INTERNATIONAL STANDARD

ISO 15181-4

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Paints and varnishes — Determination of release rate of biocides from antifouling paints —

Part 4:
Determination of pyridine-triphenylborane (PTPB) concentration in the extract and calculation of the release rate

Peintures et vernis — Détermination du taux de lixiviation des biocides contenus dans les peintures antisalissures —

Partie 4: Détermination de la concentration de pyridine-triphénylborane (PTPB) dans l'extrait et calcul du taux de lixiviation

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15181-4 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

ISO 15181 consists of the following parts, under the general title *Paints and varnishes — Determination of release rate of biocides from antifouling paints*:

- *Part 1: General method for extraction of biocides*
- *Part 2: Determination of copper-ion concentration in the extract and calculation of the release rate*
- *Part 3: Calculation of the zinc ethylene-bis(dithiocarbamate) (zineb) release rate by determination of the concentration of ethylenethiourea in the extract*
- *Part 4: Determination of pyridine-triphenylborane (PTPB) concentration in the extract and calculation of the release rate*
- *Part 5: Calculation of the tolylfluanid and dichlofluanid release rate by determination of the concentration of dimethyltolylsulfamide (DMST) and dimethylphenylsulfamide (DMSA) in the extract*

The following part is under preparation:

- *Part 6: Determination of tralopyril release rate by quantification of its degradation product in the extract*

Introduction

By using standard conditions of temperature, salinity and pH at low biocide concentrations in the surrounding artificial seawater, a repeatable value of the release rate under the specified laboratory conditions can be determined using the method given in this part of ISO 15181, which can be used for quality assurance and material selection purposes. The actual release rate of biocides from antifouling paints on ships' hulls into the environment will, however, depend on many factors, such as ship operating schedules, length of service, berthing conditions, paint condition, as well as the temperature, salinity, pH, pollutants and biological community in a particular area.

The results of this test do not reflect environmental biocide release rates for antifouling products and are not suitable for direct use in the process of generating environmental-risk assessments, producing environmental-loading estimates or for establishing release rate limits for regulatory purposes. In comparison with copper and organotin release rates obtained either by direct or indirect measurements of the copper and organotin release rate from ships' hulls and from measurements made on panels exposed in harbours, all available data indicate that the results of this generic test method significantly overestimate the release rate of biocides under in-service conditions. Published results demonstrate that the results of this test method are generally higher than direct *in situ* measurements of copper and organotin release rates from in-service ship hulls by a factor of about 10 or more for several commercial antifouling coatings [1, 2]. A similar relationship is expected to be found for other biocides. Realistic estimates of the biocide release rate from a ship's hull under in-service conditions can only be obtained from this test method if this difference is taken into account.

Where the results of this test method are used in the process of generating environmental-risk assessments, producing environmental-loading estimates or for regulatory purposes, it is most strongly recommended that the relationship between laboratory release rates and actual environmental inputs be taken into account to allow a more accurate estimate of the biocide release rate from antifouling coatings under real-life conditions to be obtained. This can be accomplished through the application of appropriate correction factors [2].

Paints and varnishes — Determination of release rate of biocides from antifouling paints —

Part 4:

Determination of pyridine-triphenylborane (PTPB) concentration in the extract and calculation of the release rate

1 Scope

This part of ISO 15181 specifies the apparatus and analytical method for determining the amount of pyridine-triphenylborane (PTPB) that has been released from an antifouling paint into artificial seawater in accordance with the procedure given in ISO 15181-1. It determines the PTPB concentration in the artificial seawater extract and gives the final calculation for the release rate of PTPB under the specified laboratory conditions.

This part of ISO 15181 is designed to allow the concurrent determination of PTPB and other biocides that can be released by a given antifouling paint (for example zineb) through the analysis of separate sub-samples of an artificial seawater extract generated in accordance with the procedure given in ISO 15181-1.

When used in conjunction with ISO 15181-1, the practical limits on the quantitative measurement of release rates by this method are from 1,8 $\mu g \cdot cm^{-2} \cdot d^{-1}$ to 500 $\mu g \cdot cm^{-2} \cdot d^{-1}$. The quantitative measurement of release rates below this range will require the concentration step described in the last paragraph in 8.2 or the use of an analytical method with a limit of quantitation for PTPB in artificial seawater which is lower than the limit specified in Clause 3 and in 5.1.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 15181-1:2007, *Paints and varnishes — Determination of release rate of biocides from antifouling paints — Part 1: General method for extraction of biocides*

ASTM D 6442-06, *Standard Test Method for Determination of Copper Release Rate from Antifouling Coatings in Substitute Ocean Water*

3 Principle

The quantity of PTPB biocide released into artificial seawater by the method given in ISO 15181-1 is determined by the use of a high-performance liquid chromatograph (HPLC) with UV detection at 220 nm after pre-concentration by solid-phase extraction (SPE), or by an alternative analytical method provided that it demonstrates a limit of quantitation for PTPB in artificial seawater of 10 µg/l or less. The release rate of the biocide under the specified laboratory conditions is then calculated as PTPB.

The use of the HPLC method in conjunction with the additional sample concentration step described in the last paragraph in 8.2 can allow a limit of quantitation for PTPB in artificial seawater of 0,5 µg/l to be achieved.

NOTE Additional information on PTPB is given in Annex B. Additional background information on the analytical method has been published by Takahashi, *et al*. [3].

4 Supplementary information

The items of supplementary information required to be able to use the general extraction procedure, described in ISO 15181-1, for PTPB are given in Annex A.

5 Apparatus

5.1 High-performance liquid chromatograph (HPLC), or **other equivalent instrument**, which has a limit of quantitation for PTPB in artificial seawater of 10 µg/l or less. The limit of quantitation shall be determined by the procedure given in Annex 2 (Determination of the LOQ for Copper in Substitute Ocean Water for the Analytical Method) of ASTM D 6442-06, suitably modified for PTPB. If HPLC is used, the system shall, where possible, include the components specified in 5.1.1 to 5.1.6.

5.1.1 Isocratic pump, capable of achieving a pressure of 150 bar and a flow-rate of 1,0 ml/min.

5.1.2 Ultraviolet detector, capable of monitoring at 220 nm.

5.1.3 Autosampler, capable of making 10 µl injections.

5.1.4 Chromatography column: A reverse-phase column with an internal diameter of 4,6 mm and a length of 250 mm, packed with a microparticulate octadecylsilane (C-18, end-capped) stationary phase (mean particle size 5,0 µm) or equivalent.

5.1.5 Column oven, capable of maintaining a constant column temperature of 30 °C.

5.1.6 Electronic data-processing system, capable of controlling the HPLC system, acquiring data and automatically integrating peak areas.

5.2 Pipettes, with disposable tips.

5.3 Volumetric flasks, made of glass.

5.4 Solid-phase extraction (SPE) cartridges, packed with 360 mg of C-18 sorbent, and sequentially conditioned prior to use with 10 ml of a solvent blend of acetonitrile and pyridine in a ratio of 99:1 parts by volume, then 10 ml of water, and finally 10 ml of artificial seawater.

5.5 Solid-phase extraction system, or equivalent system suitable for use with the extraction cartridges.

5.6 Rotary vacuum evaporator, with heating bath.

6 Reagents and materials

Suppliers' material safety data sheets should be consulted for details of any hazards associated with the reagents listed below, and the risks associated with their use should be assessed. Appropriate protective clothing and equipment should be utilized.

Unless otherwise specified, use only reagents of recognized analytical grade.

6.1 Cleaning reagents.

Use one of the following reagents for cleaning all the equipment:

6.1.1 Hydrochloric acid, concentrated aqueous solution, 37 % by mass.

6.1.2 Hydrochloric acid, aqueous solution, 10 % by volume.

6.2 Acetonitrile, HPLC grade.

6.3 Pyridine.

6.4 Water, conforming to the requirements of grade 2 of ISO 3696.

6.5 Artificial seawater, as defined in ISO 15181-1.

6.6 Pyridine-triphenylborane (PTPB), analytical grade, with a certified mass fraction of PTPB of at least 99,0 %.

6.7 Tetra-*n*-butylammonium phosphate (TBAP), ion-pair reagent, aqueous solution, 0,5 mol/l.

NOTE The source of the reagent can have an effect on the result of the test. Material supplied by Tokyo Chemical Industry Co., Ltd. (TCI) is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 15181 and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

7 Test samples

Use extracts taken from the release rate measuring containers as described in ISO 15181-1.

8 Procedure

8.1 General

Carry out all determinations on the extract in triplicate.

Clean all non-disposable or re-used apparatus by immersion in concentrated hydrochloric acid (6.1.1) for at least 30 min, or dilute hydrochloric acid (6.1.2) for at least 6 h, to remove all traces of biocide. Rinse thoroughly with grade 2 water (6.4).

Operate the chromatograh or other suitable instrument in accordance with the manufacturer's instructions.

Ensure that all treated test samples and calibration standards are at equilibrium at room temperature prior to analysis.

8.2 Sample treatment

Add pyridine to the test sample to give a concentration 1 % by volume. Pass 100 ml of this solution through a pre-conditioned C-18 solid-phase extraction cartridge at a flow rate of about 7 ml/min to 10 ml/min using the solid-phase extraction system. Wash the cartridge with 10 ml of a solvent blend comprising acetonitrile, pyridine and water in a ratio of 39,6:0,4:60 parts by volume, then discard the eluate. Elute the PTPB with 15 ml of a solvent blend comprising acetonitrile and pyridine in a ratio of 99:1 parts by volume, and collect the eluate. Make up the eluate to a final volume of 20 ml with a mixture comprising acetonitrile and pyridine in a ratio of 99:1 parts by volume.

This solution may be stored at a temperature of – 5 °C for up to 7 days, or at – 20 °C for up to 3 months, before analysis.

Where the concentration of PTPB in the final solution is below the limit of quantitation for the analysis, the solution may be further concentrated by evaporation to near-dryness on a rotary evaporator at 40 °C, drying fully under a stream of nitrogen gas, and then re-dissolving the residue in a solvent blend comprising acetonitrile and pyridine in a ratio of 99:1 parts by volume to make up to an appropriate final volume for HPLC analysis.

8.3 Preparation of PTPB stock solution

Prepare a solution of PTPB at a concentration of 1 mg/l in a solvent blend comprising acetonitrile and pyridine in a ratio of 99:1 parts by volume for the preparation of calibration standards (see 8.4).

NOTE It is advisable to prepare this solution by serial dilution of a solution of PTPB at a concentration of 1,0 g/l in the same solvent blend.

8.4 Preparation of calibration standards

Dilute the PTPB stock solution with a solvent blend comprising acetonitrile and pyridine in a ratio of 99:1 parts by volume to prepare at least five calibration standard solutions with different concentrations of PTPB, covering the range from 50 µg/l to 1 000 µg/l of PTPB.

Calculate the actual concentrations of PTPB in each calibration standard from the certified purity of the PTPB and the subsequent dilutions.

8.5 Preparation of chromatography eluent

Prepare a mixture comprising acetonitrile, water and the TBAP ion-pair reagent solution in a ratio of 65:34:1 parts by volume for use as the chromatography eluent.

8.6 Instrument calibration

At the beginning of each instrument run, determine the PTPB concentration in an artificial seawater blank and in the PTPB calibration standards using the HPLC system. Generate a calibration curve by plotting peak area as ordinate against PTPB concentration as abscissa; perform linear regression analysis and calculate the slope, intercept and correlation coefficient. If the correlation coefficient for the linear regression analysis is < 0,999, then prepare fresh calibration standards and re-calibrate.

8.7 Sample determination

Using the HPLC system or other suitable instrument (see 5.1), determine the concentration of PTPB in the treated test sample for each test cylinder and the uncoated reference blank (see 8.1 and 8.7 of ISO 15181-1:2007). For each set of triplicate analyses, if any result differs by more than 10 % from the mean, discard that result and re-analyse another sample of the extract.

The retention time for PTPB using the chromatographic equipment and conditions specified in Clause 5 is generally about 6,0 min to 7,0 min. Figure 1 shows a representative chromatogram obtained using an Inertsil®

ODS-3 column (GL Sciences). The specified equipment and conditions are typical starting points for the analysis, and the composition of the mobile phase, the flow-rate of the mobile phase, the injector volume, the column dimensions and the stationary phase may be varied, if necessary, to improve chromatographic resolution.

NOTE The exact retention time depends on the HPLC system and the chromatographic conditions. The retention time can fall outside this range without affecting the quality of the results.

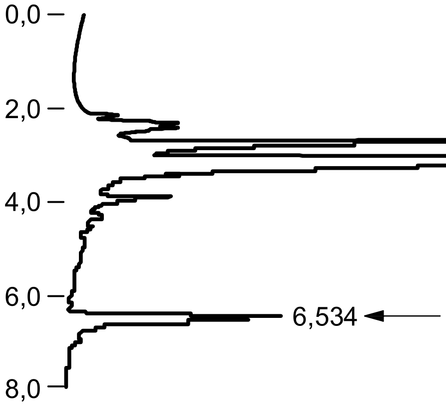


Figure 1 — Representative chromatogram for PTPB analysis by HPLC

9 Calculation and expression of results

9.1 Calculation of the PTPB concentration

Calculate the concentration of the PTPB released into the artificial seawater, c_{PTPB}, in µg/l, in each individual measuring container on each test day using the equation

$$c_{\text{PTPB}} = \frac{c_{\text{V}} \cdot E}{S}$$

where

c_{V} is the concentration of PTPB in the treated test sample derived from the calibration curve, in µg/l;

E is the final volume of the treated test sample, in ml (= 20; see 8.2);

S is the extracted volume of treated test sample, in ml (= 100; see 8.2).

9.2 Release rate for each test cylinder

Calculate the release rate of PTPB, R, in µg·cm^{-2}·d^{-1}, for each test cylinder on each test day using the equation

$$R = \frac{c_{\text{PTPB}} \times 1{,}5 \times 24}{t \times A}$$

where

c_{PTPB} is the concentration of the PTPB released into the measuring container, in µg/l (see 9.1);

24 is the number of hours in a day;

1,5 is the volume, in litres, of seawater in the measuring container, as specified in ISO 15181-1:2007, 10.1;

t is the length of time, in hours, the test cylinder was immersed and rotated in the measuring container (see Annex A, Table A.1, item 5);

A is the surface area, in cm^2, of the paint film (= 200; see Annex A, Table A.1, item 6).

This equation can be simplified if the standard volume, time and size, as given above, are used:

$$R = \frac{C_{\text{PTPB}} \times 0{,}18}{t}$$

9.3 Mean release rate for each set of three cylinders

Calculate the mean PTPB release rate, in $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$, for each set of three test cylinders on each test day (see 9.2).

9.4 Cumulative PTPB release

Calculate the 14-day cumulative release of PTPB, $R_{0,14}$, in $\mu\text{g}\cdot\text{cm}^{-2}$, using the equation

$$R_{0,14} = \sum \overline{R}_{i,j}(j-i) = \sum \frac{(R_i + R_j)}{2}(j-i)$$

where

$\overline{R}_{i,j}$ is the mean PTPB release rate, in $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$, between consecutive test days i and j for all test days up to day 14;

i and j are the times, in days, from the start of the trial for each pair of consecutive test days, specifically 0 and 1 days, 1 and 3 days, 3 and 7 days, etc. (see ISO15181-1:2007, 9.6 and 9.7);

R_i and R_j are the mean PTPB release rates for each set of three test cylinders, in $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$ (see 9.3), for each pair of consecutive test days from the start of the trial up to day 14, specifically days 0 and 1, days 1 and 3, days 3 and 7, etc., and where the release rate on day 0 (R_0) is taken as 0 $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$.

NOTE The cumulative release of PTPB for other periods of time may be calculated, if specified, using the equation

$$R_{x,y} = \sum \overline{R}_{i,j}(j-i) = \sum \frac{(R_i + R_j)}{2}(j-i)$$

where

$R_{x,y}$ is the cumulative release of PTPB, in $\mu\text{g}\cdot\text{cm}^{-2}$, from day x to day y;

$\overline{R}_{i,j}$ is the mean PTPB release rate, in $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$, between consecutive test days i and j for all test days from day x to day y;

i and j are the times, in days, from the start of the trial for each pair of consecutive test days, for example 0 and 1 days, 1 and 3 days, 3 and 7 days, etc., and, where day 0 is included, the release rate on day 0 (R_0) is taken as 0 $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$;

R_i and R_j are the mean PTPB release rates for each set of three test cylinders, in $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$ (see 9.3), for each pair of consecutive test days from day x to day y, for example days 0 and 1, days 1 and 3, days 3 and 7, etc., and, where day 0 is included, the release rate on day 0 (R_0) is taken as 0 $\mu\text{g}\cdot\text{cm}^{-2}\cdot\text{d}^{-1}$.

9.5 Mean PTPB release rate

Calculate the weighted mean PTPB release rate from day 21 to the final test day, $\overline{R}_{\text{21,end}}$, in µg·cm^{-2}·d^{-1}, using the equation

$$\overline{R}_{\text{21,end}} = \frac{\sum \overline{R}_{i,j}(j-i)}{\sum (j-i)} = \frac{\sum \frac{(R_i + R_j)}{2}(j-i)}{\sum (j-i)}$$

where

$\overline{R}_{i,j}$	is the mean PTPB release rate, in µg·cm^{-2}·d^{-1}, between consecutive test days i and j for all test days from day 21 to the final test day;
i and j	are the times, in days, which have elapsed since the start of the trial for each pair of consecutive test days, specifically 21 and 24 days, 24 and 28 days, 28 and 31 days, etc. (see ISO15181-1:2007, 9.6 and 9.7);
R_i and R_j	are the mean PTPB release rates for each set of three test cylinders, in µg·cm^{-2}·d^{-1} (see 9.3), at each pair of consecutive test days from day 21 through to the final test day, specifically days 21 and 24, days 24 and 28, days 28 and 31, etc.

NOTE 1 This equation calculates the weighted mean release rate, taking into account any differences in time between test days, and is a more valid treatment of the data than calculation of the simple arithmetic mean. The calculation can be conveniently done using a suitable computer-generated spreadsheet.

NOTE 2 The mean PTPB release rate over other periods of time can be calculated, if specified, by modifying this equation to account for the different test days used.

9.6 Pseudo-steady-state mean PTPB release rate

If the coating exhibits a pseudo-steady state, calculate the pseudo-steady-state PTPB release rate, $\overline{R}_{\text{PSS}}$, in µg·cm^{-2}·d^{-1}, using the equation

$$\overline{R}_{\text{PSS}} = \frac{\sum \overline{R}_{i,j}(j-i)}{\sum (j-i)} = \frac{\sum \frac{(R_i + R_j)}{2}(j-i)}{\sum (j-i)}$$

where $\overline{R}_{i,j}$, j, i, R_i and R_j are as defined in the Note to 9.4.

For the purposes of this part of ISO 15181, a “pseudo-steady state” is defined as being a period of at least 24 days and including four or more test days, where the mean PTPB release rate for the set of three test cylinders on each test day (see 9.3) differs from the weighted mean release rate over the calculation period by no more than 15 %, and the final day of the pseudo-steady state is the final day of the trial.

NOTE Not all coatings will exhibit a pseudo-steady state. Where a coating does exhibit a pseudo-steady state, the determined pseudo-steady-state biocide release rate should not be assumed to necessarily reflect a true steady-state release rate under the conditions of the test as the release rate of the coating can continue to change beyond the test period.

10 Validation of the method

A validation study of the HPLC analytical method for determining PTPB in seawater gave the following results:

a) The limit of quantitation (LOQ) for PTPB in artificial seawater using the HPLC method was assessed at 10 µg/litre, with a mean recovery of 94 % and a precision (standard deviation) of 8,5 % when checked against the average instrument response for seawater solutions with a known PTPB content.

b) The mean recovery rates of PTPB at 25 µg/litre and 50 µg/litre were 96 % and 97 %, respectively [3]. At 500 µg/litre (50 × LOQ), the mean recovery rate was 95 % with a precision (standard deviation) of 2,9 %.

c) The limit of quantitation for PTPB in artificial seawater was assessed at 0,5 µg/litre where the additional sample concentration step described in the last paragraph of 8.2 was followed.

The repeatability and reproducibility of the method have yet to be determined.

11 Test report

The test report shall contain at least the following information:

a) all information necessary for identification of the sample tested;

b) a reference to this part of ISO 15181 (ISO 15181-4:2008);

c) all details necessary to describe the method used, including:

- the type of analytical equipment used, the manufacturer of the equipment and the method of analysis employed,
- all details required by ISO 15181-1;

d) the results of the test, including the results of the individual determinations and their mean, calculated as specified in Clause 9, including:

- the limit of quantitation for PTPB in artificial seawater by the analytical method, determined by the laboratory performing the test method (see 5.1),
- the concentration of PTPB in the artificial seawater, in µg/l, for each test cylinder on each test day (see 9.1),
- the rate of PTPB release into the artificial seawater, in $\mu g \cdot cm^{-2} \cdot d^{-1}$, for each test cylinder on each test day (see 9.2) and the mean rate of PTPB release, in $\mu g \cdot cm^{-2} \cdot d^{-1}$, for each set of three test cylinders on each test day (see 9.3),
- a graph showing the rate of PTPB release as a function of time,
- the 14-day cumulative PTPB release and the cumulative PTPB release for other periods, if specified (see 9.4),
- the mean release rate from day 21 to the end of the trial, and the mean release rate for other periods, if specified (see 9.5),
- the pseudo-steady-state release rate, if calculated (see 9.6),
- the length of time the cylinders were rotated to extract the biocide on each test day (see Annex A, Table A.1, item 5);

e) any deviations from the test procedure specified;

f) any unusual features (anomalies) observed during the test;

g) the name of the test laboratory;

h) the dates of the test.

Annex A
(normative)

Supplementary information

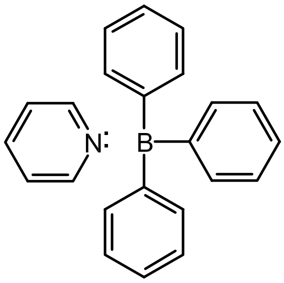
The items of supplementary information listed in Table A.1 shall be used when extracting the PTPB from the antifouling paint by the method given in ISO 15181-1.

Table A.1 — Information concerning the biocide extraction procedure

1	Test cylinder	The test cylinder shall be made of polycarbonate or glass.
2	Release rate measuring container	The release rate measuring container and baffles shall be made of polycarbonate or glass.
3	Holding tank filter type	An activated-charcoal filter, optionally combined with a styrene-supported iminodiacetic acid chelating ion-exchange resin with a typical particle-size range of about 0,300 mm to 0,850 mm, which is capable of removing transition metals from seawater. NOTE Combining an activated-charcoal filter with an ion-exchange resin can reduce the overall size of the required filter unit.
4	Maximum holding tank biocide limit	The maximum holding tank biocide limit shall be 100 µg/l (expressed as PTPB).
5	Length of time the cylinders are rotated to extract the biocide	The rotation period for the initial measurement shall be 1,0 h except that, if the biocide concentration, expressed as PTPB, in a particular release rate measuring container exceeds 200 µg/l, then the rotation period for the next measurement shall be reduced. The amount by which the rotation period is reduced shall be selected based on familiarity with the coating being evaluated and experience with the test method, and shall take into account the extent by which the measurement exceeds 200 µg/l. If the next measurement also exceeds 200 µg/l, then the rotation period shall be further reduced for the subsequent measurement. Once the rotation period has been reduced sufficiently for the biocide concentration, expressed as PTPB, to no longer exceed 200 µg/l, then the rotation period shall be incrementally increased to 1 h at the earliest possible opportunity without the concentration of biocide, expressed as PTPB, exceeding 200 µg/l. If the biocide concentration, expressed as PTPB, is greater than 200 µg/l for any measurement, record this in the test report.
6	Area of cylinder coated	The area of the cylinder surface coated shall be 200 cm^2. NOTE Alternative sample areas may be used when 200 cm^2 is not appropriate, for example where a coating is expected to show a high release rate, a sample area of 100 cm^2 may be used to avoid exceeding biocide concentrations of 200 µg/l in the measuring container.

Annex B
(informative)

Additional information on PTPB

Chemical name (IUPAC)	Pyridine-triphenylborane	
CAS Registry Number	971-66-4	
Structural formula		
Molecular formula	$C_{23}H_{20}BN$	
Molecular mass	321,2	
Melting point	210 °C (decomposition)	
Vapour pressure	< 133 Pa	
Solubility (20 °C)	Water (deionized)	0,08 mg/l
	Acetonitrile	1,0 g/l
	Acetonitrile/pyridine, 99:1 by volume	1,0 g/l
	Methanol	0,22 g/l
	Ethyl acetate	3,1 g/l
	n-hexane	< 0,1 g/l
Stability of PTPB in artificial seawater containing copper ions [3]	PTPB is unstable in artificial seawater containing copper ions. It has been found that, when 10 µg/l and 50 µg/l of PTPB were present with 10 mg/l to 50 mg/l of copper ions in artificial seawater, the PTPB concentrations decreased by 50 % to 70 % within 2 h.	

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