irish soft coral *Duva florida*.<sup>418</sup> Tuaimenal G **1015** displayed selectively potent *in vitro* cytotoxicity towards a HPV-negative cervical human cancer cell line. Soft corals continue to be an excellent source of diterpenoids with over one hundred and sixty examples reported in 2023. Lobocatalens A–G **1017, 1018–1023** are lobane skeleton diterpenoids isolated from a Xisha Is. collection of *Lobophytum catalai*.<sup>419</sup> The structure of lobocatalen A contains an unusual ether linkage between C-14 and C-18. Fifteen diterpenoids, including lobane examples related to the known MNP fuscol, were identified in extracts of *Klyxum molle* and named xishaklyanes A–O **1024–1038**.<sup>420</sup> Xishaklyanes D and K exhibited



moderate activity towards the fish pathogenic bacteria *Lactococcus garvieae* and *Streptococcus parauberis*, respectively. Lactone diterpenoids sinulatones A **1039** and B **1040** were isolated from extracts of the South China Sea soft coral *Sinularia scabra*.<sup>412</sup> Absolute configurations were assigned by TDDFT calculations of ECD spectra. Sinulatone B was a weak inhibitor of osteoclastogenesis. *Sinularia nanolobata* specimens collected in the South China Sea were the source of diterpenoids nanolobatones A–E **1041**, **1042–1045** and nanolobaperoxides A–D **1046–1049**.<sup>421</sup> The structures of nanolobatones A and B and nanolobaperoxide A were secured by XRD analysis.



Seven examples of diterpenoids containing the rare flexibilane scaffold, paraflexinols A–G **1050–1056**, were reported from a Green Is., Taiwan, collection of *Paralemnalia thyrsoides*.<sup>422</sup> The authors speculated that acetoxy analogues **1053** and **1054** may be artefacts. Fourteen verticillane diterpenoids, heterolactone **1057** and heterolactams A–M **1058–1070** were isolated from the soft coral *Heteroxenia ghardaensis*.<sup>423</sup> A further example of a verticillane, cespitulactam M **1071** was identified in extracts of *Cespitularia* sp. collected at Green Is., Taiwan.<sup>415</sup> Targeted isolation using molecular networking analysis led to the identification of twelve dolabellane diterpenoids clavirolidides J–U **1072–1083** from extracts of *Clavularia viridis* collected at Yongxing Is., South China Sea.<sup>424</sup> Clavirolidide L exhibited weak inhibition of HIV-1 but it lacked the ability to inhibit reverse transcriptase. The authors considered clavirolidide O to be an artefact derived from EtOH solvent used in the extraction process. Xisha Is. collections of *C. viridis* afforded clavusins A–E **1084–1088**, with structures and absolute configuration of clavusins A and E being secured by XRD analysis,<sup>425</sup> while a Green Is. collection of *Clavularia* sp. yielded dolabellanes clabellanes A–C **1089–1091**.<sup>417</sup> New examples of capnosane diterpenoids, sarcocrassolins A–F **1092–1097**, were isolated from a Nansha Is. collection of *Sarcophyton crassocaule*, with the structure of sarcocrassolin B being secured by XRD.<sup>426</sup> Further investigation of the biological activities of the casbane-type diterpenoid sinueracabanone D have revealed it to be capable of inducing apoptosis in HepG2 cells *via* a mechanism that in-part involves enhanced generation of reactive oxygen species.<sup>427</sup> With over fifty new examples, cembranoids remain the dominant sub-structural class of diterpenoids reported from soft corals. The structures and biological activities of cembranoids isolated from marine, and terrestrial, organisms between 2011

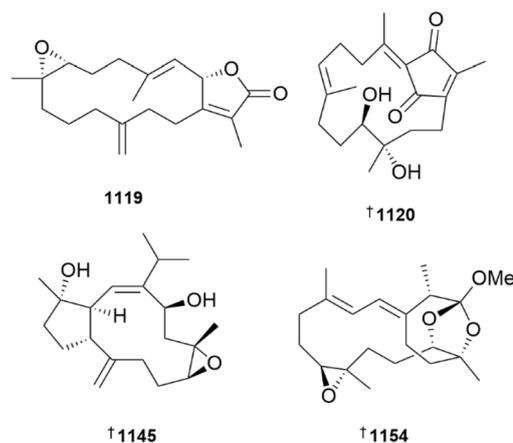
and 2022 have been reviewed.<sup>428</sup> The structures of previously reported norcembrane diterpenoids yonarolide and scabrolide B were revised and absolute configuration assigned to **1098** and **1099**, respectively, by XRD analysis.<sup>429</sup> It should be noted that the structure now assigned to scabrolide B is identical to that previously reported for sinuscalide D, isolated from *Sinularia scabra*.<sup>430</sup> A new synthetic route to (–)-scabrolide A was reported, with a subsequent dehydration step using Burgess reagent to afford (–)-yonarolide, providing further confirmation of structure and absolute configuration of the latter.<sup>431</sup>

Sinulaflexiolide Q **1100**, isolated from *Sinularia flexibilis*, was inactive in a *Bugula neritina* larvae settlement assay.<sup>432</sup> The structures and absolute configurations of *Sinularia*-sourced cembranoids sinupendunculide A **1101**,<sup>433</sup> sinulariaone A **1102** and previously reported chlorofurancembranoid B **1103** were established or confirmed by XRD analyses.<sup>434</sup> Of two related  $\alpha$ -methylene- $\epsilon$ -lactonic cembranoids, querciformolides G **1104** and H **1105**, isolated from *Sinularia querciformis*, only the former exhibited anti-inflammatory activity, weakly inhibiting the release of elastase from activated neutrophils.<sup>435</sup> The  $\alpha$ -methylene- $\delta$ -lactone flexibanone **1106**, isolated from a Taiwanese collection of *Sinularia flexibilis*, was inactive towards a panel of three HTCLs.<sup>436</sup> Additional examples of cembranoids isolated from soft corals of the genus *Sinularia* included **1107–1109**, along with casbane **1110**, from a South China Sea collection of *Sinularia nanolobata*,<sup>437</sup> the absolute configurations of which were determined using TDDFT calculations of ECD spectra, and situmulins A **1111** and B **1112** isolated from specimens of *Sinularia tumulosa*, also collected in the South China Sea.<sup>438</sup> Two  $\alpha$ -methylene- $\gamma$ -lactone-containing cembranoids, ximaolobophytolides A **1113** and B **1114** were reported from *Lobophytum* sp.<sup>439</sup> Despite the presence of electrophilic functionality, ximaolobophytolide A was inactive against the HEL tumour cell line *in vitro* while structurally-related known co-metabolites were cytotoxic. Soft corals of the genus *Sarcophyton* were a prolific source of additional examples of cembranoids including variants with unusual substitution patterns and some new examples of dimers. Cembranoids **1115–1118**, derived from an extract of *Sarcophyton trocheliophorum*, were found to be inactive against two bacterial strains



and influenza A virus H1N1.<sup>440</sup> The structurally-related sarcophine derivative **1119**, isolated from a Red Sea collection of *S. glaucum*, exhibited an interesting range of anti-oxidant and anti-inflammatory activities against indomethacin-induced gastric injury in rats.<sup>440</sup> A structurally-diverse set of cembranoid diterpenoids, sarcoelegans A–H **1120**, **1121–1130** were reported from a South China Sea collection of *Sarcophyton elegans*.<sup>441</sup> Sarcoelegans A–C, E and G were isolated as optically active MNPs while the remaining examples were isolated as racemates, subsequently being separated by chiral HPLC and absolute configuration was assigned to each of the enantiomers. The structures of sarcoelegans A, D, F and G were also secured by XRD analysis. Three cembranoids isolated from a Hainan collection of *Sarcophyton* sp., sarcophynoids A–C **1131–1133**, are variants on the simple cembran-tetraene (first) and sarcophytonolide (last two) scaffolds.<sup>442</sup> All three were inactive against a panel of three bacterial strains. Four new examples of isosarcophytoxides **1134–1137**, notable for containing a 2,5-dihydrofuran moiety, were reported from a Xiao Liuqiu Is. collection of *S. cinereum*.<sup>443</sup> Despite MeOH being used in the isolation of the metabolites, the authors did not discuss the possibility that the methoxy-containing examples were artefactual. New examples of unsaturated-lactone containing cembranoids, isoehrenbergol D **1138** and sarcoehrenolides F–L **1139–1144**, were isolated from a Weizhou Is. collection of *S. ehrenbergi*.<sup>444</sup> None of the MNPs were able to inhibit the production of TNF- $\alpha$  in a cell-based anti-inflammatory assay. While sartrocheliol A **1145** is a capnosane-type diterpenoid, the remaining diterpenoids isolated from a Ximao Is. collection of *S. trocheliophorum*, sartrocheliols B–F **1146–1150**, were cembranoids.<sup>445</sup> The structure and absolute configuration of sartrocheliol A were secured by XRD analysis. All of the MNPs were deemed inactive when evaluated against a panel of HTCLs and microorganisms. Amongst the cembranoids, sarcomililalol H **1151** isolated from a Xigu Is., South China Sea collection of *S. mililatisensis*<sup>446</sup> and two further examples, sarcoboettgerols D **1152** and E **1153**, isolated from a Weizhou Is. collection of *S. boettgeri*.<sup>447</sup> Sarcomililalol H contains a rarely encountered C-2 to C-12 ether linkage, while sarco-boettgerol E has an ether linkage between C-17 and C-12. A diverse set of terpenoids and bis-terpenoids were isolated from *S. tortuosum*, collected from Ximao Is., South China Sea.<sup>448</sup> Of the first two, sarcotortin A **1154** is a cembranoid that contains an unusual orthoester moiety, while sarcotorolide A **1155** contains a ring closure between C-2 and C-11 of the cembranoid framework, making it a eunicellane-type scaffold. The structures and absolute configurations of both metabolites were secured by XRD analysis. The structure of co-metabolite sarcostolide G was revised to the C-2 epimer **1156** also by the analysis of XRD data. In addition, biscembranoids, ximaolides M **1157** and N **1158** were isolated from the soft coral – the structure and absolute configuration of the former was again secured by XRD analysis. Of all the isolated compounds, only ximaolide M exhibited bioactivity, being found to be a weak inhibitor of PTP1B. Two additional examples of biscembranoids, sarcotroxides A **1159** and B **1160** were isolated from

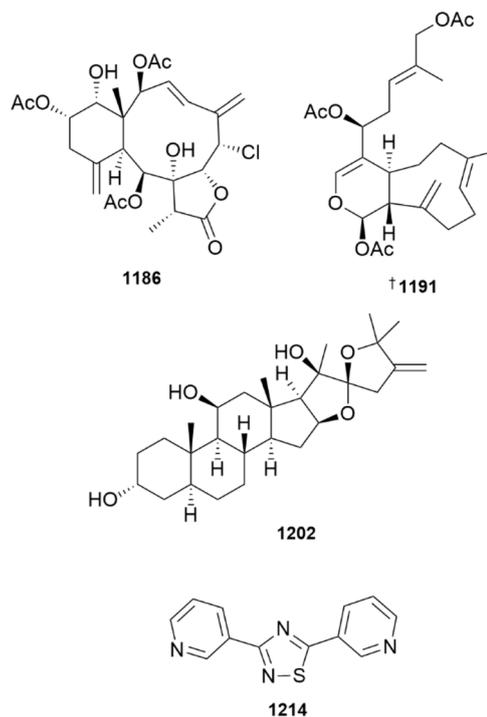
aquaculture-derived *S. trocheliophorum* – the latter MNP was found to be a weak inhibitor of superoxide generation by stimulated neurophils.<sup>449</sup>



New biological activities were reported for cnidarian-derived cembranoids including the finding that 11-*epi*-sinulariolide acetate can induce apoptosis in oral cancer cells via the PI3K/AKT/FOXO pathway,<sup>450</sup> that sinularin induces, amongst other things, ferroptosis, which is apoptosis induced in an iron-dependent mechanism,<sup>451</sup> and that crassolide can stimulate anticancer immune responses in part by blocking mitogen-activated protein kinase 14 activation.<sup>452</sup> In two publications, new congeners of the briavioid family of briarane diterpenoids, briavioids D–G **1161–1164** were reported from specimens of *Briareum violaceum* grown in aquaculture.<sup>453,454</sup> The latter study also reported an XRD structure of briavioid A **1165**, defining its absolute configuration for the first time. Briarlide S **1166** was isolated from an Okinawa Prefecture collection of *Pachyclavularia (Briareum) violacea*.<sup>455</sup> *Briareum stechei* was the source of three new briaranes, 12-*epi*-briacavatolide B **1167**, and briastecholides M **1168** and N **1169**, with the structure and absolute configuration of the last being secured by XRD analysis.<sup>456–458</sup> Of 45 briarane diterpenoids isolated from a South China Sea collection of *Junceella juncea*, 16 new analogues, juncelactones A–P **1170–1185** were characterised.<sup>459</sup> Juncelactone K exhibited weak ability to inhibit RANKL-stimulated osteoclastogenesis activity. Comprehensive biological evaluation was undertaken on the major (known) MNP in the extract, praelolide, identifying the briarane to be a potential new lead in the treatment of osteoclastogenic bone disease. Investigation of an extract of a Southern Taiwan collection of *J. fragilis* afforded a series of briaranes of which one, fragilide Y **1186**, was new.<sup>460</sup> The structure of fragilide Y contains the 3(*E*),5(16)-diene system in a *s-cis* form, the determination of which was aided by XRD analysis of two previously reported co-metabolites (–)-frajunolide H **1187** and fragilide P **1188**. These structures correct those originally reported for the diterpenoids, both of which were depicted with a *s-trans* diene. Similar changes from *s-trans* to *s-cis* were proposed for the structures of juncelolides B **1189** and C **1190**. In a major achievement, the first syntheses of the xenicin



class diterpenoids (+)-waixenicin A **1191** and (+)-9-deacetoxy-14,15-deepoxyxeniculin **1192** have been reported.<sup>461</sup> This is particularly noteworthy as the former MNP is a potent irreversible inhibitor of transient receptor potential melastatin 7 (TRPM7) channels, with potential applications in neurodegenerative disorders, cardiovascular disease and cancer chemotherapies. The authors also noted that reaction of **1192** with mild base ( $K_2CO_3$  in MeOH) induced a rearrangement to another known xenicin NP, xeniafaraunol A **1193**. In an extension to their previous work identifying and characterising the BGCs associated with cnidarian terpenoid biosynthesis, the Schmidt lab have reported that expressing the relevant terpene cyclase in the yeast *Saccharomyces cerevisiae* can yield fermentation titres of close to  $100\text{ mg L}^{-1}$  for the production of (–)-klysimplexin R.<sup>462</sup> Subsequent semi-synthetic transformations allowed for rapid preparation of analogues belonging to several different structural classes. The biosynthesis and enzymology associated with eunicellane biosynthesis has been reviewed.<sup>463</sup> Specimens of the soft coral *Sinularia depressa* collected at Xisha Is. in the South China Sea were the source of three ergosterol and one cholesterol analogue, sinulasterols D–G **1194–1197**,<sup>464</sup> while 7-hydroperoxides of gorgosterol **1198**, **1199** and campesterol **1200** were isolated from a Taiwanese collection of *Cespitularia* sp.<sup>415</sup> None of the MNPs exhibited bioactivity in a range of HTCL cytotoxicity, antibacterial and anti-inflammatory assays. The absolute configuration of the ring-A-aromatised bile acid analogue **1201**, isolated from an Irish deep-sea collection of *Duva florida*, was established by XRD analysis.<sup>418</sup> Two spiroketal steroids, 24-dehydrohippuristanol **1202** and hippuristanol 11-one **1203**, the latter being first reported as a NP, were isolated from an Orchid Is., Taiwan collection of *Isis hippuris*.<sup>465</sup> The former MNP exhibited weak cytotoxicity towards 2 HTCLs. Three steroids, one being a 7-one **1204** and two being epimeric 7-hydroperoxides **1205** and **1206** were isolated from a Xisha Is., South China Sea collection of *Lobophytum sarcophytoides*.<sup>466</sup> The steroidal 3,23-diketone dendronestadione **1207**, derived from an extract of a Red Sea *Dendronephthya* sp., exhibited weak cytotoxicity towards the prostate PC-3 cell line.<sup>467</sup> Enone and dienone steroids lobosteroids A–E **1208–1212** and  $5\alpha,8\alpha$ -epidioxy-containing lobosteroid F **1213** exhibited weak to moderate activities towards three species of pathogenic fish bacteria.<sup>468</sup> A single isomer of a previously reported cnidarian-derived sterol, 25(R)-26-acetoxy-3 $\beta$ ,5 $\alpha$ -dihydroxycholest-6-one, has been synthesised starting from diosgenin.<sup>469</sup> The semi-synthetic compound was inactive against respiratory syncytial virus, but did show weak cytotoxicity towards two HTCLs. The therapeutic potential of sea anemone neurotoxins and pore forming peptides has been reviewed.<sup>470</sup> Investigation of ethanolic extracts of the invasive anemone *Condylactis* sp. led to the characterisation of a diverse range of small molecules including bispyridinyl-1,2,4-thiadiazole **1214**, a previously reported synthetic compound, thionicotinamide **1215** and (–)-betonicine **1216**, a known higher plant NP.<sup>471</sup>



A number of reviews were published that are relevant to the chemistry and biology of metabolites derived from cnidarians. Genus or species specific reviews summarised the chemistry of the *Cladiella* genus, covering the period 2006 to August 2022,<sup>472</sup> the genus *Litophyton* covering from the 1970s until July 2023,<sup>473</sup> and the structural diversity of terpenoids reported from *Sarcophyton trocheliophorum*, covering the period 1976 to October 2022.<sup>474</sup> In addition to these, two reviews focused on biological activities, with one summarising neutrophilic anti-inflammatory NPs isolated from cnidarians covering the period 1995 to April 2023,<sup>475</sup> and the other covering NPs with enzyme inhibiting properties published between 1974 and 2022.<sup>476</sup>

While no new MNPs isolated from zoanthids and hard corals were reported, further study of zoanthid-derived NPs identified that extremely low doses (low pM) of palytoxin exhibit anti-leukaemic activity in cell-based and zebrafish xenograft assays,<sup>477</sup> and that *epi*-oxyzoanthamine, sourced from *Zoanthus vietnamensis*, reduced skin cell damage and hyperplasia, suggesting it has potential in the treatment of atopic dermatitis.<sup>478</sup> Molecular networking has been used to analyse the chemical relationships between four species of hard corals, *Pocillopora meandrina*, *Seriatopora hystrix*, *Acropora formosa* and *Fungia fungites*, collected in the South China Sea.<sup>479</sup> The study revealed notable differences in amino acid, peptide and lipid profiles that were discernible using principal component analysis. Variation of tissue lipid composition of seven species of *Porites* hard corals with changes in temperature and  $pCO_2$  concentrations identified, amongst other changes, increases in free fatty acids and campesterol with increasing temperature while seawater  $pCO_2$  concentrations generally had no significant effect.<sup>480</sup> Two reviews summarised the extensive lipid chemistry of reef-building corals and their dinoflagellate symbionts.<sup>481,482</sup>



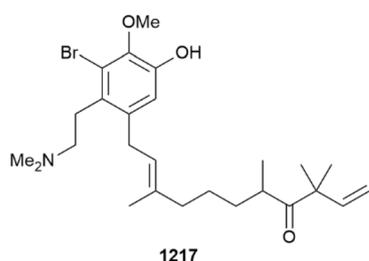
The hydrozoan-derived indole-oxazole alkaloids, breitfussin G and H, have been synthesised for the first time, using a convergent, one-pot Friedel–Crafts/Robinson–Gabriel method.<sup>483</sup>

## 8 Bryozoans

No new metabolites were reported from bryozoans over the past year but two syntheses of bryozoan metabolites were accomplished. Total synthesis of the tetrabrominated alkaloid aspidostomide G was achieved in five steps and an overall yield of 16%,<sup>484</sup> and trichlorinated alkaloid caulamidine A was prepared in 11 steps.<sup>485</sup> The potential of bryostatin-1 for treatment of neurological disorders was reviewed.<sup>486</sup>

## 9 Molluscs

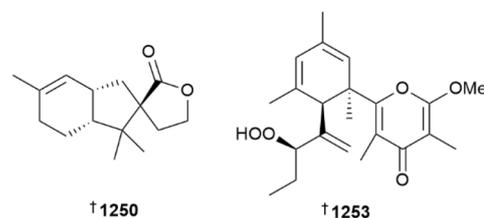
A comprehensive review of the chemical structures, chemical ecology and pharmaceutical potential of MNPs derived from marine molluscs, covering the period 2011–2021, has been published.<sup>487</sup> MS-directed investigation of bioactive extracts of the sea hare *Aplysia kurodai* collected from the Shima Peninsula, Japan, afforded new members **1217**, **1218–1222** of the aplaminone family of dopamine-terpenoid hybrids.<sup>488</sup> Cytotoxicity towards the HCT 116 HTCL ranged from inactive to moderate in potency. Amongst the set, isoplamminone **1217** is structurally unusual in that it contains a reverse prenyl substituent. LCMS analysis of alga species collected in the vicinity of the sea hare collection site identified the presence of known aplaminones A and B in the red alga *Palisada intermedia*, and that the levels of the molecules varied with location, leading the authors to speculate that the MNPs were produced by microbes.



Investigation of Vietnamese collections of *Aplysia dactylo-mela* afforded three nor-chamigrane and bisabolane sesquiterpenoids, dactylomelanins C–E **1223–1225**.<sup>489</sup> The absolute configuration of a co-metabolite, 2-chloro-3,7-epoxychamigran-9-one **1226**, previously reported from the red alga *Laurencia obtusa*, was secured by TDDFT ECD calculations.

A new example of an irieane diterpenoid, 12-hydroxypinnaterpene C **1227**, was isolated from specimens of *Aplysia argus* collected at Ikei Is. in the Okinawa Prefecture.<sup>490</sup> Syntheses of the sea hare metabolites aplysiasecosterols A and B, the latter for the first time, have been reported, using a late-stage convergent strategy.<sup>491</sup> In two separate accounts, new metabolites have been reported from South China Sea collections of the nudibranch *Hexabranchnus sanguineus*. In the first of these, nine

diterpenoids, sanyanolides A–I **1228–1236** were purified from an extract of the nudibranch's internal organs.<sup>492</sup> The majority of these metabolites were also identified in extracts of the sponge *Chelonaplysilla* sp. collected in the immediate area. Antimicrobial testing against a panel of eight human pathogens identified sanyanolide E as being weakly active against two species of *Streptococcus*, while none were found to inhibit NO production by LPS stimulated macrophages. Further investigation of extracts of the same organism from the same location afforded thirteen sesquiterpenoids sanyagunins A–H **1237–1244**, sanyalides A–C **1245–1247** and sanyalactams A **1248** and B **1249**.<sup>493</sup> The absolute configuration of known MNP herbabysidolide **1250** was secured by XRD. Sodium borohydride reduction of sanyagunin C gave two diastereomeric allylic alcohols which were identical to two previously reported MNPs. Mosher's derivatisation led to determination of the secondary alcohol configuration in **1251** and **1252**, requiring reversal of the original assignments. A number of  $\gamma$ -pyrone polypropionates were reported from the photosynthetic mollusc *Placobranchus ocellatus*.<sup>494</sup> Ocellatuspyrones A **1253** and B **1254** were both isolated as racemic mixtures; the former was resolved using chiral HPLC and the relative and absolute configuration assigned using DP4+ analysis and Mosher's derivatisation.



Ocellatuspyrone C **1255** shares close structural similarity to the known polypropionate co-metabolite tridachiapyrone J **1256**. The absolute configuration of tridachiapyrone J was established using XRD analysis, which allowed determination of that of ocellatuspyrone C. Reduction of the hydroperoxide moiety in tridachiapyrone J afforded co-metabolite tridachiapyrone G **1257**, securing its absolute configuration as well as that of the diastereomer tridachiapyrone H **1258**. Four new structurally simpler polypropionates were also characterised from the extract, ocellatuspyrones D–G **1259–1262**, with relative and absolute configurations assigned by combinations of DP4+ and ECD analysis. Three ceramides, bathymodiolamides C–E **1263–1265** were isolated from extracts of the mussel *Bathymodiolus azoricus* collected near deep-sea hydrothermal vents.<sup>495</sup> An unusual aspect of their structures is the presence of a methoxy ethylene ether substituent. The authors revealed that the structures they previously reported for congeners bathymodiolamides A and B were incorrectly drawn in their original publication<sup>496</sup> and should be represented as **1266** and **1267**, respectively. It is disappointing that such a simple error in structure drawing, compounded by a factual mistake regarding a stereochemical model, were not recognised by the authors, nor was it caught during the peer review process. Unfortunately, those errors led to a subsequent total synthesis study to target



an incorrect diastereomer of bathymodiolamides A and B which, unsurprisingly, exhibited NMR data that did not match those reported for the isolated NPs.<sup>497</sup>

The presence of the polyunsaturated fatty acid docosahexaenoic acid in marine bivalves appears to play a role in increasing the levels of esterification of diarrhetic shellfish toxins and activating the Nrf2 signalling pathway, leading to a reduction in damage to the digestive glands by the toxins.<sup>498</sup> A seven-year study of spirolide toxins in bivalve molluscs of the Galicia Coast (NW Spain) has identified the mussel *Mytilus galloprovincialis* to be the major accumulator of 13-desmethyl spirolide C (13-desmSPXC), while the presence of an isomer, likely an epimer based upon high similarity of MS/MS fragmentation patterns, was restricted to cockles and two clam species.<sup>499</sup> The highest levels of 13-desmSPXC occurred during the autumn–winter months while the isomer had peak levels in spring–summer. A second, 21 months study, also undertaken on the Galicia Coast, identified the presence of multiple lipophilic toxins in non-traditional vectors, including 13-desmSPXC and pinnatoxin G in cephalopods and ascidians, and 13-desmSPXC, OA and dinophysistoxin 2 in polychaete worms.<sup>500</sup> Overall it was concluded that the mussel *Mytilus galloprovincialis* could be used as a biological indicator for toxicity. Mice treated with sublethal concentrations of extracts of the cockle *Acanthocardia tuberculatum*, known to harbour saxitoxins, found that the toxins induced oxidative stress and affected energy metabolism, but with limited to no impairment of renal function.<sup>501</sup>

Racemic synthetic *N*-(2-ozoazepan-3-yl)-pyrrolidine-2-carboxamide, previously reported from *Octopus vulgaris* ink, was evaluated for a range of antiproliferative and anti-inflammatory activities but the responses did not reach a threshold for inclusion in this review.<sup>502</sup> Two steroidal glycosides **1268** and **1269** were reported as weakly anti-inflammatory constituents of the octopus *Cistopus indicus*.<sup>503</sup> The biological activities of 1-*O*-alkylglycerol ethers, reported from a number of different marine organisms including cartilaginous fish, cnidarians and molluscs, which can include reduction of oxidative stress, and stimulation of hematopoiesis and immune responses have been reviewed.<sup>504</sup> Dietary supplements containing crude preparations of 1-*O*-alkylglycerol ethers derived from the squid *Berryteuthis magister*, when given to obese patients with asthma, afforded improved pulmonary function and reduction in plasma levels of several inflammatory cytokines and oxylipins.<sup>505</sup> Several reviews pertaining to cone snail toxins have been published, including a bibliometric summary of toxins published from 2000 to 2022,<sup>506</sup> a perspective on those toxins that target voltage-gated sodium channels,<sup>507</sup> and an update of the *Conus*-derived toxins reported from snails collected off the coast of Brazil.<sup>508</sup> Ten cysteine-free conopeptides, based upon transcriptomes previously determined for the vermivorous snail *Conus betulinus*, were synthesised and evaluated for insecticidal activity against mealworm (*Tenebrio molitor*) larvae leading to the identification of several analogues with nanomolar potency.<sup>509</sup> Two studies reported investigation of

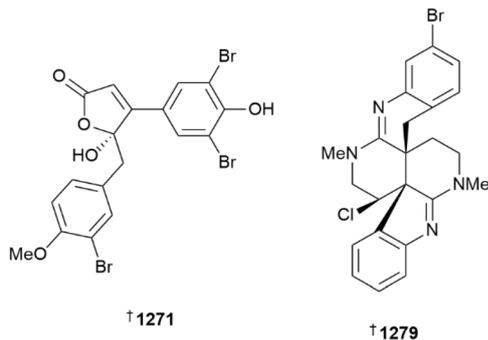
structure–activity relationships of conotoxins. In the first, a series of alanine insertion and truncated loop 2 mutants of  $\alpha$ 4/6-conotoxin TXID were prepared and demonstrated similar or weaker inhibition of  $\alpha$ 3 $\beta$ 4 nACh receptors than the parent toxin.<sup>510</sup> In the second study, it was found that swapping the position 11 L-arginine for D-arginine in  $\alpha$ -conotoxin RgIA led to reduced ability to block  $\alpha$ 9 $\alpha$ 10 nAChR activity, but instead led to introduction of the ability to block  $\alpha$ 7 nAChR activity.<sup>511</sup>

The solution structure of mono-disulfide-containing *Conus monile*-derived peptide Mo1853 has been investigated using 2D NMR spectroscopy, identifying the presence *cis* and *trans* Lys-Pro conformers.<sup>512</sup>

## 10 Tunicates (ascidians)

In addition to summaries of the biological activities reported for ascidian MNPs, the clinical status of ascidian-derived therapeutics has been reviewed.<sup>513</sup> Halorotetin A **1270** is a terpenoid isolated from the tunic of the edible ascidian *Halocynthia rotetzi*.<sup>514</sup> Although the authors reported many biological assay results for the compound, including anti-proliferative and gene expression profiles, the potency of activity did not reach the threshold used by this review. Bioassay-directed fractionation of extracts of Australian collections of the ascidian *Polycarpa procera* led to isolation of the butenolide procerolide E **1271**, methylprocerolate A **1272** and 3-bromo-4-methoxyphenylacetamide **1273**, the latter being reported as a NP for the first time.<sup>515</sup> In addition to a set of known related MNPs also isolated from the ascidian, all three new metabolites exhibited the ability to bind to  $\alpha$ -synuclein in an affinity mass spectrometry assay. Follow up testing in a biochemical amyloid aggregation assay showed the former two compounds to be inhibitors. Five examples of hexacyclic alkaloids, isocaulamidines B–D **1274–1276** and caulamidines C **1277** and D **1278** were reported from a Palau collection of *Polyandrocarpa* sp.<sup>516</sup> All five MNPs shared similar ECD spectra to the previously reported co-metabolite caulamidine B, and so were assigned absolute configurations corresponding to that of caulamidine B, which had been determined using TDDFT calculations. Within months, enantioselective syntheses of (–)-caulamidine D **1279** and (–)-isocaulamidine D **1280** were reported, leading to reassignment of absolute configuration of the isolated NPs to their respective enantiomers.<sup>517</sup> Two studies reported new biological activities for lepadin alkaloids, including the finding that lepadin A activates the intrinsic apoptosis pathway in human melanoma cells leading to induction of immunogenic cell death,<sup>518</sup> and that lepadins E and H can induce an iron-dependent accumulation of phospholipid peroxides that lead to mitochondrial shrinkage, cell membrane perforation and cell death, a process known as ferroptosis.<sup>519</sup> Lepadin H exhibited *in vivo* activity against B16F10 melanoma cells with limited to no toxicity towards internal organs.





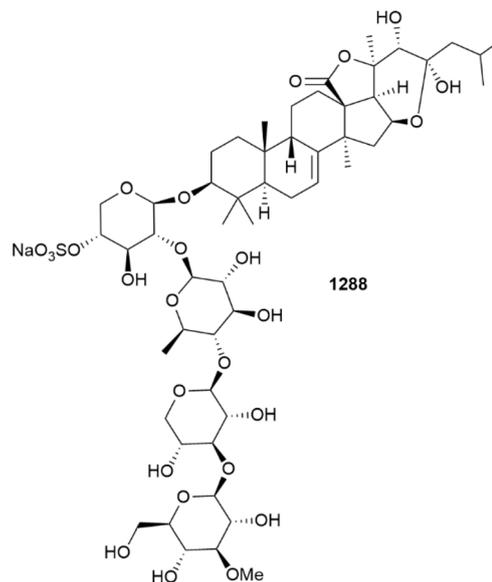
Two analogues of siladenoserinol A, that explored variation in substituent configuration about the dioxabicyclooctane central skeleton, were less active in their abilities to inhibit p53-Hdm2 interaction than the NP.<sup>520</sup> In addition to the first reported synthesis of meridianin B, a library of analogues were synthesised and evaluated for antibiofilm activity against *Acinetobacter baumannii*.<sup>521</sup> None were found to be active, but in additional testing, several analogues demonstrated the ability to synergistically improve the antibiotic activity of gentamicin and ceftriaxone. Analogues of the diarylpyrrole ascidian alkaloid lukianol A were prepared and screened for human aldose reductase inhibition properties, identifying two analogues with comparable activity to the NP.<sup>522</sup> The study noted the importance of at least two of the phenolic hydroxyl groups for bioactivity. A series of pyrazole-containing, lamellarin O-type analogues were prepared and found to exhibit variable levels of cytotoxicity, ranging from inactive to weak, against a panel of three HTCLs.<sup>523</sup> Closer analysis using HCT116 cells showed two of the compounds to promote G2/M-phase cell cycle arrest. *N*-9-Alkylated analogues of the  $\beta$ -carboline eudistomin Y are at best weakly cytotoxic towards a panel of five HTCLs.<sup>524</sup> The presence of electron-rich groups in the *N*-9 substituent restored fluorescence, enabling subcellular localisation studies that identified lysosomes as the location of accumulation.

## 11 Echinoderms

Pentaoside protonodososide **1281** was isolated from a Vietnamese collection of the starfish *Protoreaster nodosus*.<sup>525</sup> It was found to be inactive towards a panel of five HTCLs. A study exploring the saponin and fatty acid profiles of body wall extracts of the sea cucumber *Holothuria atra* led to the characterisation of the C-12 epimer of desholothurin B (desulfated holothurin B) **1282**.<sup>526</sup> Far Eastern specimens of the sea cucumber *Paracaudina chilensis* afforded chilensosides E–G **1283–1285**, all of which were inactive towards a panel of HTCLs and were also non-haemolytic.<sup>527</sup> In two studies, new triterpenoid glycosides djakonoviosides A, A<sub>1</sub> and A<sub>2</sub> **1286–1288**, B<sub>1</sub>–B<sub>4</sub> **1289–1292**, C<sub>1</sub>, D<sub>1</sub>, E<sub>1</sub> and F<sub>1</sub> **1293–1296** were reported from extracts of the Far Eastern sea cucumber *Cucumaria djakonovi*.<sup>528,529</sup> Three of the glycosides, djakonoviosides A<sub>2</sub> (**1288**), B<sub>2</sub> and B<sub>4</sub> contained an unusual 23,16-hemiketal linkage.

Palmitic acid, isolated from *Holothuria leucospilota*, is active in *Caenorhabditis elegans* models of Parkinson's disease,

improving locomotion, extending the lifespan and decreasing  $\alpha$ -synuclein aggregation.<sup>530</sup>



As with previous years, a number of studies have reported additional biological activities for echinochrome A including prevention of heart failure after myocardial infarction in mice,<sup>531</sup> preventing diabetic nephropathy,<sup>532</sup> induced inhibition of Ca<sup>2+</sup>-permeable cation channels,<sup>533</sup> and ability to prevent asthma in a mouse model *via* inhibition of inflammation and oxidative stress.<sup>534</sup> Phase II metabolic profiling of echinochrome A using rat and human hepatic preparation identified mono-methylated and mono-glucuronide conjugates as the principle phase II metabolites.<sup>535</sup> Further biological activities reported for known echinoderm MNPs include the finding that asterosaponin P1 causes significant toxicity and embryonic effects towards zebrafish embryos,<sup>536</sup> and that holothurin A inhibits the epithelial-mesenchymal transition that drives apoptosis in prostate cancer cells.<sup>537</sup> Two studies were reported using the saponin frondoside A whereby a detailed study of HTCL cytotoxicity and differential gene expression analysis provided further evidence that frondoside A regulates multiple pathways in tumour cells,<sup>538</sup> and that an immunomodulator can synergistically potentiate the antiproliferative and anti-migration properties of frondoside A against human bladder cancer cells and that the combination was significantly active in an *in vivo* xenograft model.<sup>539</sup>

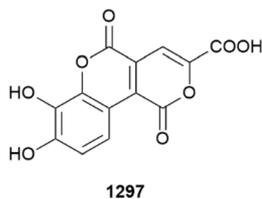
Several reviews have been published covering specific aspects of the chemistry of echinoderms, including a commentary on saponins derived from North Sea holothurians,<sup>540</sup> an *in silico* analysis of cytotoxic sea cucumber compounds,<sup>541</sup> fatty acids from sea cucumbers,<sup>542</sup> and a discussion of the synthesis and structure elucidation of dimeric hydroxynaphthazarins.<sup>543</sup>

## 12 Miscellaneous

In an excellent exposition of NP research, the structure elucidation of an ellagic acid derivative, lumnitzeralactone **1297** isolated from root extracts of the mangrove *Lumnitzera*



*racemosa*, was achieved using combinations of NMR, including 1,1-ADEQUATE and 1,*n*-ADEQUATE data, computer-assisted structure elucidation, and DFT calculated NMR chemical shifts with DP4+ analysis.<sup>544</sup> Synthesis was achieved by photo-oxidation of ellagic acid to an intermediate that was then subjected to thermal decarboxylation to afford the NP. Lumnitzeralactone was only detected in two of thirty-one *Lumnitzera* sp. samples, leading the authors to speculate that the NP is biosynthesised by associated microorganisms.



Previous studies have identified that Chinese medicinal use of Hai-Long, the fish *Syngnathus acus*, is associated with anti-tumour activity. Investigation of extracts afforded two glycerolipids, syngaculipids A **1298** and B **1299** as well as triallyl isocyanurate **1300**, with the last being reported as a NP for the first time.<sup>545</sup> The use of marine annelids to investigate the mechanism of action of toxic microalgae on marine invertebrates has been reviewed.<sup>546</sup> A racemic synthesis of a thienothiochromene-based luciferin of the polychaete worm *Odontosyllis undecimdongta* has been reported.<sup>547</sup> Tetrodotoxin and congeners are present in larvae of the toxic ribbon worm *Cephalothrix cf. simula*, with developmental time course analysis suggesting the larvae were not capable of independent production and that the toxins were obtained from the maternal organism.<sup>548</sup> A study involving the comparative amino acid sequence, 3D structure analysis and biological evaluation of BRICHOS-domain antimicrobial peptides isolated from three polychaete worm species, including known peptides alvinellacin from *Alvinella pompejana* (deep-sea, hydrothermal) and arenicin from *Arenicola marina* (temperate, coastal) and a new peptide polaricin from *Amphitritides* sp. (polar, coastal), concluded that production of the specific peptides was the result of environmental pressures to afford antimicrobial agents that would function under specific conditions and against specific microbial targets.<sup>549</sup> Temporal analysis of the fish *Lagocephalus sceleratus* collected in Antalya Bay, Mediterranean Sea, has identified the gonads to contain the highest levels of tetrodotoxin with levels peaking in the late autumn to winter (November–December) months, identifying this invasive pufferfish as being a public health risk.<sup>550</sup> A similar study of the same fish species, covering a two-month period using specimens collected from Rhodes Is., Greece, identified a wider array of tetrodotoxin analogues, with liver and gonad tissues being the most toxic and with the highest concentrations being observed in ovaries of female fish.<sup>551</sup> One of the lethal symptoms of tetrodotoxin poisoning is severe hypotension, which has now been attributed to the ability of the toxin to block voltage-gated sodium channels in resistance arteries leading to a decrease in vascular tone.<sup>552</sup> The ability of fish intestinal gut derived gangliosides to bind bacteria of the genus *Vibrio* has been

reviewed.<sup>553</sup> The linear antimicrobial peptide epinecidin-1, originally isolated from the orange-spotted grouper *Epinephelus coioides*, has been found to have good characteristics as a preservative for raw milk, preventing spoiling and reducing the incidence of milk-borne pathogens.<sup>554</sup> Phlorizin, a known dihydrochalcone glycoside isolated from the seagrass *Syringodium isoetifolium*, induces apoptosis in HepG2 cells, and exhibits *in vivo* activity against diethylnitrosamine + CCl<sub>4</sub> induced hepatocellular carcinoma.<sup>555</sup> Using Mediterranean collections of *Nanozostera noltei*, phenolic metabolites, including rosmarinic acid and zosteranoic acid appear to be useful markers of sea grass health.<sup>556</sup>

## 13 Conclusion

The decline in new prokaryote MNP discoveries in recent years may reflect the challenges involved in finding uniquely marine microbes since easily accessible prokaryotes obtained from marine environments produce chemical diversity typical of terrestrial sourced strains. This section summarises the current knowledge.

In the mid 1980s MNP researchers began to speculate that microorganisms were likely to play a key role in the production of some NPs isolated from marine invertebrates. The similarity in structures of some marine invertebrate NPs to those reported from terrestrial bacteria and marine and freshwater cyanobacteria was the basis for this hypothesis.<sup>557</sup> The isolation of diketopiperazines from both the sponge *Tedania ignis* and cultures of the actinomycete bacteria *Micrococcus* sp. isolated from the sponge in 1988 provided the first example of MNPs ascribed to the sponge being produced by an associated bacterium.<sup>558</sup>

The seminal work of John Faulkner and others in the early 1990s highlighted that, through cell separation techniques, some of the complex polyketide and peptidic NPs reported from the sponges *Theonella swinhoei* and *Dysidea herbacea* were co-located in bacterial cell fractions and this provided circumstantial evidence for their bacterial origins.<sup>559</sup> These findings intersected with the growing realisation that sustainable production of some biomedically important and complex invertebrate-derived MNPs such as ET-743, bryostatins and didemnin B would require a different approach to sourcing the compounds by wild invertebrate harvest. The promise of fermentation of microorganisms, speculated to be the true source of these compounds, resulted in growing interest in marine microorganisms for MNP discovery. Cultivation of marine bacteria led in part by the groundbreaking discoveries and methodological advancements of Fenical and Jensen at Scripps Institution of Oceanography in the early 1990s, and the impactful “Natural Products as Sources of New Drugs over the Last 25 Years” by Newman and Cragg in 2007 that urged a significant expansion in NP research focused on marine microbes, resulted in a rapid increase in interest in marine bacteria as a new source of MNP chemical diversity (Fig. 2).<sup>559–561</sup>

Over the last 20 years an exponential increase in studies aimed at discovering new MNPs from marine microbes rather than from marine macro-organisms has occurred. In contrast,



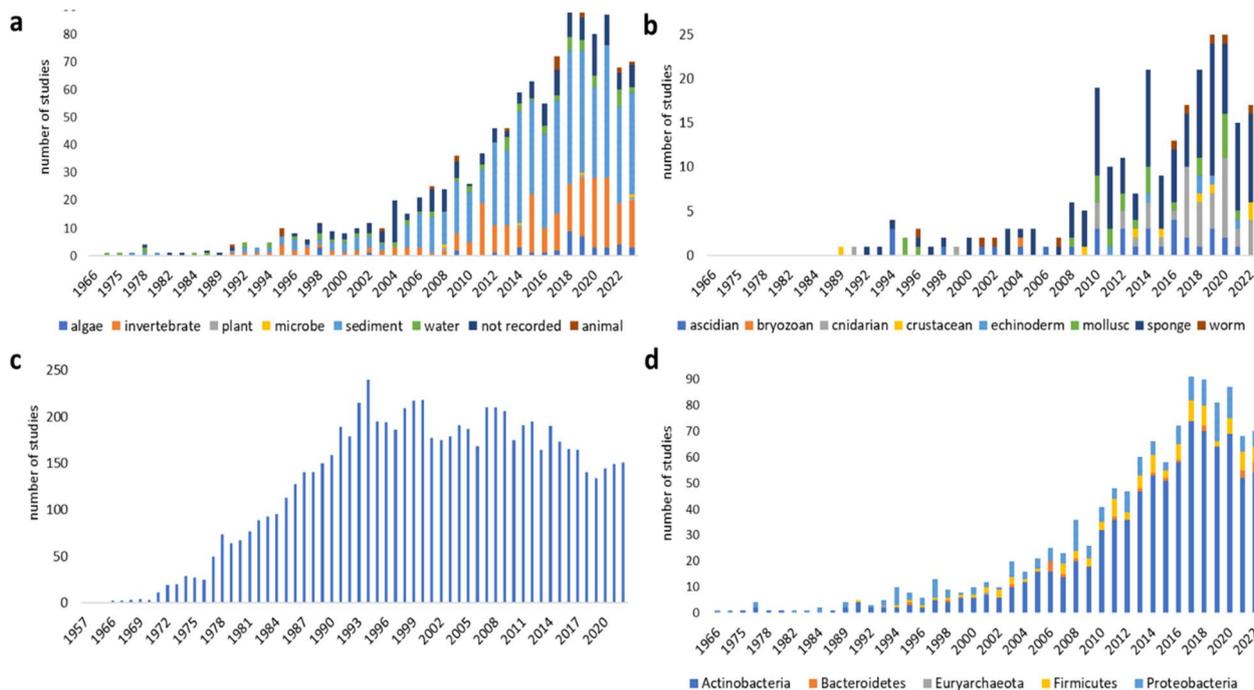


Fig. 2 Trends in marine bacteria investigations. (a) Environmental sources accessed to discover new MNPs from bacteria (b) marine invertebrate sources leading to bacterial cultures that have yielded new MNPs, (c) number of studies of marine invertebrates leading to new MNPs (d) phyla of bacteria from which new MNPs have been obtained.

marine invertebrate NP studies reached a zenith in 1994 and have been in slow decline since (Fig. 2).

The study of NPs from marine prokaryotes (excluding cyanobacteria) includes isolates obtained from the surface or within macro-organism tissues as well as from water and sediment. Over the last 67 years there have been 1151 papers that have reported new MNPs from marine bacteria. Of these, 648 have been from water or sediment samples, with corals, sponges, echinoderms, algae, ascidians, fish and bryozoans contributing to most of the remainder. In contrast, MNPs reported from cyanobacteria are almost exclusively derived from wild harvested material (Fig. 2).

The Actinobacteria are the most studied phylum of marine bacteria, and the organisms are also a prolific source of terrestrial bacterial NPs. However, the marine environment is different from terrestrial biomes and genomic data obtained from cultured, metagenomic and single amplified genome sources from marine environments (sediment and sea water) suggest that Actinobacteria only represent a small proportion of marine bacterial biodiversity with Proteobacteria being a dominant group (Fig. 3).<sup>562–564</sup>

In 2022 we highlighted the dissimilarity of marine micro-organism NP chemistry with that of marine macro-organisms and further highlighted the high similarity between terrestrial and marine microbial NP chemical diversity.<sup>565</sup>

Research efforts to discover new MNPs from marine prokaryotes are striking in that only a few of the studies on cultures obtained from marine habitats and reporting new chemistry have produced known MNPs isolated previously from

marine invertebrates. The serendipitous discoveries of prokaryotes isolated from marine habitats that have yielded didemnin B and the lobatamides that were originally isolated from ascidians are two examples but these microbes were not found in the ascidian hosts that yielded these MNPs.<sup>566,567</sup> Chemical diversity analysis using a self-organising map (Fig. 4) shows that there is little overlap between NPs reported from either sponges, cnidarians or ascidians and the microbes that have been isolated and cultured from their tissues.

The one report that stands out is that by Berlinck *et al.* of the isolation of a *Pseudovibrio* strain from a Brazilian Demospongiae sponge (*Arenosclera brasiliensis*, order: Haplosclerida) that produces bromotyrosine alkaloids that were only previously reported from sponges of the Demospongiae order Verongiida.<sup>568,569</sup> This begs the question, do Verongiida sponges also harbour *Pseudovibrio* species with the capacity of making bromotyrosine alkaloids? Previous cell localisation experiments suggested that Verongiid sponge cells accumulated the alkaloids but definitive proof of their biosynthesis within sponge cells is lacking.<sup>567</sup> Somewhat perplexing is the fact that the sponge *Arenosclera brasiliensis* harbouring *Pseudovibrio denitrificans* has not been reported to produce bromotyrosine alkaloids.

Although there is specific and circumstantial evidence to support the bacterial origin of some macro-organism NPs, the last 20 years of research has not provided many examples where bacteria isolated from marine macro-organisms have generated cultures that can be used for sustainable production of MNPs in the lab. In fact, there are only three examples (manzamine A



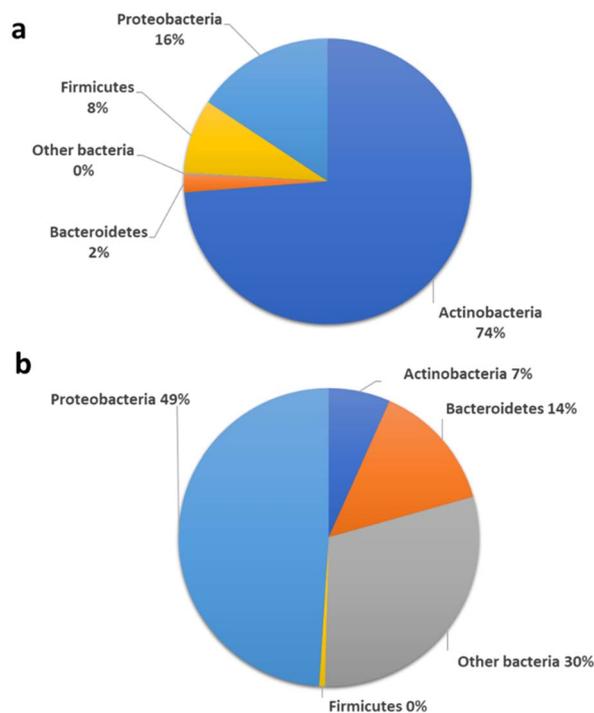


Fig. 3 Marine bacterial diversity (a) proportion of bacteria phyla yielding new MNPs (b) proportion of bacteria phyla found in marine environments based on metagenomic analysis.

*Acanthostrongylophora ingens*/Micromonospora M42; patellamides *Lissoclinum patella*/*Prochloron didemni*; and diketopiperazines *Tedania ignis*/*Minococcus* sp.) where a viable bacteria/cyanobacterial strain that produces the NP has been isolated from the macro-organism host that contains the MNP.<sup>558,570–572</sup> However, the manzamine A result must be considered provisional since more recent studies have shown that the original strain no longer produces the compound.<sup>573</sup>

Notably, an increasing number of studies have definitively identified microbial sources for NPs initially reported from marine invertebrates. The seminal studies by Piel, Schmidt and Jaspars are highlights here.<sup>571,572,574</sup> Current evidence has shown

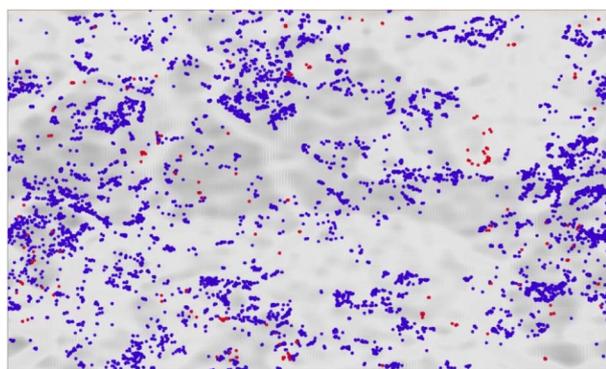


Fig. 4 Self-organising map depicting chemical diversity of sponge derived MNPs (10 267) (blue) and MNPs (272) obtained from bacteria isolated from sponge (red).

that many of the symbiotic microorganisms found in marine macro-organisms that produce the NP are obligate symbionts that are unlikely to be culturable. These obligate symbionts are mainly in bacterial phyla that, to date, remain unculturable and therefore have not been shown to produce isolable NPs.<sup>574</sup> This highlights the need to find alternative methods to exploit their biosynthetic genes through heterologous gene expression. There have been examples where bacteria isolated from sediment or seawater have yielded NPs that are the same or closely related to macro-organism NPs, but these are the exceptions rather than the rule and, in most cases, a prokaryote sourced from a terrestrial habitat has yielded NPs closely linked to a marine invertebrate-derived NP.

Analysis of data reported in reviews<sup>574–577</sup> (including previous installments of this review), and compound matching within chemical databases (MarinLit vs. NP Atlas)<sup>2,578</sup> suggests that marine invertebrate NPs from 46 compound classes have been definitively identified to be biosynthesised by prokaryotes (see Tables S1 and Fig S1 in the ESI<sup>†</sup>) (Fig. 5). These associations have been established through (i) BGC analysis which has identified 28 compound classes (one identified from two different bacteria phyla), (ii) analysis of NPs from separated prokaryote cells obtained from macro-organism hosts has identified four compound classes or (iii) culturing of randomly sourced bacteria (seven compound classes) and wild harvest of cyanobacteria (two compound classes) with a further six compound classes initially isolated from prokaryote cultures then later found in invertebrates. A further 17 invertebrate MNP structure classes have been associated with prokaryote sources by inference because similar molecules have been reported from microbes and marine invertebrates.

Within these 64 MNP structure classes, 39 have been identified as having a marine prokaryote origin, 20 are associated with terrestrial prokaryotes and two are associated with both marine and terrestrial prokaryotes. Invertebrate MNP compound classes that are also found in marine prokaryote cultures are from Cyanobacteria (patellamides, palyotoxin, swinholide), Actinomycetes (manzamine, diketopiperazine) and Proteobacteria (fistularin, dideminin). Terrestrial microbial cultures Proteobacteria (2), Actinomycete (5), Myxococcota (1),

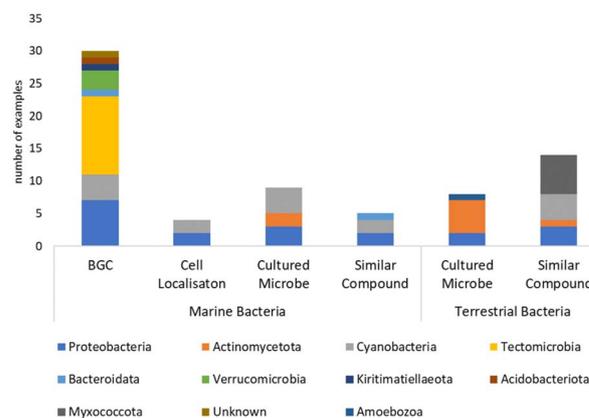


Fig. 5 Bacteria sources ascribed to marine invertebrates NPs.



and Amoebozoa (1) are the source of eight invertebrate MNP compound classes. There are four marine invertebrate compound classes that have similar compounds in marine microbes, two are from Cyanobacteria and two are from Proteobacteria. The 13 compound classes from invertebrates that are similar to terrestrial microbial compounds are found in Proteobacteria (4), Myxococota (6), Cyanobacteria (4), and Actinomycetes (2). Three structure classes are found in two different prokaryote sources. Microbial BGCs that encode enzymes that biosynthesise MNPs originally found in marine invertebrates are associated with microbes from Tectomicrobia (11) Proteobacteria (5), Cyanobacteria (5), Verrucomicrobia (3), Kiritimatiellaota (1), Acidobacteriota (1), Bacteroidata (1), and one unknown phylum.

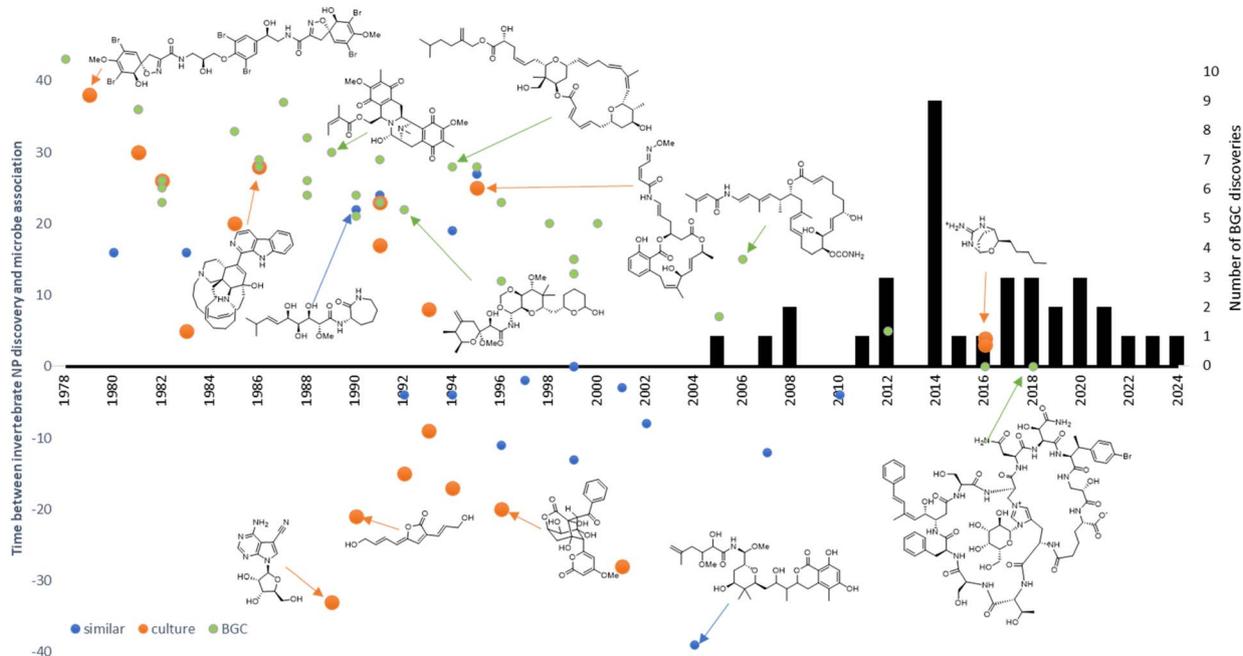
Microorganism-associated MNPs isolated from marine invertebrates have primarily been investigated within sponges and ascidians. There are 41 sponge derived structures (18 polyketides, 15 peptides, 6 alkaloids, 1 terpenoid, 1 phenol) that have been ascribed to a prokaryotic source (either through genomics, microbial culture, cell separation/imaging or structural similarity). Congeners of these structures represent 604 sponge-derived MNPs (5.8% of all sponge MNPs). Of the structures (and their congeners) definitively ascribed to prokaryotes by BGC analysis or cultivation, 8.5% of peptides, 8% of polyketides and 10.6% of alkaloids. This suggests that to date only 4.2% of all sponge NPs can be ascribed a microbial origin. A similar analysis of ascidian NPs shows that 14 structures have been ascribed to prokaryotes. When congeners of these structures are included, 101 ascidian derived NPs (7.8% of ascidian

NPs) are likely to be biosynthesised by prokaryotes and these represent 43% of peptides, 27.5% of polyketides and 3.2% of the alkaloids reported from ascidians.

The average time between the discovery of a marine invertebrate NP and ascribing its biosynthesis to a symbiotic microbe is >15 years (Fig. 6). BGC analysis has proven to be the most successful technique to identify a bacterial source within an invertebrate that produces the NP. However, this method has only been successful for PKS, NRPS and RiPP NP structure classes. This is most likely because the biosynthesis of PKS, NRPS and RiPP NPs is associated with clustered genes that produce enzymes that are co-located in the cell. Other classes of NPs appear to have more complex biosynthetic pathways, and the enzymes used to make them may not be clustered. This is particularly the case with some alkaloids. The full biosynthetic pathway to ET-743, for example remains to be discovered.<sup>579</sup>

Since the structural uniqueness of MNPs continues to be linked to wild harvested macro-organisms and cyanobacteria, but annually over half of all new MNPs are reported from marine microbes, the lessons learnt from identifying the prokaryote producers of marine macro-organism derived MNPs should be applied to aid in the selection of uniquely marine microbes for the discovery and exploitation of unique microbial MNP chemical diversity.

Furthermore, the realisation that many of the marine microorganisms responsible for macro-organism derived MNPs have eluded culturing has meant that other methods to access their rich biosynthetic diversity are required. Heterologous expression of BGCs identified from metagenomic analysis of



**Fig. 6** Timeline of discoveries of bacterial origins of marine invertebrate derived NPs. The horizontal axis is the year of discovery of the MNP from an invertebrate source and the coloured dots represent the time period between invertebrate MNP discovery and discovery of the bacterium source. Negative values represent cases where the NP was originally isolated from a bacterium and has more recently been found in a marine invertebrate. The bar chart is a histogram of number of discoveries of a bacterial BGC ascribed to marine invertebrate derived MNPs in a given year.



environmental samples (accessed from sediment, seawater or macro-organism hosts) in culturable hosts is one way to access this chemical diversity.<sup>580–583</sup>

Recent genomic studies have also started to reveal the capabilities of marine invertebrates to manufacture NPs *de novo*.<sup>584–586</sup> Both cnidarians and sponges have capabilities to biosynthesise terpenoids, a structure class that represents 89% and 65% of NPs reported from these phyla, respectively. Alkaloids possess some of the most diverse structures and play a predominant role to drug development, yet their biosynthesis and true origins remain mostly unresolved.

Finally, the number of invertebrate species that have been shown to contain NPs of prokaryote origin is also very limited. Five genera (four sponge and one ascidian) account for half of the compound classes ascribed to prokaryotes *Theonella* (structure classes: swinholid, onnamide, theopederin, konbamide, cyclotheronamide, keramamide, pseudotheonamide, nazumamide, polytheonamide, motuporin, theopalauamide, theonellamide) *Discoderma* (structure classes: calyculin, discoderamide, kasumigamide), *Mycale* (peloruside, mycalamides, pateamine), *Dysidea* (structure classes: bromodiphenyl ethers, trichloropeptides, arenastatin, dysonosin, dysidazirine), *Lissoclinum* (structure classes: patellamides, lissoclinamide, patellins trunkamides, ascidiacyclamide, patellazoles, mandelalide, lissoclinolide, haterumalide). It is difficult to know if these limited examples reflect the difficulty in studying symbiosis or if these super-producer sponge and ascidian species have a unique ability to host diverse microbiomes that manufacture a range of NPs compared to most invertebrates.

Although there is growing evidence for the contribution of marine prokaryotes to MNP production in marine invertebrates, this knowledge has not translated into widespread sustainable production of marine invertebrate-derived NPs in culturable microbes. Furthermore, the promise of targeting the culture of marine prokaryotes sampled from marine habitats as an alternative way to discover the unique chemical diversity of marine invertebrates has so far not eventuated. Metagenomics has certainly highlighted the huge potential of marine bacteria to yield new chemical diversity based on the predicted uniqueness of BGCs that they host, and heterologous gene expression appears to be the most promising way forward to harness this diversity. A complimentary approach would be to expand studies of marine invertebrates using cheminformatics tools to identify unique chemicals. Flipping the focus of outputs generated by GNPS to target the unique unknown singletons rather than the clusters of unknown analogues within well studied structure classes may be a good starting point.

## 14 Conflicts of interest

There are no conflicts to declare.

## 15 Acknowledgements

We thank Royal Society of Chemistry for the provision of data used in this review, adapted from the MarinLit database with permission from the Royal Society of Chemistry.<sup>2</sup> We thank

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