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Marine natural products‡

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This review covers the literature published in 2014 for marine natural products (MNPs), with 1116 citations (753 for the period January to December 2014) referring to compounds isolated from marine microorganisms and phytoplankton, green, brown and red algae, sponges, cnidarians, bryozoans, molluscs, tunicates, echinoderms, mangroves and other intertidal plants and microorganisms. The emphasis is on new compounds (1378 in 456 papers for 2014), together with the relevant biological activities, source organisms and country of origin. Reviews, biosynthetic studies, first syntheses, and syntheses that lead to the revision of structures or stereochemistries, have been included.

- 1 Introduction
- 2 Reviews
- 3 Marine microorganisms and phytoplankton
- 3.1 Marine-sourced bacteria (excluding from mangroves)
- 3.2 Bacteria from mangroves
- 3.3 Marine-sourced fungi (excluding from mangroves)
- 3.4 Fungi from mangroves
- 3.5 Cyanobacteria
- 3.6 Dinoflagellates
- 4 Green algae
- 5 Brown algae
- 6 Red algae
- 7 Sponges
- 8 Cnidarians
- 9 Bryozoans
- 10 Molluscs
- 11 Tunicates (ascidians)
- 12 Echinoderms
- 13 Mangroves
- 14 Miscellaneous
- 15 Conclusion
- 16 Acknowledgements
- 17 References

1 Introduction

These annual reviews of marine natural products were initiated by the late Professor D. John Faulkner in 1984^{1,2} and continued by the New Zealand group since 2003. A feature of the reviews has been the inclusion of the structures for all new MNPs, and any subsequently revised structures. The number of new MNPs reported each year has steadily grown from 332 in 1984 to 1378 in this present review of the 2014 literature. This has inevitably resulted in an increased size for each review. With the ever-increasing size creating difficulties for preparation of the annual review, the NPR Editorial Board suggested changing the format to focus on a selection of highlighted structures. To maintain the usual comprehensive coverage of all new and revised MNPs, we have prepared a ESI‡ document with links associated with this review, showing all structures, along with their names, taxonomic origins, locations for collections, and biological activities. The numbers for all highlighted structures in this review (169) are shown in non-italicised bold font, while italicised numbers refer to the remaining structures in the ESI document.‡ For structures that have their absolute configurations fully described, the compound number in the diagrams is preceded with †. In addition to the highlighted compounds in this review, we have retained the inclusion of reference to first syntheses of MNPs, and comments on new information on ecological aspects, bioactivities or other relevant data for previously reported MNPs, all as non-highlighted material. The Reviews section (Section 2) has also been reformatted to show selected highlights, with all other reviews referenced in a section of the ESI document.‡

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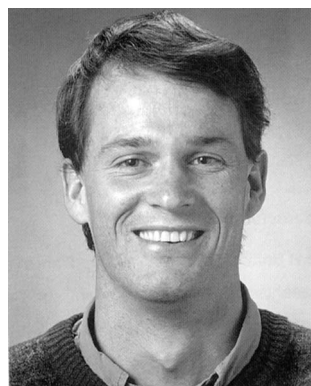
2 Reviews

There continues to be a steady increase in the number of reviews of various aspects of MNP studies. Some of the more



John Blunt obtained his BSc (Hons) and PhD degrees from the University of Canterbury, followed by postdoctoral appointments in Biochemistry at the University of Wisconsin-Madison, and with Sir Ewart Jones at Oxford University. He took up a lectureship at the University of Canterbury in 1970, from where he retired as an Emeritus Professor in 2008. His research interests are with natural prod-

ucts, the application of NMR techniques to structural problems, and the construction of databases to facilitate natural product investigations.



Brent Copp received his BSc (Hons) and PhD degrees from the University of Canterbury, where he studied the isolation, structure elucidation and structure-activity relationships of biologically active marine natural products under the guidance of Professors Blunt and Munro. He undertook postdoctoral research with Jon Clardy at Cornell and Chris Ireland at the University of Utah. 1992–93 was spent

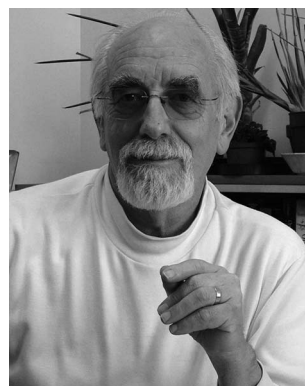
working in industry as an isolation chemist with Xenova Plc, before returning to New Zealand to take a lectureship at the University of Auckland, where he is currently an Associate Professor.



Rob Keyzers carried out his BSc (Hons) and PhD studies at Victoria University of Wellington. His thesis research, carried out under the guidance of Assoc. Prof. Peter Northcote, a former contributor to this review, focused on spectroscopy-guided isolation of sponge metabolites. He then carried out post-doctoral research with Mike Davies-Coleman (Rhodes University, South Africa) and Raymond Andersen

(University of British Columbia, Canada) before a short role as a flavour and aroma chemist at CSIRO in Adelaide, Australia. He was appointed to the faculty at his alma mater in 2009 where he is currently a Senior Lecturer.

significant reviews (16) are given here while a listing of the remainder (71) is given in the ESI section.† A comprehensive review of MNPs reported in 2012 has appeared.³ ‘*Marine-sourced anticancer and cancer pain control agents in clinical and late preclinical development*’ have been reviewed,⁴ and ‘*New horizons for old drugs and drug leads*’ were described.⁵ The implications of the Convention on Biological Diversity (1999) and its Nagoya Protocol (2010) on the collection of marine genetic resources has been discussed and should be noted by all who collect marine organisms for MNP studies.⁶ The putative microbial origin of sponge metabolites has been the subject of several reviews and articles.^{7–11} Developments in chemical ecology for fish and benthic algae and invertebrates for 2010–2012 have been reviewed, with comment on the biosynthesis of bioactive MNPs by symbiotic microorganisms.¹² Polyketide biosynthesis in dinoflagellates has been reviewed.¹³ There have been comprehensive reviews for marine nucleosides,¹⁴ saxitoxin,¹⁵ and tetrodotoxin.¹⁶ A review of ‘*Emerging strategies and integrated systems microbiology technologies for biodiscovery of marine bioactive compounds*’ provides a very useful oversight of a number of developing techniques.¹⁷ ‘*AlgaeBase: an on-line*



Murray Munro, Emeritus Professor in Chemistry at the University of Canterbury, has worked on natural products right through his career. This started with diterpenoids (PhD; Peter Grant, University of Otago), followed by alkaloids during a post-doctoral spell with Alan Battersby at Liverpool. A sabbatical with Ken Rinehart at the University of Illinois in 1973 led to an interest in marine natural

products with a particular focus on bioactive compounds which has continued to this day. In recent years his research interests have widened to include terrestrial/marine fungi and actinomycetes.



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

Zealand to take up a lectureship at the University of Waikato, where she is currently an Associate Professor.



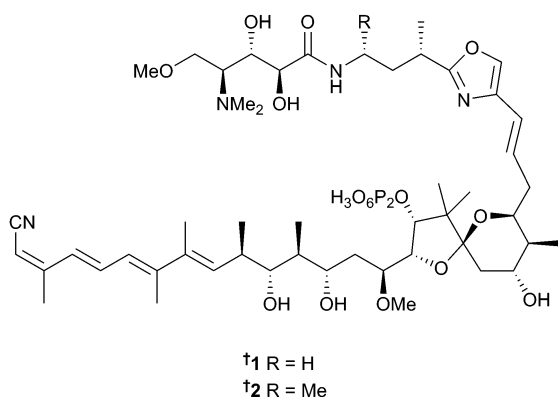
resource for algae is an article describing a very comprehensive algal database.¹⁸ As in previous years, the MarinLit database¹⁹ has been updated and used as the literature source for the preparation of this present review.

3 Marine microorganisms and phytoplankton

Even considering the trend of recent years that many marine natural products research efforts are directed towards microorganisms, there has been a sharp upward swing in the number of new metabolites reported from marine microorganisms (677 vs. 493 in 2013). Unless otherwise stated, compounds described in this section were obtained from cultures of the named microorganisms.

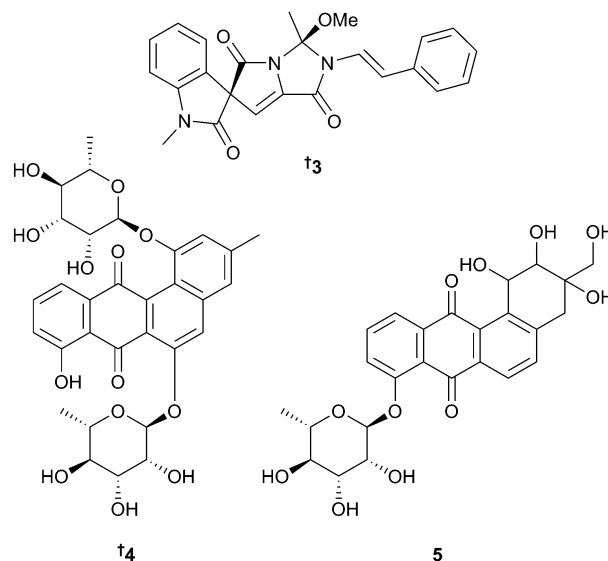
3.1 Marine-sourced bacteria (excluding from mangroves)

The number of new compounds reported from marine bacteria (164) is similar to the 158 reported in 2013. A metagenomic approach has identified the gene cassette responsible for the biosynthesis of calyculin A, originally isolated from the sponge *Discodermia calyx*,²⁰ as microbial in origin, arising from *Candidatus Entotheonella* sp. Functional analysis of the biosynthetic pathway has shown that the end product is actually a diphosphate protoxin, phosphocalyculin A **1** rather than calyculin A, suggesting a phosphorylation/dephosphorylation mechanism for the active chemical defence of the host sponge.²¹ A further diphosphate, protoxin phosphocalyculin C **2**, has been isolated from *D. calyx* but can be assumed to have arisen from the same microbial source. Phosphocalyculin C, although potent (IC₅₀ = 36 nM vs. P388), is 5000 times less toxic than calyculin C itself.²²



A Chinese sediment-derived *Actinoalloteichus cyanogriseus*²³ was the source of cyanogramide **3**, an unprecedented spirocyclic alkaloid with multidrug-resistance (MDR) reversing activity.²⁴ An interesting dereplication strategy was utilised in the study of sponge-associated *Actinokineospora* sp. Principal Component Analysis (PCA), hierarchical clustering (HCA) and orthogonal partial least square-discriminant analysis (OPLS-DA) were employed to evaluate HRFTMS and NMR data of crude extracts arising from four different fermentation regimes. Statistical analysis enabled identification of the most suitable one-strain-many-compounds (OSMAC) culture conditions and extraction

method resulting in isolation of the *O*-glycosylated angucyclines actinosporin A **4** and B **5** of which actinosporin A **4** possessed moderate activity against the causative agent of sleeping sickness, *Trypanosoma brucei* (*T. brucei*).²⁵

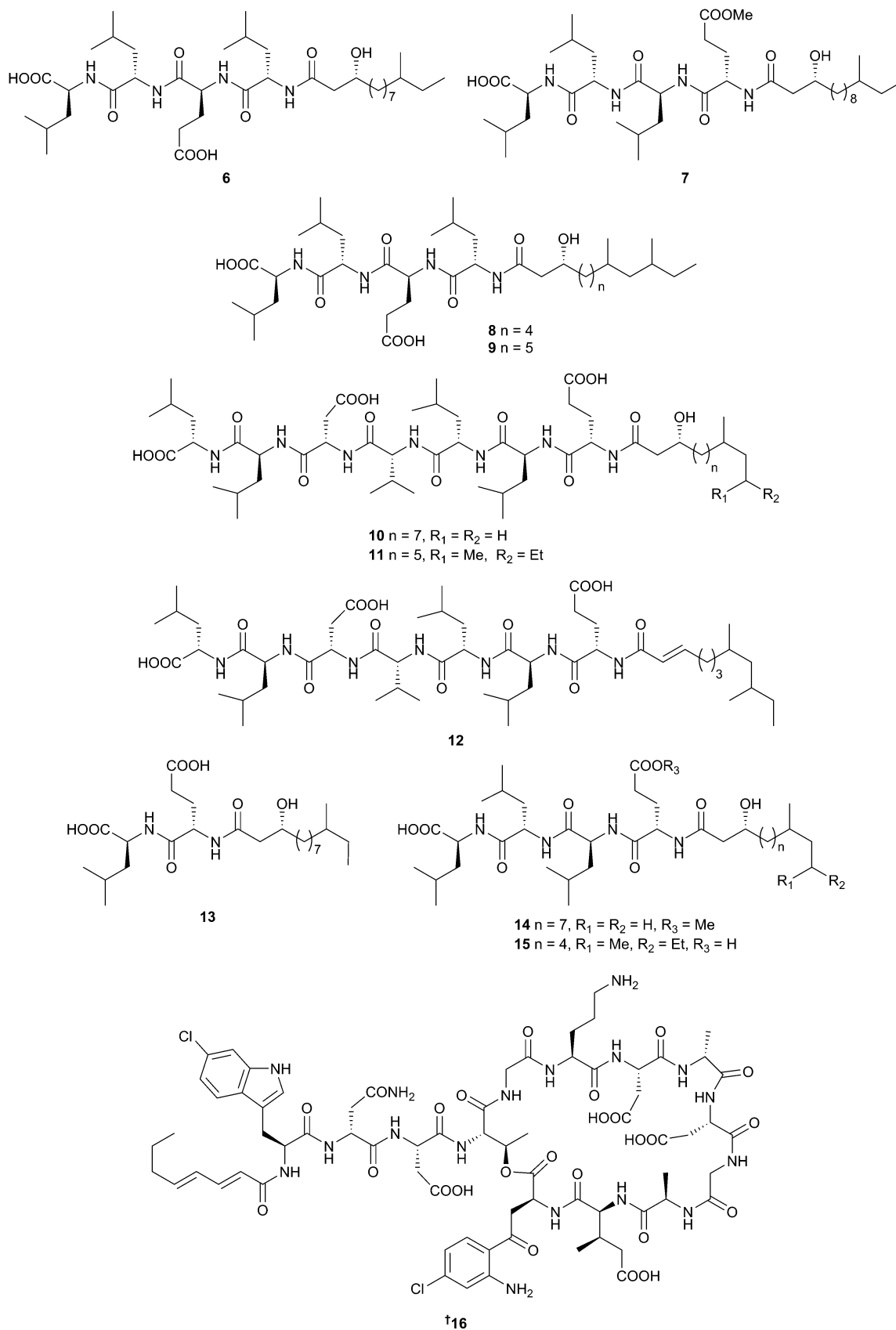


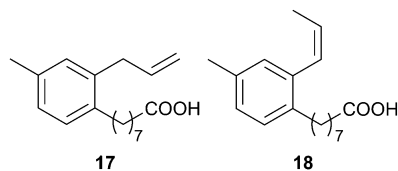
A Korean sediment-derived strain of *Bacillus subtilis* (*B. subtilis*) has yielded a variety of linear lipopeptides with differing biological properties, the gageopeptides A–D **6–9**,²⁶ gageostatins A–C **10–12**²⁷ and gageotetrins A–C **13–15**.²⁸ Most are non-cytotoxic with good antibacterial activity, but better antifungal activity especially against the late blight pathogen *Phytophthora capsici* in the case of gageotetrins A–C. A transformation-associated recombination (TAR) cloning approach was used to capture, activate and express a 67-kb non-ribosomal peptide synthetase (NRPS) biosynthetic gene cluster from *Saccharomonospora* sp., resulting in isolation of the dichlorinated lipopeptide antibiotic taromycin A **16**.²⁹

Solwaspora sp. (ascidian, *Trididemnum orbiculatum*, Florida Keys, U.S.A.) produced the trialkyl-substituted aromatic acids, solwaric acids A **17** and B **18**. Enrichment with ¹³C-labelled glucose followed by acquisition of a ¹³C–¹³C COSY enabled unambiguous determination of the position of the methyl group on the phenyl ring, an approach which could be very useful for structural determination of molecules with multiple quaternary carbons.³⁰

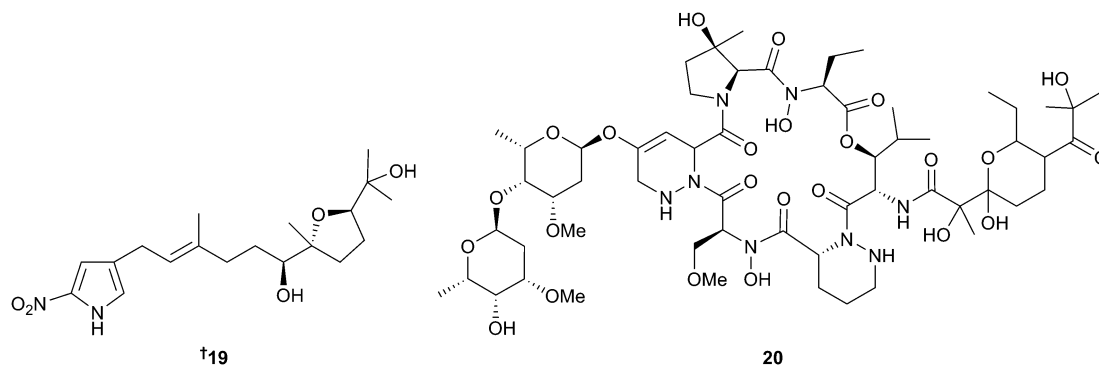
Heronapyrroles A–C are nitropyrrole metabolites with a partially oxidised farnesyl chain appended that were obtained from a *Streptomyces* sp.³¹ Biosynthetic considerations prompted the hypothesis that a mono-tetrahydrofuran-diol, heronapyrrole D might be an as yet unidentified metabolite of the bacterium. Following a putative biomimetic synthesis of heronapyrrole D **19** the metabolite was then detected in cultures of the bacterium, thus validating this approach.³² Total synthesis of heronapyrrole C³¹ has also been reported.³³ Mollemycin A **20** is a first-in-class glycol-hexadepsipeptide-polyketide from a *Streptomyces* sp. (sediment, South Molle Is., Queensland, Australia) and active against certain Gram-positive and Gram negative bacteria, in addition to extremely potent antimalarial activity







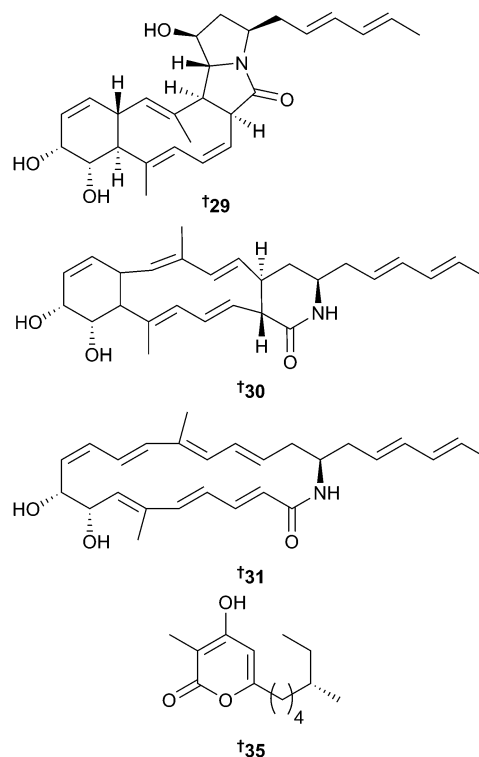
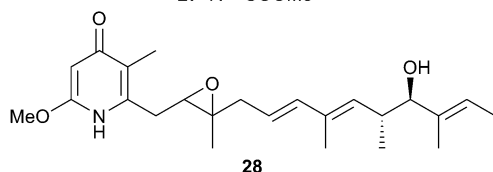
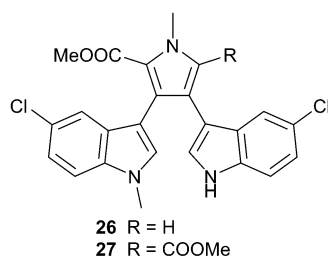
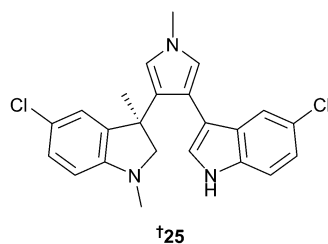
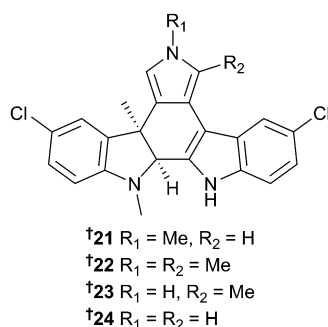
has now led to isolation of a number of bisindole alkaloids; indimicins A–E **21–25**, lynamycins F **26** and G **27**³⁷ and piericidin E1 **28**³⁶ using the same modified A1BFe + C medium. Piericidin E1 **28** was shown to be an intermediate in the biosynthesis of piericidin A1³⁸ during identification of the biosynthetic gene cluster.



against drug sensitive and MDR *Plasmodium falciparum* (*P. falciparum*) clones.³⁴

A deep-sea strain of *Streptomyces*, previously a source of spiroindimicins A–D,³⁵ lynamycins A and D³⁵ and piericidins³⁶

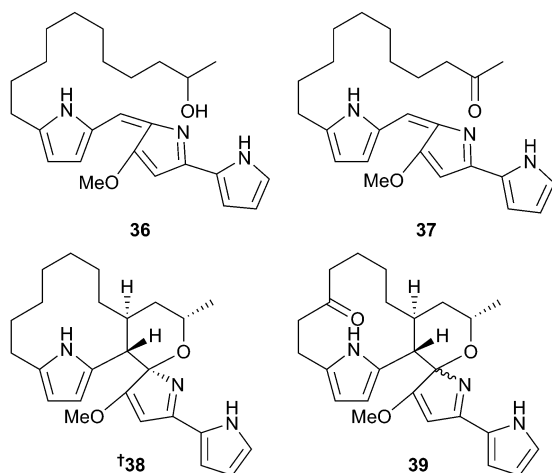
Growth of the strain in modified ISP3 medium yielded heronamides D–F **29–31**.³⁹ Violapyrones H **32**, I **33**, B **34** and C **35**⁴⁰ were obtained from *Streptomyces* sp. (starfish, *Acanthaster planci*, Chuuk, Federated States of Micronesia).⁴¹ Violapyrone C **35** has since been synthesised and the absolute configuration determined.⁴²



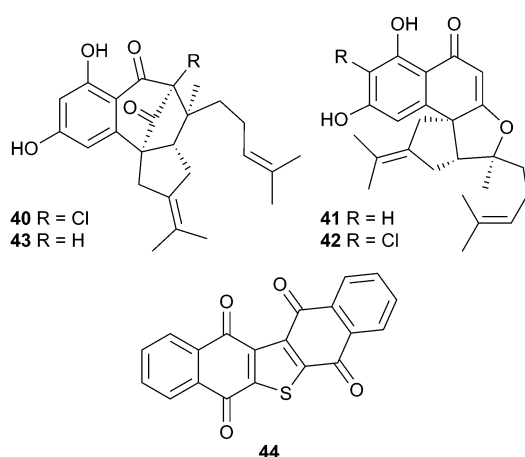
A study of the biosynthetic pathway for marineosins⁴³ in *Streptomyces* sp. led to isolation of 23-hydroxyundecylprodiginine **36**, 23-ketoundecylprodiginine **37**, premarineosin A **38** and 16-ketopremarineosin A **39**.⁴⁴ Syntheses of 23-hydroxyundecylprodiginine **36** (both enantiomers and the



perdeuterated version) and of 23-ketoundecylprodiginine **37** were reported, as was the feeding of synthetic prodiginine analogues which led to the production of novel premarineosins, although these were not fully characterised.⁴⁵



The structure of merochlorin A^{46,47} has been revised to **40**⁴⁸ and a biomimetic synthesis of (±)-merochlorin B⁴⁶ has been achieved,⁴⁹ while a study of the biosynthesis of the merochlorins revealed that just four enzymes are involved⁵⁰ and that a vanadium-dependent chloroperoxidase mediated a complex series of unprecedented transformations in the biosynthesis. Development of a chlorination method paralleling the biocatalytic process led to the identification of previously undiscovered merochlorins **41–43**.⁵¹ The known synthetic compound seriniquinone **44**⁵² has now been obtained as a natural product from *Serinicoccus* sp. and displayed potent and selective anti-tumour activity.⁵³



Investigation of the biosynthesis of sulfur-containing roseobacticides^{54,55} produced by *Phaeobacter inhibens* indicated that these compounds arise from three subunits – dimethylsulfiopropionate, phenylacetic acid and *p*-coumaric acid and that roseobacticides regulate the symbiosis between *P. inhibens* and the microalga *Emiliana huxleyi*.⁵⁶ Further new metabolites **45–103** were obtained from the genera *Actinoalloteichus*, *Actinomadura*, *Actinokineospora*, *Amycolatopsis*, *Bacillus*, *Dermaococcus*,

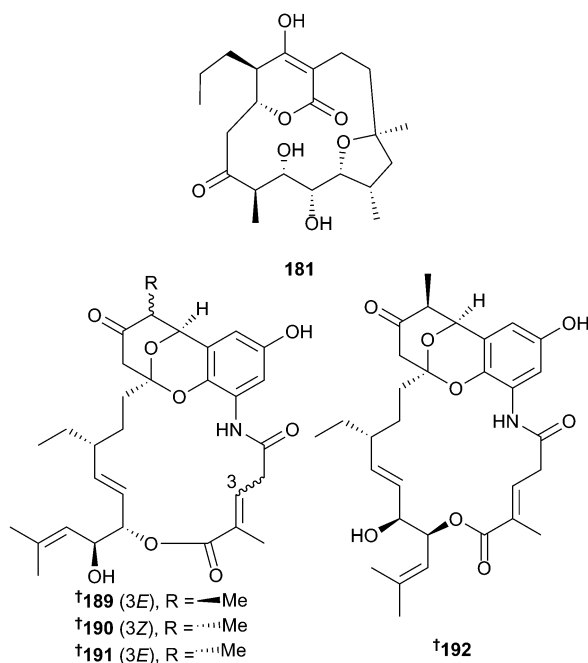
Escherichia, *Jejua*, *Micrococcus*, *Micromonospora*, *Nocardiopsis*, *Pelomonas*, *Pseudoalteromonas*, *Rapidithrix*, *Salinispora* and *Shewanella*.^{57–80} As usual, many new metabolites, and some with revised structures, were also obtained from the genus *Streptomyces* including **104–172**.^{81–103} The genera *Verrucosipora* and *Vibrio* also yielded new metabolites **173** and **174**.^{104,105} Investigation of an octocoral-associated *Pseudoalteromonas* sp. by MALDI-imaging mass spectrometry (IMS) and molecular network analyses indicated that the strain produces higher levels of the antifungal polyketides, alteramides^{106,107} in the dark than in the light and also led to revision of the configuration at C-6 for **175** and **176**.¹⁰⁸ In the light, these compounds were inactivated through a photoinduced intramolecular cyclisation and production of higher levels of these antifungal metabolites in the dark was proposed as a strategy to protect the host corals during night feeding, when they are more exposed.¹⁰⁸ Synthesis of the proposed structure of heronamide C¹⁰⁹ has indicated that the actual structure of the natural product needs to be re-examined¹¹⁰ and the structure of anthracimycin¹¹¹ has also been corrected to **177**.¹¹² Total syntheses of indoxamycins¹¹³ has led to stereochemical revision for the indoxamycins B,¹¹⁴ D¹¹³ and E¹¹³ to **178–180**.¹¹⁵ A number of other total syntheses of bacterial metabolites have been reported. These include syntheses of the depsipeptides, solanamides A¹¹⁶ and B^{116,117} and arenamides B¹¹⁸ and C,^{118,119} and synthesis¹²⁰ of the nucleoside antibiotic A201A.^{121,122} Total synthesis of dixiamycin B¹²³ was achieved utilising electrochemical oxidation.¹²⁴ The alkaloid mansouramycin D^{125,126} was synthesised, as was 5-deoxytetrodotoxin.^{127,128} New biological activities have been reported for sporolide B¹²⁹ from *Salinispora tropica*,¹³⁰ for some butenolides^{131,132} and undecylprodigiosin¹³³ from *Streptomyces* strains.^{134,135} Biosynthetic studies have been conducted into various bacterial metabolites including arachidonic acid (in *Aureispira marina*),^{136,137} macrolactins¹³⁸ and bacillaene¹³⁹ (in *Bacillus marinus*),¹⁴⁰ polybrominated aromatics¹⁴¹ (in *Marinomonas mediterranea*¹⁴² and *Pseudoalteromonas* spp.¹⁴³), lomaiviticins¹⁴⁴ (in *Salinispora pacifica*^{145,146} (formerly *Micromonospora lomaivitiensis*)), tropodithietic acid¹⁴⁷ (in *Phaeobacter inhibens*),¹⁴⁸ sulfur volatiles¹⁴⁹ (in the *Roseobacter* clade)¹⁵⁰ and avaroferrin¹⁵¹ and putrebactin¹⁵² (in *Shewanella* sp.).¹⁵³ Biosynthetic studies within the genus *Streptomyces* include those on thiocoraline¹⁵⁴ (in *S. albus*),¹⁵⁵ ikarugamycin,^{156–158} antimycins^{159,160} and polyhydroxylated saturated fatty acids,¹⁶¹ marinophenazines^{162–164} and the isoprenylated phenazines,^{165,166} JBIR-46, JBIR-47 and JBIR-48.¹⁶⁷

3.2 Bacteria from mangroves

There has been an increase in the number of new metabolites reported from bacteria associated with mangroves (23 in 2014 vs. 10 in 2013). *Lechevalieria aerocolonigenes* yielded the cyclopentadecane metabolites, mangromicins A **181**, B **182**¹⁶⁸ and D-I **183–188**,¹⁶⁹ with mangromicin A **181** exhibiting potent antitrypanosomal and radical scavenging (DPPH) activities.^{168,169} New divergolide congeners **189–192** were obtained from an endophytic *Streptomyces* strain¹⁷⁰ and the biosynthetic gene cluster for divergolides¹⁷¹ identified and characterised.¹⁷²

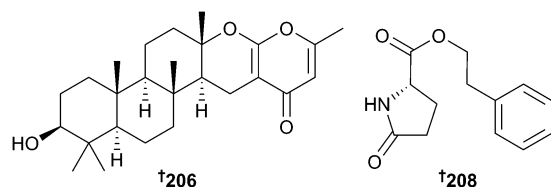


Other metabolites **193–203** were isolated from the genera *Fishengella*, *Micromonospora*, *Streptomyces* and *Verrucosisspora*,^{173–176} and a bio-inspired total synthesis of the indole sesquiterpenoid sespenine¹⁷⁷ completed.¹⁷⁸



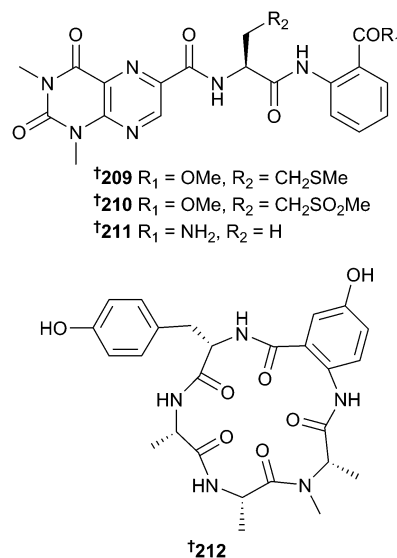
3.3 Marine-sourced fungi (excluding from mangroves)

Studies of fungi continue to rise with 318 new compounds reported in 2014 compared to 223 in 2013. Sponge-associated *Aspergillus similanensis* yielded two isocoumarin derivatives **204** and **205**, and a deacetyl analogue of chevalone C,¹⁷⁹ chevalone E **206**, in addition to pyripyropene S **207**.¹⁸⁰ While chevalone E **206** itself did not display significant antibacterial activity, it did exhibit synergy with the antibiotics oxacillin and ampicillin against MRSA.¹⁸¹ Ultrasonication of *Aspergillus versicolor* spores led to the isolation of a mutant with neomycin resistance from which six metabolites not present in the parent strain, including **208** were obtained. Although several patents exist for synthesis of the planar structure of this compound,^{182–184} this is the first natural product (NP) isolation and establishment of stereochemistry.

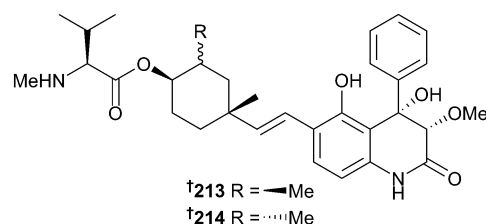


Cyclo(D-Tyr-D-Pro) was also claimed as a first NP isolation but this has previously been obtained from both terrestrial¹⁸⁵ and marine¹⁸⁶ sources.¹⁸⁷ Lumazine peptides, penilumamide B–D **209–211** and a cyclic pentapeptide, asperpeptide A **212** were obtained from a gorgonian-derived *Aspergillus* sp. The presence of the sulfone penilumamide¹⁸⁸ and the derived sulfoxide

penilumamide C **210** led to speculation that these compounds were derived from oxidation of a putative metabolite containing a methionine residue. This led to a feeding experiment with L-methionine resulting in isolation of the sulfide, penilumamide B **209**. Yields of penilumamide B **209** and penilumamide increased with increasing concentration of L-methionine and when penilumamide B **209** was exposed to air, penilumamide was detected after a few days whilst penilumamide C **210** was formed several days later.¹⁸⁹

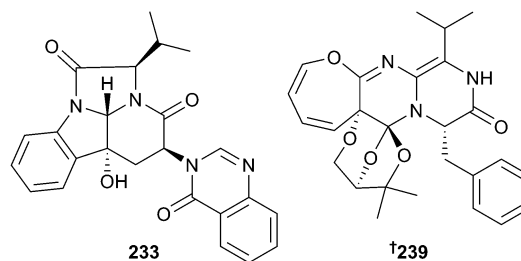
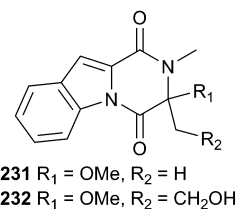
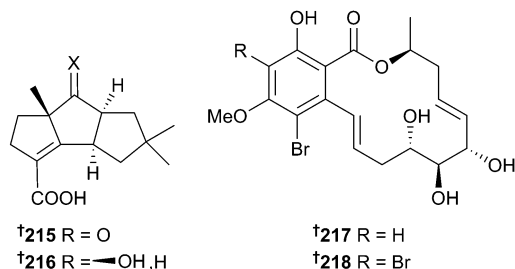


Of the two prenylated hydroquinone derivatives **213** and **214** obtained from a gorgonian-derived *Aspergillus* sp., **214** exhibited very potent activity against respiratory syncytial virus (RSV).¹⁹⁰ These two metabolites differ only in the configuration of a methyl group on a cyclohexane ring yet given that **214** is an extremely potent anti-RSV agent whilst its epimer **213** is completely inactive, indicating the importance of the configuration of this ring for anti RSV-activity.

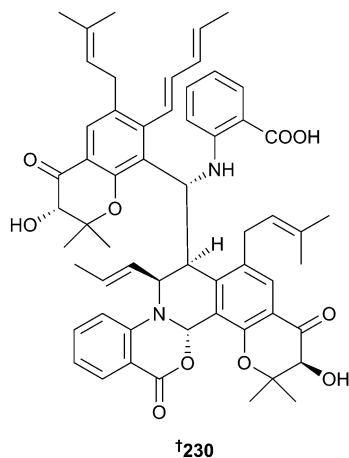
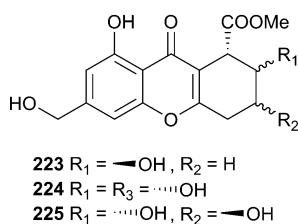


Soft-coral associated *Chondrostereum* sp. has previously been reported to produce hirsutane-framed sesquiterpenes.^{191–193} Cultivation of the fungus in a medium with glycerol as the carbon source led to the isolation of the sesquiterpenes chondrosterin I **215** and J **216** of which chondrosterin J **216** displayed potent activity against HTCLs.¹⁹⁴ Chemical epigenetic modification of *Cochliobolus lunatus* (sea anemone *Palythoa haddonii*) with inhibitors of histone deacetylase (HDAC) resulted in isolation of two brominated 14-membered resorcylic acid lactones **217** and **218**, but only in the presence of an HDAC inhibitor.¹⁹⁵



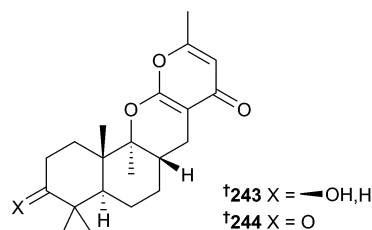
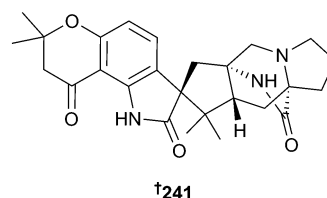
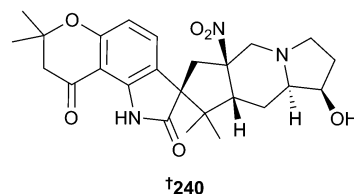


The chromones engyodontiumone A–H **219–226** and the phenol derivatives **227–229** were derived from deep-sea derived *Engyodontium album*. Of these, engyodontiumones E–G **223–225** were obtained as racemates.¹⁹⁶ The known polyketide aspergillus one B¹⁹⁷ was also isolated and was a potent inhibitor of settlement of *Balanus amphitrite* (*B. amphitrite*) larvae.¹⁹⁶ Fermentation of a filamentous fungus of the Eurotiomycetes class (ascidian, *Lissoclinum patella*, Papua New Guinea) produced the pentacyclic oxazinin A **230**, derived from a combination of benzoxazine, isoquinoline and pyran rings. Oxazinin A **230** occurred as a racemate and was antimycobacterial.¹⁹⁸



Neosartorya pseudofischeri (starfish *Acanthaster planci*, Hainan, China) produced different suites of metabolites when cultured in different media. When cultured in glycerol-peptone–yeast extract (GlyPy), the diketopiperazines neosartin A **231** and B **232** were produced along with six known diketopiperazines and a precursor alkaloid but when fermented in glucose–peptone–yeast extract (GluPy), a tetracyclic-fused alkaloid neosartin C **233** was produced along with a known meroterpenoid and five known gliotoxin analogues **234–238**, obtained here for the first time as NPs.¹⁹⁹ Endophytic *Paecilomyces variotii* (red alga, *Grateloupia turuturu*, Qingdao, China) yielded varioxepine A **239**, an alkaloid with an unprecedented 3,6,8-trioxabicyclo[3.2.1]octane unit.²⁰⁰

Biosynthetic feeding experiments on *Penicillium citrinum* using ¹³C labelled glucose, anthranilic acid and ornithine resulted in isolation of two new citrinalins, 17-hydroxycitrinalin B **240** and citrinalin C **241** and supported the proposition that the citrinalins arise from a bicyclo[2.2.2]diazaoctane precursor. Also in this investigation, synthesis of the enantiomer of citrinalin B²⁰¹ led to revision of the structure of citrinalin B to **242**.²⁰² Penicillipyrones A **243** and B **244** are meroterpenoids obtained from a sediment-derived *Penicillium* strain and represent a new skeletal class with a unique linkage between the drimane sesquiterpene and pyrone moieties. Penicillipyron B **244** elicited significant induction of quinone reductase in murine hepatoma cells, indicating a possible cancer preventative role.²⁰³



Metabolites **245–254** were also obtained from the genera *Acremonium*, *Arthrinium*, *Ascotricha* and *Astrocystis*.^{204–207} As usual, a large number of metabolites were obtained from species of the *Aspergillus* genus. Two indole diterpenoids **255** and **256**, and an isocoumarin **257** were obtained from *A. flavus*. The known compounds β -aflatoxin,²⁰⁸ paspalinine^{209,210} and leporine B²¹¹ were

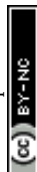
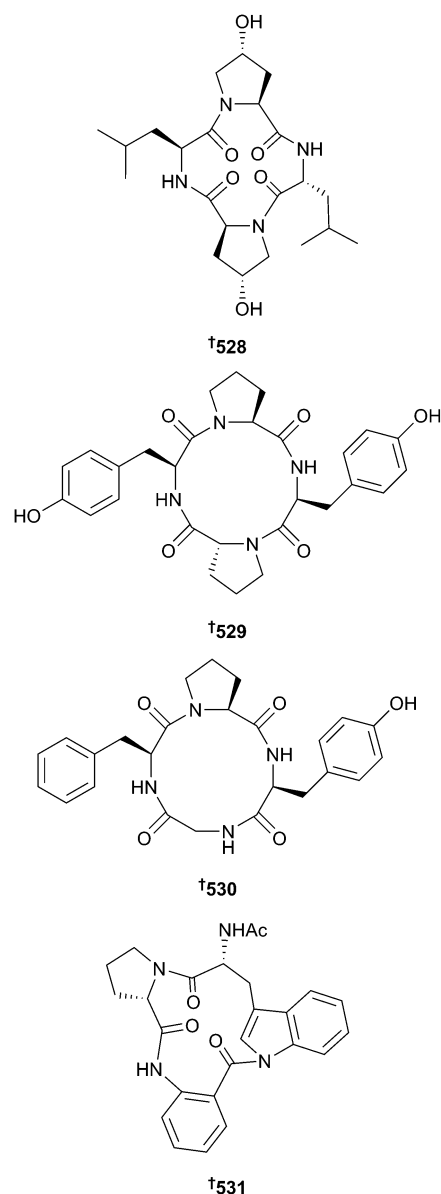


isolated and leporine A²¹² was prepared from the last of these. Configurations were established for each as **258–261** respectively and β -aflatrem and leporine B were isolated for the first time as MNPs.²¹³ Other metabolites isolated from the genus *Aspergillus* include **262–327** (the last obtained from co-culture with an unidentified bacterium).^{214–237} New metabolites **328–371** were also obtained from the genera *Beauveria*, *Cladosporium*, *Cochliobolus*, *Curvularia*, *Dendrodochium*, *Diaporthaceae*, *Dichotomomyces*, *Emericella* and *Eurotium*.^{238–250} A pyrrolidinoindoline diketopiperazine dimer, cristatumin E, isolated from *Eurotium herbariorum*,²⁵¹ appears to be identical to the previously reported eurocristatine.²⁵² The genera *Hansfordia*, *Humicola*, *Isaria*, *Neosartorya*, *Nigrospora*, *Paecilomyces* and *Paraconiothyrium* also yielded the new metabolites **372–399**.^{179,253–258} The genus *Penicillium* was the source of many other metabolites **400–461**.^{259–278} Other genera to yield new the metabolites **462–520** were *Pseudallescheria*, *Spicaria*, *Spirromastix*, *Stachybotrys*, *Talaromyces*, *Trichoderma*, *Xylaria* and *Xylariaceae*,^{279–291} while **521** was obtained from a strain of the order Xylariales and also synthesised.²⁹² A number of syntheses of fungal metabolites have resulted in structural revisions of the natural products, including syntheses of (–)-protubonine A **522**, (–)-protubonine B **523** (*Aspergillus* sp.)^{293,294} and (+)-cristatumin C **524** (*Eurotium cristatum*).^{295,296} Synthesis of the racemate of oxalicumone C (*Penicillium oxalicum*)²⁹⁷ followed by resolution by chiral HPLC and examination of experimental and calculated ECD data, resulted in the configuration of the natural product being assigned as (S)-**525**.²⁹⁸ Culture of a strain of *Aspergillus clavatus* isolated from the hydrothermal vent crab *Xenograpsus testudinatus* in the presence of the abiotic stress agent and hydrothermal vent fluid component zinc (as zinc sulfate), elicited production of a known synthetic cyclic peptide²⁹⁹ that was isolated for the first time from a natural source and named as clavatuside C **526**. The fungus did not produce clavatuside C **526** when cultured in the absence of zinc.³⁰⁰ Pericosine E³⁰¹ (*Periconia byssoides*) occurs as an enantiomeric mixture in nature and synthesis of the preferred enantiomer (–)-pericosine E **527** has been reported.³⁰² First syntheses of a number of fungal metabolites achieved include those of secalonic acids A³⁰³ (*Paecilomyces* sp.)³⁰⁴ and D³⁰⁵ (*Gliocladium* sp.)^{306,307} ($\pm$)-sorbiterrin A³⁰⁸ (*Penicillium terrestre*),³⁰⁹ (–)-auronamide C³¹⁰ (*Penicillium aurantiogriseum*),³¹¹ calcaripeptides A–C³¹² (*Calcarisporium* sp.),³¹³ (–)-aspergilazine A³¹⁴ (*Aspergillus taichungensis*),³¹⁵ aspirin³¹⁶ (*Aspergillus versicolor*),³¹⁷ cochliomycin B³¹⁸ (*Cochliobolus lunatus*),³¹⁹ dendroides³²⁰ (*Dendrodochium* sp.) A,³²¹ B³²² and E,³²² paecilocin³²³ (*Paecilomyces variotii*),³²⁴ penicimonoterpene³²⁵ (*Penicillium chrysogenum*)³²⁶ and (+)-penostatin E³²⁷ (*Penicillium* sp.).³²⁸ The benzaldehyde derivative isotetrahydro-auroglauin^{329,330} has been shown to exhibit anti-inflammatory activity, inhibiting the NF- κ B pathway through suppressing production of both pro-inflammatory mediators and cytokines.³³¹ Oxirapentyns A^{332,333} and B³³³ exhibited growth stimulatory effects on seedling roots of barley and wheat³³⁴ while iso-echinulin A^{335,336} was a strong inhibitor of settlement of larvae of the barnacle *B. amphitrite*.³³⁷ Methylpenicillin³³⁸ also displayed anti-inflammatory activity, suppressing expression of pro-inflammatory mediators through the NF- κ B and MAPK pathways.³³⁹ Heavy metal stress of two strains of hydrothermal vent fungi (*Aspergillus sclerotiorum* and *A. clavatus*) induced biosynthesis of

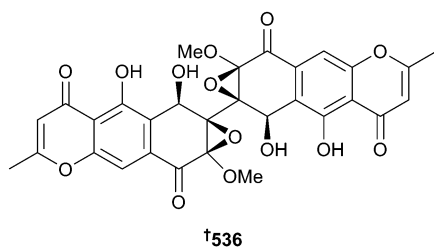
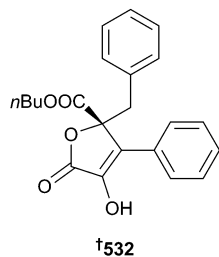
metabolites that were not produced under normal culture conditions: *A. sclerotiorum* produced aspochracin^{340,341} when stressed with copper and produced stephacidin A³⁴² and notoamides B³⁴² and F³⁴³ under normal culture conditions whilst *A. clavatus* produced the acetophenone derivative clavato^{344,345} under stress conditions and deoxytryptoquivaline³⁴⁶ and tryptoquivaline A³⁴⁶ in metal-free culture.³⁴⁷

3.4 Fungi from mangroves

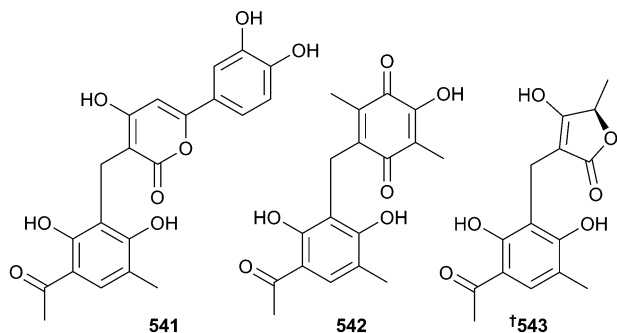
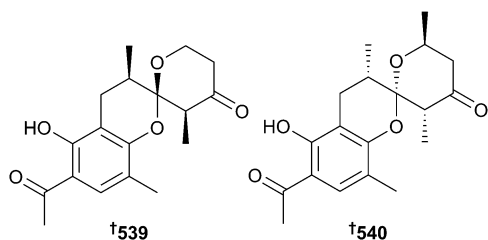
There has been a considerable increase in the number of new metabolites reported from mangrove-associated fungi (108 in 2014 vs. 75 in 2013), with the majority coming from endophytic species. Co-culture of *Alternaria* and *Phomopsis* species led to isolation of three cyclic peptides **528–530**, all of which exhibited significant activity against a range of plant pathogenic fungi,^{348,349} whilst co-culture of two brown alga (*Sargassum*)-derived *Aspergillus* species also produced a cyclic peptide, psychrophilin E **531**.³⁵⁰



Aspergillus flavipes was the source of the aromatic butyrolactones, flavipessin A **532** and B **533** and the previously synthesised **534**³⁵¹ and **535**,³⁵² of which flavipessin A **532** exhibited moderate to good antibacterial activity. Unlike penicillin, it was able to penetrate the biofilm matrix to kill live bacteria inside mature *Staphylococcus aureus* biofilm.³⁵³ An endophytic *Diaporthe* sp. was the source of diaporine **536**, an unprecedented symmetric polyketide which induces conversion of tumour associated macrophages from the M2 to the M1 phenotype in both cellular and animal models.³⁵⁴

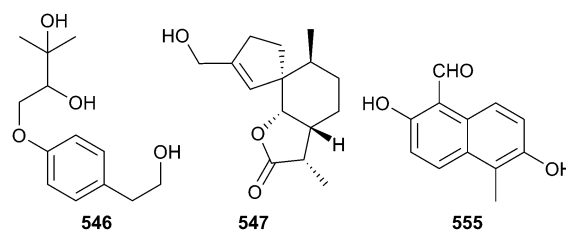


The polyketides dothiorelone F **537** and I **538** were obtained from endophytic *Dothiorella* sp. along with three known analogues.³⁵⁵ Of these analogues, dothiorelone G³⁵⁶ is actually the same as the previously reported cytosporone R,³⁵⁷ also obtained from a mangrove associated species (*Leucostoma persoonii*). Peniphenones A–D **539**–**543** were obtained from *Penicillium dipodomycicola*. Of these, peniphenone A **539**, **540** occurs as a racemate and was separated into its enantiomers by chiral HPLC while peniphenones B **541** and C **542** strongly



inhibited *Mycobacterium tuberculosis* protein tyrosine phosphatase B (MptpB).³⁵⁸

The polyketides **544** and **545** were obtained from co-culture of mangrove soil derived *Penicillium* sp. with the sediment derived bacterium *Streptomyces fradiae* and were not produced in discrete bacterial and fungal control cultures, suggesting the activation of silent gene clusters by co-cultivation. These also occurred as a racemate and were separated by chiral chromatography into **544**³⁵⁹ a known terrestrial fungal metabolite (9*R*,14*S*)-epoxy-11-deoxyfunicone, but now isolated as a first-time MNP, and the enantiomer (9*S*,14*R*)-epoxy-11-deoxyfunicone **545**.³⁶⁰ An endophytic *Penicillium* sp. was the source of a phenyl ether derivative **546** and a spiroax-4-ene-12-one derivative **547** with the spiroax-4-ene-12-one derivative **547** more potent to the MG-63 cell line with *in vivo* activity and significant inhibition of human osteosarcoma in nude mice upon oral administration.³⁶¹ The prenylated phenols vaccinol A–G, **548**–**554** and the naphthalene derivative vaccinal A **555** were isolated from *Pestalotiopsis vaccinii*. Vaccinal A **555** exhibited potent COX-2 inhibition.³⁶² Other genera or families of fungi associated with mangroves to yield the new metabolites **556**–**635** were *Acremonium*, *Alternaria*, *Daldinia*, *Guignardia*, *Penicillium*, *Pestalotiopsis*, *Phoma*, *Phomopsis*, *Pseudolagarobasidium*, *Rhytidhysterium*, *Stemphylium* and *Xylariaceae*.^{363–388}

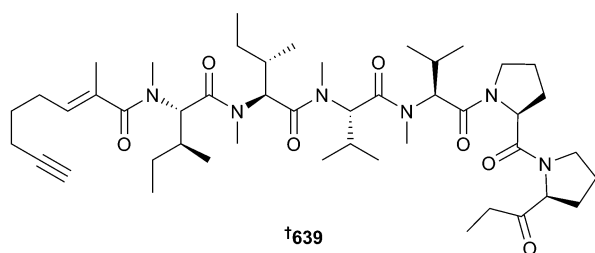
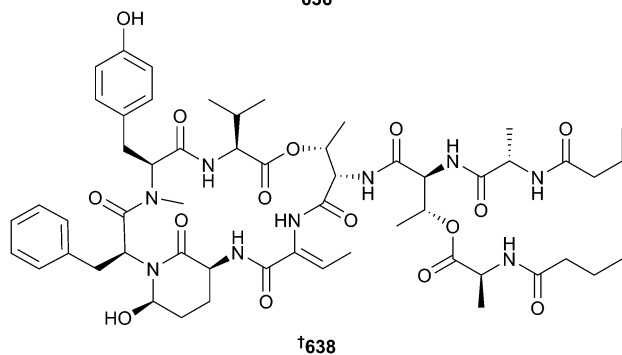
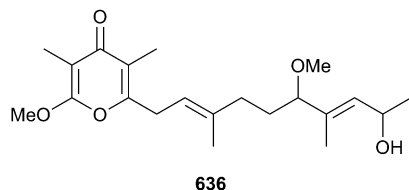


3.5 Cyanobacteria

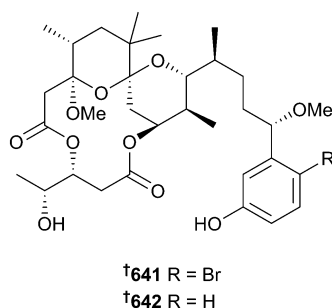
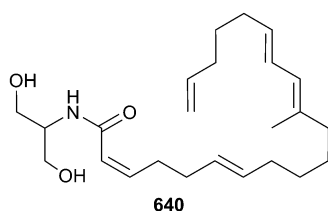
There has been an increase in the number of new metabolites reported from cyanobacteria since 2013, but the total numbers are still low overall (20 in 2014 vs. 9 in 2013), seemingly continuing an overall downward trend. Typical of the phylum, the vast majority of metabolites reported were peptidic in nature. Although not peptidic, yoshinone A **636** and the diastereoisomers yoshinone B1 and B2 **637** were isolated from *Leptolyngbya* sp. Yoshinone A **636** inhibited differentiation of 3T3-L1 cells into adipocytes without accompanying cytotoxicity suggesting potential as an anti-obesity lead.³⁸⁹ A cyanobacterial assemblage, consisting mostly of *Lyngbya* sp. (now renamed as *Moorea* sp.) yielded the dolastatin 13 analogue, kurahamide **638**, a strong inhibitor of the proteases elastase and chymotrypsin³⁹⁰ and the acetylenic lipopeptide kurahyne **639** an inhibitor of the HeLa cell line and inducer of apoptosis.³⁹¹

Mooreamide A **640** is a cannabinomimetic lipid obtained from *Moorea bouillonii* and is the most potent marine-derived inhibitor of the neuroreceptor CB₁ reported to date.³⁹² Two new aplysiatoxin analogues, 3-methoxyaplysiatoxin **641** and 3-methoxydebromoaplysiatoxin **642** were isolated from *Trichodesmium erythraeum* and **642**, along with the co-isolated known debromo analogues debromoaplysiatoxin³⁹³ and





anhydrobromoplysiatoxin,³⁹⁴ were inhibitors of the Chikungunya virus.³⁹⁵



Other new metabolites **643–655** were obtained from the genera *Anabaena*, *Lyngbya*, *Moorea*, *Oscillatoria* and *Symploca*.^{396–402} The absolute configurations of coibacins A and B⁴⁰³ have been determined as **656** and **657** respectively by total synthesis.⁴⁰⁴ Syntheses of biselyngbyolide A⁴⁰⁵ (*Lyngbya* (*Moorea*) sp.),⁴⁰⁶ apratoxin C⁴⁰⁷ (*Lyngbya* (*Moorea*) sp.),⁴⁰⁸ malevamide D⁴⁰⁹ (*Symploca hydroides*),⁴¹⁰ micromide⁴¹¹ (*Symploca* sp.),⁴¹² and 12-*epi*-hapalindole Q isonitrile⁴¹³ (*Hapalosiphon laingii*),⁴¹⁴ have also been achieved. Total syntheses of the proposed structures

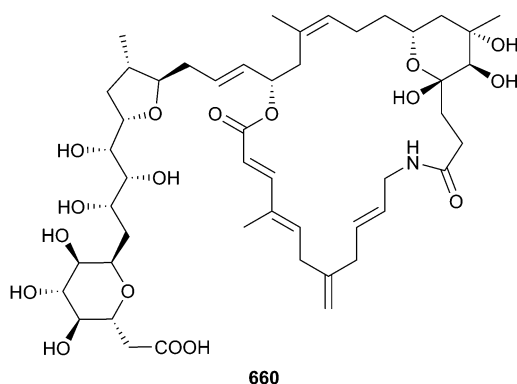
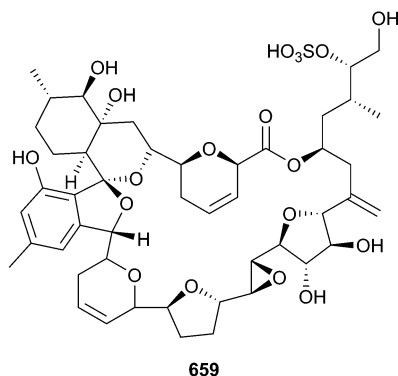
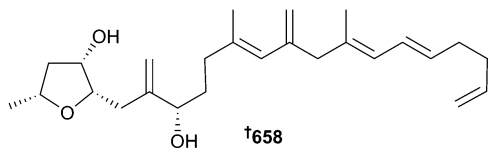
for itralamide B⁴¹⁵ and coibamide A⁴¹⁶ have indicated that the structures of these natural products require revision.^{417,418} Grassypeptolides A–C⁴¹⁹ were shown to selectively inhibit the dipeptidyl peptidase (DPP8) protease and molecular docking studies indicated that grassypeptolide A binds to the enzyme at two different sites.⁴²⁰ Gallinamide A⁴²¹ was shown to be a potent and selective inhibitor of the human cysteine protease cathepsin L.⁴²² A genome mining approach was utilised to identify three proteusin rSAM epimerases, enzymes which install multiple D-amino acids in genetically encoded peptide chains, from three strains of cyanobacteria including a marine-derived *Pleurocapsa* strain.⁴²³ A genome mining approach was also used to show that LanA peptides, linear precursor peptides of lanthionine-containing peptides (lanthipeptides),⁴²⁴ are highly diverse among different systems and that closely related lanthipeptide synthetases can be associated with quite different substrate sets.⁴²⁵

3.6 Dinoflagellates

The number of new metabolites reported from dinoflagellates has remained about the same as for 2013 with 19 compounds reported in 2014. The linear polyketide, amphirionin-4 **658**, from an *Amphidinium* species displayed very potent and selective growth promotion activity on murine bone marrow stromal ST-2 cells. Feeding experiments with ¹³C single and double labelled acetate indicated that polyketide **658** comprises a linear C22 chain with two irregular C1 sites (in the tetrahydrofuran moiety) and four C1 branches.⁴²⁶ *Dinophysis acuminata* was the source of the macrolide acuminolide A **659** which was non-toxic to human tumour cell lines (HTCLs) but a potent stimulator of actomyosin ATPase activity.⁴²⁷ Belizentrin **660**, a 25-membered macrolactam obtained from *Prorocentrum belizeanum* is the first member of its class of polyunsaturated and polyhydroxylated macrocycles and displayed potent effects on neuronal network integrity in cerebellar cells, ultimately resulting in cell death.⁴²⁸

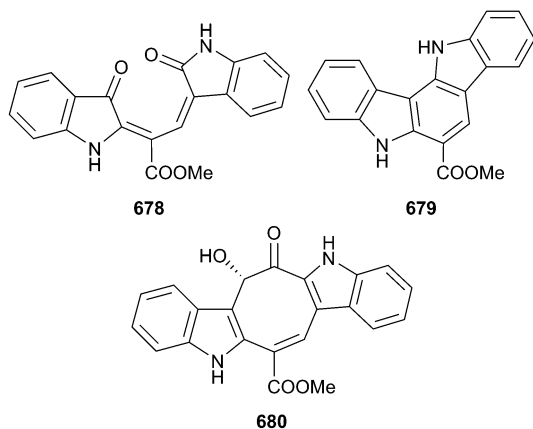
Other genera of dinoflagellates from which the new metabolites **661–675** were isolated include *Amphidinium*, *Azadinium*, *Karenia* and *Vulcanodinium*.^{429–436} Tentative structures were assigned to ovatoxin-g **676** and an isomer of palytoxin, the so-called isobaric palytoxin.⁴³⁷ The absolute configuration of amphidinol A⁴³⁸ was determined as **677**.⁴³⁹ Synthesis of genetically predicted saxitoxin intermediates with identification and quantification in the dinoflagellate *Alexandrium tamarense* and the cyanobacterium *Anabaena cicalis* supports the genetically proposed biosynthetic route⁴⁴⁰ to saxitoxin.⁴⁴¹ Metabolomic and proteomic analyses indicated that *Karenia brevis* exhibited allelopathy against two competitor diatoms *Asterionellopsis glacialis* and *Thalassiosira pseudonana*. The former, which co-occurs with *K. brevis*, exhibited somewhat more robust metabolism while in the latter, energy metabolism was disrupted and cellular protection mechanisms were impeded.⁴⁴² Other studies examined the biosynthesis of brevetoxin,^{443,444} brevisamide^{445,446} and the genetics of toxin production in *Alexandrium catenella*.⁴⁴⁷





4 Green algae

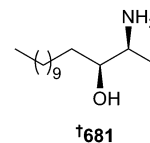
Research into green algal chemistry continues at low ebb with just 13 relevant articles published and just three new compounds published for 2014. The new compounds were the racemosines A–C **678–680**,^{448,449} bisindole alkaloids isolated from *Caulerpa racemosa* which are biosynthetically related to the well-established green algal metabolite caulerpin, also from a *Caulerpa* sp.⁴⁵⁰ Caulerpin featured in studies of anti-nociception mechanisms⁴⁵¹ and the antituberculosis activities of caulerpin and synthetic analogues.⁴⁵² Included in the green



algal literature for 2014 was a study on the effect of natural lycopene (*E/Z* mixture) on a human prostate cancer cell line,⁴⁵³ while a thought-provoking paper tackled a problem all marine natural product chemists should be alert to – the misuse of taxonomic descriptors and the implications that this has. Misuse of such descriptors appears to be a particular problem with marine algae.⁴⁵⁴

5 Brown algae

The output of new compounds (**17**) from brown algae in 2014 was again relatively low and although dominated by terpenoid chemistry saw the emergence of a new class of brown algal metabolite. This was the characterisation of a 1-deoxy-sphingoid, 3-*epi*-xestoaminol C **681**, from a New Zealand collection of *Xiphophora chondrophylla* following a *M. tuberculosis*-guided fractionation. A genome-wide screening against a library of non-essential gene deletion mutants of *Saccharomyces cerevisiae* established the cellular processes that **681** disrupted.⁴⁵⁵

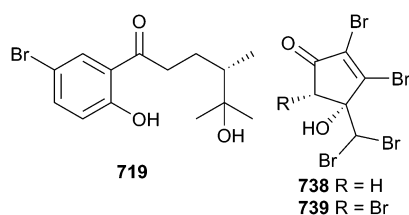


Some 27 diterpenoids ranging across six classes were isolated from a Chinese collection of *Dictyota plectens*. These included seven new diterpenes, belonging to the dolabellanes **682–684**, prenylated guaianes **685–687**, and a xenicane **688**, 19 known analogues and an ethoxylated artifact as well.⁴⁵⁶ Three new diterpenoids, a dolabellane **689** a xenicane **690**, and a prenylated guaiane **691** with five previously characterised compounds were characterised from Mediterranean collections of several *Dictyota* spp.⁴⁵⁷ Three new, **692–694**, and three known dolabellane diterpenoids,^{458,459} were isolated from a Brazilian *D. pfaffi*, and a single crystal X-ray analysis established the absolute configuration of the known 10,18-diacetoxy-8-hydroxy-2,6-dolabelladiene.^{458,460} The meroterpenoids sargachromanol Q **695** and R **696** along with the known sargachromanol J⁴⁶¹ resulted from re-examination of the original extract from *Sargassum siliquastrum*,⁴⁶² while thunberol, **697**, is the only new sterol of five isolated from the Chinese *Sargassum thunbergii*.⁴⁶³ Typical brown algal phlorotannins such as eckol and dieckol have stimulated research into a wide range of topics – heme oxygenase-1 expression,⁴⁶⁴ oxidative stress,⁴⁶⁵ anti-adipogenic activity,⁴⁶⁶ anti-HIV-1⁴⁶⁷ and antibacterial activity.⁴⁶⁸ Among the studies on the anti-inflammatory properties of phlorotannins^{469,470} was the synthesis of a rhodamine-labelled dieckol. Confocal laser microscopy determined that the labeled dieckol was mainly located in the endoplasmic reticulum and studies showed that the anti-inflammatory activity of the conjugate was considerably greater than that of dieckol itself.⁴⁷¹ The potential therapeutic properties of fucoxanthin and derivatives have been studied,^{472,473} as have those of polyphenols such as octaphlorethol,^{474–476} and mono- and diacylglycerols.⁴⁷⁷



6 Red algae

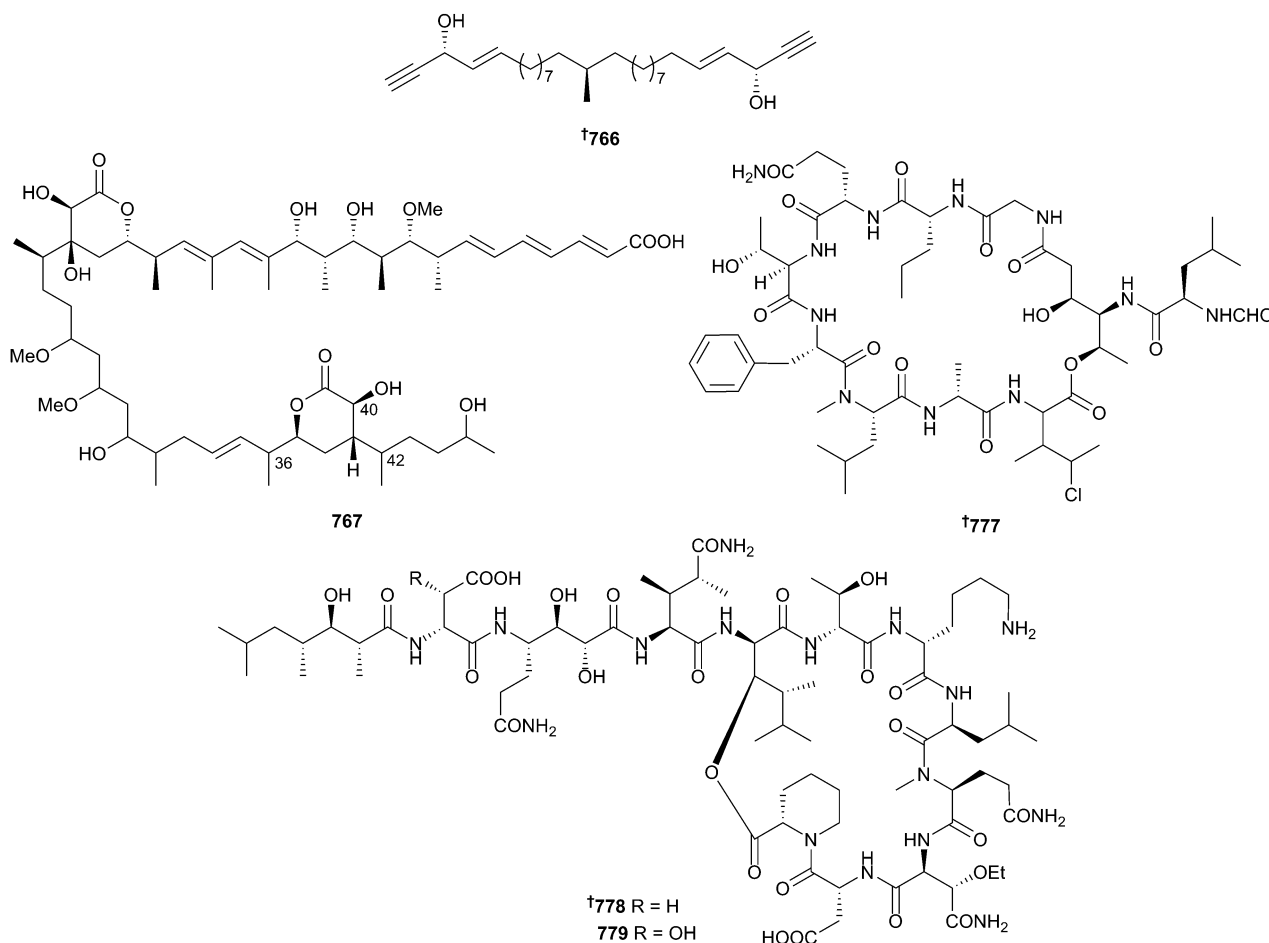
There continues to be a marked variation in the number of new compounds obtained from red algae and reported each year, with 42 for 2014 compared to 9 for 2013. There was the usual range of structural types with glycolipids **698**⁴⁷⁸ and **699–701**,⁴⁷⁹ halogenated allenes and alkynes **702–704**,⁴⁸⁰ **705–709**,⁴⁸¹ **710**⁴⁸² and **711**,⁴⁸² halogenated monoterpenes **712–718**,⁴⁸³ sesquiterpenes **719–725**,⁴⁸⁴ **726**,⁴⁸⁵ **727**,⁴⁸⁵ and **728–730**,⁴⁸⁶ (including the unusual *seco*-laurokamurone **719**), diterpenes **731–733**,⁴⁸⁷ brominated aromatics **734**⁴⁸⁸ and **735–737**⁴⁸⁹ and the mahoronones **738–739**, unusual 2,3-dibrominated 2-cyclopentenones.⁴⁹⁰ The use of rapid dereplication tools (UHPLC–PDA–HRMS, 2D HSQC NMR, software tools and databases) for identification of the allenes **710** and **711**⁴⁸² and LC–UV–MS–SPE–NMR for the monoterpenes **712–718**⁴⁸³ were notable features.



A synthesis⁴⁹¹ of the brominated sesquiterpene aldingenin C⁴⁹² showed that the original structure was incorrect, and it was suggested that aldingenin C was probably the known compound caespitol.⁴⁹³ A synthesis⁴⁹⁴ of the proposed structure of prevezol B⁴⁹⁵ has shown that the original structure was incorrect. Of the ~70 polyhalogenated acyclic monoterpenes isolated from *Plocamium* spp., surprisingly none had been synthesised until the use of a broadly applicable approach generated four of the naturally occurring compounds and a number of analogues.⁴⁹⁶ An asymmetric total synthesis of (+)-bermudenynol⁴⁹⁷ has been accomplished,⁴⁹⁸ while a total synthesis of the snyderane (+)-luzofuran⁴⁹⁹ has also been made.⁵⁰⁰ The anti-inflammatory potential of 5 β -hydroxypalisadin B⁵⁰¹ from *Laurencia snackeyi* has been demonstrated in LPS-stimulated RAW 264.7 macrophages⁵⁰² and LPS-induced zebrafish embryo.⁵⁰³

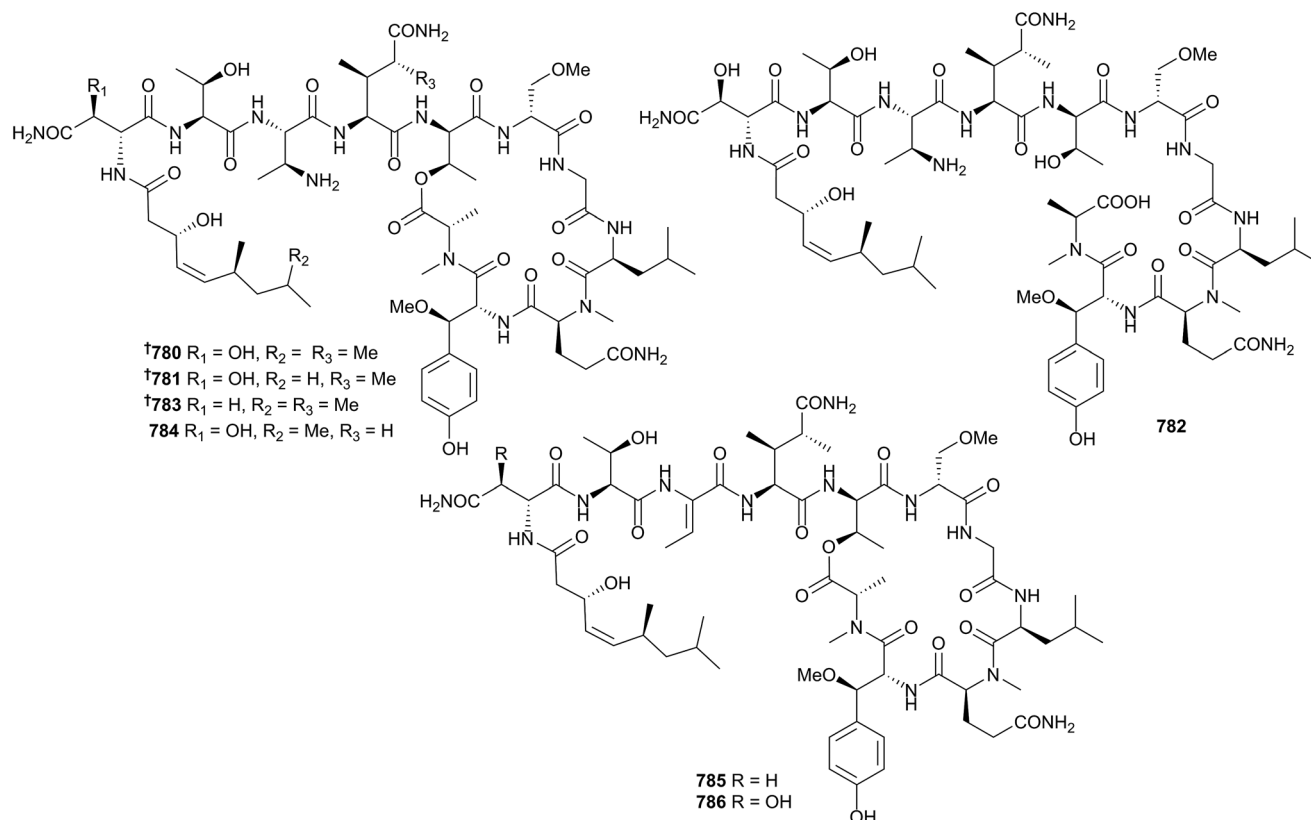
7 Sponges

With 283 new structures reported from the phylum Porifera in 2014, sponges have returned again to be a dominant source of new bioactive metabolites, although the growing realisation that microbial symbionts are the real producers of “sponge” specialised metabolites highlights the need for more detailed metagenomic and biosynthetic analyses of sponge matrices.



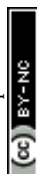
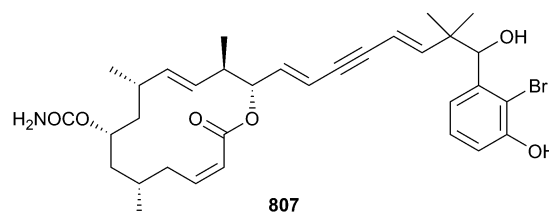
Sphingoids **740**⁵⁰⁴ and **741**,⁵⁰⁵ taurinated fatty acids **742–745**^{506,507} and a large number of polyacetylenes **746–765**^{508–513} have been reported from the genera *Callispongia*, *Coscinoderma*, *Echinoclathria*, *Petrosia*, *Placospongia*, *Siphonochalina* and *Xestospongia*. The synthesis of a series of miyakosyne A (*Petrosia* sp.)⁵¹⁴ diastereomers followed by RP-HPLC separation at -56°C has revealed that the natural product **766** is actually a mixture of two stereoisomers in $\sim 96:4$ (14*R*):(14*S*) ratio.⁵¹⁵ A comprehensive blend of synthesis with NMR, IR and vibrational

The stellatolides A–G **780–786** are a family of cyclopeptides from a Madagascan *Ecionemia acervus*. The full stereochemical analyses of several variants were established by Marfey's analysis, and the solid-phase peptide total synthesis of **780** was also achieved. The unexplained racemisation of L-Thr during the Marfey's analysis is a salient warning to all experimentalists using this technique. Several of the stellatolides were cytotoxic in the nM range against three HTCLs.⁵²⁵



circular dichroism (VCD) spectroscopy has allowed for the relative configurational assignment of the C-36–C-42 segment of hemicalide **767** (*Hemimyscale* sp.), as well as securing the absolute configuration of C-42. This potent (sub-nM) inhibitor of mitosis was sourced from a deep water sponge collected at the Torres Islands, Vanuatu, but to date has only been reported in a patent.^{516,517} A series of peroxides **768–773**,⁵¹⁸ halogenated alkenes **774** and **775**,⁵¹⁹ and an amide **776**⁵²⁰ have been reported from *Plakortis simplex*, *Dysidea* sp. and *Anoxycalyx* (*Scolymastra*) *joubini*, respectively. Sponges continue to be a prolific source of peptide natural products. Reisolation of cyclolithistide A from a deep sea *Discodermia japonica* (~ 200 m, Sagami Bay, Japan), originally reported in 1998,⁵²¹ allowed in-depth LC-MS/MS and Marfey's analyses that necessitated a revision of the amino acid sequence and configurations as shown here **777**.⁵²² The simple substitution of a side-chain Asp for hydroxy-Asp in pipicolidepsins A **778** and B **779** (*Homophymia lamellosa*, Saint Marie Is., Madagascar) resulted in a greater than ten-fold increase in activity against three HTCLs.⁵²³ The synthesis of **778** had already been reported in 2013.⁵²⁴

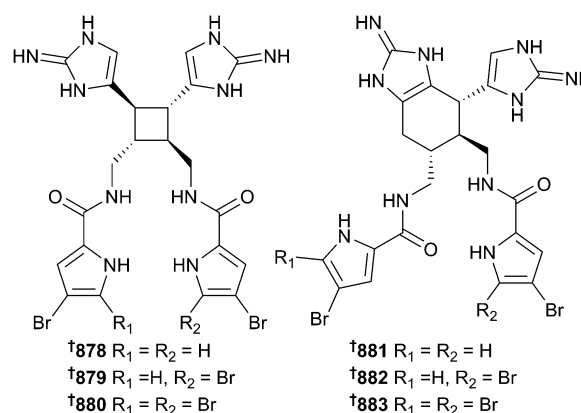
The genera *Asteropus*, *Discodermia*, *Pipestela*, *Reniochalina*, *Stylissa*, *Suberites* and *Theonella* have also yielded a large number of peptides **787–806**.^{526–532} Callispongolide **807** is an unusual carbamate macrolide with an unprecedented conjugated dienyne side chain isolated from *Callispongia* sp. (Ambon, Indonesia). Evaluation of **807** against three HTCLs indicated potent cytotoxicity ($\text{IC}_{50} = 60\text{--}320$ nM). Notably, the viability of cell lines treated with callispongolide was not affected by QVD-OPh, a known caspase-inhibitor, suggesting the test compound induces cellular toxicity in a caspase-independent manner.⁵³³



N[C@@H](Cc1c[nH]c2ccccc12)C(=O)O

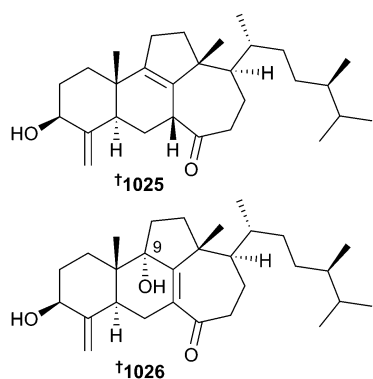
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inhibition of both Gram positive and negative bacteria (MIC 0.5–8 $\mu\text{g mL}^{-1}$ vs. eight bacterial cell lines), even though the initial isolation study reported minimal activity. Further detailed investigation could not determine a specific mode of action but did suggest secondary membrane-disruption as the likely route to activity.⁵⁶³ The comprehensive total synthesis of sceptrin, ageliferin and massadine congeners *via* single electron cycloaddition reactions has several wide-ranging consequences. Firstly, it necessitates the revision of the absolute configuration of sceptrin and its brominated analogues **878–880**^{564–566} and ageliferin and congeners **881–883**⁵⁶⁷ as shown here, based upon X-ray crystal structures. Moreover, the massadines had been thought to be rearrangement products from the ageliferins,^{568,569} however the absolute configurations of the two series of compounds are antipodal, hence indicating enantiodivergent biosynthetic pathways *in vivo* to these bromopyrrole alkaloids and invalidating the so-called “Scheuer-hypothesis” for their formation.^{570,571}

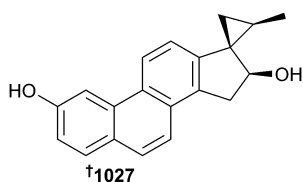


Chemical structures of compounds 931 and 932 are shown. Compound 931 is a complex polycyclic molecule with a central benzene ring fused to a cyclohexene ring, which is further fused to a cyclohexane ring. It has a carboxylic acid group, a hydroxyl group, and a methyl group. Compound 932 is a similar polycyclic molecule, but with a sulfonamide group instead of a carboxylic acid group.

A merotetraterpenoid **933** has been reported from *Sarco-tragus spinosulus*.⁵⁸⁶ Similarly, *Axinyssa*, *Dysidea*, *Ircinia* and *Topsentia* sponges were the sources of a number of sesquiterpenoids **934–964**,^{587–591} some of which had been cleaved, while diterpenoids were obtained from *Diacarnus megaspinorhabdosa* **965–970**,⁵⁹² *Spongia* sp. **971–973**⁵⁹³ and *Darwinella oxeata* **974–982**.⁵⁹⁴ A large number of sesterterpenoids **983–1011** were reported in 2014 from the sponge genera *Clathria*, *Coscinoderma*, *Hippospongia*, *Hyrtios*, *Petrosaspongia*, *Phorbas* and *Scalarispongia*.^{506,595–601} As always, sponges yielded a variety of steroids **1012–1019**,^{504,505,602,603} steroidal amines **1020** and **1021**,⁶⁰⁴ and degraded steroids **1022–1024**.^{605,606} from species of *Echinoclathria*, *Corticium*, *Haliclona*, *Ircinia*, *Petrosia*, *Plakortis* and *Theonella*. *T. swinhoei* (Xisha Is., S. China Sea) yielded swinhoeisterols A **1025** and B **1026** with unprecedented 6/6/5/7 tetracyclic frameworks. The absolute configurations of the compounds were secured by a combination of X-ray diffraction, TDDFT ECD calculations and Mosher's method. Detailed *in silico* analysis of both compounds against 211 potential protein targets of relevance for cancer therapy revealed **1025** should exhibit activity against the histone acetyltransferase (h)p300. This was validated *in vitro* with $IC_{50} = 2.9 \mu M$ while **1026** was almost 100 times less potent ($IC_{50} = 240 \mu M$), indicating the steric importance of the 9-OH in abrogating activity.⁶⁰⁷



Cinanthrenol A **1027** is the first example of a phenanthrene-containing steroid. It was sourced from *Cinachyrella* sp. collected by dredging at -160 m (Oshima-Shinson, Japan) where the unique phenanthrene-fluorescence signature was used to select the sponge from amongst detritus. As well as moderate activity against various HTCLs, **1027** was a potent estrogen-receptor binder, displacing estradiol in a competitive manner with $IC_{50} = 10$ nM, as well as altering estrogen-responsive gene expression.⁶⁰⁸



A series of triterpenoids **1028–1032** have been reported from *Jaspis stellifera*⁶⁰⁹ and *Siphonochalina siphonella*.⁶¹⁰ An artificial “sponge” has been developed for the non-destructive

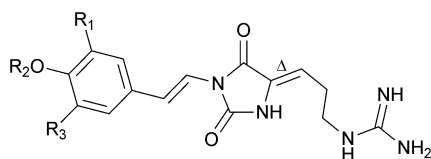
concentration of marine natural products in sensitive marine ecosystems. A pump is used to suction sponge-matrix particles that are filtered into a hollow-fibre bioreactor. This is used to cultivate microbial symbionts that produce secondary metabolites which are, in turn, trapped on reversed-phase cartridges for concentration. The artificial sponge system was used in three expeditions across 11 locations, two in the Pacific and one in the South China Sea, with the deployment at Pulau Lakei (Sarawak, Malaysia) yielding jasplakinolide, jasplakinolide B and C in a 18 : 1 : 1 ratio, the same proportions as found from *Jaspis splendens* collected near Vanuatu.^{611,612} Metagenomic analyses continue to rise in their importance to sponge natural product biosyntheses. Detailed metagenomic profiling of the “High Microbial Abundance” (38–57% microbial biomass) sponge *Plakortis halichondrioides* (Little San Salvador Is., Caribbean Sea) found characteristic gene sequences of poribacteria (a bacterial phylum found exclusively in sponges) but the known *supA* and *swfA* biosynthetic clusters observed were absent from the poribacteria genomes. Further investigation showed similarity between these clusters and protist type I polyketide synthetase (PKS) genes hence protists could be a previously overlooked reservoir of novel bioactive metabolites.⁶¹³ A comprehensive metagenomic survey of the microbial community associated with the metabolite-rich sponge *Theonella swinhoei* has uncovered a new bacterial genus, *Entotheonella*. This genus is a member of the new candidate phylum Tectomicrobia and appears to be a widespread sponge endosymbiont across many diverse sponge species. Moreover, biosynthetic gene cassettes suitable for production of many “*Theonella swinhoei*” NRPS and PKS compounds, including the onnamide,⁶¹⁴ polytheonamide,⁶¹⁵ keramamide⁶¹⁶ and cyclotheonamide⁶¹⁷ classes, have been identified in the microbial genome.^{8,9} A metagenomic approach identified the gene cassette for calyculin A (*Discodermia calyx*)²⁰ biosynthesis as being microbial in origin, as described in 3.1 Bacteria, and resulted in the identification of two diphosphate protoxins (see 1 and 2),^{21,22} again underscoring the importance of symbiotic microbial communities in the production of “sponge” metabolites. The microtubule-stabilising mode of action of laulimalide⁶¹⁸ and peloruside A⁶¹⁹ has been established through the use of high-resolution crystal structures of the compounds bound to tubulin,⁶²⁰ while aaptamine⁶²¹ has been shown to have protective effects against cisplatin-induced kidney damage,⁶²² and also prevents photoageing.⁶²³ A polybrominated diphenylether⁶²⁴ (*Dysidea granulosa*) has been shown to inhibit hepatitis C non-structural protein NS3 helicase, a validated antiviral target.⁶²⁵ The four bromotyrosine compounds ianthelliformisamine A–C (*Suberea ianthelliformis*)⁶²⁶ and spermatinamine (*Pseudoceratina* sp.)⁶²⁷ were selective and potent inhibitors of human carbonic anhydrase IX ($IC_{50} = 0.20–0.36 \mu M$), with implications for maintaining cellular pH homeostasis and hence application in a variety of disease states.⁶²⁸ The sesterterpenoid heteronemin (*Hyrtios* sp.)⁶²⁹ inhibited *trans*-activation response DNA-binding protein 43 kDa (TDP-43), a key factor of several neurodegenerative states. Heteronemin binds to TDP-43 ($k_i = 270$ nM) and altered the aggregation state and localisation of this important neurochemical target.⁶³⁰ The first total syntheses of an all-(*Z*) octadeca-pentene-3-one (*Callyspongia* sp.),^{631,632} strongylodiol G (*Strongylophora*



sp.),^{633,634} bitungolide B (*Theonella swinhoei*),^{635,636} plakortone L (*Plakortis clathrata*),^{637,638} epiplakinic acid F (*Plakinastrella* sp.),⁶³⁹ and its methyl ester (*Plakortis halichondrioides*),^{640,641} gracilioether F (*Plakinastrella mamilaris*),^{642,643} taumycins A and B (revised **1033** and **1034**, *Fascaplysinopsis* sp.),^{644,645} polydiscamides B–D (*Ircinia* sp.),^{646,647} callipeltin M (*Latrunculia* sp.),^{648,649} topsentolide A2 (revised **1035**, *Topsentia* sp.),^{650,651} penarolide sulfate A2 (*Penares* sp.),^{652,653} haliclorensin C (*Haliclona tulearensis*),^{654,655} nakinadine A (revised **1036**, *Amphimedon* sp.),^{656,657} thiaplakortone A (*Plakortis lita*),^{658,659} (+)-cylindradine A (*Axinella cylindratus*),^{660,661} ianthelli-formisamines A–C (*Suberea ianthelliformis*),^{626,662,663} tokaradine C (*Pseudoceratina purpurea*),^{663,664} (+)-hemifistularin 3 (revised **1037**, *Verongia* sp.),^{665,666} trachycladines A and B (*Trachycladus laevispirulifer*),^{667–669} cyclospongiaquinone-1 (*Stelospongia conulata*),^{670,671} deoxyspongiaquinol (revised **1038**) and deoxyspongiaquinone (revised **1039**, *Euryspongia* sp.),^{672,673} plakinamine B (*Plakina* sp.),^{674,675} and clionamine D (*Cliona celata*)^{676,677} have all been completed. The total syntheses of the putative structures of 15-oxopuuphehoic acid (*Hyrtios* sp.)^{678,679} and astakolactin (*Cacospongia scalaris*)^{680,681} have also been achieved but the spectroscopic data call into question the original identified structures, with no alternative suggestions provided.

8 Cnidarians

The number of new compounds reported from cnidarians in 2014 (201) is similar to the previous decadal average. The importance of chemical cues in coral reef remediation have been highlighted in a study that found that coral and fish juveniles were repelled from reefs that were overfished and seaweed-dominated.⁶⁸² A method of recovery of such degraded habitats was proposed to involve reduction in fishing harvest of critical species of herbivorous fishes. The chemistry of both hard and soft corals are dominated by terpenes – alkaloids are only rarely isolated. In 2014 there were seven examples of alkaloids isolated from soft corals, comprised of an unusual diketopiperazine **1040** from *Menella kanisa*,⁶⁸³ and six tetraprenylated purines, malonganenone L–Q **1041–1046**, from *Echinogorgia pseudossapo*.⁶⁸⁴ In contrast, cnidarians of the order Zoantharia (zoanthids) were the sources of five new parazoanthine congeners **1047–1051** (*Parazoanthus axinellae*).⁶⁸⁵ These structures were established using a metabolomics model, with LCMS/MS fragment analysis being used to define structures and LC-ECD employed to assign absolute configuration (to **1047**, **1048** and **1050**).



- †**1047** R₁ = R₃ = H, R₂ = Me, Δ saturated
 †**1048** R₁ = Br, R₂ = R₃ = H, Δ saturated
 †**1049** R₁ = Br, R₂ = R₃ = H
 †**1050** R₁ = R₃ = Br, R₂ = Me, Δ saturated
 †**1051** R₁ = R₃ = Br, R₂ = Me

Other zoanthamine analogues, **1052–1058**, were isolated from *Zoanthus* sp.^{686,687} and *Zoanthus kuroshio*⁶⁸⁸ as well as a new

42-hydroxypalytoxin diastereomer **1059** from *Palythoa tuberculosa*.⁶⁸⁹ Previous studies of an extract of *Palythoa toxica* identified the major toxin to be a 42-hydroxy analogue of palytoxin **1060** however the configuration at positions C-41 and C-42 remained unresolved.⁶⁹⁰ More recent J-based analysis has defined the configuration at these stereocentres as (41*S*) and (42*S*). The same study also established the absolute configuration of a related palytoxin analogue isolated from *P. tuberculosa* as being (42*S*)-hydroxy-(50*S*)-palytoxin **1059**. The relative affinity of palytoxin, **1059** and **1060** towards a mouse anti-palytoxin monoclonal antibody identified palytoxin to have the most potent K_D (nM), while **1059** and **1060** were approximately one and three orders of magnitude less potent respectively. Similar relative levels of cytotoxicity towards keratinocytes were also observed, with palytoxin being the most potent. Palytoxin is known to be capable of disrupting mechanisms of cellular ion homeostasis: NMR studies have defined the C-25–C-33 and C-47–C-53 fragments of palytoxin as being involved with Ca²⁺ coordination.⁶⁹¹

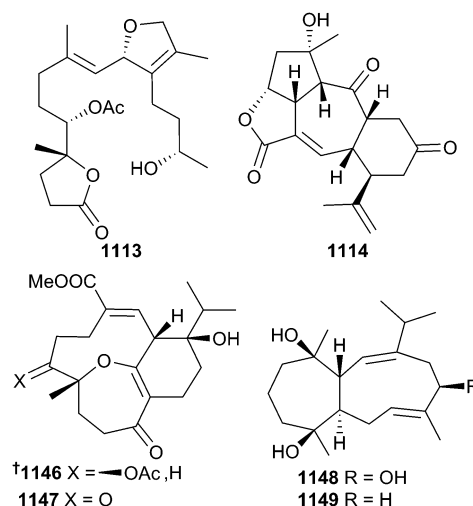
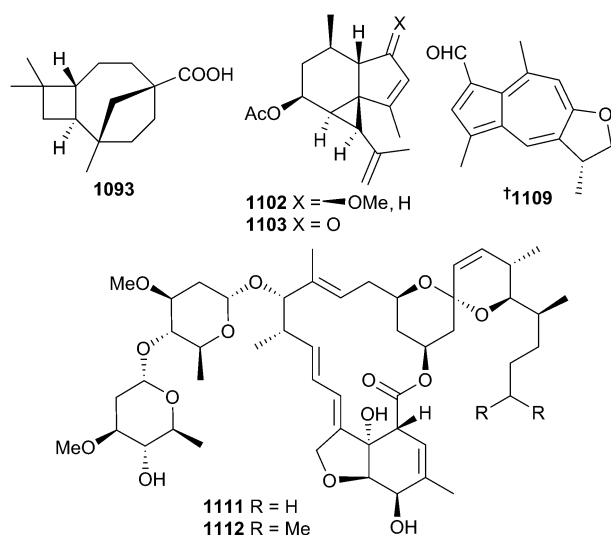
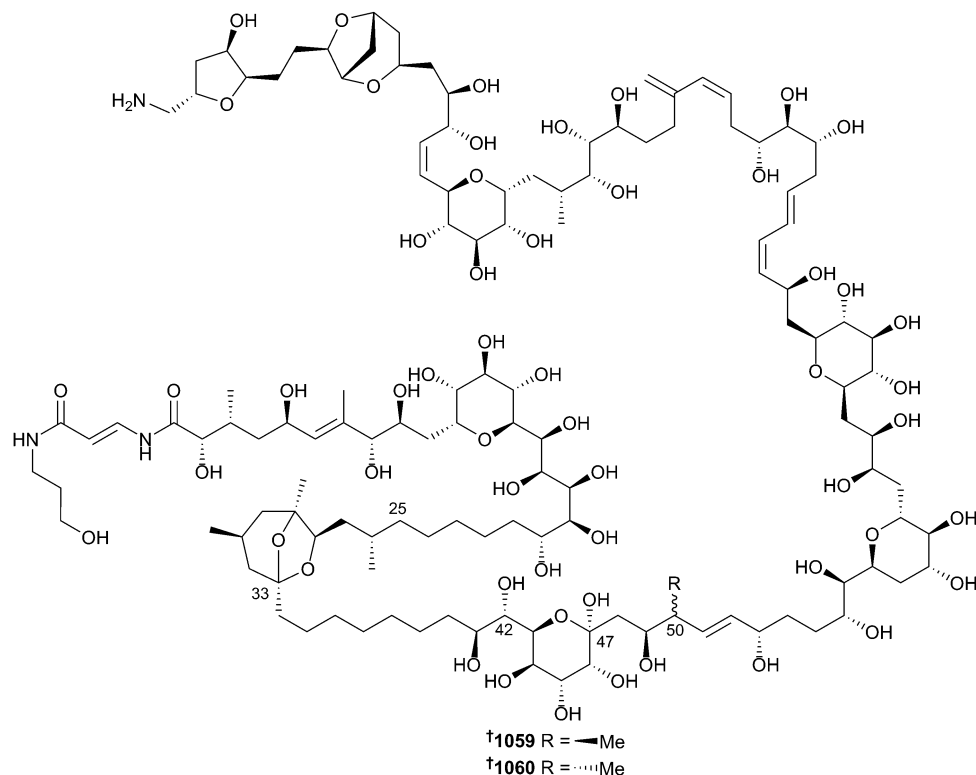
A series of butenolide and cyclopentenones **1061–1072** were reported from *Subergorgia suberosa* and *Sinularia* sp. soft corals.^{692–694} A comprehensive investigation of a Bahamian collection of *Pseudopterogorgia rigida* identified a chamigrane **1073** and seven bisabolanes **1074–1079** as new sesquiterpenes.⁶⁹⁵ The study also reported an extensive number of co-metabolites **1080–1083** that had been previously reported from terrestrial plants or as semi-synthetic derivatives **1084–1090**. Other sesquiterpenes **1091–1110** have been reported from *Rumphella antipathies*,^{696–698} *Lemnalia philippinensis*,⁶⁹⁹ *Menella kanisa*,⁷⁰⁰ *Nephthea erecta*,⁷⁰¹ an unidentified gorgonian,⁷⁰² *Dendronephthya* sp.,⁷⁰³ *Sinularia kavarattensis*,⁷⁰⁴ *Echinogorgia saspapo reticulata*⁷⁰⁵ and *Anthrogorgia ochracea*.⁷⁰⁶ Of these sesquiterpenes, rumphellaic acid A **1093**⁶⁹⁷ contains a unique skeleton, while shagenes A **1102** and B **1103**⁷⁰² and ochracenoid A **1109**⁷⁰⁶ contain rarely reported skeletons. In the case of the ochracenoid A, absolute configuration was assigned by use of TDDFT calculated ECD data.

A South China Sea collection of *Anthrogorgia caerulea* yielded, in addition to two known avermectin macrolides, avermectin B_{2a} and 22,23-dihydroavermectin A_{1a},⁷⁰⁷ two new congeners B_{1c} **1111** and B_{1e} **1112**.⁷⁰⁸ All four MNPs exhibited moderate antifouling activities.

Of the thirty-six cembrane related metabolites **1113–1149** reported from cnidarians in 2014,^{709–722} secocrassumol **1113** (*Lobophytum crassum*)⁷⁰⁹ is notable as it is derived from the cembrane skeleton via an unusual C-11–C-12 bond cleavage, sinugrosanolide A **1114** (*Sinularia gyrosa*) is an unprecedented C-4 norcembranoid,⁷¹⁰ tortuosenes A **1146** and B **1147** (*Sarcophyton tortuosum*)⁷²¹ represent the first examples of cembranoids containing a C-2–C-20 ring closure, and sinularbols A **1148** and B **1149** (*Sinularia arborea*) contain a rare C-3–C-9 ring closure.⁷²²

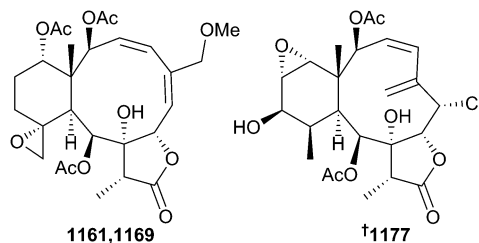
Cnidarian chemistry is dominated by metabolites derived from terpene biosynthesis, with particularly large numbers falling into the diterpenoid classification. Thirty-eight new briarane-skeletoned MNPs (**1150**, gemmacolides AS–AY **1151–1157**, briarenolide J **1158**, junceollolides M–P **1159–1162**, fragilisins A–L **1163–1174**, dollfusilins A **1175** and B **1176**, briaviolides A–J **1177–1186** and anthrogonoid A **1187**) were reported





from octocoral species *Pennatula aculeata*, *Dichotella gemmacea*, *Junceella gemmacea*, *J. fragilis*, *Ellisella dollfusi*, *Briareum violacea*, and *Anthrogorgia caerulea*.^{723–730} As usually happens each year, there is duplication of structure/trivial name amongst a small number of MNPs. In the set of briaranes reported from *Junceella gemmacea* (South China Sea) and *J. fragilis* (also South China Sea), junceollolide O **1161**⁷²⁶ and fragilisinin G **1169**⁷²⁷ share the same assigned structure, however spectroscopic and photometric data reported for the two compounds are quite different clearly indicating that one of the structures is in need of revision. The structure and absolute configuration of briarviolide A **1177**⁷²⁹ was secured by single crystal X-ray analysis.

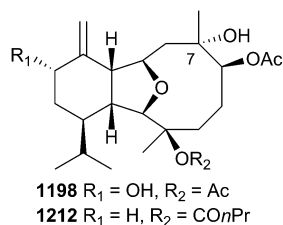
The second dominant class of diterpenoid metabolites reported from soft corals contain the eunicellin skeleton.



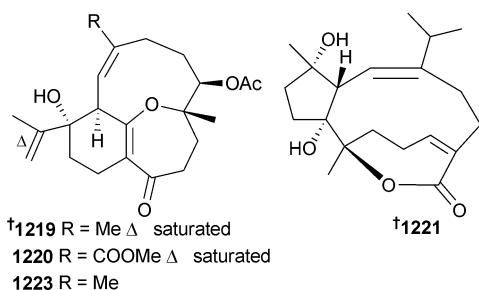
Examples of eunicellins were reported from *Anthrogorgia caerulea* (antsimplexin A, **1188**),⁷³⁰ *Muricella sibogae* (sibogins A **1189** and B **1190**),⁷³¹ *Cladiella* sp. (cladieuniceillin J, **1191**),⁷³² *Klyxum molle* (klymollins T–X, **1192–1196**),⁷³³ *Cladiella* sp. (cladieuniceillin M–Q, **1197–1201**),⁷³⁴ *Cladiella krempfi* (krempfielins N–R,



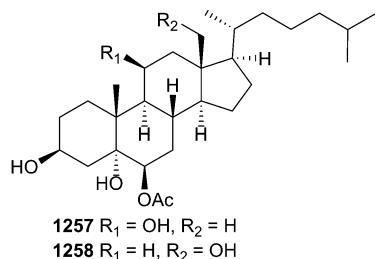
1202–1206),^{735,736} and *Cladiella hirsuta* (hirsutalin N-R, **1207–1211**).⁷³⁷ Readers should note that cladienicellin N **1198** is a structural duplicate of the previously reported *Klyxum molle* metabolite klymollin Q,⁷³⁸ exhibiting identical spectroscopic, spectrometric and chiroptical properties and that the structure elucidation of cladienicellin M–Q also led to revision of configuration at C-7 of the structure of lithophynin I diacetate to that shown in **1212**.



Further examples of diterpenoids, **1213** and **1214**,⁷³⁹ **1215** and **1216**,⁷⁴⁰ cespitulones A **1217** and B **1218**,⁷⁴¹ dihydrosarsolenone **1219**, methyl dihydrosarsolenoneate **1220**, and sarsolilides B **1221** and C **1222**,⁷⁴² have been isolated from soft corals of the genera *Cespitularia*, *Xenia*, and *Sarcophyton*. Structure elucidation and determination of absolute configuration of **1219** and **1220**⁷⁴² led the authors to propose a revision of the relative configuration at C-2 of the previously reported MNP sarsolenone (to that shown **1223**).⁷⁴³ Absolute configuration was assigned to **1219** and **1220** using TDDFT calculations of ECD data.



Sarcophyton ehrenbergi was the source of a number of prostaglandins including sarcoehrendins A–J **1224–1233**.⁷⁴⁴ In addition, three prostaglandins previously reported as synthetic compounds **1234–1236** were reported as natural products for the first time. Relatively potent activity was observed towards phosphodiesterase-4, a target for CNS, inflammatory and respiratory diseases. Soft corals also yielded a variety of steroids including pregnanes **1237** and **1238**,⁷⁴⁵ *seco*-sterols **1239–1244**,^{746,747} and hydroxylated/polyhydroxylated sterols **1245–**



1265^{747–753} from species of *Schleronephthya*, *Subergorgia*, *Sarcophyton*, *Sinularia*, *Verrucella*, *Echinogorgia*, and *Leptogorgia*. Noteworthy amongst these compounds, was the enhanced (synergistic) cytotoxicity observed for paclitaxel in the presence of punicinols A **1257** and B **1258** (*Leptogorgia punicea*) and that the two sterols were also able to inhibit A549 tumour cell growth in a clonogenic assay over a sustained period of ten days.⁷⁵²

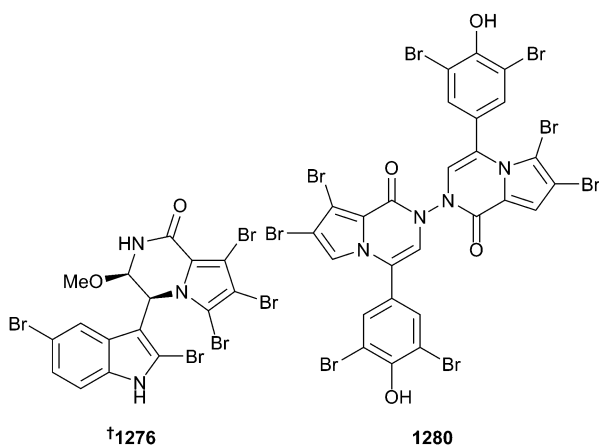
In addition to the mildly antiproliferative epoxyergosterols **1266–1271** (*Anthopleura midori*),⁷⁵⁴ investigation of MNP chemistry of sea anemones and hydrozoa has identified two new cytotoxins (3013 and 3375 Da) from the tentacles of the hydrozoan *Olindias sambaquiensis*⁷⁵⁵ and PhcrTx1, a 32 amino acid residue acid-sensing ion channel inhibiting peptide from the sea anemone *Phymanthus crucifer*⁷⁵⁶ that represents the first example of a peptide containing an Inhibitor Cysteine Knot scaffold to be isolated from a cnidarian. Further study of the biological activities of previously reported anemone toxins has identified Av3 (*Anemonia viridis*) to show specificity towards arthropod voltage-gated sodium channels by binding to one of the transmembrane clefts of the channel α -subunit,⁷⁵⁷ while AdE-1 (*Aiptasia diasphana*) prolongs cardiomyocyte action potential duration while lowering peak amplitude *via* slowing inactivation of sodium channels and enhancing the transient K⁺ current.⁷⁵⁸ Mutation of the pore-forming toxin sticholysin I (*Stichodactyla helianthus*) residue tryptophan 111 to cysteine reduced the toxins affinity for membranes by an order of magnitude,⁷⁵⁹ cysteine mutants of phenylalanine 15 or arginine 52 did not alter pore-forming activity but did protect the toxin from peroxynitrite oxidative damage,⁷⁶⁰ while a third study, using monolayers of phosphatidylcholine and sphingomyelin, determined that the toxin preferentially binds and penetrates membranes which have moderate enrichment in sphingomyelin and membrane fluidity.⁷⁶¹ The first total syntheses of alkaloids *N*-(3-guanidinopropyl)-2-(4-hydroxyphenyl)-2-oxoacetamide (*Campanularia* sp.)^{762,763} and ($\pm$)-tubastrindole B (*Tubastraea* sp.),^{764,765} butenolide (+)-hydroxancepsenolide (*Pterogorgia anceps*),^{766,767} and diterpenes sandresolide B,⁷⁶⁸ amphilectolide⁷⁶⁹ and (+)-ileabethoxazole⁷⁷⁰ (*Pseudopterogorgia elisabethae*)^{771,772} have been completed. Conversion of bipinnatin J (*Pseudopterogorgia bipinnata*)⁷⁷³ to intricarene (*Pseudopterogorgia kallos*)⁷⁷⁴ *via* a photochemical pathway has been demonstrated,⁷⁷⁵ while a photochemical (*E/Z*) olefin isomerisation was a critical step in the total synthesis of the natural product analog 17-deoxyprovidencin.⁷⁷⁶ Clarification of the structure of the cladiellin diterpene sclerophytin F (*Sclerophyllum capitalis*)⁷⁷⁷ has been an ongoing issue, with Friedrich and Paquette in 2002 proposing the structure should be revised to be the (3*S*) diastereomer of sclerophyton A.⁷⁷⁸ Synthesis of this proposed revised structure gave a product whose spectroscopic data differed from those reported for the natural product, suggesting further investigation is needed to resolve this issue.⁷⁷⁹ A modular approach to a number of cladiellin MNPs using a gold-catalysed tandem reaction of 1,7-diynes has been reported.⁷⁸⁰ Hippuristanol (*Isis hippurris*)⁷⁸¹ analogues have been evaluated as inhibitors of eukaryotic translation initiation,⁷⁸² cembranoid and ergosterol MNPs from Vietnamese cnidarians were found to exhibit



selective *in vitro* activity against *T. brucei*,⁷⁸³ a number of diterpenes (*Sinularia maxima*) were found to be modest to poor inhibitors of NF- κ B transcriptional activation,⁷⁸⁴ structurally simpler analogues of fuscocaulon exhibit comparable or better anti-inflammatory activity in the mouse ear edema assay,⁷⁸⁵ semi-synthetic oxygenated dolabellane diterpenes exhibit *in vitro* anti-HIV activity,⁷⁸⁶ further semi-synthetic examples of hydroxysterols were found to induce pregnane X receptor transactivation,⁷⁸⁷ flexibilide⁷⁸⁸ exhibits *in vivo* anti-neuroinflammatory activity in rats,⁷⁸⁹ 11-*epi*-sinulariolide acetate inhibits carcinoma cell migration and invasion by suppressing a number of phosphorylation-dependent pathways,⁷⁹⁰ 13-acetoxysarcocrassolide induces apoptosis in carcinoma cells by activation of p38/JNK and suppression of PI3K/AKT pathways,⁷⁹¹ and cembranoids from *Sarcophyton glaucum* exhibit cytotoxicity towards a murine melanoma cell line.^{792,793}

9 Bryozoans

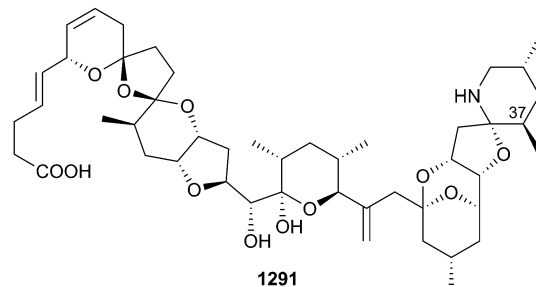
There were only three reports (containing 19 compounds) of new metabolites isolated from bryozoans in 2014, which is a large increase on 2013 when only one new metabolite was reported from this understudied phylum. The bromopyrrole alkaloids aspidostomides A–H **1272–1279** and aspidazide A **1280** were isolated from the Patagonian bryozoan *Aspidostoma giganteum*. Aspidostomide E **1276** exhibited moderate inhibition of the 786-O renal carcinoma cell line. Aspidazide A **1280** is a rare asymmetrical diacylazide and NOE NMR correlations and chemical transformations were utilised in determination of the structure.⁷⁹⁴ A series of ceramides neritinacamide A–E **1281–1285** were obtained from *Bugula neritina* and exhibited selective but weak activity against two HTCLs.⁷⁹⁵ The same sample yielded several sterols **1286–1290**.⁷⁹⁶ Convolutamydine A⁷⁹⁷ displayed significant anti-inflammatory activity both *in vitro* and *in vivo*.⁷⁹⁸



10 Molluscs

The 23 new metabolites reported from molluscs is the average number reported per year over the past decade. 37-*epi*-

Azaspiracid-1 **1291** was isolated from contaminated raw shellfish – the epimer forms spontaneously *via* an equilibrium and formation, as expected, is accelerated by heating.⁷⁹⁹ Implication of the latter in relationship to the cooking of shellfish is important as 37-*epi*-AZA1 was 5-fold more toxic towards Jurkat T lymphocyte cells *in vitro* than AZA1. Corresponding epimers of the related toxins AZA2 and AZA3 were also detected.



Specimens of the Mediterranean sacoglossan mollusc *Thuridilla hopei* afforded new nor-diterpene aldehydes **1292–1294**⁸⁰⁰ in addition to known congeners thuridillin A, B and C.⁸⁰¹ The mollusc feeds on the green alga *Derbesia tenuissima*, extracts of which only contain a known epoxylactone,⁸⁰² supporting the assumption that **1292–1294** are mollusc transformation products. Acetylenic alcohols **1295–1299** were isolated from both the Mediterranean dorid nudibranch *Peltodoris atromaculata* and one of the nudibranch's common dietary prey, the sponge *Haliclona fulva*.⁸⁰³ In a similar manner, renieramycin-related alkaloids fennebricin A **1300** and B **1301** were isolated from both the nudibranch *Jorunna funebris* and the sponge *Xestospongia* sp.⁸⁰⁴ The substructurally-related isoquinoline **1302** was only identified in the sponge extract. New diketopiperazines **1303–1306** were reported from *Pleurobranchus areolatus* – related metabolites have been reported from the ascidian *Didemnum* sp., a suspected prey of *P. areolatus*.⁸⁰⁵ Further investigation into the origin of tetrodotoxin in *Pleurobranchaea maculata* demonstrated that the mollusc can accumulate the toxin through its diet however there was no identifiable tetrodotoxin source in the molluscs local environment.⁸⁰⁶ Two studies of sea hares identified two moderately cytotoxic sesquiterpenes oculiferane **1307** and *epi*-obtusane **1308** (*Aplysia oculifera*)⁸⁰⁷ and an anti-neuroinflammatory diterpene dactylo-diterpenol acetate **1309** (*A. dactylomela*).⁸⁰⁸ 6-Bromoisatin, typically isolated from muricid molluscs, is weakly antiproliferative towards HT29 cells, and enhances apoptosis in an *in vivo* colon cancer model.⁸⁰⁹ Further structure–activity relationship studies of the cyclic peptide sanguinamide B (*Hexabranchus sanguineus*)⁸¹⁰ have found that antiproliferative activity is dependent upon both the location of specific amino acids in the macrocycle and their configuration.^{811,812} New synthetic auristatin analogues, being related to the original MNP dolastatin 10 (*Dolabella auricularia*),⁸¹³ bearing changes at the *N*-terminus showed pronounced antitumour activity.⁸¹⁴ The binding of three examples to tubulin was investigated by co-crystal X-ray studies, identifying an interesting structural feature whereby in the solid state the valine-dolaisoleucine fragment exists in

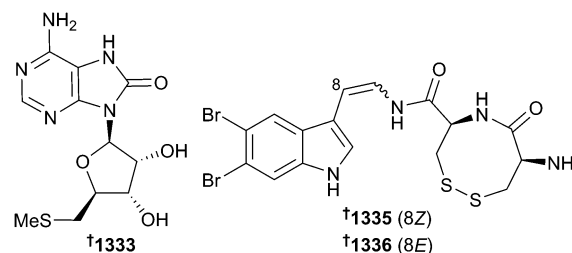


the *cis*-configuration, whereas in solution it exists solely in the *trans*-configuration. In further studies, cholesterol and other simple sterols purified from mussels showed anti-aging and neuroprotective properties,⁸¹⁵ synthetic tambjamine analogues show enhanced tumour cell antiproliferative and chloride transport properties,⁸¹⁶ and lamellarin D (*Lamellaria* sp.)⁸¹⁷ induces senescence in cancer cells, in a process that includes the generation of intracellular ROS and requires the presence of topoisomerase I.⁸¹⁸ Biosynthesis of long chain polyunsaturated fatty acids in the scallop *Chlamys nobilis* was investigated, revealing the presence of a new elongase, that can elongate 20:4*n*-6 and 20:5*n*-3 to C22 and C24 acids, and a Δ^8 -desaturase.⁸¹⁹ A new example of an α 4/7-conotoxin, α -BnIA **1310** was isolated from crude venom of the molluscivorous cone snail *Conus bandanus*.⁸²⁰ Peptides with the same sequence, Mr1.1 and Bn1.1, were previously identified by PCR amplification of venom duct cDNA from molluscs *C. marmoreus*⁸²¹ and *C. bandanus*.⁸²² The peptide reversibly inhibited the human α 7 nicotinic acetylcholine receptor (nAChR) and blocked nerve-evoked skeletal muscle contractions. *Conus bandanus* (Vietnam) was also the source of BnIIID **1311**, a 15 residue M-1 family peptide containing six cysteines (disulfide connectivity not determined) and three post-translational modifications comprised of a bromotryptophan, γ -carboxy glutamate and amidated aspartic acid residues.⁸²³ An unusual α 5/5 conotoxin AusIA **1312** was purified from the venom of *C. australis* – both synthetic globular (natural) and ribbon (different disulfide linkages) configurations inhibited the α 7 nAChR.⁸²⁴ A short cyclic hexapeptide Vi804 **1313** was isolated from crude venom of *C. virgo* and the solution structure of it and the ^DW3 synthetic analogue explored by NMR spectroscopy.⁸²⁵ The high hydrophobicity of γ -conotoxins make them difficult to synthesise by standard peptide synthesis techniques. A recent report describes the use of a Lys₄ solubilising C-terminus tag to enable the synthesis and Na_v subtype selectivity of three previously reported *C. consors* γ -conotoxins, γ -CnVIB, γ -CnVIC and γ -CnVID.⁸²⁶ Further studies have reported on the structure and activity of dicarba analogues of α -RgIA,⁸²⁷ the influence of disulfide connectivity on structure and bioactivity of α -TxIA,⁸²⁸ the influence of acetylcholine to affect the binding of α -MII at nAChRs,⁸²⁹ the neuronal target selectivity of the *Conus textile* T-superfamily peptide TxVC,⁸³⁰ and the Ca²⁺-activated K⁺ (BK) channel selectivity of the unusual M superfamily conotoxin Vt3.1.⁸³¹

11 Tunicates (ascidians)

The 22 new tunicate-derived natural products presented in this review is the second lowest number reported in one year over the last decade. A structurally-diverse range of metabolites were reported, with examples of glycerides **1314** and **1315**,⁸³² amino alcohols **1316–1322**,⁸³³ new didemnaketal congeners **1323**, **1324**⁸³⁴ and **1325**, **1326**,⁸³⁵ halogenated alkaloids **1327** and **1328**,⁸³⁶ a new rubrolide (R) **1329**⁸³⁷ (that unfortunately shares the same letter designation as a related metabolite reported from *Aspergillus terreus*²¹⁷), pyridoacridine cnemidine A **1330**⁸⁵² and sulfated sterols **1331** and

1332.⁸³⁸ Noteworthy was the isolation, structure elucidation and synthesis of a rare example of a modified nucleoside bearing a 5'-thiomethyl substituent **1333**.⁸³⁹ The same study also led to the reassignment of structure of a known sponge metabolite hamiguanosinol⁸⁴⁰ from the enol tautomer to the guanosine keto tautomer **1334**. The tanjungides A **1335** and B **1336**,⁸⁴¹ novel dibromoindole enamide alkaloids isolated from *Diazona cf. formosa* exhibit potent cytotoxicity (<1–2 μ M) towards a panel of HTCLs. An eleven-step linear synthesis utilising Buchwald vinyl iodide amidation and peptide synthesis established the absolute configuration of the alkaloids.



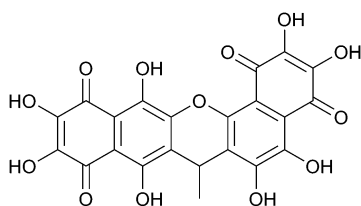
Over the years a number of cyclic ribosomal peptides, now known as the cyanobactins, have been reported from ascidians, typically of the genus *Lissoclinum*. The true producers of these natural products are photosynthetic symbiotic cyanobacteria of the genus *Prochloron*. The apparent random distribution of cyanobactins isolated from ascidians has now been credited to host phylogeny, with genetic analysis revealing that '*Lissoclinum patella*' falls into three phylogenetic groups that in turn may contain further cryptic species.⁸⁴² The implications that local extinctions of such cryptic species may reduce marine natural product diversity is indeed food-for-thought in the context of climate change. A very interesting demonstration of the use of the biosynthetic machinery of cyanobactin production was recently reported, whereby engineered enzymes of the patellamide pathway, in combination with enzymes from other cyanobactin-related pathways, were used for the *in vitro* production of a small library of cyclic peptides.⁸⁴³ This proof of principle allowed for the preparation of 1–2 mg of each peptide. Total synthesis has led to revision of the original structure proposed for didemnaketal B (*Didemnum* sp.)⁸⁴⁴ to **1337**, requiring stereochemical inversion of the C-10–C-20 spiroacetal domain of the MNP.⁸⁴⁵ Such a revision may be of relevance to the ongoing revision of the (stereo)structure of didemnaketal A.⁸⁴⁶ Syntheses of the reported structures of polycitorols A and B (unidentified ascidian)^{847,848} and (+)-didemniserinolipid C (*Didemnum* sp.)^{849,850} suggest that the structures of all three MNPs require revision. Total synthesis has also led to revision of the structure of mandelalide A (*Lissoclinum* sp.)⁸⁵¹ to **1338**⁸⁵² and confirmation of the revised structure of haouamine B (*Aplidium haouarianum*),^{853–855} while the structures of distomadines A and B (*Pseudodistoma aureum*)^{856,857} and synoxazolidinones A and B (*Synocicum pulmonaria*)^{858,859} have been confirmed by synthesis. Synoxazolidinones A and C and *S. pulmonaria* co-metabolites pulmonarins A and B exhibited



variable levels of anti-biofouling activity against a panel of test organisms, with synoxazolidinone C being particularly potent as both a growth and adhesion inhibitor.⁸⁶⁰ The effects of ascidian extracts on the estrogen receptor-negative breast cancer cell line MDA-MB-231 were investigated by content-rich screening, leading to the identification of eusynstyelamide B (*Didemnum candidum*, originally isolated from *Eusynstyela latericus*⁸⁶¹) as a moderate cytotoxin (IC₅₀ 5 μ M) causing cell cycle arrest in G2/M and inducing apoptosis.⁸⁶² The potently cytotoxic macrolide iejimalide C (*Eudistoma cf. rigida*)⁸⁶³ joins congeners iejimalides A and B as being identified as an inhibitor of the vacuolar-type ATP-driven proton pump (H⁺-ATPase).⁸⁶⁴ After 24 h treatment, cells also exhibited actin aggregates, but as the MNP does not inhibit actin polymerisation *in vitro*, it was concluded that actin activity was a consequence of disruption of pH homeostasis. Preliminary data suggesting that trabectedin (Et 743) exhibits anti-angiogenic activity towards breast cancer cell lines has been reported.⁸⁶⁵

12 Echinoderms

The 35 new metabolites reported from echinoderms in this review is 25% lower than the average number reported per annum over the last decade. Beyond the simple sulfonic acid derivative **1339** isolated from the sea urchin *Brisaster latifrons*⁸⁶⁶ and the highly substituted unsymmetrical binaphthoquinone mirabiquinone **1340** (sea urchin *Scaphechinus mirabilis*),⁸⁶⁷ the natural product chemistry of echinoderms is dominated by steroidal tri- (**1341**⁸⁶⁸ and **1342**⁸⁶⁹), tetra- (**1343–1352**^{870–873}), penta- and hexaoses (**1353–1371**).^{873–875} The aglycone **1372** was also isolated.⁸⁷⁴ In many cases these MNPs exhibited biological activity including anti-inflammatory,⁸⁶⁶ cytotoxic,^{870,872,874} hemolytic⁸⁷⁰ and antifungal^{873,875} properties.



1340

A number of new saponins were identified in extracts of the Australian sea cucumber *Holothuria lessona* using solely LC-MS/MS metabolomic techniques.⁸⁷⁶ In a conceptually similar manner, the chemical diversity of saponins present in different organs of the starfish *Asterias rubens* was investigated by combinations of MALDI-TOF and LC-MS(/MS) techniques.⁸⁷⁷ The latter study concluded that different organs are characterised by different saponin mixtures and inter-specimen variability exists suggesting influence of sex and/or collecting season on saponin profile. A short octapeptide echinometrin **1373** (sea urchin *Echinometra lucunter*) was found to exhibit ability to degranulate mast cells leading to an inflammatory reaction.⁸⁷⁸ The sequence of the peptide is an internal fragment

of vitellogenin, a nutrient protein present in sea urchin gametogenic cells, suggesting the possibility that echinometrin is a cryptide.⁸⁷⁹

LRKLMLQR

1373

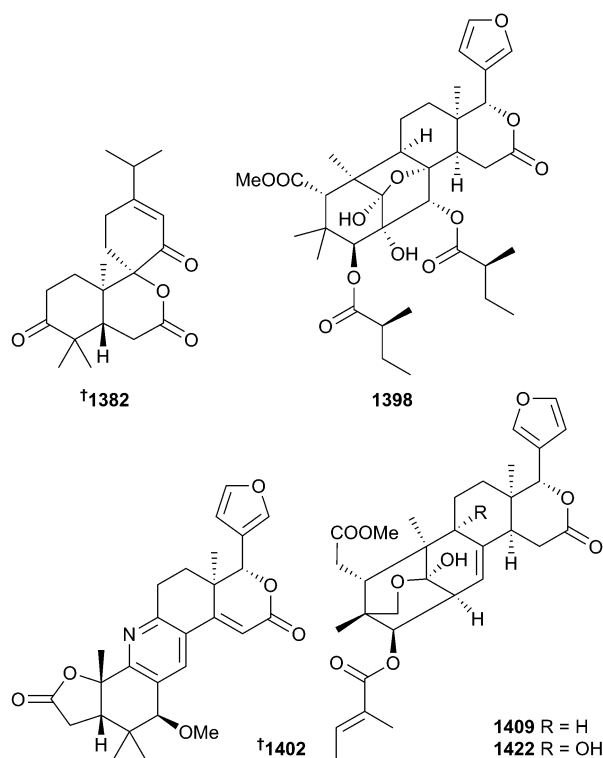
The first synthesis of a pyranonaphthazarin pigment isolated from the sea urchin *Echinothrix diadema*⁸⁸⁰ has been reported.⁸⁸¹ Further studies using purified pentahydroxynaphthoquinone echinochrome A^{882,883} have identified it to enhance mitochondrial biogenesis,⁸⁸⁴ to protect cardiomyocyte mitochondria from the effects of cardiotoxic drugs⁸⁸⁵ and to inhibit acetylcholinesterase in an irreversible and uncompetitive mode.⁸⁸⁶ The mechanisms of antioxidant reactivity of the structurally related echinamines A and B⁸⁸⁷ has been investigated using DFT methods.⁸⁸⁸ The benzochromenone comaparvin (*Comanthus bennetti*)⁸⁸⁹ exhibits anti-inflammatory activity in the carrageenan-induced rat model.⁸⁹⁰ A simple synthesis of ovothiol A⁸⁹¹ from L-hisidine has been reported,⁸⁹² the role of a non-heme iron oxidase enzyme in the biosynthesis of ovothiol A investigated,⁸⁹³ NMR-pH titrations used to investigate the microspeciation of the amino acid,⁸⁹⁴ and the antiproliferative activity towards Hep-G2 cell line *via* an autophagy mechanism identified.⁸⁹⁵ Sterols and fatty acids from the spiny sea-star *Marthasterias glacialis* exhibit both anti-inflammatory⁸⁹⁶ and cell cycle arrest properties,⁸⁹⁷ while sterols from an urchin and a starfish exhibit selective antiparasitic activity towards *T. brucei*.⁷⁸³ Investigation of the structure and bioactivity of chimeric analogues of the starfish cardiac-stomach relaxing SALMFamide neuropeptides S1 and S2 has highlighted the importance of the C-terminus for bioactivity and that the N-terminus confers structural stability.⁸⁹⁸ Treatment of pancreatic cancer *in vivo* in athymic mice using a combination of frondoside A (*Cucumaria frondosa*)⁸⁹⁹ and the nucleoside anticancer agent gemcitabine was significantly more effective than with either drug alone.⁹⁰⁰ The cytotoxic and apoptotic-inducing abilities of steroids from the cold water starfish *Ctenodiscus crispatus*⁹⁰¹ and the triterpene glycosides, including echinoside A, from *Holothuria scabra* and *Cucumaria frondosa* have been reported.⁹⁰² Echinoid A and related saponin holothurin A inhibit dietary fat absorption and decrease the adipose tissue accumulation in mice,⁹⁰³ and typicosides (*Actinocucumis typica*)⁹⁰⁴ exhibit a strong immunostimulatory effect on mouse macrophages with marked increase in lysosomal activity and ROS formation.⁹⁰⁵ Complete NMR assignments have been reported for four pentaoside asterosaponins: thornasteroside A, versicoside A, anasteroside B and asteronylpentaglycoside sulfate (*Asterias amurensis*).⁹⁰⁶

13 Mangroves

In addition to a series of mildly antioxidant glycosides, marinoids F–M **1374–1381**, reported from the whole fruits of the mangrove *Avicennia marina*,^{907,908} seco-diterpene **1382** (*Ceriops decandra*),⁹⁰⁹ cinnamides **1383–1387** (*Micromelum falcatum*, mangrove associate),⁹¹⁰ protolimonoids **1388–1394** (*Xylocarpus granatum*),⁹¹¹ and limonoids **1395–1401**,⁹¹² **1402–1404**,⁹¹³ **1405–**



1408,⁹¹⁴ **1409–1418**,⁹¹⁵ **1419–1422**⁹¹⁶ and **1423**⁹¹⁷ (*X. rumphii* and *X. granatum*) were also isolated from mangroves or their associates. The absolute configuration of the unusual spiro-*seco*-abietane decandrinin **1382** was secured *via* analysis of experimental and calculated ECD and ORD chiroptical data⁹⁰⁹ while of the limonoids reported, the structures of xylorumpihiin G **1398**,⁹¹² xylogranatopyridine A **1402**,⁹¹³ granatumin L **1409**⁹¹⁵ and granatumin Y **1422**⁹¹⁶ were established by single crystal X-ray diffraction studies.

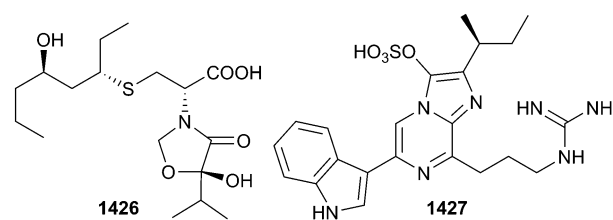


Further investigation of previously reported mangrove MNPs has identified avicequinone C (*Avicennia marina*)⁹¹⁸ as a 5 α -reductase-type 1 inhibitor,⁹¹⁹ catunaregin (*Micromelum falcatum*)⁹²⁰ as an angiogenesis inhibitor,⁹²¹ the cardiac glycoside neriifolin (*Cerbera manghas*)⁹²² as the acaricidal component of *Panonychus citri*,⁹²³ and that leaf extracts and purified components of *Avicennia marina* exhibit antibacterial activity with relevance to urinary tract infections.⁹²⁴ Finally, a new biomimetic synthesis⁹²⁵ and a structure–activity relationship study of the protein tyrosine phosphatase 1B inhibiting properties⁹²⁶ of the unusual dimeric alkylbutenolide paracaseolide A (*Sonneratia paracaseolaris*)⁹²⁷ have been reported.

14 Miscellaneous

Two studies of sea grass from the Egyptian Red Sea led to the identification of flavone xyloside **1424** (*Thalassia hemprichii*)⁹²⁸ and dihydrochalcone diglycoside **1425** (*Thalassodendron ciliatum*)⁹²⁹ as antimicrobial and anti-influenza A virus MNPs respectively. Thelepamide **1426** is an unusual ketide-amino acid isolated from the annelid worm *Thelepus crispus*.⁹³⁰ The

relative configuration of the alkaloid was determined by a combination of heteronuclear J-based configurational analysis and GIAO calculated chemical shifts and DP4 probability analysis. Mild cytotoxicity towards a leukemia cell line was also observed. The arenicins, antimicrobial peptides produced by the polychaete worm *Arenicola marina*,⁹³¹ are constitutively expressed in a range of tissues in the organism suggestive that the peptides play a front-line role in defense against infections.⁹³² Cypridina luciferyl sulfate **1427** appears to be a more stable storage form of cypridina luciferin, the luminescence precursor of the ostracod *Cypridina (Vargula) hilgendorffii*.⁹³³ The luciferin could be converted to luciferyl sulfate by action of crude extract of the organism, presumably containing a sulfotransferase, in the presence of 3'-phosphoadenosine 5'-phosphosulfate (PAPS, a sulfate donor), while the reverse reaction took place in the presence of adenosine 3',5'-diphosphate, a sulfate acceptor.



Extracts of ovary tissue from *Takifugu pardalis* yielded a new tetrodotoxin analogue, 6-deoxyTTX **1428**.⁹³⁴ The potent voltage-gated sodium channel blocker was also detected by LC-MSMS in other marine animals including snail and octopus. Alanine scanning of the hagfish (*Myxine glutinosa*)⁹³⁵ 12-amino acid residue antimicrobial peptide myxinidin has identified a number of critical residues for the observed biological activities and that judicious substitution with arginine led to the identification of more potent analogues.⁹³⁶ The 33-amino-acid residue peptide pardaxin (flatfish *Pardachirus marmoratus*) exhibited *in vivo* activity towards MRSA dermal infection and enhanced wound healing.⁹³⁷ A range of arsenic-containing lipids have been synthesised, providing valuable reference standards for future studies directed towards understanding the uptake, biotransformation and toxicity of these unusual MNPs.⁹³⁸

15 Conclusion

In a recent review the number of new MNPs reported from a range of countries was analysed based on the location of the principal author for each publication.³ What is not apparent from this analysis is the location of the collecting sites for the organisms. This can be problematical as shown in an extreme example: one prominent MNP chemist based in the Mid-West of the USA, thousands of km from any coastal resource, collected widely (see Fig. 1).

With the biogeographic aspect of the MarinLit database¹⁹ now widely available an alternative perspective is available for viewing the collection data. It is now possible to search by region showing who is collecting where and hence the



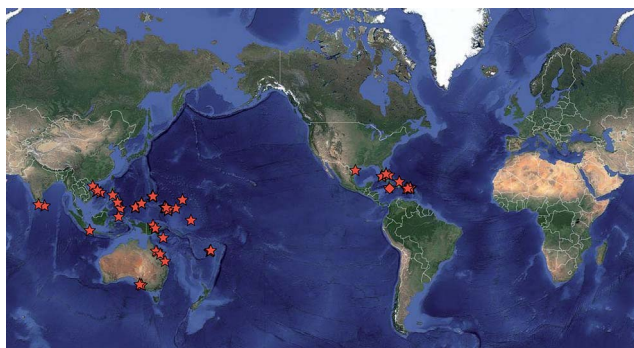


Fig. 1 Collection sites (red stars) for a US MNP chemist.

interests that MNP chemists have in the various regions of the globe. The 50 years of collecting effort globally is depicted in Fig. 2. The red stars in Fig. 2 represent the collecting sites, described in ~9000 papers, from which ~25 700 new compounds have been obtained. The actual number of unique collection sites is less than the number of papers as multiple collections have often been made at the same sites. Fig. 2 displays those areas that have had high collecting pressures and those where there has been, for whatever reason, lower collecting pressures. An analysis has been made of the collecting effort globally by semi-decade since 1965. For convenience the globe was divided up into 22 regions or countries (see Table 1) and the published papers for each region compiled. Totals for papers (citing collection sites) describing new compounds from 1965 are also listed in Table 1 while Fig. 3 shows the incidence of discovery of new compounds in each region or country by semi-decade since 1965. It is worth noting that since 1965–280 papers contained no biogeographic data, not even a country or an ocean, to describe the origin of new compounds being reported. Fortunately, recent years have seen the increasing use of exact coordinates. The Japanese region, including Okinawa, has been the most productive region with 3877 compounds described. This was

Table 1 The 22 oceanic regions or countries used in the survey giving numbers of publications and new compounds discovered in each region/country over the period 1965–2014

Country or Oceanic region	#Compounds	#Papers
Pacific Russia, sea of Japan	377	167
China	2915	942
South China sea and Yellow sea	525	203
Taiwan	1350	376
Japan, including Okinawa	3877	1570
South Korea	848	239
North Pacific islands and atolls	1539	576
South Pacific islands and atolls	1548	522
Maritime SE Asia, Papua-New Guinea	1340	502
Australia	1854	677
Mainland SE Asia (including East Malaysia)	457	173
South Asia	714	333
Indian ocean and islands	326	155
Mediterranean, Arabian Peninsula, Black sea	2358	876
Other African countries	456	154
Atlantic Europe and the Baltic sea	476	210
Atlantic ocean and islands	361	140
South American countries	538	210
Central America	245	86
Gulf of Mexico, Caribbean sea and islands	1524	571
North America	1382	520
Arctic and Antarctica	330	105

followed closely by China with 2915 compounds from the mainland with a further 525 compounds to be included if the regions of the South China Sea and Yellow Sea are also considered. But what is remarkable here is the rapid emergence of MNP chemistry using Chinese-based collections as seen in Fig. 3. Other regions where there have been extensive collections are in the Mediterranean (2358) (which includes the Mediterranean coasts of Spain and France, Italy, the islands of the Mediterranean, Greece, Turkey, Israel, Egypt, the Arabian Peninsula, Black Sea and the Mediterranean African coastlines), Australia (1854), the North (1539) and South (1548) Pacific islands and atolls, and the Gulf of Mexico, Caribbean Sea and islands (1524). Other regions that have been well explored include Taiwan (1350) and Maritime SE-Asia and Papua-New Guinea (1340).

The graph shows very clearly those countries that were most closely studied in the early years. Compounds of Japanese origin were prominent from the 1960s, but the search for MNPs was quickly taken up with compounds from the Mediterranean, North Pacific and North American regions appearing in the early 1970s, followed by Australian and Maritime SE-Asian compounds later in that decade. Prospecting activity in other parts of Asia was relatively slow to start and it was not really until the 1990s that compounds of Mainland SE-Asian, South Korean, Taiwan and Chinese origin started to appear. The output from these regions has rapidly accelerated since, particularly so in the case of compounds of Chinese origin. Fig. 2 and 3, taken in combination, provide a snapshot of the past efforts in marine natural products as well as current endeavours and highlights those areas of the globe that are currently under-explored.

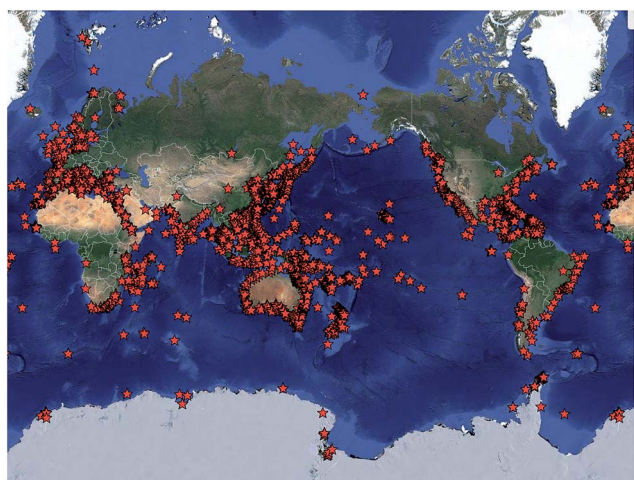


Fig. 2 All collection sites for MNP discovery, 1965–2014.



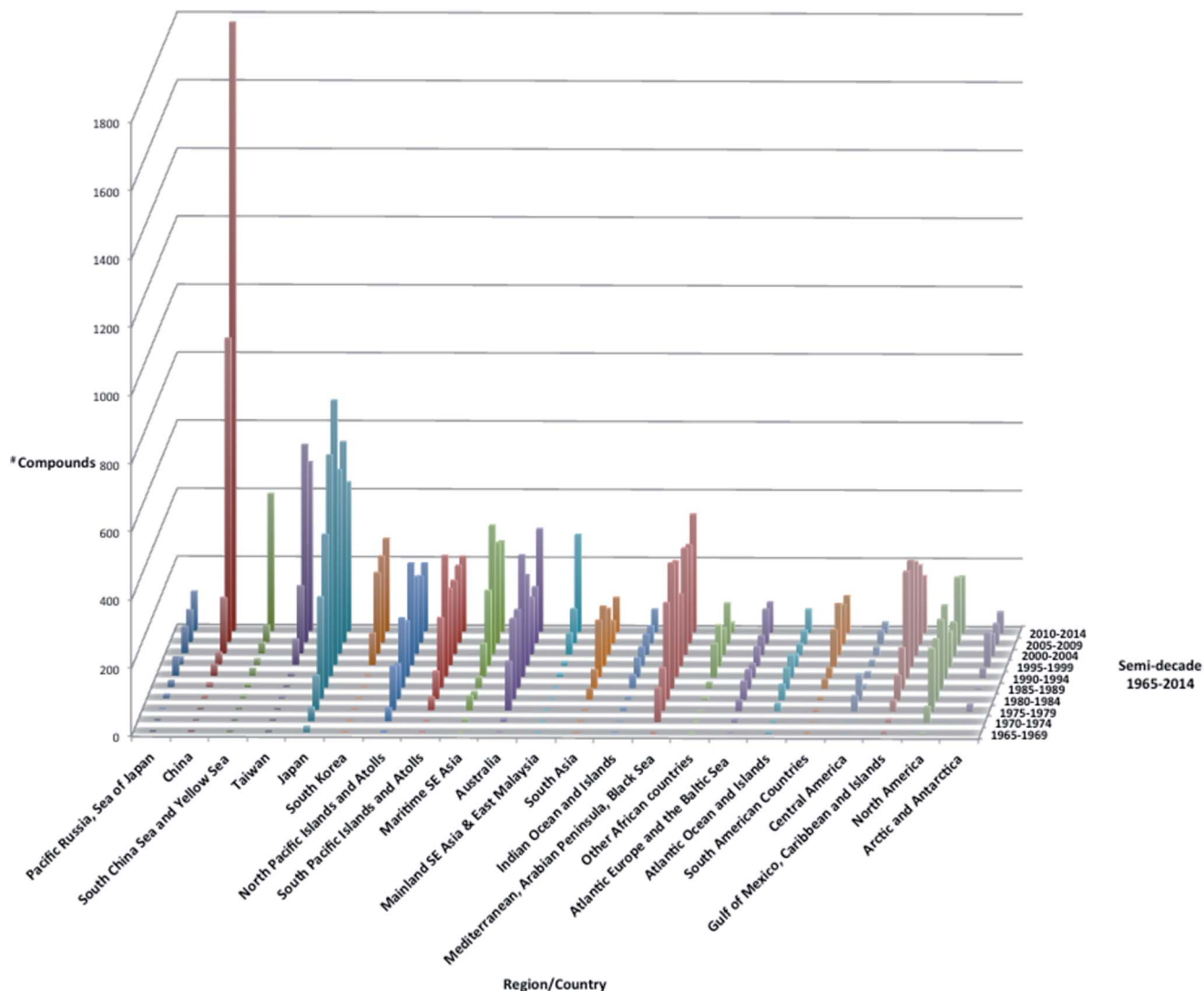


Fig. 3 The count of new compounds by region/country over the period 1965–2014, by semi-decade.

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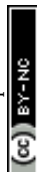
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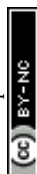
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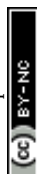
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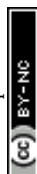
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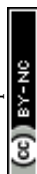
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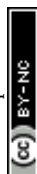
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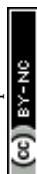
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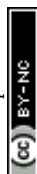
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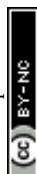
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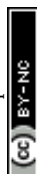
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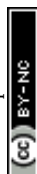
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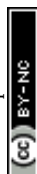
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