

Chemical and Biological Aspects of the Sponge Genus *Dysidea*, A Review^ϕ

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Abstract – The chemical constituents and their biological activities of the sponge genus *Dysidea* has been reviewed.

Key words – *Dysidea*, polybrominated diphenyl ethers, chlorinated amino acid derivatives, sesquiterpenes, aromatic sesquiterpenes, polyhydroxy steroids.

Introduction

The chemistry of marine natural products has been reviewed time to time covering the literature on isolation and biological activity of secondary metabolites from marine flora and fauna (Faulkner, 1997). Literature survey revealed that the sponges (Porifera) have received increased attention from organic chemist, biochemist and pharmacologist, as sponges yielded variety compounds with unique structural features and high biological activity. The sponges of the genus *Dysidea* (family *Dysididae*, order *Dictyoceratida*) are cosmopolitan in nature, widely distributed throughout the tropical and subtropical waters and can be collected from inter tidal zones to deep waters. To the best of our knowledge, thirteen species of *Dysidea* have been subjected for chemical examination. The chemical examination of the genus *Dysidea*, so far afforded, polybrominated phenyl ethers, chlorinated amino acid derivatives, furano sesquiterpenes and polyhydroxy steroids.

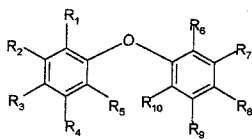
Polybrominated phenyl ethers – The early studies on the sponge *D. herbacea* resulted in isolation of five [1-5] Gram

positive and Gram negative antibacterial poly brominated diphenyl ethers [1-5] (Sharma *et al.*, 1969 and 1972). Subsequently, Carte and Faulkner reported that the sponge *D. herbacea* afforded compounds 1, 2 and a new compound 6 and *D. chlorea* contained only compound 3 (Carte and Faulkner, 1981). The structures of these compounds were determined by the study of spectral data. In the same year R. J. Wells group discovered five new poly brominated diphenyl ethers [7-12] from the sponges *D. herbacea* of Great Barrier Reef and Fijian coast (Norton *et al.*, 1981). Compounds 7-12 are related to the diphenyl ethers 1-6, but differ in that they are all oxygenated on both of the phenyl rings instead of only one. The structures of the new compounds 7-12 have been determined by ¹³C-spin lattice relaxation data for quaternary carbons (Norton and Wells, 1980). The sponge *D. fragilis* afforded a new hexabromodiphenyl ether 13 and two known brominated diphenyl ethers 1 and 11 (Utkina *et al.*, 1987). An unidentified species of *Dysidea* collected from the Philippines coast afforded a major new diphenyl ether [14] (Salva and Faulkner, 1990). A specimen of *Dysidea* sp. from Indo-Pacific Ocean afforded seven known diphenyl ethers [1, 2, 3, 6, 7, 8 and

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11], five new diphenyl ethers [15-19] and simple brominated phenols **25** and **26** (Fu *et al.*, 1995). All new compounds characterized by spectral studies. The sponge *D. herbacea* from Indian Ocean afforded a new tetra bromodiphenyl ether [20] (Anjaneyulu *et al.*, 1996). Very recently, four new polybrominated diphenyl ethers [21-24] were isolated from the sponge *D. herbacea* from west Sumatra coast, Indonesia (Handayani *et al.*, 1997).

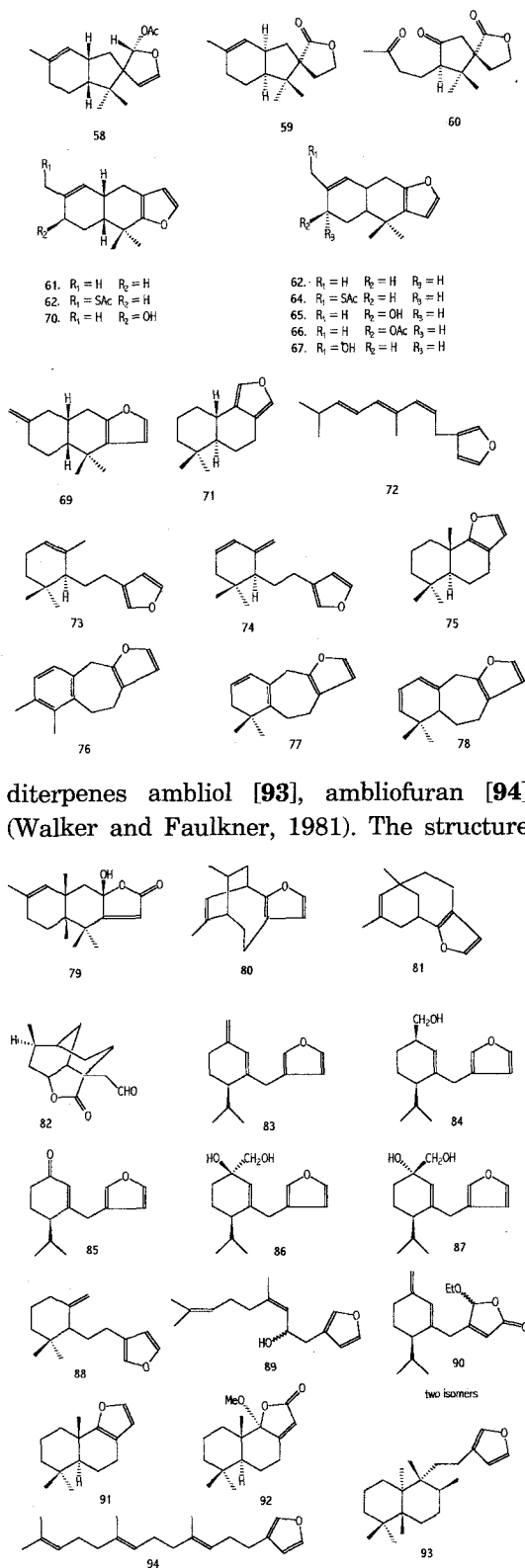
Chlorinated Amino acid derived metabolites and cyclic peptides—The chlorinated metabolites from *Dysidea* show the strongest resemblance to metabolites of blue-green algae. This is particularly true of dysidin [27] (Hofneing and Oberhansli, 1977). From *D. herbacea* collected at Townsieille, which is molecule, that bears a striking resemblance to the tetramic acid portion of malyngamide A from *Lyrgbya majuscula* (Cardellina II *et al.*, 1979). The structure of dysidin [27] was established by X-ray diffraction analysis. A sample of *D. herbacea* from Cooktown, Australia gave the hexachlorinated metabolite dysidenin [28],



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which was reported without stereochemical assignments (Kazlauskas *et al.*, 1977). The sponge *D. herbacea* from Papua-New Guinean, yielded isodysidenin [29] (Charles *et al.*, 1978a). The structure of isodysidenin was determined by X-ray analysis and was proposed that dysidenin [28] and isodysidenin [29] differ in stereochemistry at C-5. The absolute configurations of dysidenin [28] and isodysidenin [29] were established in 1984 (Biskupiak and Ireland, 1984). Erickson and Wells reported three new isodysidenin derivatives (**30**, **31**, **32**) and a new dysidenin derivative (**33**) from sponge *D. herbacea* collected near Bowen, Australia (Erickson and Wells, 1982). A diketopiperazine [34] derived from trichloro leucine was obtained from a specimen of *D. herbacea* that has been collected from near Gladstone, Australia (Kazlauskas *et al.*, 1978a). Two new hexachloro metabolites dysidamide [35] (Gebreyesus *et al.*, 1988), and its isomer **36** (Carmely *et al.*, 1990) have been isolated from a Red Sea sponge *Dysidea* sp. and the structure was determined by spectral studies and X-ray crystallography. Compounds **35** and **36** differ in stereochemistry of hydroxyl and trichloro isobutyl groups. A novel and unique azacycloprane lipid dysidazine [37] has been isolated (Molinski and Ireland, 1988) from *D. fragilis* and its structure was determined by using spectral data. Dysidazine is cytotoxic to L 1210 cells at 6.27 $\mu\text{g}/\text{ml}$. Subsequently, four new azacyclo propene derivatives [38-41] were isolated by Faulkner *et al.*, from the sponge *D. fragilis* collected from Pohnpei (Salomon *et al.*, 1995). A specimen of *D. herbacea* from the Great Barrier Reef has yielded herbaceamide [42] and was identified by chemical and spectroscopic methods (Lee and Molinski, 1992). Three new diketopiperazines, dysamines A [43], B [44] and C [45] were isolated from Hainan Island China (Su *et al.*, 1993). The structure of dysamine A [43] was established by X-ray

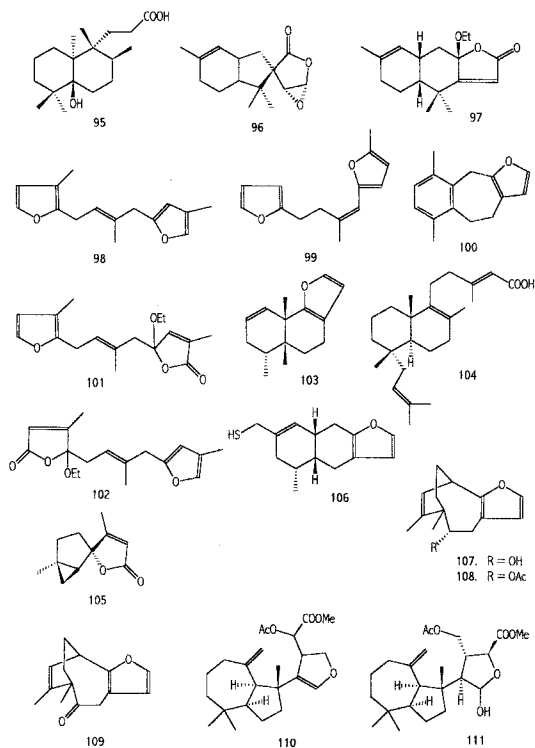
key reaction in the structure elucidation of spirodysin [58] was the $\text{BF}_3 \cdot \text{Et}_2\text{O}$ catalyzed elimination of acetic acid with concomitant rearrangement to obtain 1:1 mixture of furodysin [61] and furodysinin [62], (optical rotation of the both compounds are not reported) (Kazlauskas *et al.*, 1978c). Subsequently Kazlauskas *et al.* isolated compounds 61 and 62 from the sponge *Dysidea* collected near Sydney, Australia, together with thiofurodysin acetate [63] and furodysinin acetate [64]. The structures of 61 and 62 were determined by X-ray analysis. The structures of 63 and 64 were confirmed by chemical conversion to 61 and 62 respectively (Kazlauskas *et al.*, 1978c). In 1982, Dunlop *et al.* reported several sesquiterpenoids including furodysinin [62], five furodysinin derivatives [65-69], a furodysin derivative [70], euryfuran [71], and a linear sesquiterpene furan [72] (Dunlop *et al.*, 1982). Cortes *et al.*, reported absolute configuration of (-) euryfuran (Cortes *et al.*, 1987). The pallescensins, pallescensin-1 [73], pallescensin-2 [74], pallescensin-A [75], (Matsumoto and Usui, 1978), pallescensin-E [76] (Baker and Sims, 1981), pallescensin-F [77] and pallescensin-G [78] (Matsumoto and Usui, 1983) are group of sesquiterpene furans from *D. pallescens*. The structures and absolute configurations were confirmed by synthesis. A collection a *D. etheria* from Bermuda contained furodysinin [62] and furodysinin lactone [79] (Stephen and Cardellina-II, 1984). Nakafuran-8 [80] and nakafuran-9 [81] are two furanosesquiterpenes from Hawaiian sponge *D. fragilis* (Schulte *et al.*, 1980a). Further, *D. fragilis* yielded a novel sesquiterpene aldehyde upial [82] (Schulte *et al.*, 1980b) and penlanfuran [83] (Guella *et al.*, 1983). In continuation of work on *D. fragilis* from Brittany (Guella *et al.*, 1985a) afforded six sesquiterpenes [84-89] and two related butenolide [90 and 90a]. A sample of *D. ambliia* afforded pallescensin-A [91], pallescensolide [92], together with



diterpenes ambliol [93], ambliofuran [94] (Walker and Faulkner, 1981). The structure

of ambliol [93] was determined based on X-ray crystal structure of related compound 95 (Walker *et al.*, 1984).

An unusual β , γ -epoxy γ -lactone, dysetherin [96] has been isolated from *D. etheria* (Schram, 1985). The Mediterranean sponge *D. tupha* contain ent-furodysin, is optical enantiomer of furodysin [62] and related ethoxy lactone tuphabutenolide [97] (Guella *et al.*, 1985b). A mixed collection of the sponge consists of *Pleraph spinifera* and *D. avara* was found to contain longifuran [98] as major metabolite, tavaacuran [99], tavaacpalescensin [100], tavaacbutinolide-1 [101], tavaacutinolide-2 [102] as minor metabolites (Guella *et al.*, 1985a). Since the sponges were extracted with ethanol, it is possible that 83, 87 and 88 were all artifacts. Herbacin [103] is a new furanosesquiterpene that is a major metabolite of *D. herbacea* collected in India (Sarma *et al.*, 1986). A new sesterterpene acid [104] has been isolated from Palavan sponges of *Dysidea* (Nakagawa *et al.*, 1986) along with known compounds 2 and 135. All these were showed aldose reductase inhibitory activity at 400 $\mu\text{g/ml}$ and can be used in the treatment of galactosemic cataracts. The structure of 104 has been elucidated by spectroscopic data. A monoterpenoid adriadyliolide [105] has been isolated from a North Adriatic species of *Dysidea* (Mancini *et al.*, 1987). Thiofurodysin [106] was the first sesquiterpene mercaptan isolated from an Australian specimen of *D. avara* (Capon and Macleod, 1987). Oxidized nakafuran-8 sesquiterpenes 5-hydroxynakafuran-8 [107], 5-acetoxynakafuran-8 [108], 5-ketonakafuran [109] have been isolated from *D. etheria* from Bermuda (Cardellina II and Barnekow, 1988). Ten new rearranged spongian diterpenes have been isolated from two Red Sea specimens of *Dysidea* (Carmely *et al.*, 1988). One species of *Dysidea* contained shahamines A-G [110-116] in addition to the known diterpenes macfarlandin E [120]

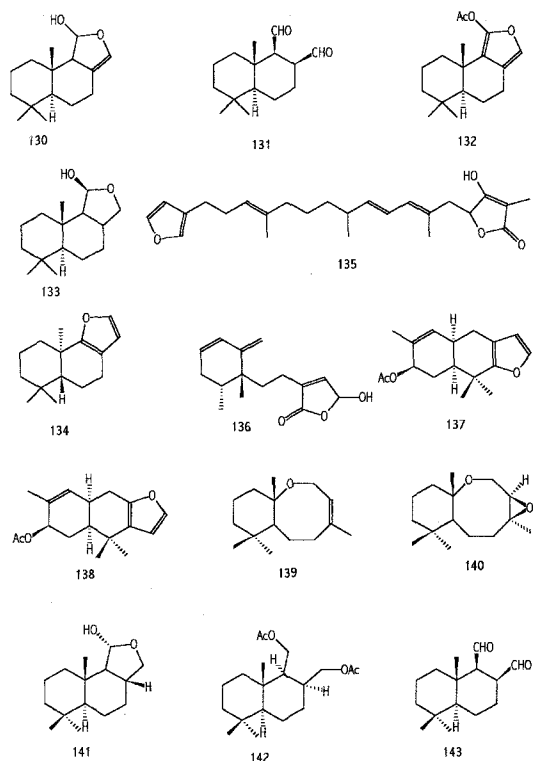
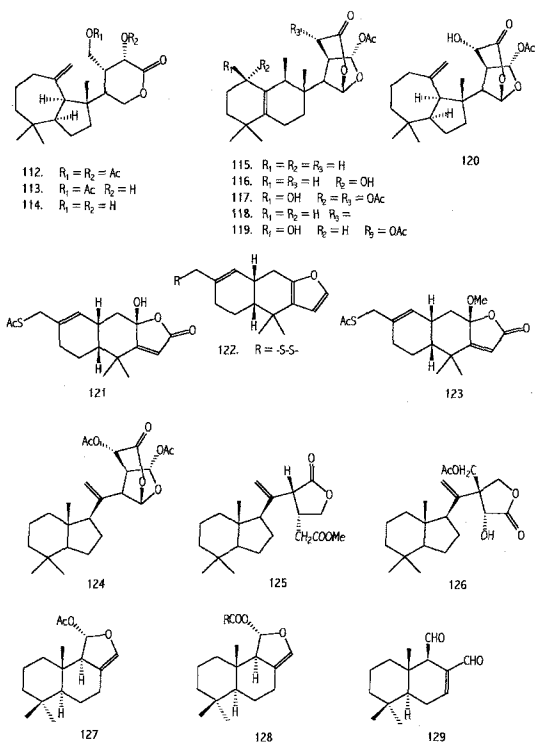


(Molinski *et al.*, 1986). The second *Dysidea* sp. contained shahamine-F [115], shahamine-H [117], shahamine-I [118] and shahamine-J [119]. The structures of the shahamines were proposed on the basis of their spectral data and supported by chemical interconversions (Carmely *et al.*, 1988).

A Palauan species of *Dysidea* contains 15-acetylthioxyfurodysin lactone [121], which binds to the human leukotriene B₄ receptor. The structure of compound 121 was determined interpretation of spectral data and confirmed by synthesis (Carte *et al.*, 1989). The absolute stereochemistry (-)-6R, 11R-thiofurodysin acetate [64] (Kazlauskas *et al.*, 1978c), (Capon and Macleod, 1987), (-)-6R, 11R-furodysin disulfide [122] (Ksebati and Schmitz, 1988) and (+)-6R, 11R-methoxythiofurodysin acetate lactone [123] which were isolated together with (-)-6R, 11R-furodysin (62) (Kazlauskas *et al.*, 1978c), (Capon and Macleod, 1987), (Grode and Cardellina-II, 1984) from a Fijian specimen of *D. herbacea*, were determined

by chemical interconversions (Harton *et al.*, 1990). A Red Sea species of *Dysidea* contained three new diterpenes norslandin [124], seco-norrisolide B [125] and seco-norrisolide C [126] (Rudi and Kashman, 1990). A new bioactive terpene 7-deacetoxy-olepupane [127] (Garson *et al.*, 1992) has been isolated from the temperate marine sponge *Dysidea* sp. along with three known compounds euryfuran [71], sesquiterpene ester [128] (Aliva *et al.*, 1991), polygodial [129] (Cimino *et al.*, 1983). The structure of 127 has been established by using spectral data. Four new drimane sesquiterpenes [130-133] were isolated from southern Australian sponge *Dysidea* sp., together with (+)-euryfuran [71] (Dunlop *et al.*, 1982) and (-)-palescensin [134] (Cimino *et al.*, 1975) which are antipodal (Butler and Capon, 1993). The mild protein phosphatase inhibitor isopalninurin [135] was isolated from *Dysidea* sp. from the Bass Strait (Murray *et al.*, 1993). A new sesquiterpene 136 has been isolated from an Indian Ocean specimen of *D. herbacea*

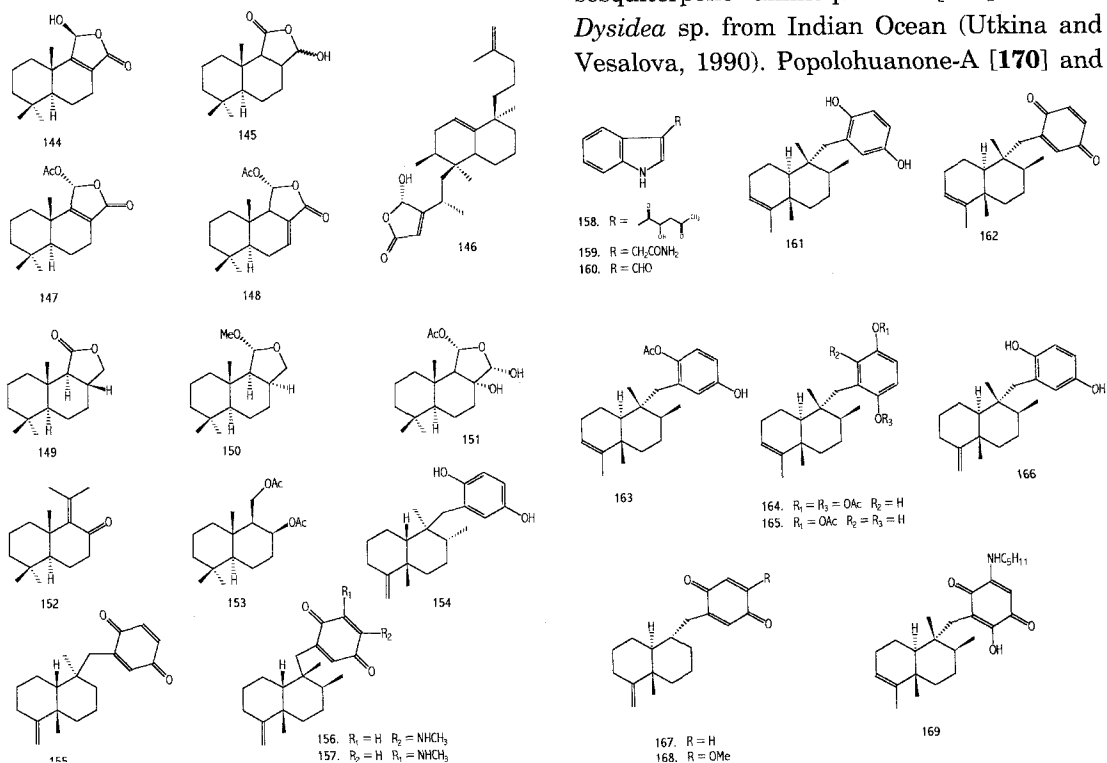
(Venkateswarlu *et al.*, 1994). Two new furo-sesquiterpenes 137 and 138 were isolated from *D. herbacea* (Searle *et al.*, 1994) and structures were determined by the study of spectral data. Two cyclic sesquiterpene arenarian-A [139] and arenarian-B [140] have been isolated from the sponge *D. arenaria* (Horton and Crews, 1995). Arenarian-A was *in vitro* active against several types of cancer cell lines. Five drimane sesquiterpenes [141-145] were isolated from the sponge *D. fusca* (Montagnac *et al.*, 1996). The structures were determined by interpretation of spectral data. Dysidiolide [146], a rearranged sesquiterpene lactone has been isolated from Caribbean sponge *D. etheria* (Gunasekara *et al.*, 1996). The structure of dysidiolide [146] was established by single crystal X-ray diffraction and compound 146 was found to inhibit protein phosphatase and total synthesis of dysidiolide [146] has been achieved (Corey and Roberts, 1997). Recently, Paul *et al.* report-



ed the isolation of seven new sesquiterpenoids of the drimane class [147-153] from a sponge of the genus *Dysidea* (Paul *et al.*, 1997) along with three known compounds 127, 129, and 142.

Aromatic Sesquiterpene – Prenylated aromatic compounds are not particularly common in sponges but they are important for their pharmacological activity. Among the simple hydroquinones and quinone are arenarol [154] and arenarone [155] from *D. arenaria* and were found to be cytotoxic (Schmitz *et al.*, 1984). The sponge *D. avara* yielded two new biologically active sesquiterpenoid amino-quinones 156 and 157 (Cimino *et al.*, 1982) along with avarone [162]. The structures of 156 and 157 are established by spectral data, the ethanol extract of the same sponge inhibits the cell cleavage of the fertilized eggs from the sea urchin *Sphaerechinus granularis*. *D. etheria* produce a new plant growth regulator 3-hydroxy-1-(indol-3-yl)pentane-1,4-dione [158]

and also contains indole 3-actamide [159] and indole-3-carboxyaldehyde [160] (Cardellina-II *et al.*, 1986). Two new aromatic sesquiterpenes avarol [161] and avarone [162] have been isolated from *D. avara* (Minale *et al.*, 1974) were found to inhibit Human Immunodeficiency Virus (HIV) *in vitro* (Sarin *et al.*, 1987). Avarol monoacetate [163], which is a minor metabolite of *D. avara*, shows cytotoxicity in brine shrimp assay (Crispino *et al.*, 1989). Recently, avarol [161] and avarone [162] were found to exhibit anti-inflammatory activity (Ferrandiz *et al.*, 1994). Diacetylavaryl [164] and 6'-hydroxy-5'-acetyl avarol [165] have been isolated as minor metabolites of *D. avara* (Giulio *et al.*, 1990), Neoavarol [166], which is a double bond isomer of avarol, and two new quinones neoavarone [167], 4'-methoxyneoavarone [168] were isolated as minor metabolites of an Okinawan species of *Dysidea* (Iquchi *et al.*, 1990) and the structures were established by interpretation of spectral data. A new sesquiterpene aminoquinone [169] from a *Dysidea* sp. from Indian Ocean (Utkina and Vesalova, 1990). Popolohuanone-A [170] and

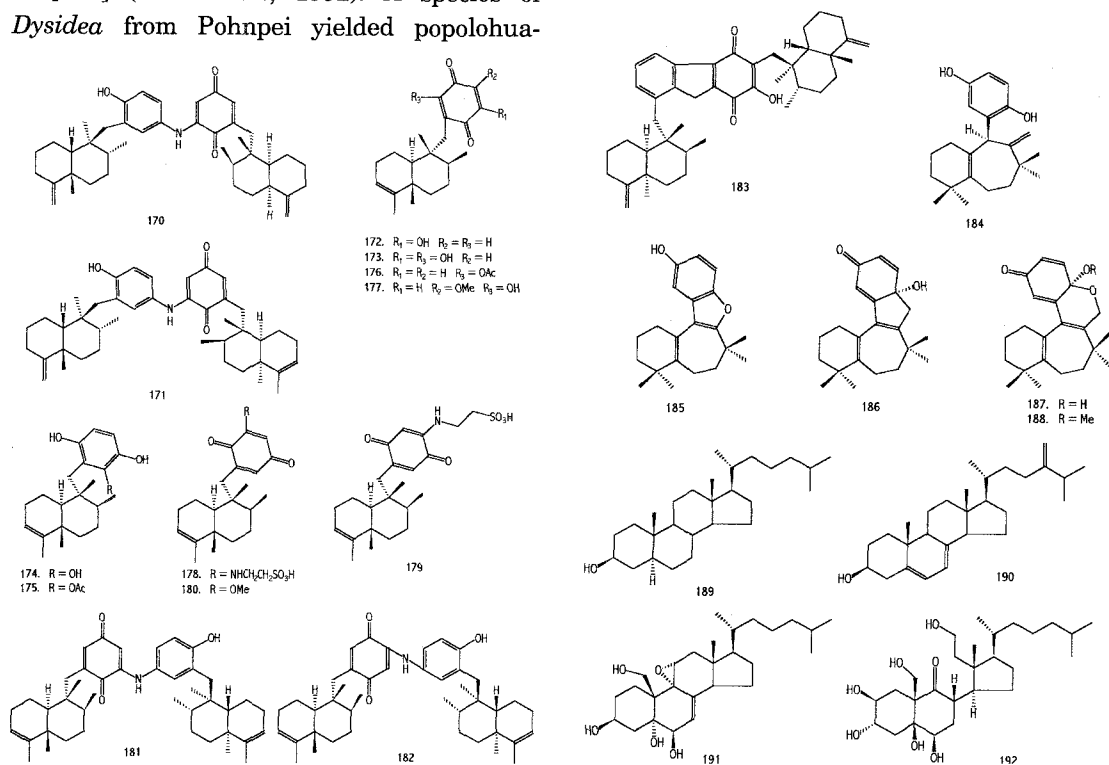


popolohuanone-B [171] are two isomeric quinones were isolated from Papua New Guinea species of *Dysidea* (Rodriguez *et al.*, 1990).

An Australian species of *Dysidea* contains both avarol [161] and the exocyclic double bond isomer isoavarol [166] (Shubina *et al.*, 1990). *D. cinerea* from the Red Sea yielded six new meroterpenoids, 3'-hydroxyavarone [172], 3',6'-dihydroxyavarone [173], 6'-hydroxyavarol [174], 6'-acetoxyavarol [175], 6'-acetoxyavarone [176] and 6'-hydroxy-4'-methoxyavarone [177] (Hirsch *et al.*, 1991). The absolute configurations of 172-177 were found to be the same as that of avarol [161]. Further the cytotoxicity, the antimicrobial activity and the anti-HIV-1 reverse transcriptase activities of new compounds were described. A specimen of *D. avara* from the Solomon Islands yielded four new compounds melemeleone-A [178], melemeleone-B [179], 18-methoxyavarone [180], popolohuanone-C [181] and popolohuanone-D [182] (Alvi *et al.*, 1992). A species of *Dysidea* from Pohnpei yielded popolohua-

none -E [183] (Carney and Scheuer, 1993) which was found to inhibit topoisomerase-II with selective lung tumor cytotoxicity. Very recently, Patil *et al.* reported five new frondosins A-E [184-188] from bioassay guided fractionation of the EtOAc extract of the sponge *D. frondosa* collected from Pohnpei. The structures and relative stereochemistry of the frondosins were established by interpretation of spectral data and were found to be inhibitors of interleukin-8 receptors and protein kinase-c in the low macromolar range (Patil *et al.*, 1997).

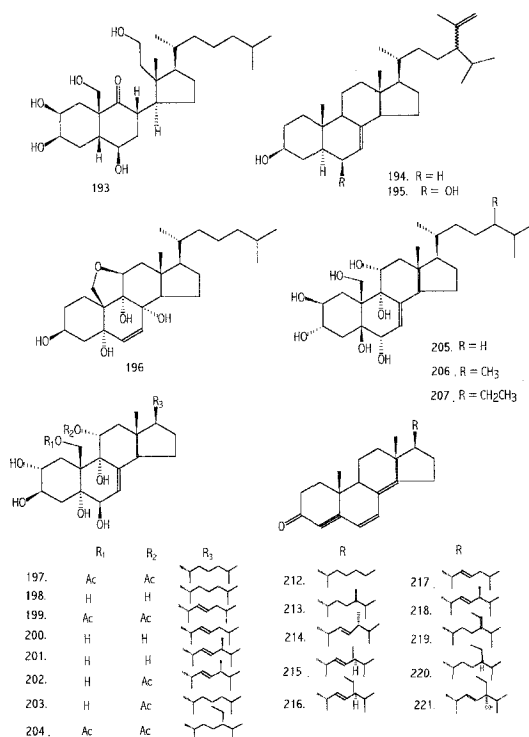
Sterols—In addition to terpenoids and alkaloids, sterols also play an important role as secondary metabolites produced by marine organisms. Several excellent reviews on the structures, biosynthesis and distribution of marine sterols have been published. (D'Auria *et al.*, 1993, Goad, 1978). Delseth *et al.* reported two new sterols 189 and 190 together with some minor and trace sterols from pacific sponges *Terpis zetirei* and *D.*



herbacea (Delseth *et al.*, 1979). Gunasekera *et al.* reported a polyhydroxylated epoxy steroid [191] from *Dysidea* sp. was found to be weakly cytotoxic (Gunasekera and Schmitz, 1983). Subsequently, the stereochemistry at C-6 in 191 and related compounds from *Dysidea* was revised (Fujimoto *et al.*, 1985) using $^1\text{H-NMR}$ method that involves pyridine induced deshielding.

A specimen of the sponge *D. herbacea* from Townsville, Australia contained two-ichytotoxic polyhydroxylated sterols herbastanol [192] and 19-nor herbastanol [193] (Capon and Faulkner, 1985). Two unusual C-30 sterols [194 and 195] have been isolated from the sponge *D. herbacea* (Rambabu and Sharma, 1987). Sica *et al.* reported the $\Delta^{5,7}$ sterols from the sponges *Ircinia pipetta* and *D. avara* (Sica *et al.*, 1987). A novel toxic polyhydroxylated sterol [196] has been isolated from the Mediterranean sponge *D. tupa* (Braekman *et al.*, 1988). West *et al.* isolated a group of eight new polyhydroxylated sterols [197-204] which were showing a common 5α -cholest-7-en- $2\alpha,3\beta,5,6\beta,9\alpha,11\alpha$, 19-heptol frame work and various conventional side chains from *D. etheria* (West and Cardellina-II, 1988). Subsequently the same group reported three new polyhydroxylated sterols [205-207] from *D. etheria* (West and Cardellina-II, 1989). The structures of 197-207 were determined by spectral analysis. Isaacs *et al.* reported a group of polar sterols 208-211 from *D. herbacea* collected near Massawa, Ethiopia. The stereochemistry of 211 was determined by using extensive NOE measurements and the structures were determined by study of spectral data (Isaacs *et al.*, 1991). Kobayashi *et al.* isolated ten 3-oxo-4,6,8 (14)-triene sterols [212-221] from the sponge *D. herbacea* (Kobayashi *et al.*, 1992). The structures of 212-221 were elucidated from spectroscopic evidences.

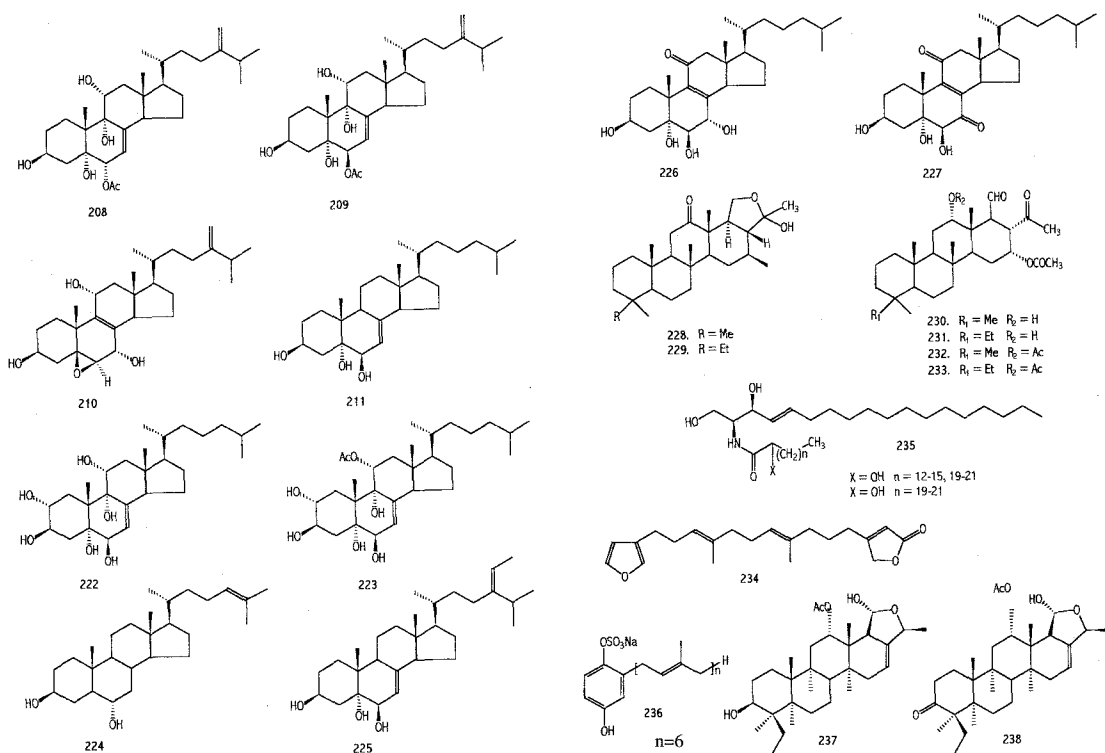
Milkova *et al.* reported two new polyhydroxylated sterols 222 and 223 from the sponge *D. fragilis* (Milkova *et al.*, 1992).



Yongli *et al.* isolated a novel sterol 224 from the South China Sea sponge *Dysidea* sp. (Yongli *et al.*, 1992). Subsequently, Yongli *et al.* reported a new sterol 225 along with 224 from the south china sponge *D. fragilis* (Yongli *et al.*, 1993). The structures of 224 and 225 were established by spectral analysis. Elenkov *et al.* reported the sterol composition and biosynthesis of the Black Sea sponge *D. fragilis* (Elenkov *et al.*, 1994). Casapullo *et al.*, reported two cytotoxic polyoxygenated sterols 226 and 227 from the sponge *D. incrustans* (Casapullo *et al.*, 1995).

Miscellaneous - In addition to above classified compounds from the genus *Dysidea* the following compounds have been isolated.

Six new biologically interesting alkylated scalarins, scalar dysin A [228], scalar dysin B [229], scalarherbacin A [230], scalarherbacin B [231] and acetates of scalarherbacin A [232], B [233] have been isolated from *D. herbacea* (Kashman and Zviely, 1979). A



new C₂₁-furanoterpene furospongolide [234] has been isolated from the marine sponge *D. herbacea* collected in the Gulf of Suez, Red Sea. (Kashman and Zviely, 1980). The sponge *D. etheria* yielded ceramide [235] (Grode and Cardellina-II, 1983) and the structure was determined by interpretation of spectral data. The sponge *D. herbacea* yielded hemagglutinins (Kamiya *et al.*, 1985). The major component DHA-I is a protein with a mol wt of 26,000, which dissociates into subunits of equal size (14,000). It contains large amounts of glutamic acid and aspartic acid, but no half-cystine, methionine or histidine. The sponge *Dysidea* sp. yielded a new hexaprenyl hydroquinone sulphate [236] (Fusetani *et al.*, 1987) and was found to inhibit proton-potassium ATPase. The Fatty acid composition of *D. fragilis* from Black Sea has been determined (Chirstie *et al.*, 1992) by analytical GC, Silver ion HPLC and GC-MS. More than a hundred different fatty acids were identified, which are similar to those tropical Seas and two new fatty

acids 13-methyl-tetradec-4-enoic and 14-methyl-hexadec-6-enoic acids, together with demospongic acids. *i.e.*, 5,9,17-tetracosatrienoic, 5,9,17-pentacosatrienoic and 5,9,19-pentacosatrienoic acids. Jaspars *et al.*, isolated two new scalaranes, 3-hydroxy-20, 22-dimethyl-20-deoxosclarin [237] and 3-oxo-20, 22-dimethyl-20-deoxosclarin 8 [238] from two different sponges and examined their taxonomic anomaly (Jaspars *et al.*, 1997).

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