

Efficacy and production of disinfection by-products of ozone treated ballast water

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Introduction

AirTree is preparing for land-based testing at the NIOZ/IMARES test facility. As the Ballast Water Management System (BWMS) developed by AirTree uses ozone as active substance, IMO Guideline G9 applies and Basic and Final Approval are also required. In preparing the Basic Approval dossier, it appeared that too little is known about the development of disinfection by-products (DBP) during the treatment process. The BSH, representing the German authorities as intended flag state for the Type Approval process, has indicated that more information is needed on the dynamics of specific DBP, in order to be able to judge environmental and human risks of the discharged ballast water after treatment.

The currently envisaged ballast water treatment procedure of AirTree consists of a two-steps approach: mechanical treatment by using filtration (50 µm), followed by chemical treatment via semi-continuous in-tank ozone injection. The ozone concentration is dosed on a level of 750 mV ORP (Oxygen Reduction Potential), which then is maintained by semi-continuous in-tank ozone injection. It was uncertain if this dosage level would be sufficient to effectively disinfect different water types.

To evaluate the maximum risks for DBP formation in the Basic Approval, the highest treatment dose was recommended to AirTree. Application of a lower dose in the final BWMS will reduce the risk, so additional risk analyses will not be necessary. On the other hand, an increase of the dose in the final BWMS would require a new risk analysis procedure and approval process. However, in order to avoid unnecessary risk mitigation procedures, the dose used for risk analysis should not be unrealistically high. Therefore, the first step was to assess a minimum required treatment dose in different water types. Based upon these results, a treatment dose was selected that was used for the final DBP testing and risk analysis. For this a treatment period of 4 weeks was proposed to AirTree.

The BSH required the following DBPs to be evaluated:

Chemical name	formula	CAS
Bromate Ion	NaBrO ₃	7789-38-0
Iodate ion	NaIO ₃	7681-55-2
Trichloroethylene	C ₂ HCl ₃	79-01-6
Tribromomethane	CHBr ₃	75-25-2
Dibromoacetic acid	CHBr ₂ COOH	631-64-1
Dibromoacetonitrile	CHBr ₂ CN	3252-43-5
Dichloroacetonitrile	CHCl ₂ CN	3018-12-0

Additionally, the BSH requested to analyse nitrate and nitrite, because oxidizing agents are suspected to influence the equilibrium of these nutrients in water.

The current report presents the results of the study into the minimum required treatment dose and DBP concentrations.

1 Assignment

This project had two main research questions that needed to be answered:

1. Assess the required minimum ozone dose for effective ballast water treatment by AirTree's BWMS in different water types.
2. Describe the DBP formation due to continuous in-tank dosing with ozone during a 4 week treatment period.

After successful commissioning of the pilot scale Ozone treatment system of AirTree, IMARES assessed the required minimum ozone dose for two water types (one freshwater and one marine water) with challenging test water conditions regarding concentrations and diversity of organisms <50 µm in minimum dimension. It was assumed that larger organisms would be filtered out in the first (filter) treatment step of the client and, therefore, they were not included in these tests.

The level of success or failure to meet the D-2 standards on the average discharge after 1 day (aiming at USCG ETV protocols) and 7 days (for IMO) were recorded and are presented in this report. The effective dose was chosen from the first tests and the formation of disinfection by-products was monitored during a 4 week treatment period with semi continuous in-tank dosing with ozone.

2 Description of the pilot scale Ozone Treatment System from AirTree

The pilot scale Ozone Treatment System (OTS) decimates aquatic organisms in the ballast water by means in-tank ozonation using the W-5 Ozone Generator in combination with the AM-20 Air Dryer. The OTS was specially designed for these pilot tests and ORP levels were controlled using Evoqua™ ORP monitors in combination with a control unit. Ozone was dosed in 640 litre IBC tanks using ceramic aeration stones. Excess gaseous ozone was destructed using an ozone destructor. For safety reasons, the system was equipped with an alarm system which monitored the ozone concentration in air.

3 Materials and Methods

3.1 Minimum effective treatment dose

3.1.1 Objective

To assess the required minimum dose for effective ballast water treatment in different water types by AirTree's Ozone Treatment System (OTS).

3.1.2 Procedure

Fresh and marine water was cultured in several ponds outdoor at the IMARES laboratories. The cultures represent local phytoplankton and zooplankton communities. To encourage phytoplankton growth, nutrients (N, P and Si) were added appropriate for the type of water. Test batches of approx. 500 litres were prepared by mixing water from different cultured ponds with the same salinity. In this way the test batches obtained sufficient organism concentrations and diversity. Organisms >50µm were removed by filtration using a Hydrobios™ 50 µm plankton net. For each water type, a test batch with three tanks (640 litre IBC container) was prepared.

After instalment of a test batch (*Table 1*), two tanks were treated with target dose of 750 mV and 800 mV. One tank served as untreated control. To assess the initial treatment effect the concentrations of vital ≥ 10 to < 50 µm organisms were measured after 1 day. ORP was maintained at the start level by additional 'in-tank' dosing of ozone during one week. The system was set to allow an ORP drop of 15%. After one week, vital organism concentrations were counted again to assess the treatment efficacy after prolonged 'in-tank' treatment.

Table 1 Ozone treatment in three tanks for two water types.

Freshwater	Marine water
I: Control	I: Control
II: 750 mV (640-750 mV)	II: 750 mV (640-750 mV)
III: 800 mV (680-800 mV)	III: 800 mV (680-800 mV)

The tests were conducted in one of the large climate controlled laboratories from IMARES set at a temperature of $15 \pm 4^\circ\text{C}$. During ozonation, the tanks were kept closed as much as possible. Grab samples were drawn from the tanks via the ozone destruction line in the tanks. For comparison reasons the same method was used for the control tank. During the experiment, there was no active mixing of the tanks.

3.1.3 Analyses

Different analysis techniques were applied to assess treatment efficacies focussing on organisms ≥ 10 to < 50 µm in minimum dimension.

For ozone measurements, the Evoqua™ redox monitors provided by the client, were used and data from the monitors were recorded manually. It was intended to use a Mettler™ Toledo LE510 electrode as check for the Evoqua™ redox monitors. However, the Mettler™ electrode was unable to give stable results. Indirect measurements of the ozone concentration in the water were provided by analysing the oxidant (TRO) levels using the colorimetric DPD method (WTW method 250420).

General water quality parameters (temperature, oxygen content, pH, salinity and turbidity) were measured on a daily basis (excl. weekends) using handheld electrodes (HACH™ for temperature and oxygen content, IMARES procedure E_4_26; Mettler™-Toledo for pH, IMARES procedure E_4_24) and a Eutech Instruments™ Turbidimeter (IMARES procedure E_4_41). Total Suspended Solids (TSS) was analysed at the start of the experiment and on the discharge water after 7 days (IMARES procedure E_4_91).

Phytoplankton (dominating the ≥ 10 to < 50 μm group) and microzooplankton were counted using a fluorescence microscope (Zeiss) after FDA/CMFDA staining of the living organisms according to the IMARES procedure E_4_92. The vitality of the phytoplankton community was monitored using PAM-fluorometer (Algae Lab Analyser, BBE Moldaenke™, IMARES procedure E_4_49).

3.2 DBP-formation test

3.2.1 Objective

To describe the DBP formation due to 'continuous' in-tank treatment with ozone during a 4 week treatment period.

3.2.2 Procedure

One water type was used for the DBP formation test. The water was extracted from the outdoor IMARES cultures to ensure a comparable water quality with the treatment doses tests. The test water was dosed with an ozone concentration of 800 mV, which was maintained at that level with an accepted drop of 15%. The treatments were performed in duplicate, resulting in 4 tanks: two untreated controls and two treatments. Each tank (640 litre IBC container) was filled with approximately 500 litres of test water.

Samples for DBP analyses were taken before dosing (T0), after 1 Day (conform ETV), 5 Days (conform IMO), and 7, 14, 21 and 28 Days. At T0 and T28 the controls were also sampled. In total 18 samples were stored until transportation conform prescriptions of Labor IBEN GmbH (Bremerhaven, Germany). Nitrate and nitrite were analysed by our partner institute NIOZ ('t Horntje, The Netherlands).

Organism concentrations were assessed prior to the test initiation (starting conditions) and after 1 Day (conform ETV) and 5 Days (conform IMO) in order to verify if the treatment efficacy was comparable to that established in the minimum effective dose test. Organism concentrations were not assessed at the termination of the test (T28).

The tests were conducted in one of the large climate controlled laboratories from IMARES set at a temperature of $15 \pm 4^\circ\text{C}$. During ozonation the tanks were kept closed as much as possible. Grab samples were drawn from the test tanks via the ozone destruction line in the tanks. For comparison reasons the same method was used for the control tank. During the experiment, there was no active mixing of the tanks.

3.2.3 Analyses

For ozone measurements, the Evoqua™ redox monitors provided by the client, were used and data from the monitors were recorded manually. Indirect measurements of the ozone concentration in the water were provided by analysing the oxidant (TRO) levels using the colorimetric DPD method (WTW method 250420).

General water quality parameters (temperature, oxygen content, pH, salinity and turbidity) were measured on a daily basis (excl. weekends) using handheld electrodes (HACH™ for temperature and oxygen content, IMARES procedure E_4_26; Mettler™-Toledo for pH, IMARES procedure E_4_24) and a Eutech Instruments™ Turbidimeter (IMARES procedure E_4_41). Total Suspended Solids (TSS) was analysed at the start of the experiment and on the discharge water after 7 days (IMARES procedure E_4_91).

TOC, DOC and total nitrogen analyse were performed by our partner institute NIOZ in samples representing the start of the experiment, discharge after 5 days (IMO G8 guideline) and discharge after 28 days (maximum duration of DBP-formation test).

Phytoplankton (dominating the ≥ 10 to < 50 μm group) and microzooplankton were counted using a fluorescence microscope (Zeiss) after FDA/CMFDA staining of the living organisms according to the IMARES procedure E_4_92. The vitality of the phytoplankton community was monitored using PAM-fluorometer (Algae Lab Analyser, BBE Moldaenke™, IMARES procedure E_4_49).

Disinfection by-products (DBP) analysis were outsourced to Labor IBEN GmbH. Unfortunately, this laboratory (but also three other laboratories that were asked) could not analyse iodate ions in seawater with a sufficiently low detection limit and high confidence. Therefore, it was decided not to analyse this component and focus on the other DBPs. The method and corresponding detection limits for analysis of the DBPs in seawater are given in *Table 2*.

Table 2 Method and detection limits of disinfection by-products analysis in seawater.

Chemical name	formula	CAS	Method	Detection limit	Units
Bromate Ion	NaBrO ₃	7789-38-0	LC-MS-MS	100	$\mu\text{g/l}$
Iodate ion	NaIO ₃	7681-55-2	Unable to analyse in seawater		
Trichloroethylene	C ₂ HCl ₃	79-01-6	DIN EN ISO 10301 (F 4)1997-08	0.1	$\mu\text{g/l}$
Tribromomethane	CHBr ₃	75-25-2	DIN EN ISO 10301 (F 4)1997-08	0.1	$\mu\text{g/l}$
Dibromoacetic acid*	CHBr ₂ COOH	631-64-1	GC-MS after derivation	0.5	$\mu\text{g/l}$
Dibromoacetonitrile	CHBr ₂ CN	3252-43-5	GC-MS	0.5	$\mu\text{g/l}$
Dichloroacetonitrile	CHCl ₂ CN	3018-12-0	GC-MS	0.1	$\mu\text{g/l}$

*This analysis was outsourced by Labor IBEN GmbH.

3.3 Sub-contractors

3.3.1 Water Quality testing – TOC/DOC, total nitrogen, nitrate and nitrite

Royal Netherlands Institute for Sea Research (NIOZ)
Landsdiep 4
1797SZ 't Horntje (Island of Texel)
The Netherlands

3.3.2 Water Quality testing – Disinfection by-products

Labor IBEN GmbH (Institut für Lebensmittel- und Umweltanalytik)
Am Lunedeich 157
D-27572 Bremerhaven
Germany

4 Results

The results given in this report apply only to the samples analysed.

4.1 Minimum effective treatment dose

4.1.1 Test water quality

The salinity in the first test (*Table 3*) was above 32 psu indicating IMO marine water. All environmental parameters met the IMO G8 requirements. No distinction was made between organisms ≥ 10 to $< 50 \mu\text{m}$ and organisms $< 10 \mu\text{m}$. However, the total number of organisms were well above the IMO G8 requirement. Based on counting's made for the DBP formation test (4.2.1) which used water from the same cultures, it is very likely that sufficient organisms ≥ 10 to $< 50 \mu\text{m}$ were present. The measured chemical parameters should be seen as background levels and show the minor interferences due to the composition of the test water.

Table 3 Test water quality for Marine water test at uptake (T0). Test initiated 21 July 2014. NA: not analysed. Measured concentration is average of three replicates with standard deviation.

Parameter	Units	IMO G8 requirements	Measured concentration
Environmental parameters			
Salinity	psu	>32	34.4 ± 0.1
Conductivity	mS/cm	-	49.7 ± 0.2
TSS	mg/l	>1	45 ± 5
DOC	mg/l	>1	NA
TOC	mg/l	-	NA
POC (TOC-DOC)	mg/l	>1	NA
Temperature	°C	-	22.2 ± 0.1
Acidity (pH)	-	-	8.9 ± 0.0
Dissolved oxygen (DO)	mg/l	-	6.6 ± 0.0
Dissolved oxygen saturation	%	-	93.5 ± 0.1
Turbidity	NTU	-	14.1 ± 0.3
Biological parameters			
Organisms ≥ 10 to $< 50 \mu\text{m}$ + Organisms $< 10 \mu\text{m}$	n/ml	$\geq 1,000$	$4,420 \pm 87$
Chlorophyll-a	$\mu\text{g/l}$	-	806.0 ± 7.4
Activity of chlorophyll-a	%	-	37.4 ± 1.5
Chemical parameters			
Oxygen reduction potential (ORP)	mV	-	191 ± 6
TRO (as free chlorine)	mg/l Cl_2	-	0.82 ± 0.01
TRO (as total chlorine)	mg/l Cl_2	-	0.82 ± 0.02

The salinity in the second test presented in *Table 4* was below 3 psu indicating that freshwater was used for this test according to the definitions of IMO. The concentration of TSS did not reach the IMO G8 requirement, but as this test was focussed on biological efficacy, it was chosen not to amend the test water. The biological parameters were well above the IMO G8 requirements. The measured chemical parameters should be seen as background levels and show the minor interferences due to the composition of the test water.

Table 4 Achieved test water quality for Freshwater test at uptake (T0). Test initiated 31 July 2014. NA: not analysed. Measured concentration is average of three replicates with standard deviation.

Parameter	Units	IMO G8 requirements	Measured concentration
Water quality parameters			
Salinity	psu	<3	1.1 ± 0.0
Conductivity	mS/cm	-	2.1 ± 0.4
TSS	mg/l	>50	17 ± 1
DOC	mg/l	>5	NA
TOC	mg/l	-	NA
POC (TOC-DOC)	mg/l	>5	NA
Temperature	°C	-	19.1 ± 0.3
Acidity (pH)	-	-	8.5 ± 0.0
Dissolved oxygen (DO)	mg/l	-	8.5 ± 0.1
Dissolved oxygen saturation	%	-	98.6 ± 0.9
Turbidity	NTU	-	18.4 ± 0.3
Biological parameters			
Organisms ≥10 to <50 µm	n/ml	≥1,000	11,750 ± 1,069
Organisms <10 µm	n/ml	-	3,388 ± 871
Chlorophyll-a	µg/l	-	69.3 ± 5.0
Activity of chlorophyll-a	%	-	37.7 ± 5.4
Chemical parameters			
Oxygen reduction potential (ORP)	mV	-	274 ± 8
TRO (as free chlorine)	mg/l Cl ₂	-	0.45 ± 0.03
TRO (as total chlorine)	mg/l Cl ₂	-	0.49 ± 0.04

4.1.2 Results of the marine minimum effective treatment dose test

During the test multiple recordings of the ozone concentration as ORP in each test tank were made (Figure 1). As this was only done during working hours, this graph does not show the complete ozone pattern during the experiment. The graph does give indication about the time needed to build up the ozone concentration in the test water to reach the target value. After approximately 3.5 hours of continuously dosing, the first tank reached the set limit of 750 mV. It took another 1.5 hour before the second tank reached the limit of 800 mV. After gradual building up the ozone concentration, the ORP levels remained within the set limits for the next days. After approximately 7 days (161 hours) the system was stopped allowing the ozone to disappear from the tank. The ozone disappeared more rapidly from the tank with the lowest set value (750 mV).

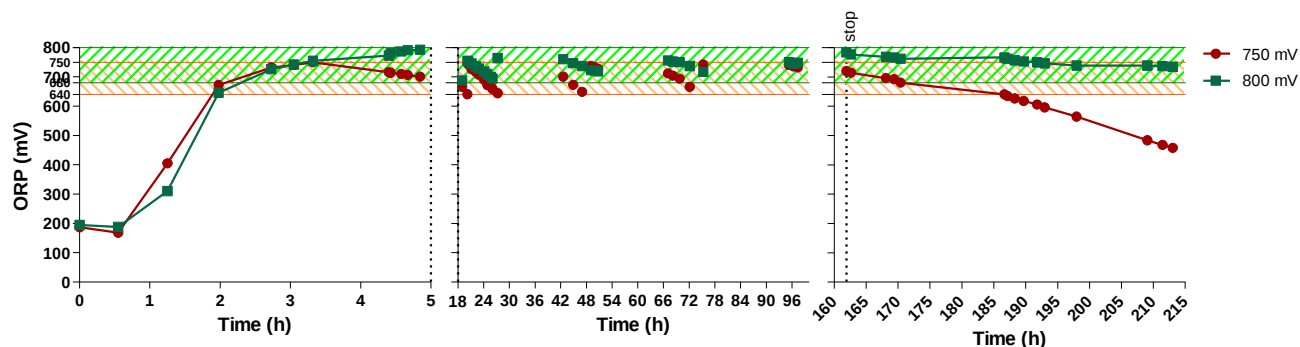


Figure 1 Ozone concentrations as oxygen reduction potential (ORP) over time in each test tank holding marine water (start-mid-end). The graph includes the allowed range based on a drop of 15% for each of the aimed ORP levels (orange for 750 mV and green for 800 mV).

In the control test tank the oxidant (TRO) concentrations remained at stable levels throughout the experiment (Figure 2). After starting the ozonation system, TRO ranged between 1.4-2.9 mg/l as total Cl_2 for the test tank with the lowest set value (750 mV) of which 67-79% were analysed as free oxidants. In the test tank with the highest set value (800 mV) TRO ranged between 1.9-6.3 mg/l as total Cl_2 of which 75-89% were analysed as free oxidants. After shutting down the ozonation system, the TRO levels dropped in both tanks reaching concentrations of 1.4 and 2.9 mg/l as total Cl_2 after two days for test tanks 750 mV and 800 mV respectively.

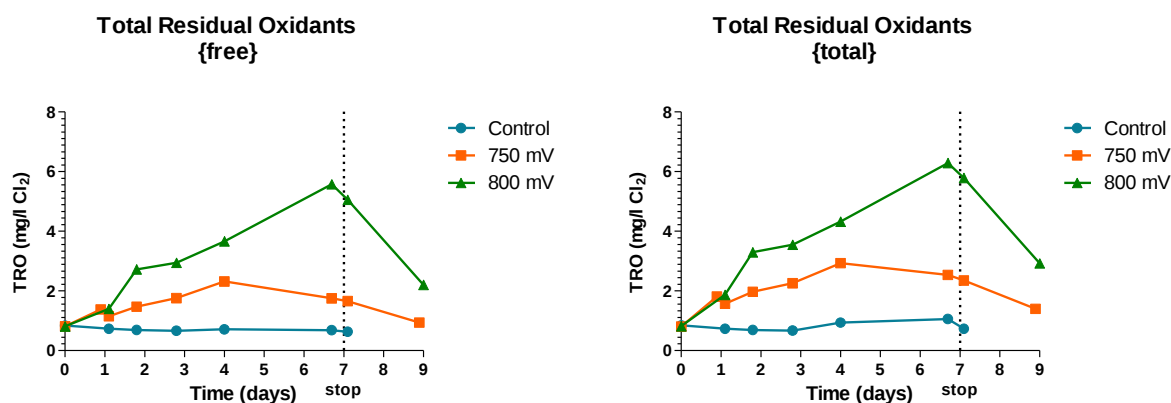


Figure 2 The oxidant levels over time in each test tank for Marine test. Left: free oxidant concentrations. Right: total oxidant concentrations.

The environmental parameters and the biological parameters chlorophyll-a and activity of the chlorophyll-a, measured on working days during the exposure time of the marine experiment are listed in Table 5. Salinity, conductivity and acidity were stable during the 7 day experiment. The temperatures ranged between 13.5 to 22.4°C. The high temperatures measured during the first three days were a result of sample handling and temperature of the outside cultures in the beginning. The measured temperatures during the last four days of the experiment represent the test tank conditions (13.5 to 14.8°C). Oxygen levels in the control tank declined from 93.4% to 63.6% saturation and the levels in the test tanks increased to a maximum of 114.3% for 750 mV and 119.0% for 800 mV. Turbidity was most stable in the control tank and differed randomly in the test tanks.

The chlorophyll-a content (indicator for amount of algae) was stable in the control tank with a measured average of 799 ± 63 µg/l and an activity (indicator of viability) of $44 \pm 5\%$. The chlorophyll-a content in the test tank 750 mV was still measurable for two days, but could not be detected from the third day till the last day of treatment. Activity of the algae decreased to 10.8% and 13.4% after one day and two days respectively followed by not detectable in the remaining period. Chlorophyll-a content and activity in the test tank 800 mV was not detectable anymore after one day and remained not detectable in the test.

Table 5 *Environmental parameters and Chlorophyll-a results for Marine test. Test initiated 21 July 2014. NA: not analysed, dl: below detection limit of analysis.*

#	Sample	Date	Salinity (psu)	Conductivity (mS/cm)	Temperature (°C)	Acidity pH (-)	Dissolved oxygen (mg/l)	DO saturation (%)	Turbidity (ntu)	Chlorophyll-a (µg/l)	Activity chlorophyll-a (%)
I	Control	21 Jul 14	34.4	49.8	22.4	8.9	6.6	93.4	14.0	814.3	37.2
I	Control	22 Jul 14	34.2	46.7	19.8	8.7	6.1	83.2	13.5	851.6	41.3
I	Control	23 Jul 14	34.4	48.6	21.4	8.7	5.1	71.9	14.0	831.4	43.7
I	Control	24 Jul 14	34.6	50.2	14.5	8.6	5.5	78.6	12.3	703.8	46.5
I	Control	25 Jul 14	34.1	42.7	13.9	NA	5.2	63.6	11.8	736.3	44.5
I	Control	28 Jul 14	34.4	49.0	14.0	8.7	5.4	71.7	10.6	854.0	50.5
II	750 mV	21 Jul 14	34.5	49.8	22.3	8.9	6.6	93.4	14.2	803.6	38.0
II	750 mV	22 Jul 14	34.8	51.0	20.1	8.7	8.1	114.3	9.2	43.4	10.8
II	750 mV	23 Jul 14	34.3	48.9	21.8	8.3	7.8	112.8	14.7	6.5	13.4
II	750 mV	24 Jul 14	34.7	51.8	14.8	8.4	7.8	112.9	6.7	dl	dl
II	750 mV	25 Jul 14	33.8	42.7	13.9	NA	8.8	108.0	7.5	dl	dl
II	750 mV	28 Jul 14	34.5	49.4	13.6	8.5	8.3	110.5	6.2	dl	dl
III	800 mV	21 Jul 14	34.4	49.5	22.1	8.9	6.7	93.6	14.1	800.2	40.1
III	800 mV	22 Jul 14	34.7	51.0	20.4	8.7	8.0	114.5	7.3	dl	dl
III	800 mV	23 Jul 14	34.2	47.7	20.7	8.4	8.3	118.1	16.7	dl	dl
III	800 mV	24 Jul 14	34.6	51.5	14.3	8.4	7.8	113.8	7.6	dl	dl
III	800 mV	25 Jul 14	34.3	42.9	13.9	NA	9.8	119.0	9.2	dl	dl
III	800 mV	28 Jul 14	34.5	49.3	13.5	8.3	8.6	114.1	14.1	dl	dl

The results of the biological parameters regarding organism density in the marine test are presented in *Table 6*. Sufficient organisms survived the holding period of 7 days in the control tank. Both treatment levels (750 mV and 800 mV) achieved the D-2 discharge standard after a holding period of 7 days. In fact, an effective treatment was already seen in the samples taken after one day. There were still a few viable organisms left in the 750 mV treatment after a treatment period of one and 7 days. No viable organisms were counted in the higher treatment of 800 mV after a treatment period of 7 days and less than one after the first day.

Table 6 Treatment efficacy for Marine test based on the organism density of organisms ≥ 10 to $< 50 \mu\text{m}$ and organisms $< 10 \mu\text{m}$ in minimum dimension. Test initiated 21 July 2014. Discharge samples is average of two replicates with standard deviation.

#	Sample	Type of water	Date	Org. ≥ 10 to $< 50 \mu\text{m}$ + org. $< 10 \mu\text{m}$ (n/ml)	D-2 standard	D-2 standard achieved?
I	Control	Uptake	21 Jul 2014	4,460	$> 1,000$	Yes
I	Control	Discharge day 1	22 Jul 2014	$6,300 \pm 796$	-	-
I	Control	Discharge day 7	28 Jul 2014	$5,871 \pm 468$	> 100	Yes
II	750 mV	Uptake	21 Jul 2014	4,320	$> 1,000$	Yes
II	750 mV	Discharge day 1	22 Jul 2014	3.0 ± 2.0	-	-
II	750 MV	Discharge day 7	28 Jul 2014	1.0 ± 1.0	< 10	Yes
III	800 mV	Uptake	21 Jul 2014	4,480	$> 1,000$	Yes
III	800 mV	Discharge day 1	22 Jul 2014	0.67 ± 0.58	-	-
III	800 mV	Discharge day 7	28 Jul 2014	0 ± 0	< 10	Yes

4.1.3 Results of the freshwater minimum effective treatment dose test

During the test period for the freshwater efficacy test multiple recordings of the ozone concentration as ORP in each test tank was recorded (Figure 3). As only recordings were made during working hours, this graph does not show the complete ozone flux during the experiment. The graph does give indication about the time needed to build up the ozone concentration in the test water to reach the set maximum value.

During the first hours of continuous dosing, very slight changes in the ORP readings were notable in the test tanks. After six hours the ORP levels increased and the set values were reached. Ozone was dosed on regular bases after reaching the maximum drop value for each tank. It was noted that the ORP levels measured reached higher values than the set values of 750 and 800 mV. After approximately 7 days (163 hours), the ozonation system was stopped allowing the ozone to disappear from the tank. Both tanks showed a similar decline in ORP readings.

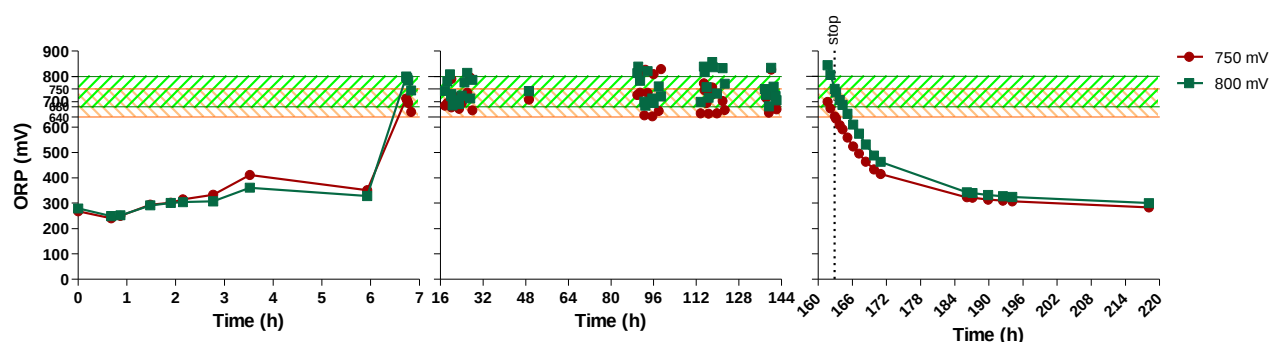


Figure 3 Recorded ozone concentrations as oxygen reduction potential (ORP) over time in each test tank holding freshwater (start-mid-end). The graph includes the allowed range based on a drop of 15% for each of the aimed ORP levels.

Indirect measurements of the ozone concentration in water by analysing the oxidant (TRO) levels using the colorimetric DPD method (WTW method 250420) are presented in *Figure 4*. TRO concentrations ranged between 0.30 to 0.65 mg/l as total Cl_2 in the control tank. The measured TRO concentrations in both test tanks fell in the range of the measured concentrations in the control tank.

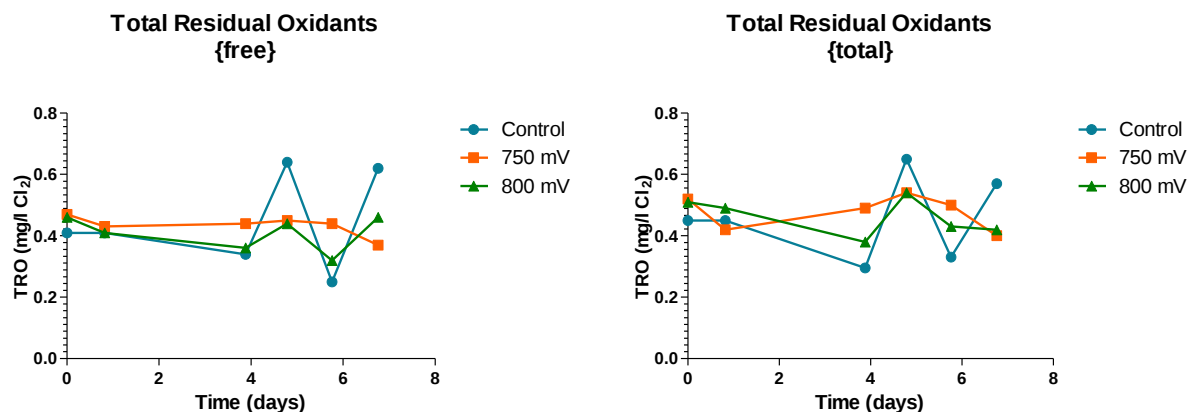


Figure 4 The oxidant levels over time in each test tank for Freshwater test. Left: free oxidant concentrations. Right: total oxidant concentrations.

The environmental parameters and the biological parameters chlorophyll-a and activity of the chlorophyll-a, measured on working days during the exposure time of the marine experiment are listed in *Table 7*. Salinity, conductivity and acidity were stable during the 7 day experiment. The temperatures started at approximately 19°C at the start of the experiment, measured after filling the tanks with cultured test water located outdoor at the IMARES facility. The temperatures measured in the following days ranged between 12.9 to 16.9°C. Oxygen levels in the control tank ranged from 81.5 to 99.0% saturation with a gradual decrease over time. The oxygen saturation in the test tanks increased to a maximum of 109.9% for 750 mV and 111.0% for 800 mV. Turbidity was most stable in the control tank and decreased in the test tanks.

The chlorophyll-a content (indicator for amount of algae) was stable in the control tank with a measured average of 63 ± 18 $\mu\text{g/l}$ and an activity (indicator of viability) of $33 \pm 7\%$. The chlorophyll-a content in the test tank 750 mV was still measurable after one day (3.8 $\mu\text{g/l}$), but could not be detected from the second day till the last day of treatment. Activity of the algae decreased to 27.6% after one day followed by not detectable in the remaining period. Chlorophyll-a content and activity in the test tank 800 mV was not detectable anymore after one day and remained not detectable in the test.

Table 7 Environmental parameters and Chlorophyll-a results for Freshwater test. Test initiated 31 July 2014. dl: below detection limit of analysis

#	Sample	Date	Salinity (psu)	Conductivity (mS/cm)	Temperature (°C)	Acidity pH (-)	Dissolved oxygen (mg/l)	DO saturation (%)	Turbidity (ntu)	Chlorophyll-a (µg/l)	Activity chlorophyll-a (%)
I	Control	31 Jul 14	1.1	2.1	19.5	8.5	8.5	99.0	18.4	66.6	31.7
I	Control	1 Aug 14	1.2	2.1	16.9	8.5	8.5	94.8	36.4	93.7	37.3
I	Control	4 Aug 14	1.1	2.0	13.9	8.4	8.0	87.2	23.5	51.4	30.1
I	Control	5 Aug 14	1.1	1.8	13.7	8.5	7.8	82.4	26.2	60.8	29.8
I	Control	6 Aug 14	1.1	1.8	14.5	8.4	7.4	81.5	14.9	41.4	25.5
I	Control	7 Aug 14	1.1	1.9	12.9	8.4	6.8	85.0	27.1	63.8	44.1
II	750 mV	31 Jul 14	1.1	2.0	18.9	8.5	8.5	97.6	18.1	66.3	42.3
II	750 mV	1 Aug 14	1.2	2.0	16.4	8.3	9.2	103.2	7.7	3.8	27.6
II	750 mV	4 Aug 14	1.1	1.9	13.3	8.3	9.9	108.9	4.5	dl	dl
II	750 mV	5 Aug 14	1.1	1.9	13.1	8.3	10.4	109.9	5.2	dl	dl
II	750 mV	6 Aug 14	1.1	1.9	15.4	8.2	9.6	105.7	4.6	dl	dl
II	750 mV	7 Aug 14	1.1	1.9	13.7	8.2	8.6	108.5	4.0	dl	dl
III	800 mV	31 Jul 14	1.1	2.0	19.0	8.5	8.6	99.3	18.8	75.1	39.0
III	800 mV	1 Aug 14	1.2	2.1	16.5	9.4	9.4	106.3	7.4	dl	dl
III	800 mV	4 Aug 14	1.1	1.9	13.1	8.3	10.2	111.0	6.1	dl	dl
III	800 mV	5 Aug 14	1.1	1.9	12.8	8.3	10.4	110.5	5.5	dl	dl
III	800 mV	6 Aug 14	1.1	1.9	15.3	8.2	9.9	107.4	4.5	dl	dl
III	800 mV	7 Aug 14	1.1	1.9	13.3	8.2	8.8	110.4	5.2	dl	dl

The results of the biological parameters regarding organism density in the marine test are presented in Table 9. At the start of the experiment, the total viable organisms <50 µm were counted without classification in the IMO group ≥10 to <50 µm in minimum dimension. From the 1st of August this distinction was made. Based on the results from the 1st of August it is known that approximately 86% of the total organisms counted fell into the category <10 µm in minimum dimension.

Sufficient organisms survived the holding period of 7 days in the control tank. Both treatment levels (750 mV and 800 mV) achieved the D-2 discharge standard after a holding period of 7 days. In fact, an effective treatment was already seen in the samples taken after one day. There were still a few viable organisms left in the 750 mV treatment after one and 7 days. On average less than one organism ≥10 to <50 µm in minimum dimension survived the treatment after one day and no viable organisms <50 µm were counted in the higher treatment of 800 mV after a treatment period of 7 days.

Table 8 Treatment efficacy for Freshwater test based on the organism density of organisms ≥ 10 to $< 50 \mu\text{m}$ and organisms $< 10 \mu\text{m}$ in minimum dimension. Test initiated 31 July 2014.

#	Sample	Type of water	Date	Org. $< 10 \mu\text{m}$ (n/ml)	Org. ≥ 10 to $< 50 \mu\text{m}$ (n/ml)	D-2 standard	D-2 standard achieved?
I	Control	Uptake	31 Jul 2014	11,163 $\pm$ 1,432		>1,000	Yes
I	Control	Discharge day 1	1 Aug 2014	13,667 $\pm$ 1531	2,150 $\pm$ 691	-	-
I	Control	Discharge day 7	7 Aug 2014	2,108 $\pm$ 530	475 $\pm$ 141	>100	Yes
II	750 mV	Uptake	31 Jul 2014	12,325		>1,000	Yes
II	750 mV	Discharge day 1	1 Aug 2014	1.7 $\pm$ 1.2	0.33 $\pm$ 0.58	-	-
II	750 mV	Discharge day 7	7 Aug 2014	1.0 $\pm$ 1.0	0.67 $\pm$ 0.58	<10	Yes
III	800 mV	Uptake	31 Jul 2014	12,350		>1,000	Yes
III	800 mV	Discharge day 1	1 Aug 2014	0 $\pm$ 0	0.33 $\pm$ 0.58	-	-
III	800 mV	Discharge day 7	7 Aug 2014	0 $\pm$ 0	0 $\pm$ 0	<10	Yes

4.2 DBP-formation test

4.2.1 Challenge water quality

The salinity in the DBP-formation test presented in Table 9 was just below 32 psu indicating that brackish water was used for this test according to the definitions of IMO. The test water was not amended to reach higher salinity. TSS, DOC and POC met the IMO G8 requirements for marine water and the levels of DOC and POC were also well above the requirements for brackish water. The biological parameters achieved the IMO G8 requirements. The measured chemical parameters should be seen as background levels and show the minor interferences due to the composition of the test water.

Table 9 Achieved test water quality for Marine DBP-formation water test at uptake (T0). Test initiated 21 August 2014.

Parameter	Units	IMO G8 requirements	Measured concentration
Environmental parameters			
Salinity	psu	>32	31.6 ± 0.0
Conductivity	mS/cm	-	39.9 ± 0.1
TSS	mg/l	>1	31 ± 5
DOC	mg/l	>1	25.3 ± 0.1
TOC	mg/l	-	34.3 ± 0.2
POC (TOC-DOC)	mg/l	>1	9.0
Acidity (pH)	-	-	8.7 ± 0.0
Temperature	°C	-	15.8 ± 0.0
Dissolved oxygen (DO)	mg/l	-	8.2 ± 0.0
Dissolved oxygen saturation	%	-	99.7 ± 0.4
Turbidity	NTU	-	8.0 ± 0.2
Biological parameters			
Organisms ≥10 to <50 µm	n/ml	≥1,000	1,313 ± 92
Organisms <10 µm	n/ml	-	24,863 ± 2,398
Chlorophyll-a	µg/l	-	184.8 ± 2.0
Activity of chlorophyll-a	%	-	33.5 ± 1.0
Chemical parameters			
Oxygen reduction potential (ORP)	mV	-	211 ± 3
TRO (as free chlorine)	mg/l Cl ₂	-	0.40 ± 0.02
TRO (as total chlorine)	mg/l Cl ₂	-	0.40 ± 0.01

4.2.2 Test results of the DBP-formation test

During the test period for the marine efficacy test multiple recordings of the ozone concentration as ORP in each test tank was recorded (*Figure 5*). As only recordings were made during working hours, this graph does not show the complete ozone flux during the experiment. The graph does give indication about the time needed to build up the ozone concentration in the test water to reach the set maximum value.

After approximately four hours of continuously dosing, both replicated test tanks reached the set limit of 800 mV. After this gradual building up of ozone concentration, the ORP levels remained within the set limits for most of the test period of 28 days. Only on day 17 and on day 20 a value was recorded below the set minimum of 680 mV. This was a result of an unexpected alarm situation according to the system. However, no ozone odour could be detected in the climate controlled room and only after a few hours the system reactivated again. After a treatment period of 28 days, the system was stopped and the decline of ozone as ORP was monitored.

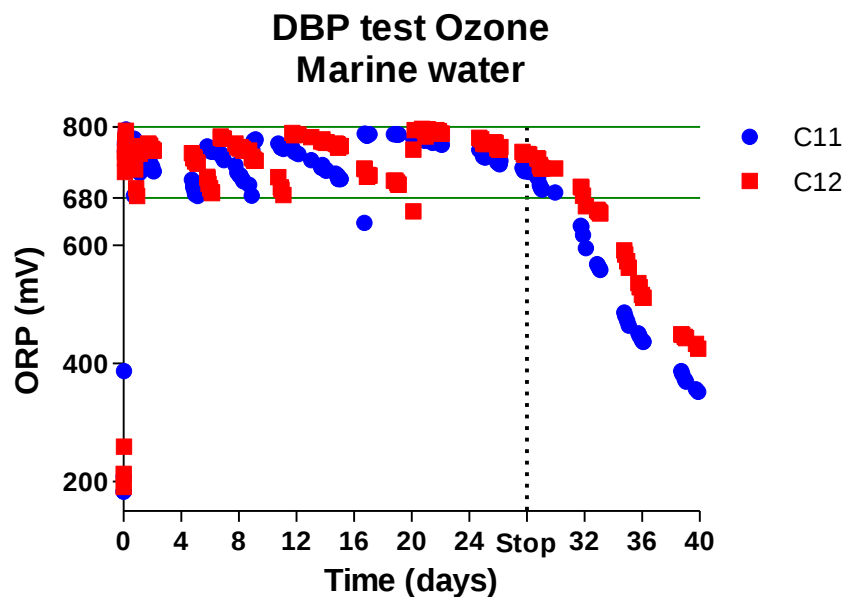


Figure 5 Recorded ozone concentrations as oxygen reduction potential (ORP) over time in each test tank during the DBP-formation test. The graph includes the allowed range based on a drop of 15% for the aimed ORP level. C11 and C12 represent the two 800 mV treatment replicates.

Indirect measurements of the ozone concentration in water by analysing the oxidant (TRO) levels using the colorimetric DPD method (WTW method 250420) are presented in Figure 6. The control test tank remained at stable levels throughout the experiment. After start of the ozonation system, the TRO ranged between 1.5-5.1 mg/l as total Cl_2 of which 53-87% were analysed as free oxidants.

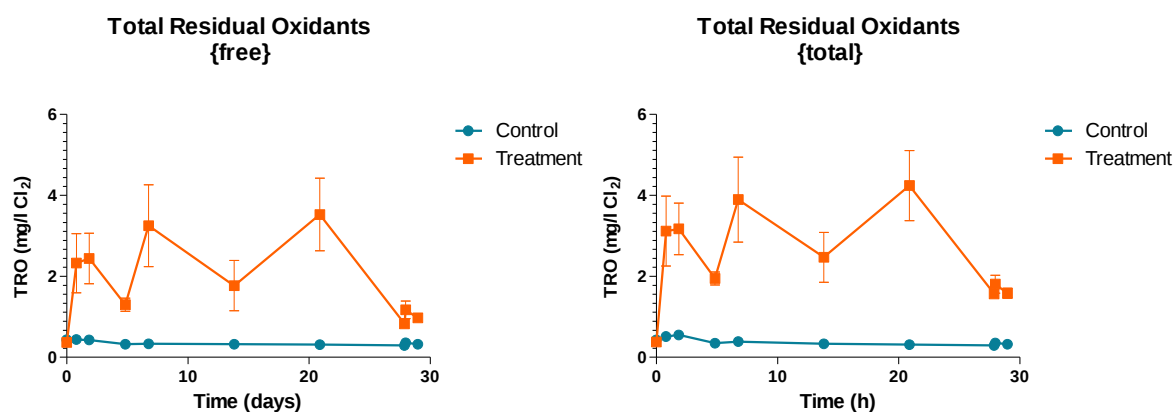


Figure 6 The oxidant levels over time in each test tank for DBP-formation test. Left: free oxidant concentrations. Right: total oxidant concentrations.

Environmental parameters and the chlorophyll-a concentration and activity were measured at each sampling event and the results are listed in Table 10. Salinity, conductivity and acidity were stable during the 28 day experiment. Also, the temperature was much more stable during the entire experiment ranging from 13.0 to 15.8°C. Oxygen saturation shows a slight decline over time in the control tanks with a minimum of 58.1% after 14 days.

Turbidity started at 8.2 ntu and declined during 28 days to 4.4 ntu for the control tanks. Turbidity levels declined more rapidly in the treatment tanks during the first day and very gradual for the remaining treatment period.

The measured chlorophyll-a content and activity remained stable in the control throughout the 28 days. No chlorophyll-a could be detected after the first day of treatment until the end of the experiment.

Table 10 *Environmental parameters and Chlorophyll-a results for DBP-formation test (average of two replicates). Test initiated 21 August 2014. NA: not analysed, dl: below detection limit of analysis.*

Sample	Date	Salinity (psu)	Conductivity (mS/cm)	Temperature (°C)	Acidity pH (-)	Dissolved oxygen (mg/l)	DO saturation (%)	Turbidity (ntu)	Chlorophyll-a (µg/l)	Activity chlorophyll-a (%)
Control	21 Aug 14	31.7	40.0	15.8	8.7	8.2	99.6	8.2	185.9	34.4
Control	22 Aug 14	31.4	41.4	14.4	NA	7.3	90.3	7.9	171.4	45.6
Control	25 Aug 14	31.1	39.6	13.4	NA	5.8	70.8	7.6	194.3	47.2
Control	26 Aug 14	31.1	40.2	13.6	NA	5.0	63.2	6.2	188.4	42.4
Control	28 Aug 14	31.1	40.5	13.5	NA	4.6	58.1	5.9	174.5	48.8
Control	4 Sep 14	31.6	41.9	13.6	8.5	5.3	66.9	5.0	183.9	42.9
Control	11 Sep 14	31.4	44.1	13.7	8.3	5.6	72.5	4.1	178.6	50.6
Control	18 Sep 14	31.4	43.9	13.6	8.2	5.9	79.2	4.4	184.4	46.7
800 mV	21 Aug 14	31.6	39.8	15.8	8.7	8.2	99.9	7.8	183.7	32.6
800 mV	22 Aug 14	31.4	41.4	14.7	NA	8.7	107.7	3.4	dl	dl
800 mV	25 Aug 14	31.1	40.5	13.7	NA	8.6	106.3	3.4	dl	dl
800 mV	26 Aug 14	31.1	40.5	13.2	NA	9.2	116.6	3.1	dl	dl
800 mV	28 Aug 14	31.4	41.0	13.0	NA	9.6	120.7	2.8	dl	dl
800 mV	4 Sep 14	31.5	41.5	13.3	8.3	9.3	117.2	2.4	dl	dl
800 mV	11 Sep 14	31.4	43.7	13.4	8.2	9.6	122.2	2.8	dl	dl
800 mV	18 Sep 14	31.4	43.6	13.1	8.1	8.5	113.4	2.0	dl	dl

The results of the biological parameters regarding organism density in the marine test are presented in *Table 11*. Sufficient organisms survived the holding period of 7 days in the two control tanks. The two treatment tanks (800 mV) achieved the D-2 discharge standard after a holding period of 7 days. In fact, an effective treatment was already seen in the samples taken after one day. None of the organisms <10 µm and organisms ≥10 to <50 µm in minimum dimension survived the treatment. The treatment was 100% effective.

Table 11 Treatment efficacy for DBP-formation test based on the organism density of organisms ≥ 10 to $< 50 \mu\text{m}$ and organisms $< 10 \mu\text{m}$ in minimum dimension. Test initiated 21 August 2014.

Sample	Type of water	Date	Org. $< 10 \mu\text{m}$ (n/ml)	Org. ≥ 10 to $< 50 \mu\text{m}$ (n/ml)	D-2 standard	D-2 standard achieved?
Control	Uptake	21 Aug 2014	24,763 $\pm$ 3,942	1,238 $\pm$ 53	>1,000	Yes
Control	Discharge day 1	22 Aug 2014	22,238 $\pm$ 2,121	1,128 $\pm$ 227	-	-
Control	Discharge day 7	28 Aug 2014	17,875 $\pm$ 124	844 $\pm$ 203	>100	Yes
800 mV	Uptake	21 Aug 2014	24,963 $\pm$ 1,290	1,388 $\pm$ 18	>1,000	Yes
800 mV	Discharge day 1	22 Aug 2014	0 $\pm$ 0	0 $\pm$ 0	-	-
800 mV	Discharge day 7	28 Aug 2014	0 $\pm$ 0	0 $\pm$ 0	<10	Yes

Phosphate, nitrite and nitrate concentrations were measured in the duplicate control and treatment samples at several sampling events during the 28 day treatment period (Table 12). Copies of the original test reports as received from NIOZ are attached in Appendix A. Phosphate concentrations were initially not planned to be analysed as no changes in the concentrations due to ozone treatment was expected. The results for phosphate as presented in Table 12 confirm no difference between control and treatment during the treatment period. On average a phosphate concentration of $194 \pm 9 \mu\text{g/l PO}_4\text{-P}$ was measured. Nitrite concentrations were stable for the control tanks and reduced in the treatment tanks over time. The average nitrite was $0.7 \pm 0.2 \mu\text{g/l NO}_2\text{-N}$ at the start of the test and decreased in the treatment tanks to $0.16 \pm 0.01 \mu\text{g/l NO}_2\text{-N}$. Nitrate concentrations were lowest at the start of the test (average of $3.8 \pm 1.4 \mu\text{g/l NO}_3\text{-N}$) and increased in the treatment tanks during the 28 days of treatment to $1,898 \pm 46 \mu\text{g/l NO}_3\text{-N}$.

Table 12 Results of the nutrients for ozone treated ballast water.

Component	Detection limit ($\mu\text{g/l}$)	Time (d)	Date	Control	Treatment
Phosphate ($\text{PO}_4\text{-P}$)	0.2	T00	21 Aug 2014	192 $\pm$ 4	195 $\pm$ 4
		T01	22 Aug 2014	-	186 $\pm$ 8
		T05	26 Aug 2014	-	194 $\pm$ 10
		T07	28 Aug 2014	-	196 $\pm$ 2
		T14	4 Sep 2014	-	203 $\pm$ 10
		T21	11 Sep 2014	-	200 $\pm$ 4
		T28	18 Sep 2014	176 $\pm$ 5	203 $\pm$ 4
Nitrite ($\text{NO}_2\text{-N}$)	0.03	T00	21 Aug 2014	0.58 $\pm$ 0.03	0.86 $\pm$ 0.07
		T01	22 Aug 2014	-	0.30 $\pm$ 0.05
		T05	26 Aug 2014	-	0.22 $\pm$ 0.01
		T07	28 Aug 2014	-	0.18 $\pm$ 0.06
		T14	4 Sep 2014	-	0.18 $\pm$ 0.06
		T21	11 Sep 2014	-	0.13 $\pm$ 0.06
		T28	18 Sep 2014	0.6 $\pm$ 0.02	0.16 $\pm$ 0.01
Nitrate ($\text{NO}_3\text{-N}$)	0.6	T00	21 Aug 2014	2.9 $\pm$ 1.4	4.7 $\pm$ 1.0
		T01	22 Aug 2014	-	979 $\pm$ 86
		T05	26 Aug 2014	-	1,328 $\pm$ 47
		T07	28 Aug 2014	-	1,420 $\pm$ 59
		T14	4 Sep 2014	-	1,692 $\pm$ 25
		T21	11 Sep 2014	-	1,766 $\pm$ 29
		T28	18 Sep 2014	5.8 $\pm$ 3.6	1,898 $\pm$ 46

The disinfection by-products measured at several sampling events during the treatment period of 28 days are presented in *Table 13*. Copies of the original test reports as received from Labor IBEN GmbH are attached in Appendix B. Bromate ion, dibromoacetonitrile, dichloroacetonitrile and trichloroethylene were analysed below detection limit at each sample event. Dibromoacetic acid and tribromomethane (bromoform) showed levels above detection limit for the treatments and the DBP-formation over time of these components are presented in *Figure 7*.

The concentration of dibromoacetic acid increased during the first 14 days, declined at day 21 and increased to $5,050 \pm 1,344 \mu\text{g/l}$ at day 28. Dibromoacetic acid was not present at the start of the test.

At the start of the experiment, tribromomethane concentrations ranged from $<0.1 \text{ (dl)}$ to $1 \mu\text{g/l}$ and increased in the treated tanks to $4,293 \pm 127 \mu\text{g/l}$ at day 28. Most of the tribromomethane was formed in the first five days of continuous dosing. After 5 days, tribromomethane concentration increased gradually and continued to increase until the last sample event at day 28. Concentrations of tribromomethane also increased in the control tanks to $105 \pm 32 \mu\text{g/l}$ after 28 days.

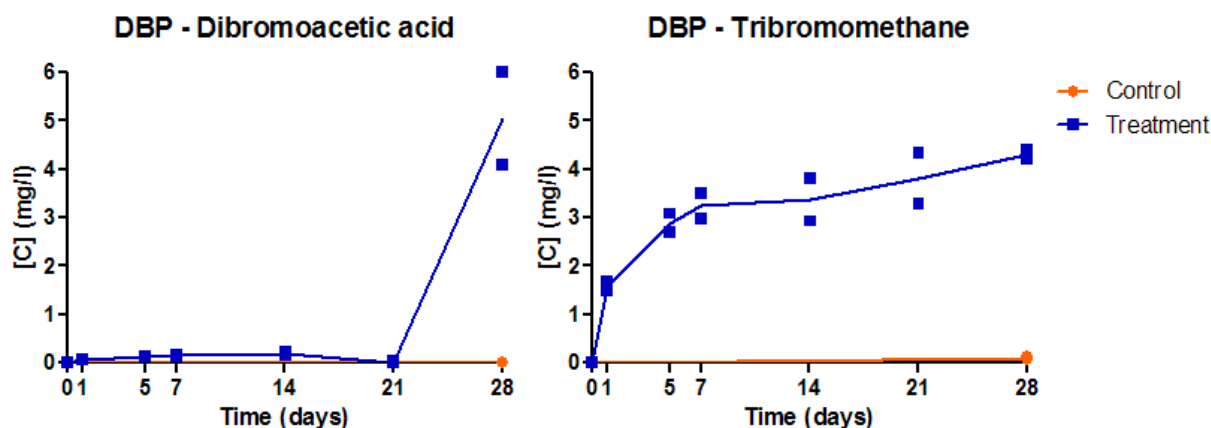


Figure 7 Formation of dibromoacetic acid (left) and tribromomethane (right) in ballast water treated with ozone with an aimed continuous ORP level of 800 mV.

Table 13 Results of the formation of disinfection by-products for ozone treated ballast water.

Component	Detection limit (µg/l)	Time (d)	Date	Control	Treatment
Bromate ion	100	T00	21 Aug 2014	dl	dl
		T01	22 Aug 2014	-	dl
		T05	26 Aug 2014	-	dl
		T07	28 Aug 2014	-	dl
		T14	4 Sep 2014	-	dl
		T21	11 Sep 2014	-	dl
		T28	18 Sep 2014	dl	dl
Dibromoacetic acid		T00	21 Aug 2014	dl	dl
		T01	22 Aug 2014	-	74 ± 8
		T05	26 Aug 2014	-	135 ± 7
		T07	28 Aug 2014	-	155 ± 21
		T14	4 Sep 2014	-	205 ± 49
		T21	11 Sep 2014	-	18 ± 18
		T28	18 Sep 2014	2 ± 1	5,050 ± 1,344
Dibromoacetonitrile	0.5	T00	21 Aug 2014	dl	dl
		T01	22 Aug 2014	-	dl
		T05	26 Aug 2014	-	dl
		T07	28 Aug 2014	-	dl
		T14	4 Sep 2014	-	dl
		T21	11 Sep 2014	-	dl
		T28	18 Sep 2014	dl	dl
Dichloroacetonitrile	0.1	T00	21 Aug 2014	dl	dl
		T01	22 Aug 2014	-	dl
		T05	26 Aug 2014	-	dl
		T07	28 Aug 2014	-	dl
		T14	4 Sep 2014	-	dl
		T21	11 Sep 2014	-	dl
		T28	18 Sep 2014	dl	dl
Tribromomethane	0.1	T00	21 Aug 2014	dl/0.14	0.59 ± 0.58
		T01	22 Aug 2014	-	1,579 ± 130
		T05	26 Aug 2014	-	2,890 ± 271
		T07	28 Aug 2014	-	3,237 ± 385
		T14	4 Sep 2014	-	3,374 ± 634
		T21	11 Sep 2014	-	3,808 ± 752
		T28	18 Sep 2014	105 ± 32	4,294 ± 127
Trichloroethylene	0.1	T00	21 Aug 2014	dl	dl
		T01	22 Aug 2014	-	dl
		T05	26 Aug 2014	-	dl
		T07	28 Aug 2014	-	dl
		T14	4 Sep 2014	-	dl
		T21	11 Sep 2014	-	dl
		T28	18 Sep 2014	dl	dl

5 Conclusions

This project had two main research questions that needed to be answered:

1. Assess the required minimum ozone dose for effective ballast water treatment by AirTree's BWMS in different water types.
2. Describe the DBP formation due to continuous in-tank dosing with ozone during a 4 week treatment period.

Three efficacy tests were performed with a pilot scale ballast water treatment system from AirTree using ozone as active substance via continuous in-tank treatment. Two of these tests focussed on assessing the required minimum ozone dose during a 7 day treatment period while the third test focussed on the DBP formation during a 28 day treatment period. The test water conditions were aimed at sufficient organisms ≥ 10 to $< 50 \mu\text{m}$ in minimum dimension for two water types and were, therefore, not augmented to reach all IMO G8 requirements. According to the IMO requirements, one freshwater, one marine water and one brackish water efficacy test was performed. However, the brackish water test should not be seen as a different water type compared to the marine water test as the salinity only differed with 3 psu and the community of species was similar (same source water). Two different ozone levels measured as ORP was chosen, 750 mV and 800 mV. Both aimed concentrations were allowed a maximum drop of 15%. Discharge samples taken after one day treatment showed an effective dose in all cases. Even though the lowest dose of 750 mV was effective in all experiment, some biological viability was measured. The dose of 800 mV showed more effective results in a shorter time period and reached 100% efficacy after 7 days for all water types tested.

DBP formation during a 4 week treatment period with continuous in-tank dosing of ozone (800 mV) showed two out of six relevant components analysed being formed in the treatment tanks. Both DBP formed are brominated, namely dibromoacetic acid and tribromomethane (bromoform). Dibromoacetic acid concentrations showed a stable formation for the first 21 days and a steep increase in the last week of the test. The concentrations of dibromoacetic acid reached the highest value after 28 days.

Most of the tribromomethane was formed in the first 5 days of dosing and a gradual increase was seen with the highest measured concentration after 28 days. Also, the control concentrations increased in time. However it is not certain what caused these relative high levels. It is possible that it is an analysis error, e.g. the column was not cleaned sufficiently between the samples. This would not affect the measurements of the treated samples.

6 Quality Assurance

IMARES utilises an ISO 9001:2008 certified quality management system (certificate number: 124296-2012-AQ-NLD-RvA). This certificate is valid until 15 December 2015. The organisation has been certified since 27 February 2001. The certification was issued by DNV Certification B.V. Furthermore, the chemical laboratory of the Fish Division has NEN-EN-ISO/IEC 17025:2005 accreditation for test laboratories with number L097. This accreditation is valid until 1th of April 2017 and was first issued on 27 March 1997. Accreditation was granted by the Council for Accreditation.

References

- EPA. Generic protocol for the verification of ballast water treatment technologies. EPA/600/R-10/146. Version 5.1, September 2010.
- IMO. International Convention for the control and Management of Ships' Ballast Water and Sediments. London. International Maritime Organization, 2004.
- MEPC. Guidelines for Approval of Ballast Water Management Systems (G8). Resolution MEPC. 174(58). Adopted on 10 October 2008.
- MEPC. Procedure for Approval of Ballast Water Management Systems that make use of active substances (G9). Resolution MEPC. 169(57). Adopted on 4 April 2008.

Justification

Report C156/14
Project Number: 430.51132.01

The scientific quality of this report has been peer reviewed by a colleague scientist and the head of the department of IMARES.

Approved: Dr. Louis Peperzak
Colleague scientist at NIOZ

Signature:



Date: November 13th 2014

Approved: F. Groenendijk
Department head

Signature:



Date: November 13th 2014

Appendices

A External Laboratory results: TOC/DOC, total nitrogen, nitrate and nitrite (Royal NIOZ)

A.1 Description of samples

In total, 18 samples were send to NIOZ with the following sample codes and descriptions. Note: the sample descriptions were not communicated with the laboratory.

Sample code	Sample description
C01-T00	C01: Control tank 1, T00: before start experiment
C01-T28	C01: Control tank 1, T28: after 28 days
C02-T00	C02: Control tank 2, T00: before start experiment
C02-T28	C02: Control tank 2, T28: after 28 days
C11-T00	C11: Treatment tank 1, T00: before treatment
C11-T01	C11: Treatment tank 1, T01: after treatment 1 day
C11-T05	C11: Treatment tank 1, T05: after treatment 5 days
C11-T07	C11: Treatment tank 1, T07: after treatment 7 days
C11-T14	C11: Treatment tank 1, T14: after treatment 14 days
C11-T21	C11: Treatment tank 1, T21: after treatment 21 days
C11-T28	C11: Treatment tank 1, T28: after treatment 28 days
C12-T00	C12: Treatment tank 2, T00: before treatment
C12-T01	C12: Treatment tank 2, T01: after treatment 1 day
C12-T05	C12: Treatment tank 2, T05: after treatment 5 days
C12-T07	C12: Treatment tank 2, T07: after treatment 7 days
C12-T14	C12: Treatment tank 2, T14: after treatment 14 days
C12-T21	C12: Treatment tank 2, T21: after treatment 21 days
C12-T28	C12: Treatment tank 2, T28: after treatment 28 days

A.2 Test reports



Koninklijk Nederlands Instituut voor Onderzoek der Zee

Klaas Kaag
IMARES
Den Helder

Landsdiep 4, 1797 SZ 't Horntje, Texel
P.O. Box 59, 1790 AB Den Burg, Texel

Telefoon 0222 369 440 /369 300
Fax 0222 319 674
E-mail jan.van.ooijen@nioz.nl
www.nioz.nl

Betreft: Monsteranalyse rapport
Uw Ref:

Datum: 29-10-2014
Onze Ref: NA271014

Geachte heer Kaag,

Hierbij stuur ik u het analyserapport van de door u aangeleverde zeewater monsters.

De monsters zijn gemeten volgens de spectrofotometrische methoden van Grashoff.

De apparatuur die gebruikt is, is een TrAAcs autoanalyser van Seal Analytical.

Voor de monsters werden gemeten, hebben wij ze gefiltreerd over een 0.2 um acrodisc filter om micro-organisme die in het water zaten te verwijderen.

De monsters werden geanalyseerd op Fosfaat (zonder extra kosten), Nitraat en Nitriet.

De accuracy van de meting is als volgt:

Voor de monsters C01T0, C01T28, C02T0, C02T28, C11T0 en C12T0

PO4 = 0.02 uM, NO3 = 0.08 uM, NO2 = 0.02 uM

Voor de overige monsters geldt een accuracy van:

PO4 = 0.02 uM, NO3 = 0.4 uM, NO2 = 0.1 uM.

ANAL PO4NO3NO2
 RUN 141027PNIMARES-AR1.RUN
 DATE 28-10-2014
 TIME 15:03
 OPER Jan

Recalculate from					
Run141027PNIMARES-A.run					
COMM	PO4	NO3+NO2	NO2	NO3	
METH					
UNIT	µmol/L	µmol/L	µmol/L	µmol/L	
C01T0	6.10	0.32	0.043	0.28	
C01T28	5.59	0.64	0.042	0.60	
C02T0	6.27	0.18	0.040	0.14	
CO2T28	5.80	0.28	0.044	0.24	
C11T0	6.37	0.34	0.058	0.28	
C11T1	6.16	74.3	0.02	74.3	
C11T5	6.50	97.3	0.02	97.2	
C11T7	6.38	104.4	0.01	104.4	
C11T14	6.76	122.1	0.01	122.1	
C11T21	6.53	124.7	0.01	124.6	
C11T28	6.64	137.9	0.01	137.8	
C12T0	6.19	0.45	0.065	0.39	
C12T1	5.82	65.6	0.02	65.6	
C12T5	6.03	92.5	0.02	92.5	
C12T7	6.30	98.5	0.02	98.4	
C12T14	6.31	119.6	0.02	119.5	
C12T21	6.36	127.6	0.01	127.6	
C12T28	6.47	133.3	0.01	133.2	

Met vriendelijke groet,

Jan van Ooijen
 Hoofd Analytisch Laboratorium
 NIOZ

NIOZ TOC-DOC / TN Analyzes
Santiago Gonzalez
Afdeling Biologische Oceanografie
23-sep-14

CRM = Consensus Reference Material
from Univ. of Miami
μM C in crm 44-46 (± 2)
μM N in crm 28-30 (± 2)

	μM C	μM N
crm	44 ± 3	27 ± 1
uitgaang DOC 21-08-14 T00	2108 ± 12	135 ± 2
uitgaang TOC 21-08-14 T00	2858 ± 16	163 ± 2
DOC C01-TO5 26-08-14	1580 ± 8	102 ± 1
TOC C01-TO5 26-08-14	2315 ± 12	148 ± 2
DOC C02-TO5 26-08-14	1939 ± 13	125 ± 1
TOC C02-TO5 26-08-14	2322 ± 15	167 ± 1
TOC C11-TO5 26-08-14	2092 ± 5	190 ± 2
TOC C12-TO5 26-08-14	2201 ± 10	203 ± 2
DOC C11-TO5 26-09-14	1944 ± 12	186 ± 2 op etiket 26-09-14 !!
DOC C12-TO5 26-09-14	2914 ± 11	180 ± 2 op etiket 26-09-14 !!
DOC C11-T28 18-8-14	2012 ± 13	246 ± 1
DOC C12-T28 18-8-14	1916 ± 8	236 ± 0
DOC C01-T28 18-9-14	1473 ± 6	120 ± 0
TOC C01-T28 18-9-14	1808 ± 9	165 ± 1
TOC C02-T28 18-9-14	1847 ± 15	162 ± 1
DOC C02-T28 18-9-14	1380 ± 14	112 ± 0
TOC C11-T28 18-9-14	2026 ± 16	255 ± 1
TOC C12-T28 18-9-14	2047 ± 10	237 ± 1
crm	47 ± 2	30 ± 4

B External Laboratory results: Disinfection by-products (Labor IBEN)

B.1 Description of samples

In total, 18 samples were send to Labor IBEN with the following sample codes and descriptions. Note: the sample descriptions were not communicated with the laboratory.

Sample code	Sample description
C01-T00	C01: Control tank 1, T00: before start experiment
C01-T28	C01: Control tank 1, T28: after 28 days
C02-T00	C02: Control tank 2, T00: before start experiment
C02-T28	C02: Control tank 2, T28: after 28 days
C11-T00	C11: Treatment tank 1, T00: before treatment
C11-T01	C11: Treatment tank 1, T01: after treatment 1 day
C11-T05	C11: Treatment tank 1, T05: after treatment 5 days
C11-T07	C11: Treatment tank 1, T07: after treatment 7 days
C11-T14	C11: Treatment tank 1, T14: after treatment 14 days
C11-T21	C11: Treatment tank 1, T21: after treatment 21 days
C11-T28	C11: Treatment tank 1, T28: after treatment 28 days
C12-T00	C12: Treatment tank 2, T00: before treatment
C12-T01	C12: Treatment tank 2, T01: after treatment 1 day
C12-T05	C12: Treatment tank 2, T05: after treatment 5 days
C12-T07	C12: Treatment tank 2, T07: after treatment 7 days
C12-T14	C12: Treatment tank 2, T14: after treatment 14 days
C12-T21	C12: Treatment tank 2, T21: after treatment 21 days
C12-T28	C12: Treatment tank 2, T28: after treatment 28 days

B.2 Description of methods

Analysed chemical substances, Levels of Quantification (LoQ), standardised test method identification numbers (Method ID), and the certified test laboratory.

Chemical substance	Molecular formula	CAS-No.	LoQ (µg/l)	Method ID	Certified test laboratory
Bromate	BrO ₃		100	LC-MS-MS	Labor IBEN
Trichlorethylene	C ₂ HCl ₃	79-01-6	0.1	EN ISO 10301 1997-04	Labor IBEN
Tribrommethane	CHBr ₃	75-25-2	0.1	EN ISO 10301 1997-04	Labor IBEN
Dibromoacetic acid	CH ₂ Br ₂ COOH	631-64-1	1.0	GC-MS after derivatisation	GBA, Hamburg
Dibromacetonitril	C ₂ HBr ₂ N	3252-43-5	0.1-0.5	EN ISO 10301 1997-04	Labor IBEN
Dichloracetonitril	C ₂ HCl ₂ N	3018-12-0			

Lightly volatile halogenated hydrocarbons and Trihalogenmethans has been analysed according to DIN EN ISO 10301 1997-04 with a gas-phase chromatograph with mass spectrometric detection and headspace sampler of Messrs. Agilent. The advantage of detection with a mass spectrometer is the definite identification and classification of the detected substance. The application of the SIM method, which accounts for the specific masses of the detected substances only, allows for very small LoQ. For these parameters the laboratory also regularly participates in interlaboratory tests. The quality assurance methods for these analyses are determined by DIN. That is besides the blank value of the instrument, the blank value is carried out for the analysis including a regular calibration by means of external

standards. For the day-to-day control a control standard is analysed and control readings shall detect methodical problems.

The laboratory IBEN GmbH analysed Acetonitrils according to the EN ISO 10301 1997-04. With regard to the sound solubility of the Acetonitrils the salt sodium sulphate was added and analysed with a gas-phase chromatograph with mass spectrometric detection and headspace sampler of Messrs. Agilent. The Acetonitrils are measured with a LoQ of 0,1 µg/l - 0,5 µg/l.

Bromate has been analysed with a liquid chromatograph with mass spectrometric detection. The difficulty in the analysis of bromate is the high salt content of the samples. With the mass spectrometric detection this may be something neglected. Unfortunately, the limit of determination must still be raised compared to less saline samples. For these parameters the laboratory also regularly participates in interlaboratory tests.

Determination of HAA was performed following the guideline EN ISO 23631:2006. The analytical range was within 0.5-10 µg/l according to the standard solutions employed, depending on the matrix of the samples, respectively. Our estimated limit of quantification was 1µg/l, which is appropriate for the analytical range stated above. Description of method and analytical constraints: 200 ml of aqueous test solution is adjusted to pH 1 and extracted after addition of 20g sodium chloride with 20 ml MTBE. Subsequently, the organic extract is removed and concentrated to 1 ml. After derivatisation of the analyte with diazomethan, concentrations were assessed by means of GC-MS. However, since the compounds under study are very polar extraction is rather difficult and will eventually be not complete. Furthermore, it will not be possible to decrease the limit of quantitation by increasing the sample volume thus leading to higher concentrations of the analyte in the organic extract. This is due to the fact the matrix will also be concentrated in this way, leading to unfavourable disturbances of the detection by GC-MS.

B.3 Quality assurance (G9: 4.2.4)

For all laboratories the Analytic Quality Assurance (AQS) is of utmost importance. It is the prime aim to deliver reliable analytic results of defined quality. Therefore one of their main targets is the quantitative assessment of all applied analysis methods, if necessary the improvement of these methods, and to ensure and to document the achieved quality throughout the analytic routine.

To meet this challenging target a quality management system (QM) is implemented. This system governs all quality related routines and provides the pre-conditions for continuous quality improvement according to DIN EN ISO 17025 and "Good Laboratory Practice" (GLP).

To comply with the high demands regarding analyses in the water pertaining enforcement the existing AQS measures have been aligned to the "Rahmenempfehlungen der Länderarbeitsgemeinschaft Wasser (LAWA) für die Qualitätssicherung bei Wasser-, Abwasser- und Schlammuntersuchungen" (basic recommendations of the working group water for the quality assurance of water, waste water and sludge analyses). On this basis the laboratory IBEN is accredited according to DIN EN ISO 17025 and holds several other concessions for legally regulated sectors. That means that in addition to internal quality assurance external controls in form of mandatory and periodic interlaboratory tests have to be carried out. This in turn guarantees the permanent control of services. Participation in interlaboratory tests is obligatory by means of the accreditation, that is that these tests have to be performed to maintain the accreditation.

B.4 Test reports

Labor IBEN GmbH, Postf. 290219, 27532 Bremerhaven

IMARES, Wageningen UR
Institute for Marine Resources and Ecosystem Studies
Department Maritime
Ambachtsweg 8a

NL-1785 AJ Den Helder / The Netherlands

Vorläufiger Prüfbericht 14082322

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C01-T00
Sample date: 21-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Probeneingang: 26.08.2014 durch: DHL
Prüfbeginn: 27.08.2014
Prüfende: 30.09.2011

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloroacetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	< 0,50	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14082322

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
Ust.-Id.-Nr.: DE 114706980
Steuer-Nr. 75/529/19720

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NL-1785 AJ Den Helder / The Netherlands

Vorläufiger Prüfbericht 14082323

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C02-T00
Sample date: 21-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Probeneingang: 26.08.2014 durch: DHL
Prüfbeginn: 27.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	0,14	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	< 0,50	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14082323

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Geschäftsführer:
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Ambachtsweg 8a

NL-1785 AJ Den Helder / The Netherlands

Vorläufiger Prüfbericht 14092247

Bremerhaven, 28.10.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C12-T28
Sample date: 18-9-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
schriftlich durch: Frau Sneekes
Probeneingang: 19.09.2014 durch: DHL
Prüfbeginn: 19.09.2014
Prüfende: 27.10.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	4204	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	6000	µg/l	GC-MS nach Derivatisierung

Bemerkung/Remark:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

The test of Dibromoacetic acid was done by another admitted laboratory.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14092247

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
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Vorläufiger Prüfbericht 14092246

Bremerhaven, 28.10.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C11-T28
Sample date: 18-9-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
schriftlich durch: Frau Sneekes
Probeneingang: 19.09.2014 durch: DHL
Prüfbeginn: 19.09.2014
Prüfende: 27.10.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	4383	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	4100	µg/l	GC-MS nach Derivatisierung

Bemerkung/Remark:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

The test of Dibromoacetic acid was done by another admitted laboratory.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14092246

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
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Vorläufiger Prüfbericht 14092245

Bremerhaven, 28.10.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C02-T28
Sample date: 18-9-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
WUR778620
Auftragsnummer:
schriftlich durch: Frau Sneekes
Probeneingang: 19.09.2014 durch: DHL
Prüfbeginn: 19.09.2014
Prüfende: 27.10.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	82,3	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	< 1,0	µg/l	GC-MS nach Derivatisierung

Bemerkung/Remark:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

The test of Dibromoacetic acid was done by another admitted laboratory.

Beurteilung:

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14092245

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
Ust.-Id.-Nr.: DE 114706980
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Department Maritime
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NL-1785 AJ Den Helder / The Netherlands

Vorläufiger Prüfbericht 14092244

Bremerhaven, 28.10.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C01-T28
Sample date: 18-9-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
schriftlich durch: Frau Sneekes
Probeneingang: 19.09.2014 durch: DHL
Prüfbeginn: 19.09.2014
Prüfende: 27.10.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	127	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	3,0	µg/l	GC-MS nach Derivatisierung

Bemerkung/Remark:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

The test of Dibromoacetic acid was done by another admitted laboratory.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14092244

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
Ust.-Id.-Nr.: DE 114706980
Steuer-Nr. 75/529/19720

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Vorläufiger Prüfbericht 14091532

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C12-T21
Sample date: 11-9-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
Ihr Auftrag vom: 25.08.2014
schriftlich durch: Frau Sneekes
Probeneingang: 12.09.2014 durch: DHL
Prüfbeginn: 12.09.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	3276	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	5,0	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14091532

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
Ust.-Id.-Nr.: DE 114706980
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Vorläufiger Prüfbericht 14091531

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C11-T21
Sample date: 11-9-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
Ihr Auftrag vom: 25.08.2014
schriftlich durch: Frau Sneekes
Probeneingang: 12.09.2014 durch: DHL
Prüfbeginn: 12.09.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	4339	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	31	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14091531

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
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Vorläufiger Prüfbericht 14090825

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C11-T14
Sample date: 4-9-2014
Verpackung: PE-Flasche / plastic bottle
Auftragsnummer: WUR778620
Ihr Auftrag vom: 25.08.2014
schriftlich durch: Frau Sneeke
Probeneingang: 29.08.2014 durch: DHL
Prüfbeginn: 29.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	3822	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	170	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14090825

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
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Vorläufiger Prüfbericht 14090823

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C12-T14
Sample date: 4-9-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
Ihr Auftrag vom: 25.08.2014
schriftlich durch: Frau Sneekes
Probeneingang: 05.09.2014 durch: DHL
Prüfbeginn: 05.09.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	2926	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	240	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14090823

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Geschäftsführer:
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Vorläufiger Prüfbericht 14082646

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C12-T07
Sample date: 28-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
Ihr Auftrag vom: 25.08.2014
schriftlich durch: Frau Sneekes
Probeneingang: 29.08.2014 durch: DHL
Prüfbeginn: 29.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	2964	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	170	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14082646

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
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Vorläufiger Prüfbericht 14082645

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C11-T07
Sample date: 28-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
Probeneingang: 29.08.2014 durch: DHL
Prüfbeginn: 29.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	3509	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	140	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14082645

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
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Vorläufiger Prüfbericht 14082644

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C12-T05
Sample date: 28-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
Probeneingang: 29.08.2014 durch: DHL
Prüfbeginn: 29.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	2698	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	130	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14082644

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
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Vorläufiger Prüfbericht 14082643

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C11-T05
Sample date: 28-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Auftragsnummer: WUR778620
Probeneingang: 29.08.2014 durch: DHL
Prüfbeginn: 29.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	3081	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	140	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14082643

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
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Vorläufiger Prüfbericht 14082327

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C11-T00
Sample date: 21-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Probeneingang: 26.08.2014 durch: DHL
Prüfbeginn: 27.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	1,00	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloroacetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	< 0,50	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
Abteilungsleiterin Umwelt

Seite 1 von 1 zum Prüfbericht Nr.: 14082327

Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

Geschäftsführer:
Dipl.-Ing. H.-J. Iben
Dr. rer. nat. E. Schuirmann
Amtsgericht Bremen Nr. 2195
Ust.-Id.-Nr.: DE 114706980
Steuer-Nr. 75/529/19720

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Vorläufiger Prüfbericht 14082326

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C12-T00
Sample date: 21-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Probeneingang: 26.08.2014 durch: DHL
Prüfbeginn: 27.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	0,18	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloroacetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	< 0,50	µg/l	GC-MS nach Derivstisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
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Seite 1 von 1 zum Prüfbericht Nr.: 14082326

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Vorläufiger Prüfbericht 14082325

Bremerhaven, 30.09.2014

Daten: Ballastwasser / Ballast Water; Sample Code: C11-T01
Sample date: 22-8-2014
Verpackung: Glasflasche / glass bottle; PE-
Flasche / plastic bottle
Probeneingang: 26.08.2014 durch: DHL
Prüfbeginn: 27.08.2014
Prüfende: 30.09.2014

Chemisch/physikalische Untersuchungen

Parameter	Befund	Einheit	Methode
Bromat / Bromate Ion	< 100	µg/l	LC-MS-MS*
Trichlorethylen / Trichloroethylene	< 0,1	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Tribrommethan / Tribromomethane	1671	µg/l	DIN EN ISO 10301 (F 4)1997-08*
Dibromoacetonitril / Dibromoacetonitrile	< 0,5	µg/l	GC-MS
Dichloracetonitril / Dichloroacetonitrile	< 0,1	µg/l	GC-MS
Dibromessigsäure / Dibromoacetic acid	79	µg/l	GC-MS nach Derivatisierung

Beurteilung:

Die Untersuchung auf Dibromessigsäure wurde untervergeben.

Dr. rer. nat. E. Schuirmann
staatl. geprüfter Lebensmittelchemiker
Geschäftsführer/Laborleiter

Susanne Graubner
Diplom Chemikerin
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Auszüge aus dem Bericht dürfen nur mit vorheriger Genehmigung vervielfältigt werden. Beurteilungen der Proben beziehen sich nur auf die durchgeführten Untersuchungen. Die Ergebnisse beziehen sich ausdrücklich auf die jeweils aufgeführte(n) Probe(n). Die akkreditierten Prüfverfahren sind mit * gekennzeichnet.

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