Field and mesocosm test methods to assess the performance of biodegradable plastic under marine conditions

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Keywords: plastic pollution, standard test methods, biodegradability, PHA, 3-tier test scheme

Abstract

The pollution of the natural environment, especially the world's oceans, with conventional plastic is of major concern. Biodegradable plastics are an emerging market bringing along potential chances and risks. The fate of these materials in the environment and their possible effects on organisms and ecosystems has rarely been studied systematically and is not well understood. For the marine environment, reliable field test methods and standards for assessing and certifying biodegradation are lacking. In this work we present newly developed field tests to assess the performance of biodegradable plastics under natural marine conditions. These methods were successfully applied and validated in three coastal habitats (eulittoral, benthic and

pelagic) and in two climate zones (Mediterranean Sea and tropical Southeast Asia). Additionally, a stand-alone mesocosm test system which integrated all three habitats in one technical system at 400-L scale independent from running seawater is presented as a methodological bridge. Films of the positive control test material polyhydroxyalkanoate copolymer (PHA) and the negative control low density polyethylene (LD-PE) were used to validate the systems. While LD-PE remained intact, PHA disintegrated with speed depending on the habitat and the climate zone. Together with the existing laboratory standard test methods, the field and mesocosm test systems presented in this work provide a 3-tier testing scheme for the reliable assessment of the biodegradation of (biodegradable) plastic in the marine environment. This toolset of tests can be adapted to other aquatic ecosystems.

Introduction

Biodegradable plastic materials are being introduced into the market with a growing share in recent years [1], with common uses including food packaging, very lightweight bags and agricultural applications. The possible environmental benefit of biodegradable plastics is dependent on the application in which they are used. For example, certified soil-biodegradable mulch films [2] are utilized to substitute conventional plastic polyethylene films, especially for those applications in which complete recollection of the film is not possible. After use, certified soil-biodegradable mulch films are plowed into the soil and biodegraded by microbes, avoiding the accumulation of non-degradable plastic fragments in the fields. It is fundamental to keep in mind that biodegradation is the remineralization of the material to CO_2 (and/or CH_4 under anoxic conditions) and water, and the conversion to biomass by the metabolic action of microbes. The proof of environmental biodegradability, i.e. beyond

controlled lab test conditions, is the first prerequisite for a polymer to be considered as a sustainable alternative to non-biodegradable materials in a specific environment. Furthermore, biodegradable substitute materials are opted for as a mitigation against marine plastic pollution (e.g. [3]). However, while there exist certification schemes for industrial compostability (e.g., based on standards [4-6]) or soil biodegradability [2], there is a lack of standards and methods for assessing marine biodegradability. It is well recognized that positive experimental results on biodegradation of polymers in one environment (e.g. in industrial compost) do not automatically imply comparable or sufficient biodegradation rates of that polymer in another system (e.g. in the marine environment) [7-8].

In fact, the knowledge on the biodegradability of polymers in freshwater and marine environments is still very limited and experimental data vary strongly. Hence, there is a clear need (and also clear international political call, e.g. The European Green Deal, 12.11.2019 [9]) for reliable environmentally relevant standardized test methodologies to produce comparative and scientifically valid results and for the verification of claims based on sound knowledge rather than assumptions [10]. Data from such tests will help to assess environmental benefits and potential risks and will allow for their comprehensive life cycle assessment.

The number of tests on biodegradable plastics that have been conducted directly in the marine environment is increasing (e.g. [11-15]), however the comparison of the results remains difficult. *In-situ* experimentation under sometimes harsh marine conditions involves several risks including theft, sabotage, conflict with other activities like fisheries and boating, and natural forces such as strong currents and wave action. Loss of samples by anthropogenic impact or natural forces [13, 15,16] could substantially jeopardize the outcome of experiments. Also, most studies have only tested in one habitat (e.g. [16]) or were too short-termed (e.g. [12]) to produce reliable

results on the full biodegradation of a certain material under marine conditions. Many studies also lack the application of a positive control to assess the general microbial activity under the given test conditions and the potential of the microbial community to biodegrade organic materials within the experimentation time at all (e.g. [17]).

The main goal of this study, partly conducted during the EU project Open-Bio [18, 19, 20], was to develop robust, reliable *in-situ* test systems to assess the performance of biodegradable plastic materials under natural marine conditions that: (a) withstand natural forces for extended times of exposure, (b) allow for testing in different marine habitats without the loss of samples and (c) generate samples ideally deteriorated by mere biological processes rather than physical destruction. Additionally, the aim was to create a basis for an EN or ISO standard test method.

So far, standard test methods for *in-situ* testing of plastic materials only exist for the sea surface [21] and are now under development for the seafloor and beach scenario [22]. For laboratory testing several standards addressing different habitat scenarios are available (water: [23]; sediment-water interface: [24, 25]; beach sand: [26, 27]) and more are under development for the water column scenario [28, 29]. Mesocosm (or tank) tests have been used to examine the degradation of plastics [30, 31] under controlled conditions; these mimic an environment which better approximates nature than is possible in small-scale laboratory tests. Thus, mesocosm tests can play an important role as the methodological bridge between field and lab tests (3-tier approach), allowing better understanding of the fundamental parameters controlling biodegradation. Currently, only one standard test exists [32] which describes the degradation of plastic materials in mesocosm tests; however it requires the supply of running seawater. For this reason, we developed a stand-alone mesocosm system independent of the direct access to seawater.

The marine environment is extremely complex and different compartments cover a large variety of conditions. Therefore, a set of tests is required to address the different aspects. Considering that 80% of plastic pollution comes from land [33], we focused mainly on the coastal regions. We chose three scenarios that cover different ecological aspects: the eulittoral, where plastic is washed to the beach and eventually buried in the sand, the pelagic, where plastic is floating neutrally buoyant in the water and the benthic, where plastic is sunken to the seafloor. Additionally, two different locations were investigated, where tests are technically and economically feasible and environmentally relevant. The field test systems were first developed in the Mediterranean Sea (at the islands of Elba and Pianosa, Italy) and further refined in Southeast (SE) Asia (Pulau Bangka, NE Sulawesi, Indonesia).

For biodegradable plastics, biodegradation is typically assessed with respirometric methods in closed lab test systems, where oxygen consumption or CO_2 evolution (and/or CH_4 under anoxic conditions) is measured over time (e.g. [23]). Disintegration occurs as a consequence of biodegradation, however fragmentation and disintegration of plastic, especially under field conditions, can also have purely physical (light, abrasion, mechanical stress) and chemical (hydrolysis) causes. In this study we used the degree of disintegration of the test material as a proxy for the biodegradation under the condition that (a) the material has been proven to biodegrade under laboratory conditions, hence is inherently biodegradable and (b) under the assumption that our test systems are able to exclude physical forces efficiently enough that disintegration is primarily caused by the metabolic action of microorganisms.

Materials and methods

Test materials

To validate the newly developed test systems, the disintegration of a polyhydroxyalkanoate copolymer (PHA, MIREL® P5001, Metabolix, Cambridge, USA) - a bio-based, bacteria-derived, biodegradable material with a film thickness of 85 µm - was used as a positive control. A non-additivated low-density polyethylene (PE-LD, LUPOLEN 2420K, LyondellBasell, USA) film with a thickness of 30 µm was used as a negative control. PE is the most common and most abundant material for light packaging and shopping bags and is regarded as non-biodegradable [34].

Test systems and settings

HYDRA test frame

The materials were mounted in a holder termed the 'HYDRA test frame' (Fig 1) to protect the specimens during exposure against physical stress and to prevent loss of larger fragments after the onset of eventual disintegration. In the Mediterranean Sea tests, the film specimens were covered on both sides with a plastic grid of PE-LD (POLY-NET, NSW-General Cable, Nordenham, Germany), with diamond-shaped meshes of 4 x 4 mm size. To be in accordance with standard methods for compost [4], after the successful experiments in the Mediterranean Sea a mesh size of 2 x 2 mm square polyester mesh (PES, SEFAR, Heiden, Switzerland) was chosen for the next generation of HYDRA test frames and their subsequent application in another climate zone (tropical SE Asia). This had also the advantage of a better retention of smaller fragments. The exposed area of test material not covered by the filaments of the mesh remained almost the same (51% vs. 52%) and the mesh was not tightly adhering to the film to ensure the complete exposure to water. The specimens covered in mesh were fixed between two plastic frames (PE-HD 300, Technoplast, Lahnstein, Germany) with 3 mm thickness and of 260 x 200 mm external and 200 x 160 mm internal

dimensions leaving a surface of 320 cm^2 of material directly exposed. The frames, the mesh and the test material were then assembled with plastic bolts and nuts (Nylon 6.6, www.kunststoffschraube.de, Singen, Germany) and each test frame was individually tagged with a code made from cable markers (HellermannTyton, Tornesch, Germany) fixed with a cable tie. The dimensions of the test items were chosen to provide enough material for subsampling (Fig 1, left) for further analyses.

Fig 1. HYDRA® test frame. Left panel: Scheme of test frame. (1) upper frame, (2) holes for fixing the frame to sample holders, (3) plastic nuts, (4) mesh covering the test material, (5) test material (film), (6) area for subsamples. Right panel: photo of HYDRA test frame, cut open to show different layers. (7) upper frame, (8) upper protective polyester mesh (modified to 2 x 2 mm), (9) test material (plastic bag), seen through film: lower mesh, lower frame.

Field tests

Three habitats (eulittoral, pelagic and benthic, Fig 2) were chosen to test the disintegration of biodegradable polymers in the sea, reflecting three common scenarios where (conventional) plastic litter is found in the ocean [33, 35].

Fig 2. Schematic illustration of coastal habitats. (1) Eulittoral: intertidal beach scenario, (2) Pelagic: water column scenario, (3) Benthic: seafloor scenario. Dots below samples symbolize sediment.

For each habitat a specific support structure for the test frames was developed (see below).

Field test locations

Fig 3. Maps of the field locations. Left: Mediterranean field sites at the islands Elba and Pianosa (Italy): (x) eulittoral, (+) pelagic and benthic test sites. Right: tropical field site at Pulau Bangka (NE Sulawesi, Indonesia) (*) pelagic and benthic test sites.

Matrices: Seawater and sediment as incubation media for the eulittoral (field and mesocosm) and benthic (mesocosm) tests

Natural Mediterranean seawater was taken at the boat slip at Seccheto, Isola d'Elba, Italy **(**42°44'06.5"N 010°10'33.5"E**)** and was used to wash the sediment and to fill the mesocosms. Natural marine sediment of siliclastic origin for the eulittoral (field and mesocosm) tests was retrieved at about 0.1 m water depth from the beach of Fetovaia, Isola d'Elba, Italy, **(**42°44'00.1"N 010°09'15.3"E) and is called "beach sediment". Carbonate sediment for the benthic mesocosm tests was collected from the seafloor at 40 m depth off Isola di Pianosa, National Park Tuscan Archipelago, Italy, **(**42°34'41.4"N 010°06'30.6"E**)** and is called "seafloor sediment". The sediment was wet sieved with seawater through a 10 mm mesh in order to eliminate coarse particles such as stones and shells and was resuspended several times to flush out very fine particles.

Study 1: Field experiments Mediterranean Sea

From April 2014 to October 2016 the eulittoral test system as described below was set up in a former saline basin on the Island of Elba, Terme di San Giovanni, Portoferraio (42°48'12.1"N 010°19'01.0"E, Fig 3). This location was chosen because of its unique structure: it is protected against waves and storms by a seawall while still interacting with the open sea by means of several inlets. There is a small irrigation trench that occasionally brings some freshwater runoff to the basin. The ecological character of

the site is that of a typical Mediterranean coastal lagoon with weak estuarine influence. Beach sediment was prepared for the eulittoral tests as described above.

The pelagic and benthic tests were performed from June 2014 to October 2016 in the marine protected area of the National Park Tuscan Archipelago off the island of Pianosa (42°34'41.4"N 010°06'30.6"E, Fig 3). The test system was set up approximately 500 m from the south-eastern shore close to Punta Secca. The ecological character of this site is that of a typical Mediterranean coastal habitat with low anthropogenic impact and the risk of loss or disturbance by human activities was regarded as very low.

Eulittoral field test system

This test system mimicked an intertidal sandy beach in which plastic was buried and experienced changing conditions of being wetted and falling dry with the fluctuations of the sea level caused by tides and waves (Fig 4). A plastic (PP) bin of approx. 60 L volume was used to confine the sediment and the samples. The bottom of the bin was perforated with holes of 25 mm diameter to allow the ambient water to infiltrate. Two layers of plastic mesh (PVC-covered glass fiber 1 x 1 mm and polyester gauze 280 x 280 μm were placed above the holes to prevent the loss of sediment. A layer of 17 cm of beach sand was filled into the bin and a plastic pipe of 32 mm diameter, covered by fine mesh at the top opening, was installed as a short-cut for the rising and falling water to facilitate the drainage of the sediment. Three test frames were buried at a sediment depth of 10 cm with an inclination of about 11° from horizontal to allow the overlaying water to run off after each flood event. The filled bins were placed in groups of five on a wooden rack in order to position the test specimens in the mid-water line in a coastal lagoon. The rack structures and bins were covered by wire barbs to prevent disturbance from birds.

Fig 4. Eulittoral test system. Top left: cross section through bin containing beach sand and samples. (1) wire barbs to deter birds, (2) 60-L plastic bin, (3) sand from a local beach, (4) test item frame, (5) fine mesh (280 µm), (6) coarse mesh (1000 µm), (7) perforations in bottom of the bin, (8) equilibration pipe, (9) fine mesh (280 µm) on the top of the pipe to allow for seawater outflow at high tide. Top center: 3 specimens in the test bin before covering with an additional layer of sand. Top right: test bin at high tide. Bottom left: wooden rack with 5 test bins at midwater. Bottom center: wooden rack with test bins at lower tide. Bottom right: schematic illustration of the eulittoral test system. (1) wooden posts, (2) wooden plank to support the bins, (3) metal chain to adjust the plank, (4) test bin, (5) high water level, (6) mid-water line, (7) low water level.

Pelagic field test system

This test system represented the habitat of the free water column where the plastic was neutrally buoyant and was in contact with only seawater as a matrix (Fig 5). The test frames were fixed with cable ties perpendicularly on top of each other to a plastic bar, four of which were mounted to a plastic cylinder (PE-HD 300, Technoplast, Lahnstein, Germany) with stainless steel bolts and nuts to form 4 radial wings. The cylinder was suspended upright at a water depth of about 20 m from a float with a lift of approximately 5 L and kept in place by a concrete anchor weight (50 kg) at the seafloor; this anchor was connected by a stainless steel cable with stainless steel bolts and nuts, or shackles. To prevent corrosion the steel cables were equipped with zinc anodes, fixed with stainless steel bolts and nuts.

Fig 5. Pelagic test system. Left: schematic illustration. (1) test materials in frames with mesh, (2) plastic pipe suspended from a float at 20 m water depth with specimens radially attached, (3) float, (4) steel cable to anchor stone. Upper left: detail of cylindrical rack with specimens attached radially, upper right and lower left: divers at pelagic test system at 20 m water depth. Lower right: detail of specimen in situ, note biofilm after 2 weeks of exposure (Mediterranean Sea).

Benthic field test system

This test system represented the habitat of the seafloor where the plastic was in contact with seawater and sediment as matrices (Fig 6). 3 x 5 test specimens were mounted on a rack of plastic bars and placed on the seafloor at approximately 40 m (Mediterranean Sea) or 32 m (SE Asia) water depth. Each rack was tied to the anchor block of the pelagic test system with a stainless steel cable and fixed to the sediment surface with U-shaped iron bars.

Fig 6. Benthic test system. Right: schematic illustration. (1) test materials in frames with mesh, (2) automated temperature logger attached to rack, (5) anchor stone, also for pelagic test system (see fig 4), (6) plastic rack with specimens in test frames attached, (7) steel cable to anchor stone. Upper row: divers at benthic test system at 40 m water depth (Mediterranean Sea), note sediment and debris on specimens. Lower left: divers detach test frame, fixed with cable ties to the rack. Lower right: specimens are packed individually into PE plastic bags for further treatment in the laboratory.

Study 2: Field experiments Indonesia

From March 2017 to October 2018 pelagic and benthic tests were performed in Sahaong Bay, Pulau Bangka, NE Sulawesi, Indonesia (01°44’35.4”N 125°09’09.3”E, fig 3). This location was chosen because it has been used for generations by local people to permanently anchoring floating fishing huts (therefore a historical proof of good storm protection). The bay is also very narrow and not suited for fishing methods that might have interfered with the test systems such as purse seining or trawling. The benthic system was deployed at a water depth of 32 m on sand to avoid the coral reef. The pelagic test was suspended in 20 m water depth. The ecological character of the site is that of a typical coastal area in the wet tropics, with high water temperature all year round and elevated nutrient content due to terrestrial run-off.

As a modification to the experiments in the Mediterranean Sea the mesh used for all test frames was 2 x 2 mm (Fig 1). There were no eulittoral tests done.

Study 3: Mesocosm experiments with Mediterranean Sea matrices

The mesocosm experiments simulated, in a multi-100-liter tank under controlled conditions beyond laboratory flask scale (usually a few 100 mL of volume), the degradation of the polymers PHA and LDPE in the marine environment in three coastal habitats: eulittoral, pelagic and benthic (Fig 7). These habitats were mimicked in a combined tank system as follows: PE-HD plastic tanks (Dolav GmbH, Bad Salzuflen) with inner dimensions 93 x 113 x 60 cm were set up in triplicates in a climate room at 21 °C. Each set consisted of the eulittoral test tank placed on top of the tank with the benthic/pelagic test system, connected by a closed-system circuit of about 400 L seawater. Water was pumped into the upper tank in a way that simulated a semidiurnal tide, creating complete flooding every 12 h and a complete draining 6 h later. Tests were conducted twice for about 10 months each and termed year 1 (y1) and year (y2), with 4 sampling time intervals each. Before repeating the test, the matrices water and sediment were renewed.

Eulittoral mesocosm test system

The top tank (Fig 7) mimicked the eulittoral (intertidal) scenario and was filled with a layer of 15 cm of beach sediment. The incoming water was led by a hose to a perforated pipe (diam. 12 mm, L = 1 m) which was laid onto the sediment surface. The water inlet was connected to the benthic/pelagic test tank below by a EHEIM compact 600 pump (Deizisau, Germany) with adjustable flow of 250 – 600 L/h. The upper tank

had an outlet in the center of the bottom (diam. 50 mm) which was covered by gauze (polyester, 280 µm mesh, SEFAR, Heiden, Switzerland) to prevent sediment loss. The pumping rate was adjusted to balance the inflow with the outflow by passive drainage through the bottom outlet. Twice a day the water level was risen above sediment level by pumping up seawater from the benthic/pelagic test tank below. When the pump was stopped the water was allowed to passively drain through the bottom of the tank, with the consequence of sediment and samples slowly falling dry. Samples mounted in HYDRA® test frames were buried in the sand at 5-10 cm depth with an inclination of 11° from horizontal to prevent water from being trapped on the film at falling water level during the simulated tidal cycles. At the end of a test interval samples were carefully dug out of the sediment and processed as described below.

Combined benthic and pelagic mesocosm test system

The benthic/pelagic test tank below the eulittoral test tank (Fig 7) was filled with a layer of 5 cm seafloor sediment. Test frames were placed horizontally on the sediment surface and weighted down with small blocks of granite to prevent them from being moved by the water flow. The tank was filled with 400 L of natural seawater.

For the pelagic tests, samples mounted in HYDRA® test frames were hung from a rack perpendicularly in the water column. The water was continuously moved by a water EHEIM compact 300 pump with a rate of about 300 L/h. The tank was illuminated from above in a 12/12 h light/dark rhythm by 2 BIOLUX L 36W/965 fluorescent lamps (OSRAM, Munich, Germany) with a nominal luminous flux of 2300 lm each. The water bulk was connected to the eulittoral test set on top and cycled by a pump as described above. At the end of each test interval polymer samples were retrieved from the tanks and processed as described below.

Fig 7. Mesocosm tank system. Left: schematic illustration. Upper left: upper tank, which mimicked the eulittoral (intertidal beach scenario) habitat. (1) beach sand, (2) test frames, (3) drainage pipe connected to lower tank, (4) perforation to drain sediment into (3), (5) hose connected to (13) delivering water to upper tank via (6) a perforated pipe, (7) high-water line in upper tank. Lower left: lower tank which mimicked the pelagic (water column scenario) and the benthic (sublittoral, seafloor scenario, sediment-water interface) habitat. (8) sediment surface, (9) pelagic test frames pending in the water, (10) water, (11) support pipe, connecting lower and upper tank, (12) flange as connector and seal between upper and lower tank, (13) pump with timer delivering water from lower to upper tank, (14) circulation pump for lower tank, (15) aquarium lamps 12h/12h, (16) high-water level, (17) low-water level. Upper center: overview of the mesocosm tank system. Upper right: view into the upper tank with eulittoral test. Lower center: view into the lower tank with pelagic and benthic tests. The pelagic samples were hanging in the water and the benthic samples were placed on the sediment on the tank floor. Lower right: view onto a sample, which was buried in the eulittoral sediment.

Environmental conditions in the different test settings

In order to characterize the natural and the mesocosm habitats the environmental parameters such as temperature, light, salinity, pH, oxygenation were monitored. Samples of the matrices water, sediment and porewater were taken and analyzed for physical, chemical and biogeochemical properties. The data are given here in the methods section as a general characterization of the experimental conditions and the matrices used rather than in the results section which is focused on the results of the disintegration experiments.

Matrix samples: water and sediment

The physical sediment parameters grain size distribution, permeability and porosity were analysed with standard methods (e.g. [36]) and are given in Table 1.

Table 1. Physical properties of the sediments from the field and mesocosm experiments

	Mediterranean field			Mediterranean mesocosm				Southeast Asia field
	benthic	eulittoral		benthic		eulittoral		benthic
		year 1	year 2	year 1	year 2	year 1	year 2	
Grain size mean ± SD (µm)	***213 ± 26***	***214 ± 5***	***278 ± 5***	***181 ± 23***	***146 ± 47***	***278 ± 14***	***206 ± 9***	***197 ± 47***
Permeability mean ± SD (10^{-11} m^{-2})	***1.12 ± 0.9***	***7.46 ± 1.5***	***20.06 ± 2.0***	***2.60 ± 0.6***	***2.40 ± 1.2***	***17.9 ± 0.6***	***9.54 ± 2.2***	***2.26 ± 0.8***
Porosity mean ± SD	***0.67 ± 0.6***	***0.41 ± 0.2***	***0.43 ± 0.5***	***0.62 ± 0.4***	***0.51 ± 0.6***	***0.45 ± 0.2***	***0.42 ± 0.8***	***0.54 ± 0.1***

*SD = standard deviation

A selection of nutrient-related parameters (C, N, P, Si compounds), photopigments and metals as well as potentially toxic substances such as arsenic and heavy metals (cadmium, chromium, copper, lead, mercury, nickel, zinc), and a catalogue of anthropogenic persistent organic pollutants (POPs) were analyzed in water and sediment from the tanks and from the field sites by external accredited analytical services (Institute Dr. Nowak GmbH & Co. KG, Ottersberg, Germany and SGS-WLN Analytical Services, Manado, Indonesia) according to standard methods. For the detailed list of parameters, methods and the detection limits see S1 – S4 Tables.

Water temperature

Water temperature was automatically recorded with HOBO UA-002-64 Pendant temperature/light loggers (Onset Computer Corporation, Bourne, MA, USA) or TinyTag Aquatic 2 - TG-4100 data loggers (Gemini Data Loggers Ltd, Chichester, West Sussex, UK), attached directly to the test devices.

Light

The light regime at the pelagic/benthic field test systems was assessed exemplarily by measuring a depth profile of the photosynthetically available radiation (PAR) within the sensor range of 400 - 700 nm; this was done by a diver from the water surface to the

benthic test systems at the seafloor with a LiCor PAR Sensor LI-192 (LICOR Inc., USA) in an underwater housing on sunny days at midday (Mediterranean Sea) or with a HOBO Pendant temperature/light logger in SE Asia. In the Mediterranean Sea the light intensity at the pelagic test system (20 m depth) was 13 % and at the benthic test system (40 m depth) 3 % of the surface intensity. In SE Asia the light intensity at the pelagic test system (20 m depth) was varying between 1.2 and 2 % and at the benthic test system (32 m depth) it was 0.5 % of the surface intensity.

Salinity, pH and oxygen

Salinity, pH and O_2 concentrations were measured in samples from each of the three field sites: from the free water around the benthic and pelagic test systems, from the sediment porewater retrieved from the bins of the eulittoral test system at the level of the test material, and from the mesocosm tanks. Measurements were taken using a TetraCon® 925 conductivity sensor, a SenTix® 940 pH sensor SenTix® 940, and a FDO® 925 oxygen sensor attached to a Multi 3420 (WTW, Weilheim).

Mesocosm tests

The measured water temperature was 20.5±1 °C (mean±standard deviation) (set: 21 °C) and mean light intensity on the sediment surface of the benthic tests was 11.56 µmol photons·m^{-2}·s^{-1}. Salinity was about 39 with a variation of ±1 for both years (set: 38.5). The pH was stable at 8.1±0.1 and the oxygen concentration was close to air saturation (98±2 %). Water chemistry: Throughout the experiments, water in all tanks and porewater from the eulittoral sand contained low to moderate levels of nutrient related parameters (TN, NO_2, NO_3, NH_4, TP, TOC, DOC, Chl *a*, Phaeophytin, etc.; S1 Table). Compared to the field data nutrient concentrations were similar or slightly elevated (S1 Table). Dissolved aluminium was detected at low concentration. None of

the toxic substances as heavy metals, organotin compounds and POPs were detected. Sedimentology and sediment chemistry: The beach sand of the eulittoral tests was mainly of siliclastic origin and was classified using [37] as medium sand. The sediment used for the benthic experiments was composed mainly of carbonate minerals and characterized as fine sand in both years. Accordingly, porosity and permeability were slightly lower for both sands in the second year (Table 1) whereas nutrient contents were similar and low (S2 Table). Metal concentrations were low or below detection limit. None of the known anthropogenic toxins analysed in the sediments used for the experiments were detected (S4 Table).

Mediterranean Sea field test - eulittoral

The temperatures in the eulittoral test bins roughly followed the temperature of the ambient water with the daily and the tidal cycles. Daily mean temperatures of about 25 °C (yr 1) and 26 °C (yr 2) were highest between June and September and lowest mid-October and March with around 12 °C (y r1 and 2). The lowest daily minimum was -1.0 °C in January 2016. Fluctuations were pronounced, and the night temperatures were usually lower (by up to 5 °C) than in the surrounding water. Salinity of seawater in the test bins was most of the time in the range of 36 – 42; lowest values were almost 0, highest values reached 70. The pH was around 7.6 – 8.2; lowest values were below 6.8, highest values reached 9. The oxygen concentration in the test bins was mostly between 20 and 80 %, and rarely reached 100 % air saturation (see also Table 2).
Water chemistry: The porewater in the bins throughout the experiments had low to moderate levels of nutrient related parameters, however compared to the benthic and pelagic test sites they were slightly elevated (S1 Table). Dissolved iron, manganese and aluminium was detected at low concentrations (S1 Table). None of the POPs tested were detected (S3 Table). Sedimentology and sediment chemistry: The beach

sand used in the test bins was fine (mean 214±5 µm yr1) and medium (mean 278±5 µm yr2) sand. The porosity and permeability were therefore higher in the second year (Table 1). Nutrient concentrations were low or b.d.l. and of the metals analysed only zinc, nickel and chromium were detected in low concentrations. Organotin compounds, pesticides and their metabolites could not be detected in the sediment (S4 Table).

Mediterranean Sea field test - pelagic and benthic

The temperature at the two stations diverged over the course of a year due to stratification of the water column from spring to late autumn, when the temperatures in the different depths merged again. Highest daily mean temperatures were about 25 °C at the pelagic test site and 20°C at the benthic test site. Lowest temperatures were recorded early February until mid-May with around 14 °C at both sites. Fluctuations between daily minimum and maximum temperatures sometimes ranged 4 – 8 °C (pelagic) and 4 – 5 °C (benthic). At the pelagic tests at 20 m depth temperature fluctuations were more pronounced during spring and summer, reflecting an irregular mixing with the surface waters and a gradual downward migration of the thermocline through the water column. At the benthic test autumn fluctuations were more pronounced due to occasional storms that led to a mixing of the water column. At the depth of the pelagic test system (20 m) solar irradiation was about 13 % and at the benthic test system (40 m) about 3 % of the surface level. Salinity was at 38, pH was 8.2 and oxygen content was around 100 % air saturation at both sites.

Pelagic and benthic water chemistry: The water at both test sites as well as the porewater at the benthic test site had low levels of nutrient related parameters throughout the experiments (S1 Table). Dissolved aluminium could be repeatedly detected at low concentrations, as well as iron and manganese in the second year. Other metals were b.d.l. (S1 Table) No toxins or POPs were detected (S3 Table).

Benthic sedimentology and sediment chemistry: The benthic sand was fine sand (mean 213±26 µm) with low concentrations of nutrients and some metals (iron, manganese, lead, chromium) (S2 Table). Organotin compounds and pesticides and their metabolites could not be detected. No toxins or POPs were detected in the sediment (S4 Table).

SE Asia field test - pelagic and benthic

The temperature at the two stations was the same throughout the year (pelagic test: 28.6±0.4 °C, benthic test: 28.6±0.5 °C). At the depth of the pelagic test system (20 m) solar irradiation was about 1.5 % and at the benthic test system (31 m) about 0.5% of the surface level. The salinity at the two stations was similar throughout the year. The mean salinity was 34.0±0.4 at the pelagic test, and 33.9±0.2 at the benthic test. A pH of 8.1±0.1 was the same at both test sites, and oxygen concentrations were 100.8±4.6% air saturation at the pelagic test site and 101.3±6.8 % at the benthic test site.

Table 2. Water temperature, salinity, pH and oxygen concentrations of the field and mesocosm experiments.

	Mediterranean field				Southeast Asia field		Mediterranean mesocosm
	eulittoral		pelagic	benthic	pelagic	benthic	all habitats
	year 1	year 2					
Highest daily max. temp. (°C)	***34.5***	***38.4***	***25***	***20***	***28.6±0.4*****	***28.6±0.5*****	***20.5±1.0*****
Highest daily mean temp. (°C)	***25***	***30***					
Lowest daily mean temp. (°C)	***12***	***11***	***14***				
Lowest daily min. temp. (°C)	***-0.8***	***-1.0***					
Salinity range	***36 - 42****		***38***		***34.0±0.4*****	***33.9±0.2*****	***39.0±1.0*****

Minimum salinity	***~0****				
Maximum salinity	***70****				
Mean pH	***7.6 – 8.2****	***8.2***	***8.1±0.1*****		***8.1±0.1*****
Minimum pH	***6.8****				
Maximum pH	***9.0****				
Oxygen (% air saturation)	***20 – 80**** ***rarely ~100****	***~100***	***100.8±4.6*****	***101.3±6.8*****	***98.0±2.0*****

*The data for Mediterranean field eulittoral are analyzed from sediment porewater. **mean ± standard deviation.

Pelagic and benthic water chemistry: The water at both test sites as well as the porewater at the benthic test site had low levels of most nutrient related parameters throughout the experiments (S1 Table). The silica, chlorophyll *a* and phaeophytin concentrations however were higher than at the tests sites in the Mediterranean Sea. Metal concentrations were all b.d.l., only cadmium was detected once in very low concentration (S1 Table). No toxic substances or POPs were detected (S1 Table). Benthic sedimentology and sediment chemistry: The benthic sand was fine sand (mean 197±47 µm) with low nutrients and low concentrations of some metals (iron, manganese, lead, chromium; S2 Table). No toxins or POPs were detected in the sediment (S4 Table).

Plastic sampling and disintegration analysis

After a given time interval the samples were detached from their racks (pelagic, benthic) or dug out of the sediment (eulittoral), gently rinsed in ambient water, packed individually in PE plastic bags and kept wet until further treatment the same day. In the laboratory, the frame was opened, the material was carefully detached from the upper frame and mesh and photographed immersed in seawater in a tray with a digital SLR

camera (Canon EOS 5DMkII, or a SONY A7 III, with a 100 mm or 50 mm macro lens). Subsamples were taken for later optional microscopic and molecular analyses, which are not subject of this publication. The remaining sample was rinsed in deionized water, air dried and archived.

The disintegration of each specimen was determined photogrammetrically [13]. Dried samples from the Mediterranean Sea experiments were scanned on a flatbed scanner (LIDE 210, Canon Inc.). The samples from the SE Asia experiments were photographed immediately after retrieval. The images thus obtained were analyzed for area loss using ImageJ software https://imagej.nih.gov/ij/) or GIMP (http://www.gimp.org/) by outlining the boundaries of the remaining test material that had been exposed and not covered by the frame. Subsequently, the ratio of total area vs. lost area was calculated as % area loss.

Results

Study 1: Field Mediterranean Sea

Visual appearance of the samples

For LDPE, after testing the only visible material alterations were superficial discolorations, presumably from mineral precipitations, biofilm and colored organisms, e.g. sponges. Exemplary images are shown in Fig 8. In the PHA specimens, the first visible signs of disintegration were translucent areas due to progressive thinning of the polymer films, followed by the appearance of cracks, holes and finally fragmentation in all tests. Most samples showed a change in color during the time of the experiment, attributed to the formation of biofilm and mineral precipitates, rendering image analysis difficult in some cases.

Fig 8. Exemplary photos of LDPE samples from disintegration tests. Freshly retrieved samples from field and mesocosm experiments under Mediterranean Sea conditions. Note: the metal bars in the images were put onto the film to keep the sample under water for photography.

Polymer disintegration

For LDPE, disintegration was not detected in any of the tests, and will not be described further in the results section. PHA disintegration was observed in all tests (Fig 12).

Eulittoral test (intertidal beach scenario)

PHA disintegrated in both years, reaching more than 50 % after 5 months in y1 in most replicates (Fig 12, top row, center). Some replicates however showed only minimal disintegration of between 5 and 20 % after 5 to 10 months. After 22 months all five replicates were disintegrated to over 65 %. In the y2 experiment PHA disintegration was slower and reached just over 30 % after 10 months for one replicate. All replicates were disintegrated to more than 10 % after 5 months. Generally, the disintegration of all replicates of all sampling intervals was heterogeneous.

Benthic test (sublittoral seafloor scenario)

PHA showed disintegration, which however differed by year (Fig 12, top row, left). One sample showed disintegration of 31 % after 5 months and two samples 60 – 75 % after 22 months. The disintegration of all other samples in both years was below 5 %. In the y2 experiment PHA disintegration was slower and only one sample reached more than 6 %. Generally, the disintegration of all replicates was heterogeneous.

Pelagic test (water column scenario)

At sampling, all specimens were still intact after the exposure time of max. 22 months (Fig 12, top row, right). In some specimens a thinning of the material was observed, visible by more translucent areas. Small holes were detected in only a few samples, but accounted for less than 1 % area loss.

Study 2: Field tests SE Asia

Benthic test (sublittoral seafloor scenario)

In the benthic test of the SE Asia study, PHA showed rapid disintegration, with substantial material loss already visible already after 19 days of exposure (Fig 9). After 90 d almost all test material was gone in all 3 replicates (Fig 9, bottom row; fig 12, bottom row). A conspicuous longitudinal crack was observed in all specimens after exposure, in the cases where there was still material left (Fig 9). This might have been be a consequence of material shrinking when exposed to water for a longer period of time. The crack was not taken into account as loss by disintegration but ignored for the area measurement.

Fig 9. Photos of PHA samples from benthic field disintegration tests in SE Asia. Photos of all replicates from 4 exposure intervals with mean disintegration ± standard deviation per interval (left column). Disintegration in % area loss below each image. Note: the values are calculated only from the exposed area not covered by the frame.

Pelagic test (water column scenario)

Disintegration was observed in all PHA samples, however the accurate measurement of the lost area was not possible due to heavy fouling of the support structure and the polymer itself. Also, the layer of the fouling organisms (mainly encrusting red algae, sponges, hydrozoans and bryozoans) often was bonding the mesh and the polymer

together. Splitting open the test frame during post-sampling in the lab caused further fragmentation and thus a mechanical destruction of the materials as an artefact. Another factor of sample deterioration in the pelagic tests was linked to animals found in between the meshes. Presumably, crabs and clams had settled as larvae on the test material and after having grown above mesh size they had been trapped between the two layers of protecting mesh. Traces of their movements point to these animals having mechanically destroyed the polymer (Fig 10, bottom row). Because the degree of error could not be assessed a numerical quantification of the disintegration in the pelagic test in SE Asia is to be treated with caution, and rather seen as an overestimation. It is notable that outside the area of interest, i.e. under the supporting frame, almost all material was disintegrated (Fig 10, bottom row).

Fig 10. Pelagic field test SE Asia: PHA disintegration. Photos of all 3 replicates (r1 - r3) after 210 d of exposure A: specimens retrieved from the field, note the dense fouling. Images 3 and 4 show front and rear of sample r3. B: specimens unpacked, note the almost complete disintegration under the frame, and no fouling there. The light surface is mostly the crust of coralline algae remaining after the disintegration of the test material. The test material is largely disintegrated, as expressed by the percentage of area loss. Note: the values given are rather an overestimation due to likely mechanical influence of macro-organisms (see main text).

Study 3: Mesocosm tests with Mediterranean seawater and sediment as matrices

Polymer disintegration

PHA samples showed disintegration in all three tested habitats eulittoral, benthic and pelagic (Fig 11).

Fig 11. Photos of PHA samples from mesocosm disintegration tests. Scan images of all 3 replicates (r1 - r3) from mesocosm eulittoral (intertidal, beach scenario), pelagic (water column scenario) and benthic tests (sublittoral, seafloor scenario) after 151 - 152 d of exposure. Disintegration in % area loss below each image. Mean disintegration ± standard deviation in left column. Note the heterogeneous disintegration between the three replicates. Variability was low between the replicates of the samples from the eulittoral and high within the samples from the pelagic and the benthic tests.

The disintegration was gradually progressed with time, with most samples disintegrating less than 50% after 308 (y1), or 270 (y2) days of exposure. The rate of disintegration differed between habitats and was heterogeneous between replicate samples of one sampling interval. PHA disintegration was fastest in the benthic tests (Fig 12, center row, left), and slowest in the y1 experiment in the eulittoral tests (Fig 12, center row, center, empty circles), but in y2 slowest in the pelagic tests (Fig 12, center row, right, filled circles).

Fig 12. PHA disintegration data from all tests. Field tests in the Mediterranean Sea (MED) and in tropical Southeast Asia (ASIA), and mesocosm tests (MESO). Disintegration values of year 1 (empty circles) and year 2 (filled circles). Top row, left: benthic field test MED: Two replicates from the y1 experiment were kept exposed over the duration of the y2 experiment for a total of 678 days. Top row, center: eulittoral field test MED: One set of replicates from the y1 experiment exposed over the duration of the y2 (686 d). Top row, right: pelagic field test MED: Disintegration in all samples was less than 1%. Two replicates from the y1 experiment exposed over y2 (678 d). Center row: mesocosm tests: left: benthic, center: eulittoral, right: pelagic. Bottom row: benthic field test SE Asia. Note: no graph presented for pelagic field test SE Asia (data insufficient) and eulittoral field test SE Asia (not conducted).

Additional observations

Macro-organism interaction with the test materials

Several measures were taken to prevent negative impact by animals on the test system. However, some disturbances resulted from the interaction of (macro-) organisms with the exposed materials in the field, and to a lower extent also in the mesocosms.

Eulittoral field tests: The picking of birds in the eulittoral test bins was successfully prevented by wire barbs on top of the test system. Infauna like worms or clams were not observed in the eulittoral test bins. A structural problem for the racks made from untreated larch wood resulted from the activity of wood-boring bivalves ("shipworms") that had destroyed the posts after two years to the brink of collapse (Fig 13).

Fig 13. Wooden post from eulittoral test rack. Post from untreated larch wood after 26 months of exposure at the Mediterranean test site. The wood is perforated by wood-boring bivalves (white tubes) and broken. Scale: white tube lower right is about 5 mm diameter.

Pelagic field tests: Fouling, i.e. the colonization of the structures by a community of larger sessile organisms (e.g. macroalgae, sponges, hydrozoa, bryozoan, ascidians, bivalves, tubeworms), was a major issue for the maintenance of the whole structure; from the steel cable to the floats, the plastic support cylinder and the test frames (Fig 10, top row and fig 14). Regular cleaning by divers was necessary to prevent the structures from becoming too heavy. At the samples themselves the fouling amalgamated with the test materials, growing through the mesh. For heavily fouled samples it was often impossible to remove the overgrowth after sampling from the remaining polymer without creating further damage and influencing the result. Steel cables were chosen over plastic rope because turtles and some fish are known to snatch ropes while feeding on sponges and other encrusting organisms. However, on several occasions bite marks on the frames and mesh were observed, but a sample was never found bitten all the way to the specimen. On two occasions cable ties that

served to fix the codes to the samples were found cut open, and once a large puffer fish was observed grazing on the pelagic test system, having just bitten off a code tag.

Fig 14. Fouling at Pelagic test system SE Asia. Sample frames and support structure were covered almost completely by sessile animals and algae. Note the fist-size oyster in lower center (zig-zag line). Exposure time 20 months.

Benthic field tests: The test frames were laid onto the sand bottom and fixed with steel bars. The flat rigid structures were readily populated by benthic fish such as gobies which dug out dens in the underlying sediment. Octopus were observed using the benthic samples as shelter and also sea cucumbers were found digging under the panels. Bioturbation presumably by sand-dwelling worms, bivalves and shrimp under and around the specimens was also observed having changed the sediment topography at the test sites at each sampling event. On some occasions, sediment and/or seagrass leaf debris was found to have accumulated on the benthic samples, but was absent at the next maintenance dive, indicating some water dynamics at the water-sediment interface.

Discussion

Field test systems allow for long-term observation under natural conditions

Three scenarios were chosen to mimic conditions at locations where dispersed plastic debris is commonly found in coastal marine areas: the beach scenario (eulittoral), the water column scenario (pelagic) and the seafloor scenario (benthic). The field test systems have repeatably proven their stability under natural coastal marine conditions in two climate zones over many months to years of exposure. However, the 22 months

of exposure time in the Mediterranean pelagic tests (Isola di Pianosa, Tyrrhenian Sea) turned out to be too short to observe strong disintegration of the exposed materials. In experiments with the same polymer types where samples were suspended from fish farm cages, [15] found much faster disintegration, presumably due to higher nutrient content of the surrounding water.

Mesh protects well against physical impact and has little effect on disintegration

The HYDRA® test frames successfully provided protection from mechanical forces in all tests and not one sample was lost from the tests. The retrieval of almost intact sheets of the positive control material PHA after being exposed to currents and waves in the open water column for almost 2 years (Fig 12, top row, right) impressively showed the efficiency. This was also confirmed by the similar status of the test materials exposed in the mesocosm test. In all samples there was a slight effect of the mesh visible at the test material surface with a differentiated thinning below the filaments and in the voids. Although the mesh was not adhering tightly there might have been some differentiations with regard to water and gas exchange, and biofilm formation. Different micro-habitat conditions could have led to the observed disintegration pattern reflecting the geometry of the mesh. However, these minor effects are seen as an acceptable trade-off for stability and operational safety of the experiments, i.e. the retrievability of the test materials after prolonged exposure time.

Mesocosms can fill the methodological gap between lab and field

The mesocosm tests also were proven practical and generally suited to run for a longer time period in a closed circuit. The salinity could be easily adjusted, and the pH was

stable over the course of the experiment. In the pelagic and benthic tests however, photosynthetic biofilms may complicate the experiments by causing a patchy overgrowth on the samples. Being of a limited amount of water and sediment, the biotic community originating from an inoculum of random diversity can lead to the dominance of few organisms (cyanobacteria and/or algae) covering large areas in each of the mesocosm tanks. In order to avoid these differences, also between tanks, we recommend conducting the experiments in the dark. Also, higher but still physiologically relevant temperatures for the respective climate zone (e.g. 25°C in the Mediterranean tests) could be used to accelerate the processes and render the tests more efficient. The heterogeneity of the disintegration between replicate samples is not to be seen as an artefact of the mesocosm tank tests but was also observed between samples in the field tests (Fig 12). This is interpreted as a reflection of the patchiness of the microbial community according to small-scale changes of environmental conditions, as similarly observed also for example, in marine coastal sands (e.g. [38]).

Using disintegration as a proxy for biodegradation only after intrinsic biodegradability has been proven in lab tests

In an open-system biodegradation test, where metabolites like CO_2 or O_2 cannot be tracked directly with commonly available (i.e. not labelled) plastic materials, degree of disintegration is a feasible parameter to be measured in a simple and cost efficient manner. The disintegration can however only be considered a valid proxy under the assumption that: 1) the physical forces are minimized or excluded in the open system tests and, more importantly, that the test material has shown to be inherently biodegradable in a (standard) lab test (water: [23, 28, 29]; sediment-water interface: [24, 25]; beach sand: [26, 27, 39]). This is the case for the positive control/test material

PHA Mirel P5001 that was also used in lab tests during the Open-Bio project. PHAs were also proven by others to fully biodegrade (PHB by [15] in seawater according to [23]; PHA Mirel P5001 by [40] in seawater according [23]; PHBV by [41] in seawater with biofilm from fish tank as inoculum).

As we were testing thin polymer film, we chose to measure area loss by photogrammetry instead of weight attrition [32] to assess the disintegration of the test material over time. Weight attrition might be suitable for water tests but can be easily biased by a variety of factors such as fouling organisms and adhering particles - if tests are done in contact with sediment - or by additional material loss during cleaning attempts.

By the assessment of lost area via image analysis we gained a hole/no-hole ratio after a certain exposure time. The method however intentionally ignored the third dimension of the material with the disadvantage of a technical lag phase until first holes were visible. The sometimes well discernible thinning of material was not taken into account and led to an underestimation of the disintegration at the beginning, extending the biogeochemical lag phase with a methodological lag of detectability. As soon as the material is heavily fragmented small particles might escape through the mesh leading to an overestimation of the disintegration at the very end of the experiment. Both effects will be more pronounced in the case of thicker materials. For accurate photogrammetry, standardized photos of fresh samples instead of scans of dried samples are better suited and it is recommended to put some effort into obtaining good image quality.

Environmental relevance and the importance of a positive control: disintegration rate depends on the environmental setting

The disintegration rate of the same test material differed strongly between habitats and climate zones. A striking example is given by the comparison of the performance of the PHA samples: in the Mediterranean pelagic tests (Isola di Pianosa) there was only little material degradation after 678 d, whereas in the benthic of SE Asia (Pulau Bangka, Sulawesi) all three replicates of the same material were almost completely disintegrated after 90 d. This emphasizes the necessity of including positive controls in all tests. These data also are a valuable indication that the test results from one specific environmental setting have to be put into an ecological context and do not represent the performance of a given material in the whole marine realm. Metadata relevant for physiological activity of microbes such as temperature, oxygen, pH, light intensity, salinity, nutrients, metals, as well as granulometry, porosity and permeability of the sediments are also necessary to be able to interpret and compare disintegration data. These parameters should be measured on site and, ideally, throughout the experiment.

Degradation under the frame differs from exposed area of interest

Due to the design of the HYDRA test frames, parts of the test material which were not intended as area of interest were covered. Only the material exposed to the matrices was analyzed as described. However, the shielding of some of the material by the frames brought along an unintentional co-testing of the material under presumably oxygen-reduced conditions and gave hints for the anaerobic degradation of biodegradable plastic. Since some plastics may degrade faster under anoxic conditions, this observation should be addressed by a specific study.

Fouling may jeopardize measurements of pelagic field tests

The specimens exposed in the pelagic test system in SE Asia were quickly covered through heavy fouling. All the materials such as frames, mesh and support structures were already overgrown after a few months by e.g. coralline algae and sponges which could not be removed without strong physical impact on the test material - a factor that had been a priority to avoid in the experimental design. Due to rather large sampling intervals, the rapid succession of the organism community and the general impact of fouling could not be studied. A small-scale observation with regular samplings every few days to weeks would be required for a good temporal resolution to understand whether fouling would hinder or accelerate polymer degradation. A previous study in the Mediterranean Sea indicated a decrease of disintegration by fouling [15].

Pelagic test system is the least environmentally relevant, however, it should be applied for materials used directly in the marine environment

The habitats and locations reported in this work were chosen since they represent well the areas in which most plastic debris is found. The eulittoral test system is ideal to investigate materials with a specific density below that of seawater that initially float at the surface and can be washed ashore. This is the case for the most commonly used conventional polymers polyethylene and polypropylene, and also all items with a positive bulk buoyancy (e.g. bottles, foam), regardless of the material. The benthic tests well represent the seafloor scenario, the sink for most plastic materials [33, 35] due to biofilm formation. For this reason, the pelagic tests in which the materials are artificially kept from further sinking despite biofilm formation could be considered the least environmentally relevant. However, the tests provide important information about the time during which the materials float in the water column. The test remains highly

relevant to assess materials that are considered to be used directly in an aquatic environment, for example in aquaculture systems. In this case materials should be tested where they will be applied in the field.

Conclusions

Reliable systematic tests to investigate the biodegradability of plastics in nature are urgently needed to complete the environmental risk assessment of these materials entering the marine environment. The tests presented here are an efficient toolset allowing quantitative measurements under natural aquatic conditions.

Technically, all test systems were proven to be stable and are suited for use over several years to expose and test plastic films in coastal habitats. Some maintenance to the systems such as regular cleaning is required, especially in areas of high fouling. These test systems could also easily be transferred to other aquatic habitats and test sites and be adapted to test objects instead of films. The test frames sufficiently protected the samples and no mechanical damage of the specimens was detected and we are convinced that under such test conditions the observed disintegration was caused predominantly by biodegradation.

Mesocosms are well suited for manipulative experiments above lab flask scale. Controlled parameters such as temperature and also nutrient content could be set to optimum conditions to the physiological limit of the locations the matrices are collected from. More replicates with a smaller size and the exclusion of light could lead to a more consistent data set which is less affected by heterogeneity.

Eulittoral and benthic tests are considered the most realistic scenarios for biodegradable plastics most of which have a specific density >1, and from environmental observation of the fate of conventional plastic accumulating on beaches

and the seafloor. Pelagic tests are mandatory for materials which are to be used in this habitat. Testing under anoxic conditions could give a deeper insight into the performance of biodegradable polymers in aquatic environments, as most sediments are reduced in or free of oxygen.

The disintegration rate of the same test material differs between habitats and climate zones. This should be accounted for by testing in different habitats and conditions and generating a set of metadata on the ecological context of the testing site.

The outcome of this work has supported the development of a new ISO standard: “ISO/DIS 22766 (en) Plastics — Determination of the degree of disintegration of plastic materials in marine habitats under real field conditions” [22].

The assessment of marine biodegradation of plastic materials with these test systems should serve to create basic knowledge, not as an advocacy for or against a technology.

Acknowledgements

Deep thanks to the student assistants and interns of HYDRA for their tireless help in assembling and setting up several test systems over the years. We thank the National Park Tuscan Archipelago, Portoferraio for the access to the protected area of the Island of Pianosa with the research permit n.3063/19.05.2014. Dott. Emiliano Somigli and his staff are gratefully acknowledged for their support and for granting access to the Terme San Giovanni basin to perform the eulittoral tests. We thank Giorgio Vendetti from Hotel Mirage, Marina di Campo, for providing meteorological data (www.elbaexplorer.com). Research in Indonesia was conducted under the research permits no. 71 and 72/SIP/FRP/E5/Dit.KI/III/2017 and extensions granted by the Indonesian Government Ministry of Research, Technology and Higher Education,

RISTEK-DIKTI, Jakarta to C.L. and M.W.. C.L. and M.W. express their thanks to UNSRAT welcoming them as guests researchers. Thanks to Marco Segre Reinach, Ilaria Reggi, Anna Clerici, Marco Perin, and staff of Coral Eye Resort and Coral Research Outpost, Bangka Island, Sulawesi Utara, Indonesia for technical support and maintenance. Thanks also to Novamont S.p.A., Novara, Italy for providing film for tests in Italy during the Open-Bio project.

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Supporting Information

S1 Table: Metadata from water analyses from the test in SE Asia, the Mediterranean Sea and the mesocosms.

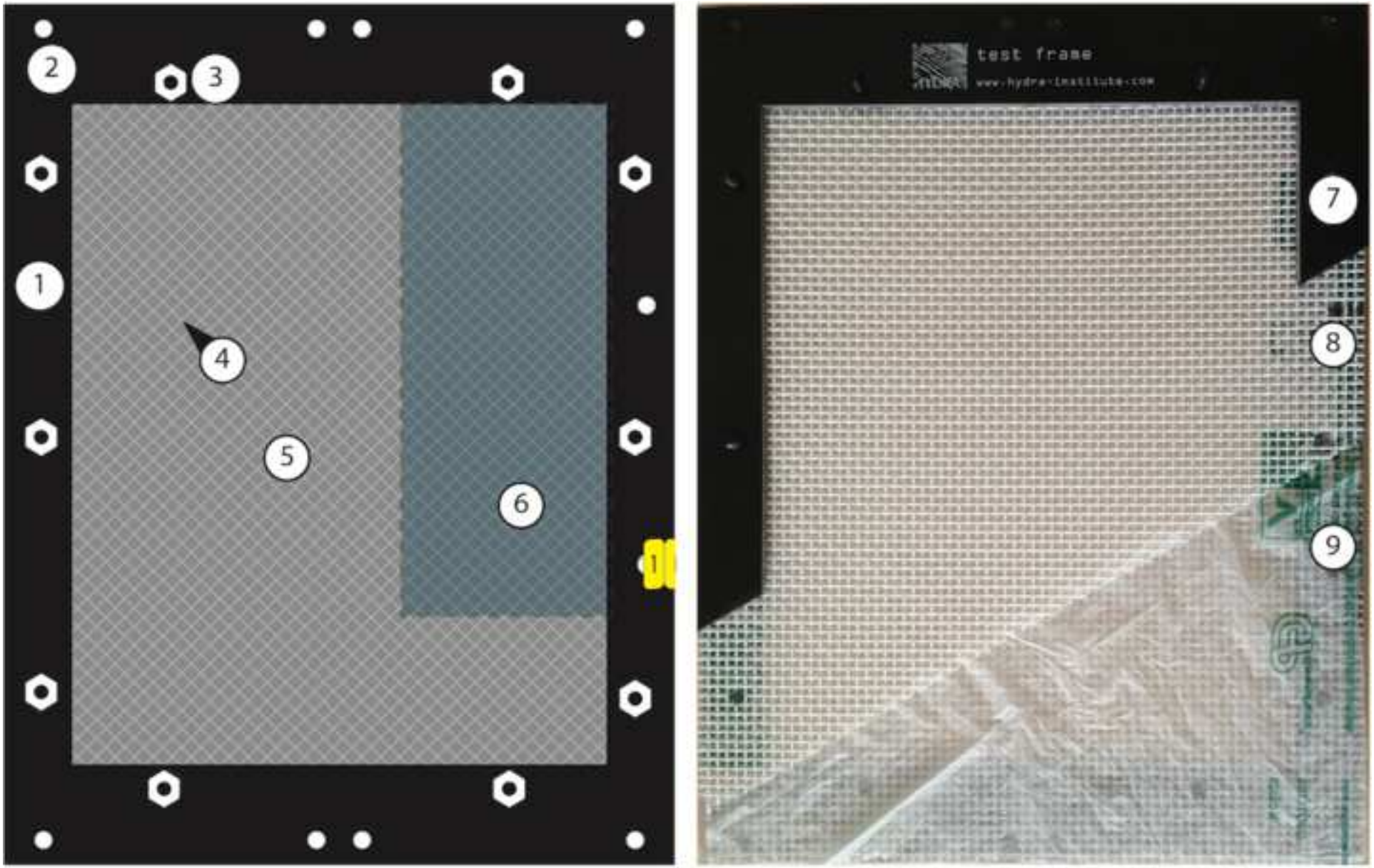
S2 Table: Metadata from sediment analyses from the test in SE Asia, the Mediterranean Sea and the mesocosms.

S3 Table: List of organic pollutants (POPs) tested in the water SE Asia, the Mediterranean Sea and the mesocosms.

S4 Table: List of organic pollutants (POPs) tested in the sediments SE Asia, the Mediterranean Sea and the mesocosms.

S5 Table: List of analytical methods for S1-S4 Tables.

Click here to access/download;Figure;Fig1.tif



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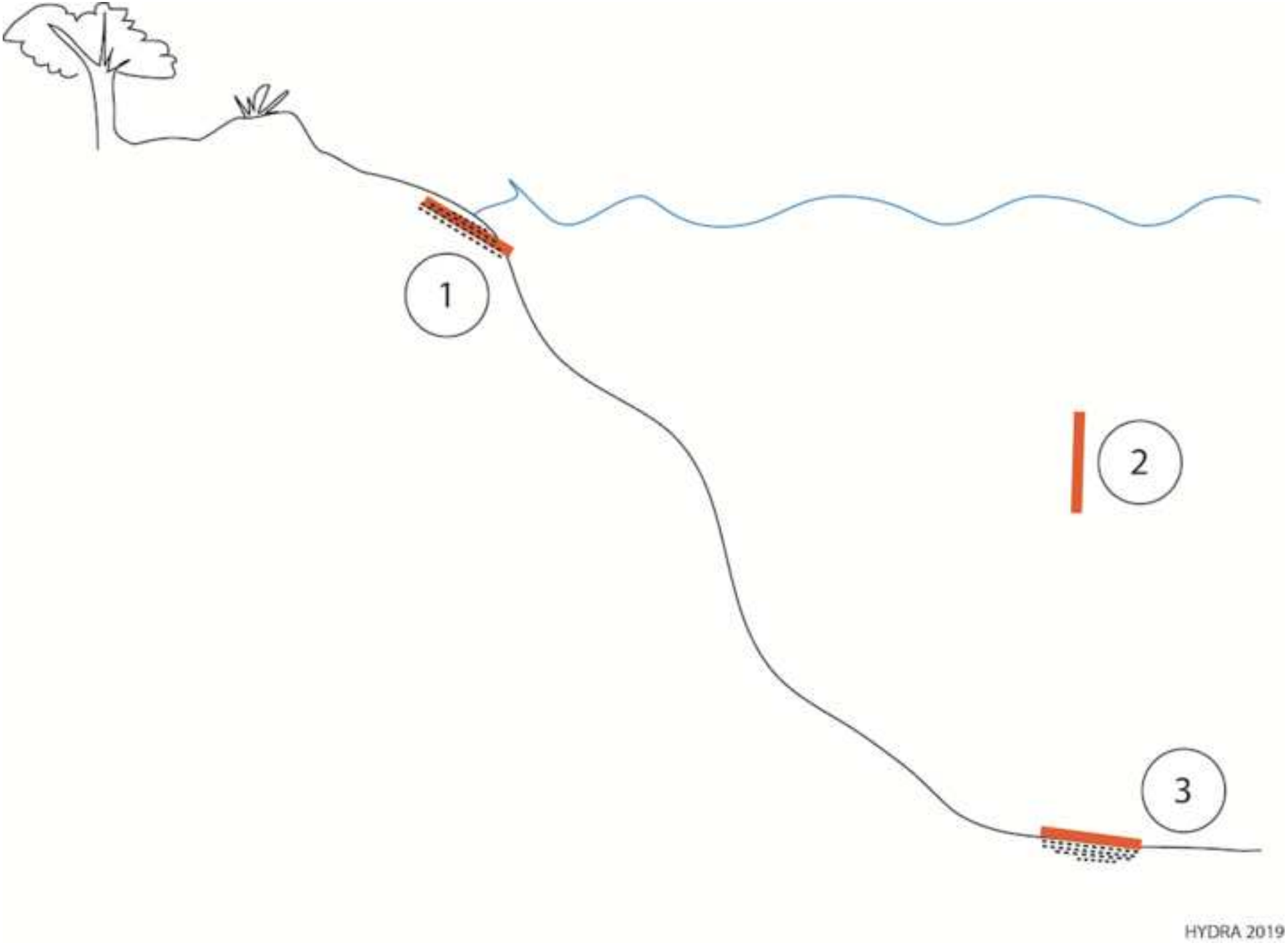

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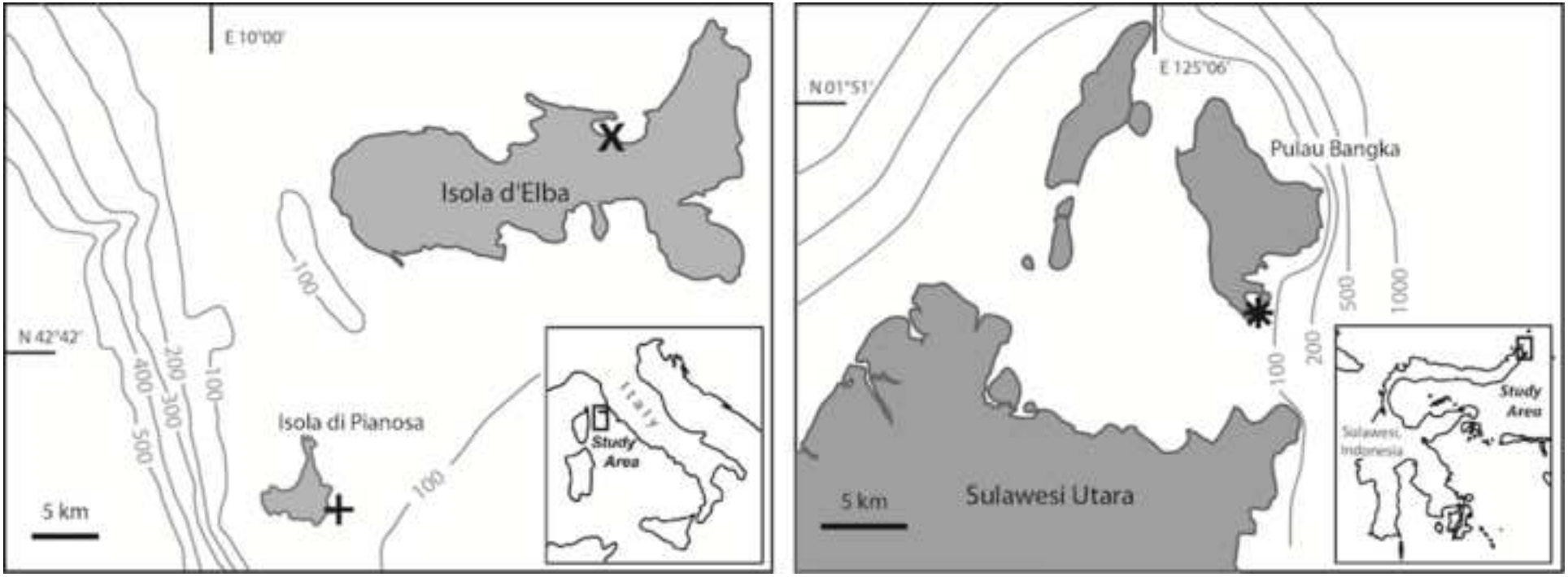

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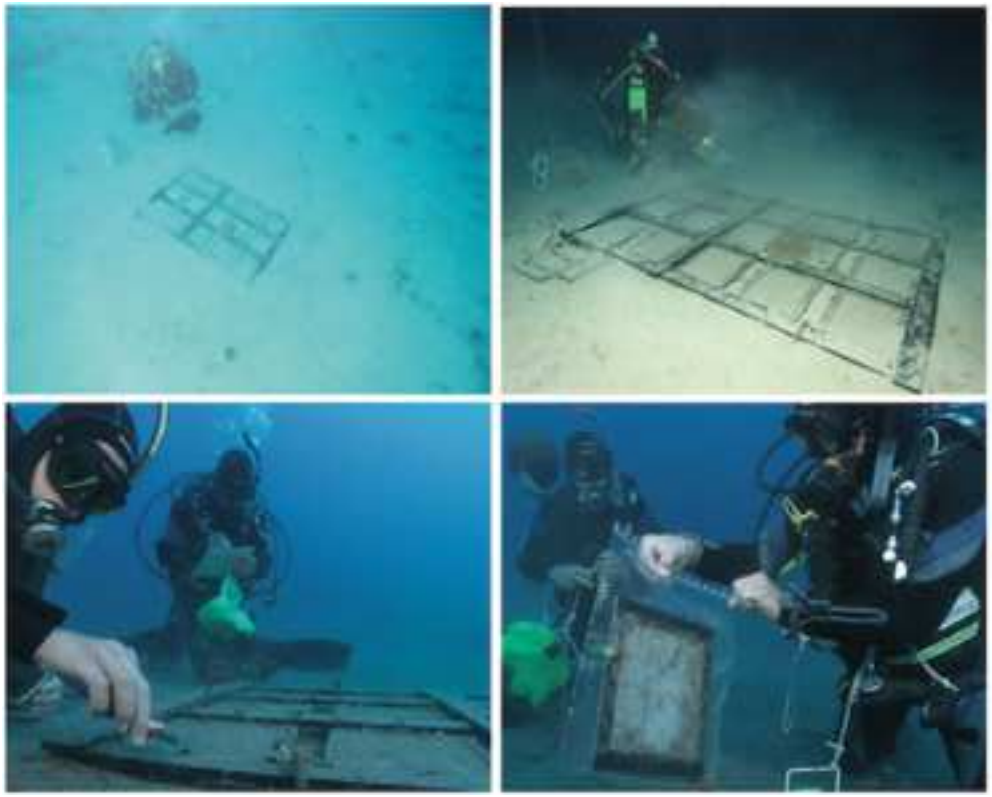

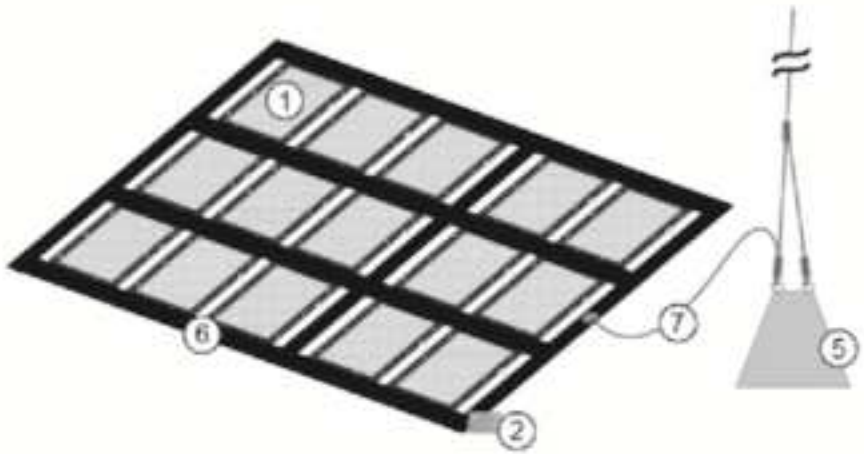

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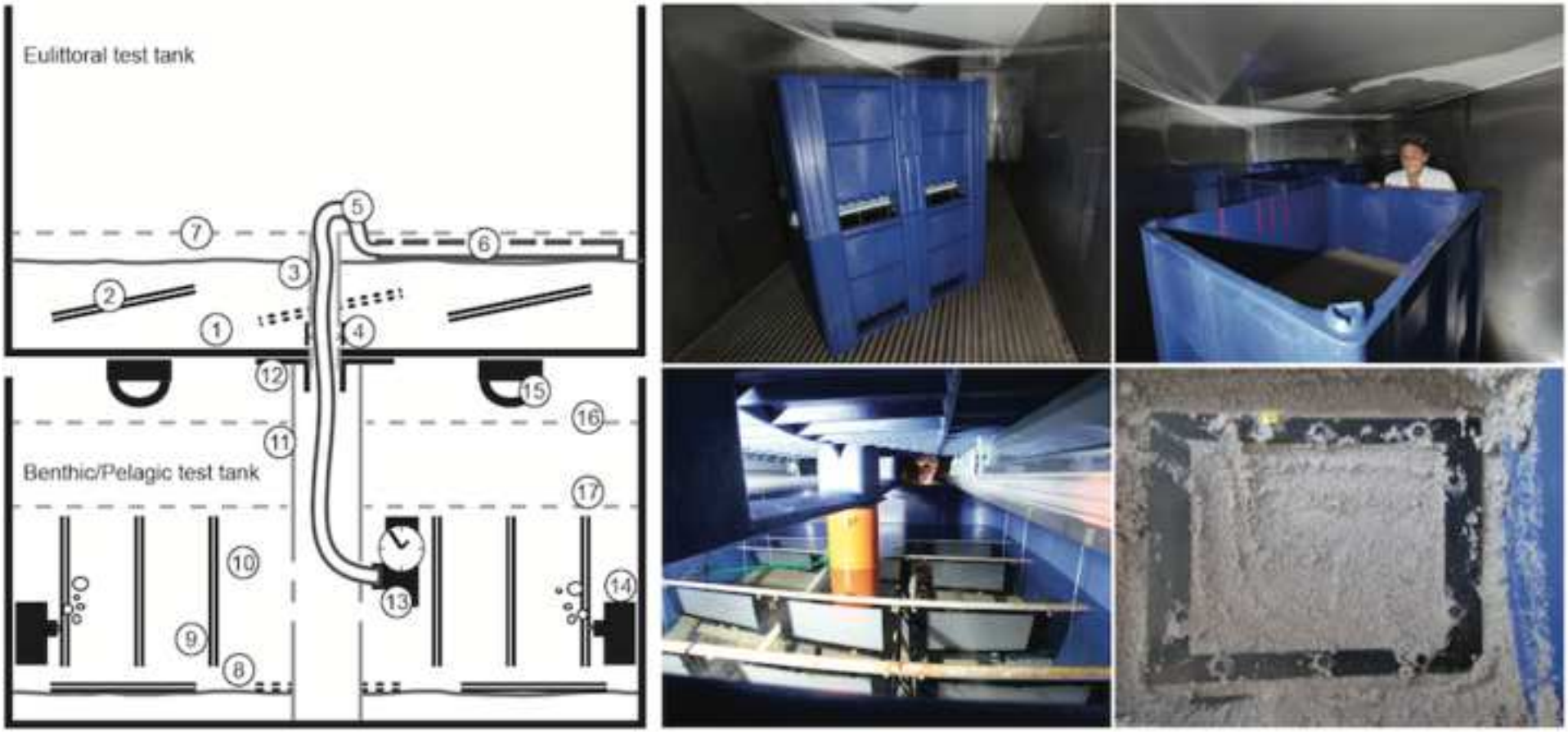

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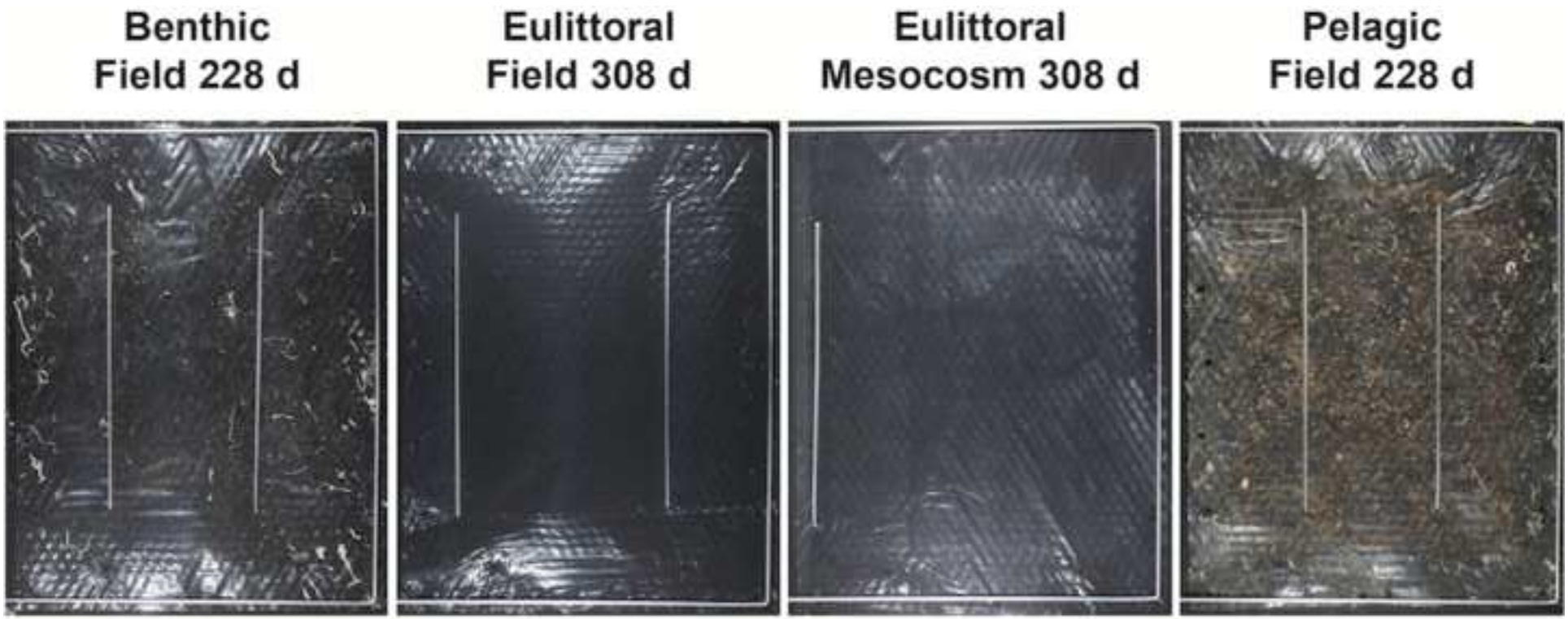

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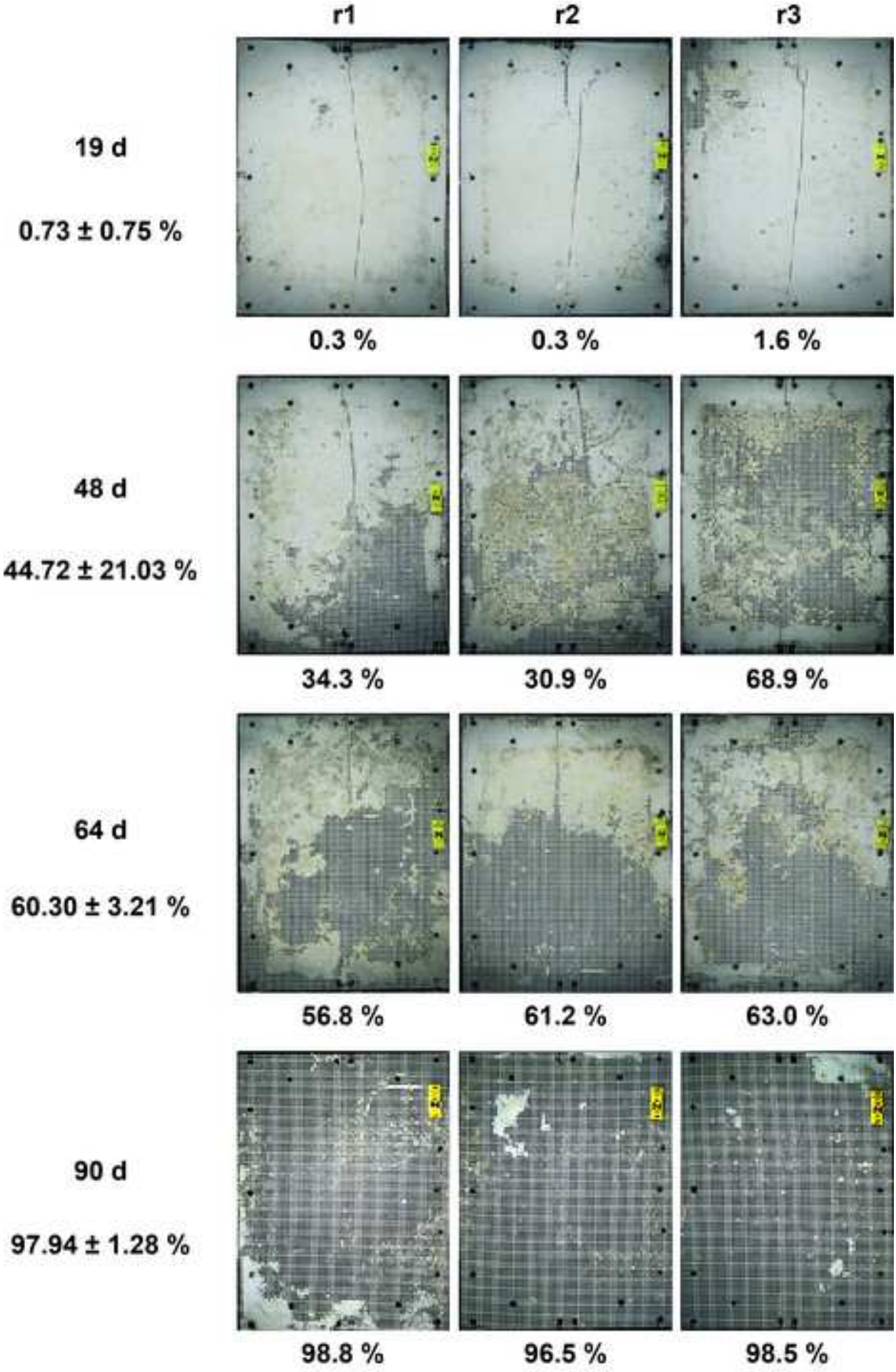

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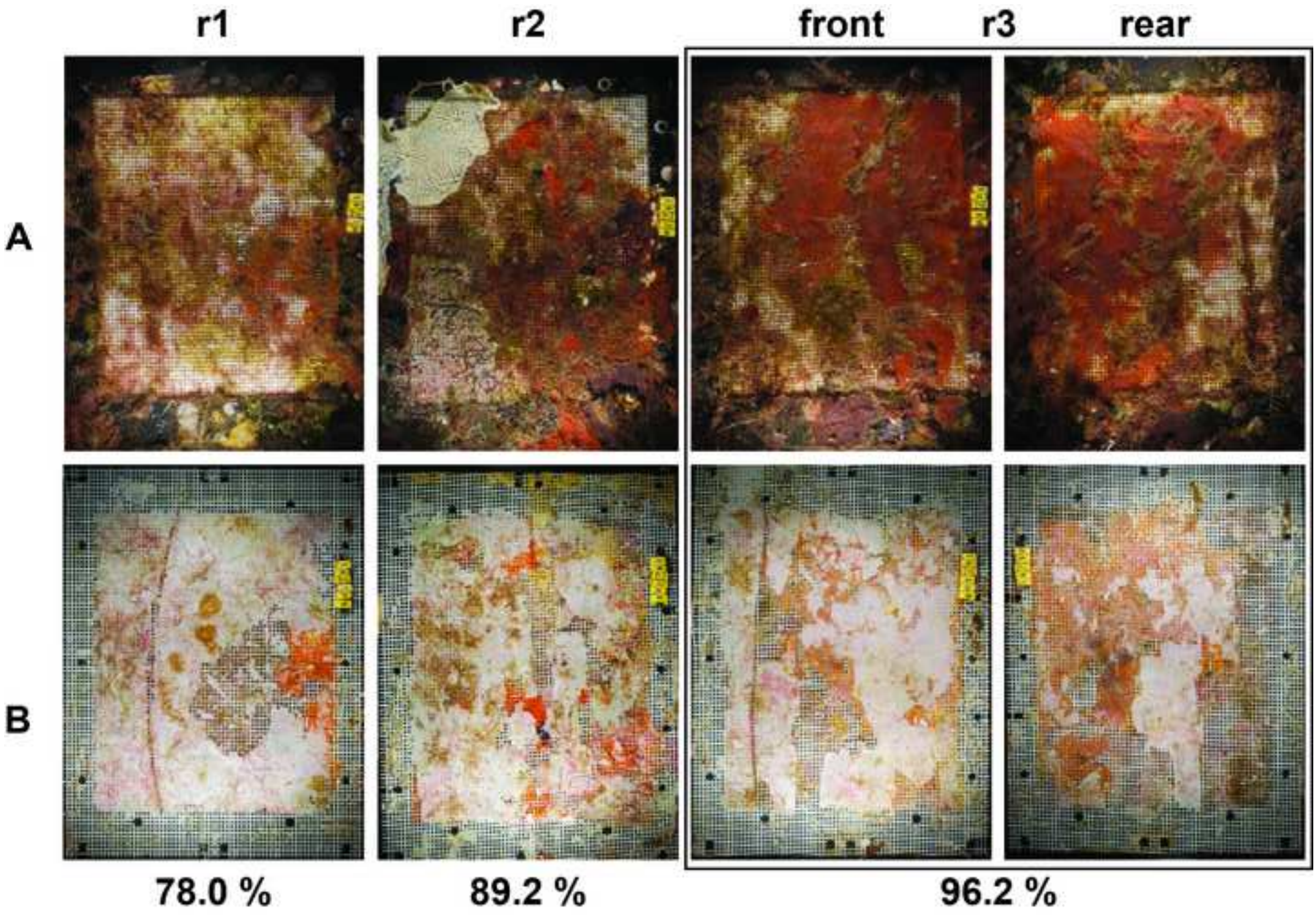

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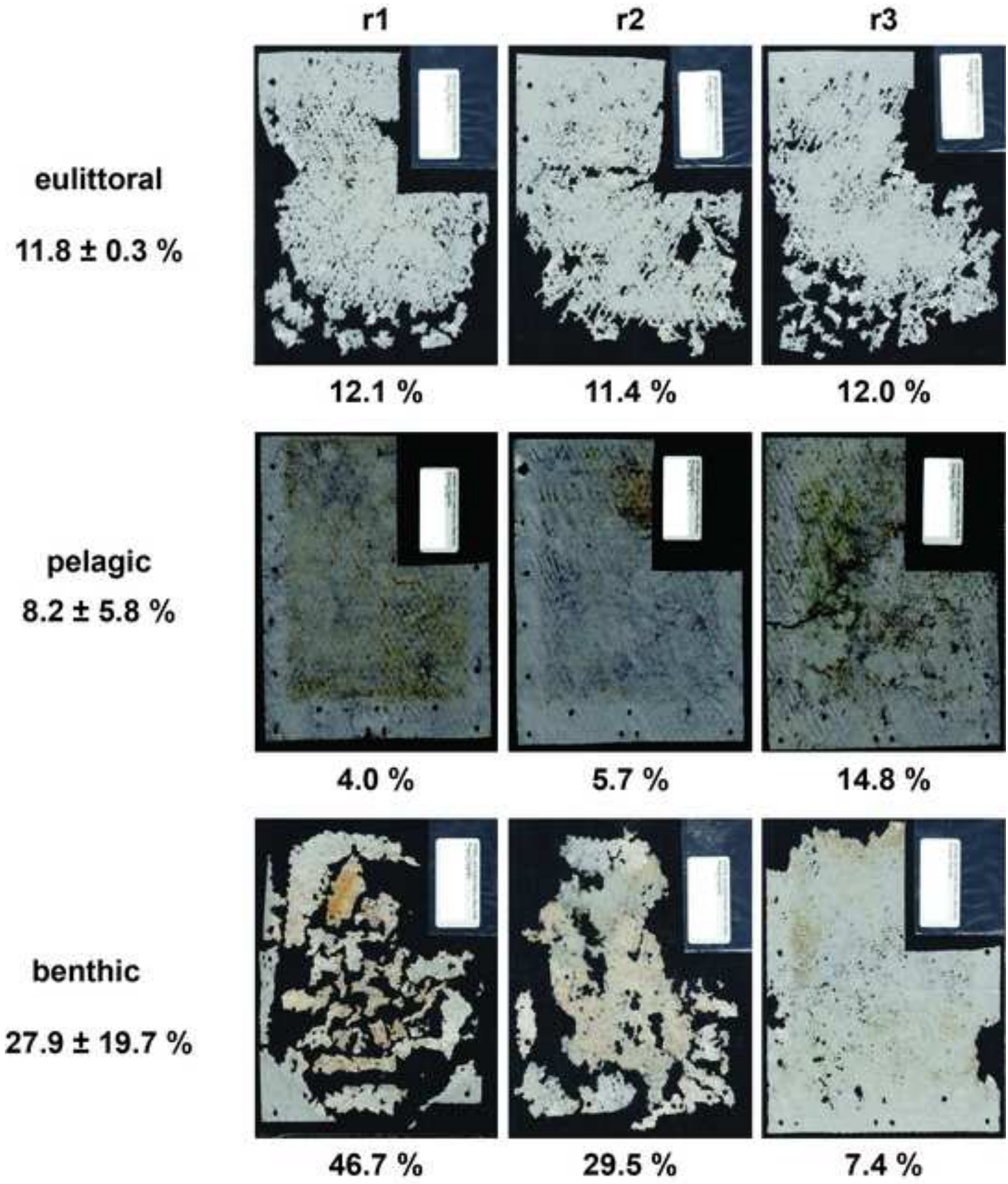

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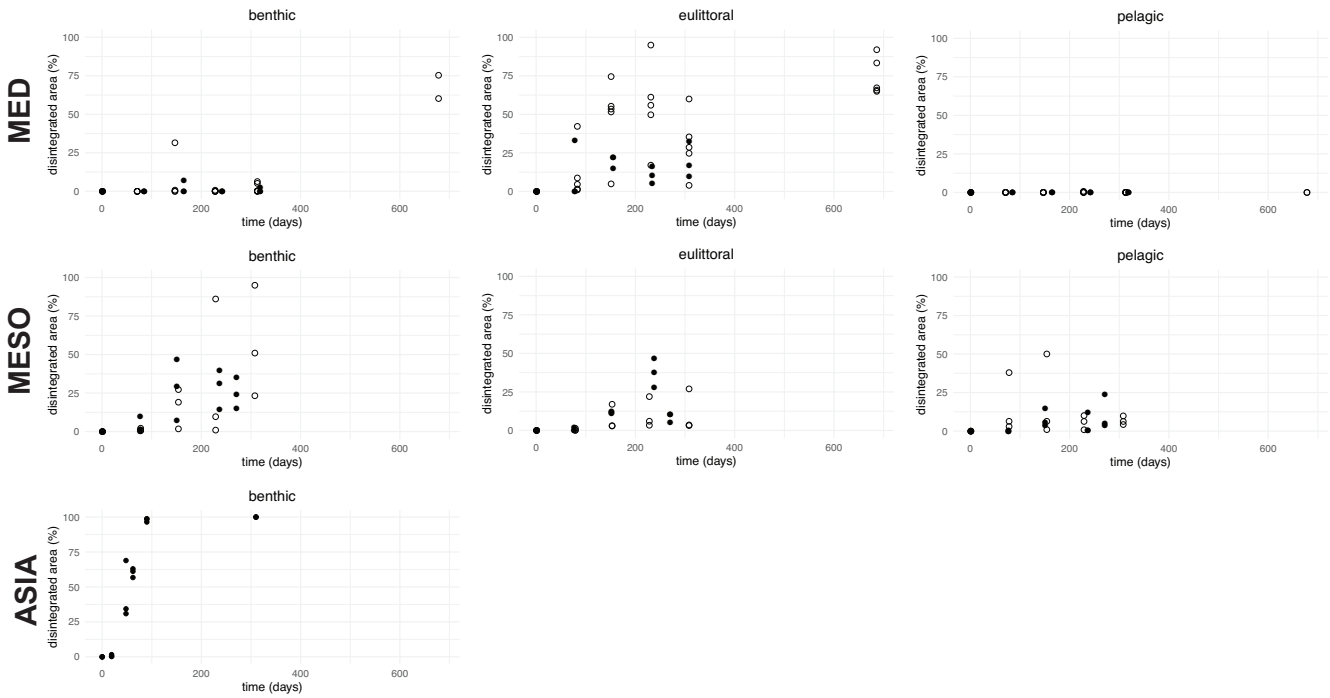

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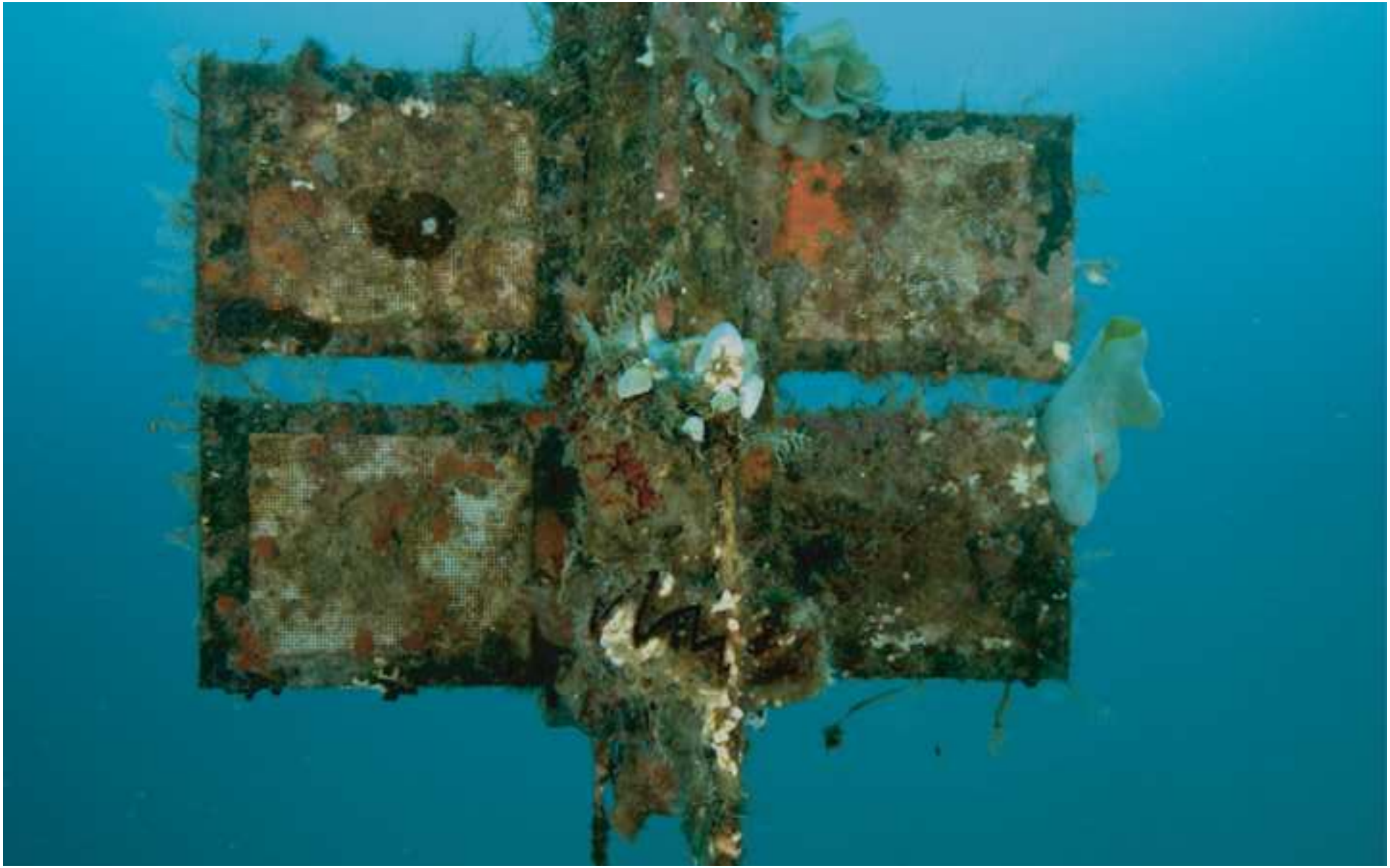

WATER samples			SE ASIA (2017-2018)								MEDITERRANEAN SEA - FIELD (2014 and 2015)																		MEDITERRANEAN SEA - MESOCOSM (2014 and 2015)					
			Water				Porewater						Field year 1		Field year 2		Field year 1		Field year 1	Field year 2			Field year 1		Field year 2		Mesocosm year 1		Mesocosm year 2		Mesocosm year 1	
			Pelagic test site		Benthic test site		Benthic test site						Pelagic		Pelagic		Benthic - water		Benthic - pore	Benthic - water			Eulittoral - porewater		Eulittoral - porewater		Pelagic / benthic - water		Pelagic / benthic - water		Eulittoral - porewater	
Test Description	unit	detection limit	mean	SD	mean	SD	mean	SD	unit	detection limit	MV	SD	MV	SD	MV	SD	MV	SD	MV	MV	SD	MV	SD	MV	SD	MV	SD	MV	SD	MV	SD
Chemical parameters, directly measured																																		
Temperature (°C)	°C	n.a.	28,6	0,4	28,6	0,5	28,9	0,0	°C	n.a.	16,5	4,5	18,2	3,8	15,4	3,2	n.m.		17,4	2,3		11,8	3,1	19,4	6,9	19,7	0,9	20,6	0,9	19,8	1
Salinity (IPSU)	PSU	n.a.	34,0	0,4	33,9	0,2	34,1	0,1	PSU	n.a.	38	0,1	38,4	0,2	38	0,1	n.m.		38,3	0,1		31,3	8,8	41,5	13,3	39,4	0,3	39,8	0,5	39,4	0,3
pH	pH	n.a.	8,1	0,1	8,1	0,1	7,9	0,0	pH	n.a.	8,19	0,02	8,15	0,08	8,18	0,05	n.m.		8,19	0,07		8,06	0,06	7,73	0,29	8,13	0,03	8,09	0,03	8,11	0,02
O2 (mg/L)	mg/L	n.a.	6,4	0,3	6,5	0,4	1,8	0,7	mg/L	n.a.	7,6	0,4	7,8	0,7	7,7	0,3	n.m.		8	0,7		7,6	2,2	4,2	1,4	7,4	0,3	7,1	0,1	7,1	0,2
O2 (%)	%	n.a.	100,8	4,6	101,3	6,8	28,2	11,2	%	n.a.	97,4	5	103,1	6,3	97,1	4	n.m.		100,9	7,7		82,7	13,7	56	13,8	101,6	2,6	99,4	1	97,3	1,6
Nutrient-related parameters, measured by analytical lab																																		
Nitrate (N-NO3)	mg/L	< 0.005	0,03	0,004	0,03	0,004	0,05	0,006	mg/L	0.02	0,07	0,07	0,08	0,06	0,05	0,04	0,10	0,03	0,01	0,15	0,06	0,10	0,09	0,10	0,15	0,04	0,02	0,11	0,13		
Nitrite (N-NO2)	mg/L	< 0.001	0,001	0,0	0,002	0,0006	< 0.001		mg/L	0.02	0,08		b.d.l.		b.d.l. and 0.067	n.a.	<0,02	b.d.l.		b.d.l. and 0,03	n.a.	b.d.l.		0,09	0,04	b.d.l.		0,08	0,03		
Ammonium (NH4)	mg/L	< 0.02	b.d.l.		b.d.l.		0,25	0,04	mg/L	0.04	0,08	0,04	0,07	n.a.	0,09	0,03	0,07	b.d.l. and 0,03	n.a.	0,10	0,03	b.d.l. and 0,09	n.a.	0,11	0,09	0,32	0,55	0,12	0,08		
Total Kjeldahl Nitrogen (TON+NH4)	mg/L	< 1	1	0,0	1	0	2,15	1,1	n.m.	n.m.																								
TON	mg/L		>1		>1		0,9525	1,3	mg/L	calculated	n.a.		0,61	0,26	b.d.l. and 0,28	n.a.	n.a.	0,53	0,14	n.a.		0,50	0,13	0,41	0,16	0,63	0,09	n.a.			
Total Nitrogen	mg/L	< 1	1	0,0	1	0	3	1,4	mg/L	0,25	0,34		0,73	0,29	b.d.l. and 0,3	n.a.	1,20	0,60	0,10	0,51	0,17	0,62	0,25	0,52	0,27	0,55	0,25	n.a.			
Total Inorganic Nitrogen	mg/L	< 0,5	b.d.l.		b.d.l.		< 0,5		mg/L	calculated	n.a.		0,10	0,03	b.d.l. and 0,02	n.a.	n.a.	0,07	0,04	n.a.		0,12	0,13	0,13	0,19	0,24	0,47	0,13	0,18		
Ortho-Phosphate (O-PO4)	mg/L	< 0.005	b.d.l.		b.d.l.		< 0.005		mg/L	0.01 resp. 0.005	b.d.l.		b.d.l.		b.d.l.		0,03	b.d.l.		b.d.l.		0,01	0,00	0,01	0,00	0,01	0,01	0,01	0,00		
Total-Phosphate (P-PO4)	mg/L	< 0.005	b.d.l.		0,007	0,0007	0,0845	0,0	mg/L	0.01	0,01	0,00	0,06	0,03	0,01	0,00	0,11	0,04	0,04	0,06	0,03	0,07	0,04	0,03	0,03	0,03	0,03	n.a.			
Dissolved Organic Phosporus (calculated)	mg/L	< 1	b.d.l.		b.d.l.		< 1		mg/L	calculated	b.d.l.		0,02		b.d.l. and 0,08	n.a.	0,08	b.d.l.		0,05	0,04	0,07	0,03	0,03	0,03	b.d.l.		n.a.			
Total Carbon	mg/L	< 1	30,9	3,6	30,4	3,4	33,5	3,5	mg/L	1	29	5	34	3	27	9	32	35	4	30	10	38	4	31	12	38	3	n.a.			
Total Organic Carbon	mg/L	< 1	5,7	3,4	5,3	3,4	8,3	1,2	mg/L	1	1,90	0,42	3,40	1,45	1,55	0,07	n.a.	3,78	1,47	b.d.l. and 9,9	n.a.	6,00	3,55	4,49	2,21	3,51	0,94	n.a.			
Total Inorganic Carbon	mg/L	< 1	25,1	0,7	25,3	1,0	25,1	2,5	mg/L	n.a.	n.a.		30	3	n.a. and 26	n.a.	n.a.	31	3	n.a.		32	6	29	3	34	3	n.a.			
Dissolved Organic Carbon	mg/L	< 1	5,3	2,1	5,0	1,9	8,3	2,3	mg/L	1	2,03	0,48	2,85	1,32	2,23	0,94	3,40	3,30	1,54	4,18	2,23	3,75	1,50	2,98	0,89	2,73	0,77	4,98	2,52		
Particulate Organic Carbon	mg/L	< 1	2,7	2,1	1,7	1,2	2		mg/L	calculated	n.a.		0,55	0,49	n.a.	n.a.	n.a.	0,48	0,69	b.d.l. and 0,0	n.a.	0,30	0,52	1,56	2,03	0,83	0,51	n.a.			
Chlorophyll-a	mg/m3	< 0.00001	1,1	0,6	1,4	0,7	n.m.		µg/l	1 resp. 0.01	b.d.l.		n.a.		b.d.l.	-	n.a.	n.m.		3,03	1,85	n.a.		1,38	1,37	n.a.		2,14	1,07		
Phaeophytin	mg/m3	< 0.00001	11,4	1,8	6,7	5,1	n.m.		µg/l	1	1,37	0,86	n.a.		1,50	1,27	n.a.	n.m.		3,22	1,39	n.a.		1,86	1,16	n.a.		5,21	2,26		
Reactive Silica (SiO2)	mg/L	< 0.07	0,3	0,04	0,3	0,1	2,4	0,1	mg/L	2 resp. 0.4	b.d.l.		b.d.l.		b.d.l.		<2	b.d.l.		b.d.l. and 0,9	n.a.	b.d.l. and 1,5	n.a.	0,45	0,09	n.a.		0,57	0,13		
Anions, metals and metalloids, measured by analytical lab																																		
Chloride	mg/L	< 0,5	18300	283	18600	707	18600	141	mg/L	1	n.m.		n.m.		n.m.		n.m.	n.m.		n.m		n.m.		n.m.		n.m.		n.m.			
Sulphate	mg/L	< 2	3060	28	2905	148	3430	0	mg/L	1	n.m.		n.m.		n.m.		n.m.	n.m.		n.m.		n.m.		n.m.		n.m.		n.m.			
Aluminum-Dissolved (Al)	mg/L	< 0,05	b.d.l.		b.d.l.		b.d.l.		mg/L	0.05	0,08	0,02	0,16	0,08	0,08	0,03	6,80	0,15	0,12	1,78	0,13	n.m.		0,10	0,02	0,22	0,01	n.m.			
Chromium-Dissolved (Cr)	mg/L	< 0,01	b.d.l.		b.d.l.		b.d.l.		mg/L	0.01	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			
Arsenic-Hydrid Dissolved (As)	mg/L	< 0.0005	b.d.l.		b.d.l.		b.d.l.		mg/L	0.01	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			
Cadmium-Dissolved (Cd)	mg/L	< 0.0001	b.d.l.		0,0001	0,0	b.d.l.		mg/L	0.001	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			
Copper-Dissolved (Cu)	mg/L	< 0.005	b.d.l.		b.d.l.		b.d.l.		mg/L	0.01	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			
Iron-Dissolved (Fe)	mg/L	< 0.02	b.d.l.		b.d.l.		b.d.l.		mg/L	0.1	b.d.l.		0,14		b.d.l.		3,80	b.d.l. and 0,2	n.a.	1,10	0,00	n.m.		b.d.l.		b.d.l.		n.m.			
Manganese-Dissolved (Mn)	mg/L	< 0.005	b.d.l.		b.d.l.		b.d.l.		mg/L	0.05	b.d.l.		b.d.l.		b.d.l.		0,13	b.d.l.		b.d.l. and 0,05	n.a.	n.m.		b.d.l.		b.d.l.		n.m.			
Nickel-Dissolved (Ni)	mg/L	< 0.001	b.d.l.		b.d.l.		b.d.l.		mg/L	0.01	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			
Lead-Dissolved (Pb)	mg/L	< 0.001	b.d.l.		b.d.l.		b.d.l.		mg/L	0.01	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			
Zinc-Dissolved (Zn)	mg/L	< 0.005	b.d.l.		b.d.l.		b.d.l.		mg/L	0.01	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			
Mercury-Dissolved (Hg)	mg/L	< 0.00005	b.d.l.		b.d.l.		b.d.l.		mg/L	0.0005	b.d.l.		n.a.		b.d.l.		n.m.	n.m.		b.d.l.		n.m.		b.d.l.		n.m.		n.m.			

SEDIMENT samples	SE ASIA (2017)			MEDITERRANEAN SEA - FIELD (2014 and 2015)						MEDITERRANEAN SEA - MESOCOSM (2014 and 2015)							
	unit	detection limit	benthic site	unit	detection limi	B - year 1	B - Year 2	E - year 1	E - year 2	Benthic - year 1		Benthic - year 2		Eulittoral - year 1		Eulittoral - year 2	
Physical parameters			mean±SD			mean±SD	mean	mean±SD	mean±SD	mean	SD	mean	SD	mean	SD	mean	SD
Dry weight	%	n.a.	55,14	% w/w	n.a.	61,5	59,2	79	78,5	56	2	56	2	75	1	75	1
Ignition loss @ 550°C	%	n.a.	0,61	% dw	n.a.	5,6	3,4	0,8	6	6	0	6	0	1	0	1	0
Ignition residue @ 800°C	%	n.a.	98,24	% dw	n.a.	57,8	56,6	99,0	99,4	57	0	57	0	99	0	99	0
Permeability	m2	n.a.	2,26E-11	m2	n.a.	1.12E-011 ± 0.91E-11	n.a.	7.46E-011 ± 1.50E-11	20.6E-011 ± 2.02E-11	2.60E-11	6.28E-12	2,40E-11	1,21E-11	1,79E-10	5,82E-12	9,54E-11	2,20E-11
Porosity	% v/v	n.a.	53,57 ± 1,31	% v/v	n.a.	66,78 ± 6,09	n.a.	41,29 ± 2,09	42,65 ± 0,45	62	4	51	6	45	2	42	8
Grain size (median)	µm	n.a.	197 ± 47	µm	n.a.	213 ± 26	n.a.	214 ± 5	278 ± 5	181	23	146	47	278	14	206	9
Nutrient-related parameters																	
Nitrogen (total-N; TNb)	%	n.a.	0,07	mg/kg	1000	<1000	<1000	<1000	<1000	<1000	0	<1000	0	<1000	0	<1000	0
Phosphorus (total-P)	%	n.a.	19	mg/kg	20	270	260	91	75	193	6	193	6	55	3	55	3
Total Carbon (TC)	%	n.a.	44,30	% dw	0.5 resp. 0.2	15	12	<0,5	<0,2	12	0	12	0	<0,2	0	<0,2	0
Total Organic Carbon (TOC)	%	n.a.	0,23	% dw	0.5 resp. 0.2	n.a.	0,33	<0,5	<0,2	0,30	0,01	0,03	0,01	<0,2	0	<0,2	0
Total Inorganic Carbon	%	n.a.	11,00	% dw	0.5 resp. 0.2	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Metals and metalloids																	
Aluminium (Al)	mg/kg dry	1	221	mg/kg	100	1620	1490	4770	4260	1100	0	1100	0	3300	100	3300	100
Arsenic (As)	mg/kg dry	1	< 1	mg/kg	5	<5	n.a.	<5	n.a.	<5	0	<5	0	<5	0	<5	0
Calcium (Ca)	mg/kg dry	20	n.a.	mg/kg	100	338000	348300	4000	3550	331000	12000	331000	12000	3300	173	3300	173
Copper (Co)	mg/kg dry	1	< 1	mg/kg	5	<5	n.a.	<5	n.a.	<5	0	<5	0	<5	0	<5	0
Cadmium (Cd)	mg/kg dry	0,02	< 0,02	mg/kg	0.5	<0.5	n.a.	<0,5	n.a.	<0,5	0	<0.5	0	<0,5	0	<0.5	0
Chromium (Cr)	mg/kg dry	0,2	0,9	mg/kg	5	6	n.a.	71	n.a.	5	1	5	1	33	3	33	3
Iron (Fe)	mg/kg dry	4	263	mg/kg	100	1880	1510	3590	3400	1133	58	1133	58	2367	58	2367	58
Potassium (K)	mg/kg dry	100	126	mg/kg	100	770	920	1790	1400	667	58	667	58	1300	100	1300	100
Magnesium (Mg)	mg/kg dry	10	3700	mg/kg	100	20200	21100	3300	2430	19167	961	19167	961	2133	58	2133	58
Manganese (Mn)	mg/kg dry	1	7	mg/kg	100	150	141	<100	<100	113	6	113	6	<100	0	<100	0
Sodium (Na)	mg/kg dry	10	3200	mg/kg	100	11200	10800	3800	3720	11767	503	11767	503	3933	379	3933	379
Nickel (Ni)	mg/kg dry	0,2	< 0,2	mg/kg	5	<5	n.a.	19	n.a.	<5	0	<5	0	9	0	9	0
Lead (Pb)	mg/kg dry	0,2	0,7	mg/kg	5	9	n.a.	<5	n.a.	5	1	5	1	<5	0	<5	0
Sulfur (S)	mg/kg dry	100	420	mg/kg	100	n.a.	3110	n.a.	330	2700	100	2700	100	333	21	333	21
Zinc (Zn)	mg/kg dry	1	< 1	mg/kg	10	<10	n.a.	10	n.a.	<10	0	<10	0	<10	0	<10	0
Mercury (Hg)	mg/kg dry	0,05	< 0,05	mg/kg	0.05	<0.05	n.a.	<0,05	n.a.	<0,05	0	<0.05	0	<0,05	0	<0.05	0

WATER samples	SE ASIA (2017-2018)							
			Water				Porewater	
			Pelagic test site		Benthic test site		Benthic test site	
Test Description	unit	detection limit	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2
Organotin compounds and Pesticides								
Aldrin	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Dieldrin	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Endrin	mg/L	< 0.00001	n.a.	< 0.00001	< 0.00001	< 0.00001	< 0.00001	n.a.
Heptachlor	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Methoxychlor	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
DDE p,p	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
p-p DDD	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
p-p DDT	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Heptachlorepoxide	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Hexachlorhexan alpha	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Hexachlorhexan beta	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Hexachlorhexan delta	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Hexachlorhexan gamma	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
alpha-HCH	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
beta-HCH	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
delta-HCH (lindane)	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
Gamma-HCH (lindane)	mg/L	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001	< 0.00001
AMPA	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Bentazon	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Bromacil	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Bromoxynil	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Chloridazon	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Chlorpyrifos	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Clethodim	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Clomazone	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Clothianidin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Carbendazim	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Captan	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Chlorpropham	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative

Chlorotoluron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Dichloroprop	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Dicamba	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Difenconazol	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Diflufenican	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Dimethachlor	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Diuron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Ethofumesat	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Flufenacet	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Flusilazol	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Fosthiazat	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Glyphosat	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Isoproturon	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Isopropyl	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Isoxaben	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Isodrin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Mecoprop	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metolachlor	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metalaxyl (Racemat)	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metamitron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metazachlor	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Methiocarb	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Methabenzthiazuron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metosulam	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metoxuron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metrafenone	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Metribuzin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Nicosulfuron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Oxadixyl	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Octachlorstyrol	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Paclobutrazol	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Picolinafen	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Prosulfocarb	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Prosulfuron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Pyraclostrobin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Pymetrozin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Pyrethrin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative

Pyridat	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Terbuthylazin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Tefluthrin	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Tetrachlorbenzol	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Trisulfuron	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative
Trifluralin (C13H16F3N3O4)	mg/L	n.a.	Negative	Negative	Negative	Negative	Negative	Negative

WATER samples	**MEDITERRANEAN SEA - FIELD (2014 and 2015)**					**MEDITERRANEAN SEA - MESOCOSM (2014 and 2015)**		
			Water		**Porewater**	**Mesocosm year 1**		
			Pelagic	**Benthic**	**Eulittoral**	**Pelagic**	**Benthic**	**Eulittoral**
Test Description	**unit**	**detection limit**	**Sample 1**	**Sample 1**	**Sample 1**	**Sample 1**	**Sample 1**	**Sample 1**
Organotin compounds	unit	detection limit	water	water	porewater	tank 1-3	tank 1-3	tank 1-3
Monobutyltin	µg/l	1	<1	<1	<1	<1	<1	<1
Dibutyltin	µg/l	0.1	<0,01	<0,1	<0,1	<0,01	<0,01	<0,001
Tributyltin	µg/l	0.001	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001
Tetrabutyltin	µg/l	0.001	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001
Tricyclohexyltin cation	µg/l	0.001	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001
Triphenyltin cation	µg/l	0.001	<0,001	<0,001	<0,001	<0,001	<0,001	<0,001
Pesticides								
AMPA	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Atrazin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Bentazon	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Bentazon-6OH	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Bentazon-8OH	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Bromacil	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Chloridazon-desphenyl (B)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Chloridazon-methyl-desphenyl (B1)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Chlortoluron	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Desethyl-Atrazin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Desethylterbutylazin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Desisoprpoylatrazin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Dicamba	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Dichlorprop (Racemat)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025

2,6-Dichlorbenzamid	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
N,N-Dimethylsulfamid (DMS)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Dimethachlor-Sulfonsäure (CGA 354742)	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Dimethachlor-Metabolit (CGA 369873)	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Dimethachlorsäure (CGA 50266)	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Diuron	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Ethidimuron	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Ethofumesat	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Glyphosat	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Isoproturon	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Mecoprop (Racemat)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metalaxyl (Racemat)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metamitron	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metazachlor	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metazachlorsäure (BH 479-4)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metazachlorsulfonsäure (BH 479-8)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metolachlor (Racemat CGA 77101/CGA 77102)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metolachlorsäure (Racemat CGA 51202/CGA 351916)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metolachlor-Sulfonsäure (Racemat CGA 380168/CGA 354743)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metolachlor-Sulfonsäure (NOA 413173)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metoxuron	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Metribuzin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Oxadixyl	µg/l	0.025	n.a.	<0,025	<0,025	<0,025	<0,025	<0,025
Simazin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Terbuthylazin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Prothioconazol	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025

Bromoxynil	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Chlofenvinphos	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Chloridazon	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Chlorpyrifos (Chlorpyrifosethyl)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Chlorpyrifosmethyl	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Diflufenican	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Methabenzthiazuron	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
MCPA	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Primicarb	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
Trifluralin	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
DDX und HCH	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
o-p DDD	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
p-p DDD	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
o-p DDE	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
o-p DDT	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
p-p DDT	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
alpha-HCH	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
beta-HCH	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
gamma-HCH (Lindan)	µg/l	0.025	<0,025	<0,025	<0,025	<0,025	<0,025	<0,025
delta-HCH (Lindan)	µg/l	0.025	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Acetamiprid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Ametroctadin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Amidosulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Azoxystrobin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Beflubutamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Benalaxyl-M	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Benthiavalicarb-isopropyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Bixafen	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Boscalid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Carbendazim	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Carfentrazon-ethyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Clethodim	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Clodinafop-propagyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Clomazone	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005

Clothianidin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Cyazofamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Cycloxydim	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Cyproconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Cyprodinil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Difenconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Diflufenican	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dimethachlor	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dimethenamid-P	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dimethoate	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dimethomorph	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dimoxystrobin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Epoxiconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fenamidone	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fenazaquin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fenhexamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fenoxaprop-P	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fenoxycarb	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fenpropidin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fenpyroxymat	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flazasulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flonicamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Florasulam	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fluazifop-P	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fludioxonil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flufenacet	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fluopicolide	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fluoxastrobin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flupyrsulfuron-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fluroxypyr-methylhepthyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flurtamone	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flusilazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flutolanil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Foramsulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fosthiazat	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fuberidazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Haloxyfop-P	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005

Hexythiazox	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Imazalil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Imazosulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Imidacloprid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Indoxacarb	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Ioxynil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Iprovalicarb	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Isoxaben	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Lenacil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Mandipropamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Mepanipyrim	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Mesosulfuron-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Mesotrione	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metalaxyl-M	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Methiocarb	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metosulam	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metrafenone	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metsulfuron-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Myclobutanil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Napropamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Nicosulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Paclobutrazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Penconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pencycuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pethoxamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Phenmedipham	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Picolinafen	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Picoxystrobin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pinoxaden	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pirimiphos-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Prochloraz	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Propamocarb	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Propaquizafop	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Propiconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Propoxycarbazone	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Propyzamid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005

Proquinazid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Prosulfocarb	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Prosulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pymetrozin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pyraclostrobin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pyraflufen-ethyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pyrimethanil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pyroxsulam	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Quinmerac	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Quinoclamin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Quizalofop-P	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Silthiopham	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Sulcotrione	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Sulfosulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tebuconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tembotrione	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tepraloxidim	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tetraconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Thiabendazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Thiacloprid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Thiamethoxam	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Thiencarbazone-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Thifensulfuron-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Thiophanat-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Topramezone	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Triasulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Triclopyr-2-buthoxyethyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Trifloxystrobin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Triflusulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Triticonazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tritosulfuron	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Warfarin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Zoxamide	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Acequinocyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Aclonifen	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Aldrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005

Bifenox	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Bromophos-ethyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Captan	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Chlorpropham	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Chlorpyrifos-ethyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Chlorpyrifos-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Chlorthalonil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Cyfluthrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Cyhalothrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Cypermethrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
DDD o,p	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
DDD p,p	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
DDE o,p	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
DDE p,p	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
DDT o,p	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
DDT p,p	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Deltamethrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dichlobenil	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dicofol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Dieldrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Endosulfan alpha	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Endosulfan beta	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Endrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Esfenvalerat	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Flumioxazin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fluquiconazol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Fluvalinat	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Folpet	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Heptachlor	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Heptachlorepoxid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Hexachlorbenzol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Hexachlorbutadien	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Hexachlorhexan alpha	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Hexachlorhexan beta	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Hexachlorhexan delta	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Hexachlorhexan gamma	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Iprodion	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005

Isodrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Kresoxim-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Methoxychlor	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Octachlornaphthalin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Octachlorstyrol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Parathion-ethyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Parathion-methyl	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pentachlorbenzol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pyrethrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Pyridat	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Quinoxyfen	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Quintozen	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tefluthrin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tetrachlorbenzol	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Tolylfluanid	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Trichlorbenzol 1,2,3	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Trichlorbenzol 1,2,4	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Trichlorbenzol 1,3,5	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Trifluralin	µg/l	0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005

n.a. = not available

Sediment samples	SE ASIA (2017-2018)			
			Benthic test site	
Test Description	unit	detection limit	Sample 1	Sample 2
Organotin compounds and Pesticides				
Tributyltin	md/kg dry	0,00001	< 0,00001	< 0,00001
Atrazin	mg/kg dry	n/a	negative	negative
MCPA	mg/kg dry	n/a	negative	negative
Simazin	mg/kg dry	n/a	negative	negative
Bentazon	mg/kg dry	n/a	negative	negative
Bromacil	mg/kg dry	n/a	negative	negative
Bromoxynil	mg/kg dry	n/a	negative	negative
Carbendazim	mg/kg dry	n/a	negative	negative
Chlortoluron	mg/kg dry	n/a	negative	negative
Chlorfenvinphos	mg/kg dry	n/a	negative	negative
Chloridazon	mg/kg dry	n/a	negative	negative
Dicamba	mg/kg dry	n/a	negative	negative
Dichlorprop	mg/kg dry	n/a	negative	negative
Dimethachlor	mg/kg dry	n/a	negative	negative
Dimethoate	mg/kg dry	n/a	negative	negative
Diuron	mg/kg dry	n/a	negative	negative
Ethofumesat	mg/kg dry	n/a	negative	negative
Fuberidazol	mg/kg dry	n/a	negative	negative
Glyphosat	mg/kg dry	n/a	negative	negative
Haloxyfop-P	mg/kg dry	n/a	negative	negative
Ioxynil	mg/kg dry	n/a	negative	negative
Isoproturon	mg/kg dry	n/a	negative	negative
Isoxaben	mg/kg dry	n/a	negative	negative
Lenacil	mg/kg dry	n/a	negative	negative
Mecoprop	mg/kg dry	n/a	negative	negative
Metamitron	mg/kg dry	n/a	negative	negative
Metazachlor	mg/kg dry	n/a	negative	negative
Methabenzthiazuron	mg/kg dry	n/a	negative	negative
Metoxuron	mg/kg dry	n/a	negative	negative
Metribuzin	mg/kg dry	n/a	negative	negative
Napropamid	mg/kg dry	n/a	negative	negative
Penconazol	mg/kg dry	n/a	negative	negative

Propiconazol	mg/kg dry	n/a	negative	negative
Propyzamid	mg/kg dry	n/a	negative	negative
Phenmedipham	mg/kg dry	n/a	negative	negative
Pyraclostrobin	mg/kg dry	n/a	negative	negative
Quintozen	mg/kg dry	n/a	negative	negative
Terbuthylazin	mg/kg dry	n/a	negative	negative
Thiabendazol	mg/kg dry	n/a	negative	negative
Triasulfuron	mg/kg dry	n/a	negative	negative
Tetrachlorobenzol	mg/kg dry	n/a	negative	negative
Tolyfluanid	mg/kg dry	n/a	negative	negative
Trichlorbenzol 1,3,5 Trifluralin	mg/kg dry	n/a	negative	negative
Monobutyhltin	mg/kg dry	n/a	negative	negative
Dibuthyltin	mg/kg dry	n/a	negative	negative
Tetrabuthyltin	mg/kg dry	n/a	negative	negative

Sediment samples	**MEDITERRANEAN SEA - FIELD (2014 and 2015)**				**MEDITERRANEAN SEA - MESOCOSM (2014 and**			
					Year 1		**Year 2**	
			Benthic	**Eulittoral**	**Benthic**	**Eulittoral**	**Benthic**	**Eulittoral**
Test Description	**unit**	**detection limit**	**Sample 1**	**Sample 1**	**Sample 1**	**Sample 1**	**Sample 1**	**Sample 1**
Organotin compounds					tank 1-3	tank 1-3	tank 1-3	tank 1-3
Monobutyltin	µg/kg	1	<1	<1	<1	<1	<1	<1
Dibutyltin	µg/kg	1	<1	<1	<1	<1	<1	<1
Tributyltin	µg/kg	1	<1	<1	<1	<1	<1	<1
Tetrabutyltin	µg/kg	1	<1	<1	<1	<1	<1	<1
Tricyclohexyltin cation	µg/kg	1	<1	<1	<1	<1	<1	<1
Triphenyltin cation	µg/kg	1	<1	<1	<1	<1	<1	<1
Pesticides								
Atrazin	µg/kg	10	<10	<10	<10	<10	<10	<10
Acetamiprid	µg/kg	2	<2	<2	<2	<2	<2	<2
Ametroctadin	µg/kg	2	<2	<2	<2	<2	<2	<2
Amidosulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Azoxystrobin	µg/kg	2	<2	<2	<2	<2	<2	<2
Beflubutamid	µg/kg	2	<2	<2	<2	<2	<2	<2
Benalaxyl-M	µg/kg	2	<2	<2	<2	<2	<2	<2
Bentazon	µg/kg	10	<10	<10	<10	<10	<10	<10
Benthiavalicarb-isopropyl	µg/kg	2	<2	<2	<2	<2	<2	<2

Bixafen	µg/kg	2	<2	<2	<2	<2	<2	<2
Boscalid	µg/kg	2	<2	<2	<2	<2	<2	<2
Bromacil	µg/kg	10	<10	<10	<10	<10	<10	<10
Bromoxynil	µg/kg	10	<10	<10	<10	<10	<10	<10
Carbendazim	µg/kg	2	<2	<2	<2	<2	<2	<2
Carfentrazon-ethyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Chlorfenvinphos	µg/kg	10	<10	<10	<10	<10	<10	<10
Chloridazon	µg/kg	10	<10	<10	<10	<10	<10	<10
Chlortoluron	µg/kg	10	<10	<10	<10	<10	<10	<10
Clethodim	µg/kg	2	<2	<2	<2	<2	<2	<2
Clodinafop-propagyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Clomazone	µg/kg	2	<2	<2	<2	<2	<2	<2
Clothianidin	µg/kg	2	<2	<2	<2	<2	<2	<2
Cyazofamid	µg/kg	2	<2	<2	<2	<2	<2	<2
Cycloxydim	µg/kg	2	<2	<2	<2	<2	<2	<2
Cyproconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Cyprodinil	µg/kg	2	<2	<2	<2	<2	<2	<2
Desethyl-Atrazin	µg/kg	10	<10	<10	<10	<10	<10	<10
Desethylterbutylazin	µg/kg	10	<10	<10	<10	<10	<10	<10
Desisoprpoylatrazin	µg/kg	10	<10	<10	<10	<10	<10	<10
Dicamba	µg/kg	10	<10	<10	<10	<10	<10	<10
Dichlorprop	µg/kg	10	<10	<10	<10	<10	<10	<10
Difenconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Diflufenican	µg/kg	2	<2	<2	<2	<2	<2	<2
Dimethachlor	µg/kg	2	<2	<2	<2	<2	<2	<2
Dimethenamid-P	µg/kg	2	<2	<2	<2	<2	<2	<2
Dimethoate	µg/kg	2	<2	<2	<2	<2	<2	<2
Dimethomorph	µg/kg	2	<2	<2	<2	<2	<2	<2
Dimoxystrobin	µg/kg	2	<2	<2	<2	<2	<2	<2
Diuron	µg/kg	10	<10	<10	<10	<10	<10	<10
Epoxiconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Ethidimuron	µg/kg	10	<10	<10	<10	<10	<10	<10
Ethofumesat	µg/kg	10	<10	<10	<10	<10	<10	<10
Fenamidone	µg/kg	2	<2	<2	<2	<2	<2	<2
Fenazaquin	µg/kg	2	<2	<2	<2	<2	<2	<2
Fenhexamid	µg/kg	2	<2	<2	<2	<2	<2	<2
Fenoxaprop-P	µg/kg	2	<2	<2	<2	<2	<2	<2

Fenoxycarb	µg/kg	2	<2	<2	<2	<2	<2	<2
Fenpropidin	µg/kg	2	<2	<2	<2	<2	<2	<2
Fenpyroxymat	µg/kg	2	<2	<2	<2	<2	<2	<2
Flazasulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Flonicamid	µg/kg	2	<2	<2	<2	<2	<2	<2
Florasulam	µg/kg	2	<2	<2	<2	<2	<2	<2
Fluazifop-P	µg/kg	2	<2	<2	<2	<2	<2	<2
Fludioxonil	µg/kg	2	<2	<2	<2	<2	<2	<2
Flufenacet	µg/kg	2	<2	<2	<2	<2	<2	<2
Fluopicolide	µg/kg	2	<2	<2	<2	<2	<2	<2
Fluoxastrobin	µg/kg	2	<2	<2	<2	<2	<2	<2
Flupyrsulfuron-methyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Fluroxypyr-methylhepthyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Flurtamone	µg/kg	2	<2	<2	<2	<2	<2	<2
Flusilazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Flutolanil	µg/kg	2	<2	<2	<2	<2	<2	<2
Foramsulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Fosthiazat	µg/kg	2	<2	<2	<2	<2	<2	<2
Fuberidazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Glyphosat	µg/kg	10	<10	<10	<10	<10	<10	<10
Haloxyfop-P	µg/kg	2	<2	<2	<2	<2	<2	<2
Hexythiazox	µg/kg	2	<2	<2	<2	<2	<2	<2
Imazalil	µg/kg	2	<2	<2	<2	<2	<2	<2
Imazosulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Imidacloprid	µg/kg	2	<2	<2	<2	<2	<2	<2
Indoxacarb	µg/kg	2	<2	<2	<2	<2	<2	<2
Ioxynil	µg/kg	2	<2	<2	<2	<2	<2	<2
Iprovalicarb	µg/kg	2	<2	<2	<2	<2	<2	<2
Isoproturon	µg/kg	10	<10	<10	<10	<10	<10	<10
Isoxaben	µg/kg	2	<2	<2	<2	<2	<2	<2
Lenacil	µg/kg	2	<2	<2	<2	<2	<2	<2
Mandipropamid	µg/kg	2	<2	<2	<2	<2	<2	<2
MCPA	µg/kg	10	<10	<10	<10	<10	<10	<10
Mecoprop	µg/kg	10	<10	<10	<10	<10	<10	<10
Mepanipyrim	µg/kg	2	<2	<2	<2	<2	<2	<2
Mesosulfuron-methyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Mesotrione	µg/kg	2	<2	<2	<2	<2	<2	<2

Metalaxyl-M	µg/kg	2	<2	<2	<2	<2	<2	<2
Metamitron	µg/kg	10	<10	<10	<10	<10	<10	<10
Metazachlor	µg/kg	10	<10	<10	<10	<10	<10	<10
Metconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Methabenzthiazuron	µg/kg	10	<10	<10	<10	<10	<10	<10
Methiocarb	µg/kg	2	<2	<2	<2	<2	<2	<2
Metosulam	µg/kg	2	<2	<2	<2	<2	<2	<2
Metoxuron	µg/kg	10	<10	<10	<10	<10	<10	<10
Metrafenone	µg/kg	2	<2	<2	<2	<2	<2	<2
Metribuzin	µg/kg	10	<10	<10	<10	<10	<10	<10
Metsulfuron-methyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Myclobutanil	µg/kg	2	<2	<2	<2	<2	<2	<2
Napropamid	µg/kg	2	<2	<2	<2	<2	<2	<2
Nicosulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Paclobutrazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Penconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Pencycuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Pethoxamid	µg/kg	2	<2	<2	<2	<2	<2	<2
Phenmedipham	µg/kg	2	<2	<2	<2	<2	<2	<2
Picolinafen	µg/kg	2	<2	<2	<2	<2	<2	<2
Picoxystrobin	µg/kg	2	<2	<2	<2	<2	<2	<2
Pinoxaden	µg/kg	2	<2	<2	<2	<2	<2	<2
Pirimiphos-methyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Primicarb	µg/kg	10	<10	<10	<10	<10	<10	<10
Prochloraz	µg/kg	2	<2	<2	<2	<2	<2	<2
Propamocarb	µg/kg	2	<2	<2	<2	<2	<2	<2
Propaquizafop	µg/kg	2	<2	<2	<2	<2	<2	<2
Propiconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Propoxycarbazone	µg/kg	2	<2	<2	<2	<2	<2	<2
Propyzamid	µg/kg	2	<2	<2	<2	<2	<2	<2
Proquinazid	µg/kg	2	<2	<2	<2	<2	<2	<2
Prosulfocarb	µg/kg	2	<2	<2	<2	<2	<2	<2
Prosulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Prothioconazol	µg/kg	10	<2	<2	<2	<2	<2	<2
Pymetrozin	µg/kg	2	<2	<2	<2	<2	<2	<2
Pyraclostrobin	µg/kg	2	<2	<2	<2	<2	<2	<2
Pyraflufen-ethyl	µg/kg	2	<2	<2	<2	<2	<2	<2

Pyrimethanil	µg/kg	2	<2	<2	<2	<2	<2	<2
Pyroxsulam	µg/kg	2	<2	<2	<2	<2	<2	<2
Quinmerac	µg/kg	2	<2	<2	<2	<2	<2	<2
Quinoclamin	µg/kg	2	<2	<2	<2	<2	<2	<2
Quizalofop-P	µg/kg	2	<2	<2	<2	<2	<2	<2
Silthiopham	µg/kg	2	<2	<2	<2	<2	<2	<2
Simazin	µg/kg	10	<10	<10	<10	<10	<10	<10
S-Metolachlor	µg/kg	10	<10	<10	<10	<10	<10	<10
Sulcotrione	µg/kg	2	<2	<2	<2	<2	<2	<2
Sulfosulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Tebuconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Tembotrione	µg/kg	2	<2	<2	<2	<2	<2	<2
Tepraloxidim	µg/kg	2	<2	<2	<2	<2	<2	<2
Terbuthylazin	µg/kg	10	<10	<10	<10	<10	<10	<10
Tetraconazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Thiabendazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Thiacloprid	µg/kg	2	<2	<2	<2	<2	<2	<2
Thiamethoxam	µg/kg	2	<2	<2	<2	<2	<2	<2
Thiencarbazone-methyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Thifensulfuron-methyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Thiophanat-methyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Topramezone	µg/kg	2	<2	<2	<2	<2	<2	<2
Triasulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Triclopyr-2-buthoxyethyl	µg/kg	2	<2	<2	<2	<2	<2	<2
Trifloxystrobin	µg/kg	2	<2	<2	<2	<2	<2	<2
Triflusulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Triticonazol	µg/kg	2	<2	<2	<2	<2	<2	<2
Tritosulfuron	µg/kg	2	<2	<2	<2	<2	<2	<2
Warfarin	µg/kg	2	<2	<2	<2	<2	<2	<2
Zoxamide	µg/kg	2	<2	<2	<2	<2	<2	<2
Acequinocyl	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Aclonifen	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Aldrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Bifenox	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Bromophos-ethyl	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Captan	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1

Chlorpropham	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Chlorpyrifos-ethyl	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Chlorpyrifos-methyl	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Chlorthalonil	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Cyfluthrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Cyhalothrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Cypermethrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
DDD o,p	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
DDD p,p	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
DDE o,p	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
DDE p,p	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
DDT o,p	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
DDT p,p	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Deltamethrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Dichlobenil	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Dicofol	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Dieldrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Endosulfan alpha	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Endosulfan beta	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Endrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Esfenvalerat	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Flumioxazin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Fluquiconazol	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Fluvalinat	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Folpet	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Heptachlor	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Heptachlorepoxid	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Hexachlorbenzol	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Hexachlorbutadien	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Hexachlorhexan alpha	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Hexachlorhexan beta	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Hexachlorhexan delta	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Hexachlorhexan gamma	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Iprodion	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Isodrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Kresoxim-methyl	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Methoxychlor	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1

Octachlornaphthalin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Octachlorstyrol	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Parathion-ethyl	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Parathion-methyl	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Pentachlorbenzol	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Pyrethrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Pyridat	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Quinoxyfen	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Quintozen	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Tefluthrin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Tetrachlorbenzol	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Tolylfluanid	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Trichlorbenzol 1,2,3	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Trichlorbenzol 1,2,4	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Trichlorbenzol 1,3,5	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
Trifluralin	µg/kg	0.1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1

SE ASIA

Water		Sediment	
Nutrient-related parameters	**Method / standard**	**Physical parameters**	**Method / standard**
Nitrate (N-NO3)	APHA-4500-NO3-E(2005)	Dry weight	Gravimetri
Nitrite (N-NO2)	APHA-4500-NO2-B(2012)	Ignition loss @ 550°C	LOI methode
Ammonium (NH4)	APHA-4500-NH3-D(2005)	Ignition residue @ 800°C	LOI methode
Total Kjeldahl Nitrogen (TON+NH4)	APHA-4500-Norg-B(2005)	Permeability	Analised by HYDRA
TON	calculated	Porosity	Analised by HYDRA
Total Nitrogen	APHA-4500-N(2005)	Grain size (median)	Analised by HYDRA
Total Inorganic Nitrogen	Calculation Method	**Nutrient-related parameters**	
Ortho-Phosphate (O-PO4)	WLN-CL-WI.15	Nitrogen (total-N; TNb)	Kjeldahl
Total-Phosphate (P-PO4)	APHA-4500-P B and E (2012)	Phosphorus (total-P)	P- K HCL 25%
Dissolved Organic Phosporus (calc)	Calculation Method	Total Carbon (TC)	Walkey and black
Total Carbon	APHA-5310-B(2012)	Total Organic Carbon (TOC)	Walkey and black
Total Organic Carbon	APHA-5310-B(2012)	Total Inorganic Carbon	calculated
Total Inorganic Carbon	APHA-5310-B(2012)	**Metals and metalloids**	
Dissolved Organic Carbon	APHA-5310-B(2012)	Aluminium	WLN-ML-WI-08(ICP-OES)
Particulate Organic Carbon	Calculation Method	Sulfur	WLN-ML-WI-08(ICP-OES)
Chlorophyl-a	APHA 10200-H (2012)	Lead	WLN-ML-WI-08(ICP-OES)
Phaeophytin	APHA-10200-H(2012)	Cadmium	WLN-ML-WI-08(ICP-OES)
Reactive Silica (SiO2)	APHA-4500-SiO2-C(2012)	Zinc	WLN-ML-WI-08(ICP-OES)
Anions, metals and metalloids		Copper	WLN-ML-WI-08(ICP-OES)
Chloride	WLN-CL-WI.05 (Spectrophotometer)	Mercury	USEPA 245.7 (2005)
Sulphate	WLN-CL-WI.17 (Spectrophotometer)	Nickel	WLN-ML-WI-08(ICP-OES)
Aluminum-Dissolved (Al)	APHA-3120-B(2012)	Arsenic	WLN-ML-WI-08(ICP-OES)
Chromium-Dissolved (Cr)	APHA-3120-B(2012)	Chromium	WLN-ML-WI-08(ICP-OES)
Arsenic-Hydrid Dissolved (As)	APHA-31140-B(2012)	Magnesium	WLN-ML-WI-08(ICP-OES)
Cadmium-Dissolved (Cd)	APHA-3120-B(2012)	Potassium	WLN-ML-WI-08(ICP-OES)
Copper-Dissolved (Cu)	APHA-3120-B(2012)	Sodium	WLN-ML-WI-08(ICP-OES)
Iron-Dissolved (Fe)	APHA-3120-B(2012)	Calcium	WLN-ML-WI-08(ICP-OES)
Manganese-Dissolved (Mn)	APHA-3120-B(2012)	Iron	WLN-ML-WI-08(ICP-OES)
Nickel-Dissolved (Ni)	APHA-3120-B(2012)	Manganese	WLN-ML-WI-08(ICP-OES)
Lead-Dissolved (Pb)	APHA-3120-B(2012)	**Organotin compounds and Pesticides**	
Zinc-Dissolved (Zn)	APHA-3120-B(2012)	All	WLN-ML-WI-05(GCMS)
Mercury-Dissolved (Hg)	USEPA 245.7(2005)		
Pesticides			
All	WLN-OL-WI-03		
Organotin compounds			
All	WLN-OL-WI-08		

MEDITERRANEAN SEA

Water		Sediment	
Nutrient-related parameters	**Method / standard**	**Physical parameters**	**Method / standard**
Nitrogen (total-N; TNb)	EN 12260-H34:2003-12	Dry weight	ISO 11465/EN 14346
Total Inorg. Nitrogen (TIN)	calculated	Ignition loss @ 550°C	DIN 38414-S3/EN 15169
Total Org. Nitrogen (TON)	calculated	Ignition residue @ 800°C	DIN 38414-S3:1985-11
Ammonium (NH4-N)	ISO 11732-E23:2005-05	Permeability	Analised by HYDRA
Nitrite (NO2-N)	ISO 13395-D28:1996-12	Porosity	Analised by HYDRA
Nitrate (NO3-N)	ISO 13395-D28:1996-12	Grain size (median)	Analised by HYDRA
Phosphor (total-P)	ISO 6878-D11:2004-09	**Nutrient-related parameters**	
Ortho-Phosphate (PO4-P)	ISO 6878-D11:2004-09	Nitrogen (total-N; TNb)	ISO 11261:1997-05
Dissolved Org. P (DOP)	calculated	Phosphorus (total-P)	ISO 11885-E22:2009-07
Total Carbon (TC)	EN 1484-H3:1997-08	Total Carbon (TC)	ISO 10694:1996-08
Total Inorg. Carbon (TIC)	calculated	Total Organic Carbon (TOC)	ISO 10694:1996-08
Total Org. Carbon (TOC)	EN 1484-H3:1997-08	**Metals and metalloids**	
Dissolved Org. Carbon (DOC)	EN 1484-H3:1997-08	Aluminium	ISO 11885-E22:2009-07
Particulate Org. Carbon (POC)	calculated	Sulfur	ISO 11885-E22:2009-09
Chlorophyll a	DIN 38412-L16:1985-12	Lead	ISO 11885-E22:2009-07
Phaeopigment	DIN 38412-L16:1985-12	Cadmium	ISO 11885-E22:2009-07
Silica (as SiO2)	DIN 38405-D21:1990-10	Zinc	ISO 11885-E22:2009-07
Anions and metals		Copper	ISO 11885-E22:2009-07
Iron	ISO 11885-E22:2009-09	Mercury	ISO 16772:2005-06
Manganese	ISO 11885-E22:2009-09	Nickel	ISO 11885-E22:2009-07
Aluminium	ISO 11885-E22:2009-09	Arsenic	ISO 11885-E22:2009-07
Anions, metals and metalloids		Chromium	ISO 11885-E22:2009-07
All	ISO 11885-E22:2009-09	Magnesium	ISO 11885-E22:2009-07
Organotin compounds		Potassium	ISO 11885-E22:2009-07
All	DIN EN ISO 1735331:2015-11	Sodium	ISO 11885-E22:2009-07
Pesticides		Calcium	ISO 11885-E22:2009-07
Most	DIN 38407 F36: 2014-09	Iron	ISO 11885-E22:2009-07
AMPA, Glyphosat	ISO 21458:2008E	Manganese	ISO 11885-E22:2009-07
Some	on the bases of DIN EN ISO 6468-F1: 1997-02	**Organotin compounds**	
		All	DIN ISO 23161:2011-10
		Pesticides	
		All	on the bases of DIN 38407 F36: 2014-09