INTERNATIONAL MARITIME ORGANIZATION

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4 ALBERT EMBANKMENT
LONDON SE1 7SR
Telephone: +44 (0)20 7735 7611 Fax: +44 (0)20 7587 3210

MSC.1/Circ.1600
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INTERIM GUIDANCE FOR CONDUCTING THE REFINED MHB (CR) CORROSIVITY TEST

1 The Sub-Committee on Carriage of Cargoes and Containers (CCC), at its fifth session (10 to 14 September 2018), considered submissions from Australia, Brazil, Canada, United States, NACE International and IIMA relating to the assessment of the Materials Hazardous only in Bulk (MHB) category "Corrosive Solids", for which section 9.2.3.7.3 of the IMSBC Code states that the UN C.1 test[1] is an acceptable test.[2] The UN C.1 test, however, was developed for liquids and solids that may become liquid during transportation and had never been validated for solids. Industry experience was that the test resulted in anomalous and highly variable results. A Global Industry Alliance (GIA) was formed under the auspices of the International Council on Mining and Metals (ICMM) in order to improve the assessment of solid bulk cargoes for the corrosive hazard.

2 After an international research programme across a range of commodities, the GIA concluded that by defining and standardizing important parameters, potential sources of variation can be removed and the degree of variability in test outcomes can be reduced to acceptable levels. On the basis of this work, Australia, Brazil, Canada, United States, NACE International and IIMA proposed:

.1 minor changes to section 9 of the IMSBC Code, specifically paragraph 9.2.3.7.3, to enhance the test method's applicability to the carriage of solid bulk cargoes; and

.2 issuance of guidance that extends and details the MHB test method for determining the corrosivity of solid bulk cargoes. The guidance should be utilized by test laboratories conducting the test in conjunction with the MHB (CR) corrosivity test description (section 9 of the IMSBC Code).

3 The Sub-Committee agreed, in principle, that the modifications to section 9.2.3.7.3 of the IMSBC Code proposed by the submitters should be recommended for adoption in amendment 06-21 to the IMSBC Code and that the proposed Interim guidance for implementation of the test protocol should be approved by the Organization.

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1 United Nations Manual of Tests and Criteria, part III, section 37.

2 The test sample shall contain at least 10% moisture by mass with water to be added to the sample in the case that the representative sample of the cargo to be shipped does not contain more than 10% moisture by mass.

4 Notwithstanding the requirements that the sample contains at least 10% moisture by mass and that aluminium test coupons are used in the test specified in section 9.2.3.7.3 of the IMSBC Code, the Maritime Safety Committee, at its 100th session (3 to 7 December 2018), approved interim guidance for conducting the refined MHB (CR) corrosivity test.

5 Member States are invited to bring the above information and the attached Interim guidance to the attention of all stakeholders, requesting that appropriate action be taken when carrying out the MHB (CR) corrosivity test, considering that the mandatory entry into force date of the mandatory amendment 06-21 to the IMSBC Code would be 1 January 2023.

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ANNEX

INTERIM GUIDANCE FOR CONDUCTING THE REFINED MHB (CR) CORROSIVITY TEST

Background

1 Based on the detailed test protocol used by the GIA for undertaking the refined MHB (CR) test and the points outlined in sections 3.1 to 3.3 of the report of the GIA (CCC 5/INF.18), general guidance for conducting the refined MHB (CR) test on any solid bulk cargo was developed. The aim was to ensure a clear understanding of the parameters that influence the test results and to provide advice on how to conduct the experimental procedure, given the range of physical and chemical properties of solid bulk cargoes.

Introduction

2 This Guidance extends and details the MHB (CR) test method for determining corrosivity of solid bulk cargo materials. The test should be conducted on representative samples of the cargo at conditions representing their as-shipped properties. It should be utilized by test laboratories conducting the test in conjunction with the MHB (CR) test description (section 9 of the IMSBC Code) and the UN C.1 test procedure (UN Manual of Tests and Criteria, section 37.4). The method is an accelerated corrosion test run at 55°C that assesses the rate of corrosion of steel test coupons exposed to a solid bulk cargo material. The test duration should be a minimum of seven days, which may be extended to 14, 21 and 28 days, in accordance with the existing UN C.1 test protocol. Each test should be conducted to ensure acceptable repeatability.

Steel coupon details

3 This section specifies the steel coupons and their preparation:

.1 Steel coupon type

At least three sets of specimens of steel coupons with nominal dimensions of 20 mm x 50 mm x 2 mm should be used in each test. The type of steel should be as specified in paragraph 9.2.3.7.3 of the IMSBC Code. Aluminium coupons should not be tested, as solid bulk cargoes are not shipped in ships with holds constructed of aluminium.

.2 Grinding and cleaning

The steel coupons should be progressively wet ground on both sides and on all four edges, to a minimum finish of 120 grit. The coupons should then undergo ultrasonic cleaning in deionised water, rinsed with high purity ethanol or acetone and immediately dried with high purity gas (e.g. nitrogen). No chemical surface preparation (pickling, etching, etc.) should be performed to prevent surface "irritations" (inhibition, passivation) and no cleaning performed with detergent, alkali or other proprietary means.

.3 Dimension measurements and weighing procedures

Coupons should be identified with a suitable marking system in accordance with relevant international standards and best practices. Any stamped or etched regions should be avoided during the assessment of localized corrosion. The dimensions of each coupon, i.e. length, width and thickness, and its weight should be recorded. The dimensions should be measured to 0.1 mm. The weight should be measured to 0.0002 g.

.4 Steel coupon storage

After recording the dimensions and weight of each coupon, the coupons should be rinsed with high purity ethanol or acetone and dried with high purity gas. If the coupons are not immediately used in a test, they should be appropriately stored prior to testing (e.g. stored in a desiccator to lower relative humidity and to avoid adventitious contamination), ensuring that coupons are kept isolated from each other. After storage and prior to testing, the coupons should again be rinsed with high purity ethanol or acetone and dried with high purity gas. Should any sign of corrosion be visible at this point, e.g. stains at the surface, the coupon should be wet ground again, using a minimum of 120 grit paper followed by the ultrasonic cleaning, rinsing, drying and measurement (size and weight) procedures described in paragraphs 3.2 and 3.3 above.

Test set-up guidance

4 The test set-up section outlines the details of how to prepare a representative sample and load it into the test cell with coupons:

.1 Sample

A representative sample of the cargo should be sourced and subsampled for testing. The representative sample should be tested at typical as-shipped cargo carriage conditions including moisture content, bulk density, particle size distribution and atmospheric conditions. The shipper should provide the typical as-shipped cargo conditions or inform the laboratory when data is not available. Where pH is known or expected to affect the testing outcome for a particular cargo material, the pH of the sample should be measured before and after testing in accordance with relevant international standards, and the pH should be reported by the laboratory. It should be ascertained that the pH during testing is representative of the material's pH in the as-shipped condition.

.2 Particle Size Distribution (PSD)

For cargoes which only contain fine particles, the material should be tested as such. For cargoes that contain a mixture of large and fine particles, where the maximum top size is greater than 25 mm, some modification of the particle size distribution may be needed in order to fit the sample into the test vessel. If the amount of +25 mm particles is small (~10%), this material can be screened at 25 mm and the finer fraction used as the representative sample for testing. If the amount of +25 mm is greater than 10%, sample reconstitution methods should be employed. Methods for both coal and bauxite are given in appendix 2 to the IMSBC Code. These should be used as guides for other cargoes that would have similar particle size distributions and therefore would need sample reconstitution approaches to be applied. For cargoes that are solely made up of coarse particles (e.g. lumpy iron ore, pig iron, hot briquetted iron, etc.) the representative sample should be crushed to achieve enough material that is -25 mm and +6.3 mm for testing. If the particle size distribution of a sample causes practical problems while executing the test, the laboratory should consult the shipper regarding its properties and the best method to modify the particle size distribution, so that it may be adequately tested.

.3 Moisture

The sample should be tested at the as-shipped moisture content provided by the shipper. Based on practical experience, the shipper should consider the possibility of moisture addition that may occur during loading and shipping, e.g. due to adverse weather conditions. The moisture content of the sample should be checked after completing any particle size distribution modifications (as outlined in paragraph 3.2). The moisture content of the sample should be measured just prior to testing and this should be reported.

.4 Bulk density

The sample, after any particle size modification has been made, should be placed into the test cell such that at least 1.5 L of material is used. For coarse or lumpy materials, the bulk density requires specific consideration. For such materials, a mark at a known volume is to be placed on the cell and the weight of sample that corresponds to its bulk density multiplied by that volume is to be added to the cell such that it fills the cell to the mark. If the shipper has no information on the as-shipped bulk density of the cargo, the laboratory should determine the bulk density in accordance with applicable ISO standards.

.5 Coupon placement

All coupons used in the test are to be orientated in the vertical position. One coupon should be fully buried in the test sample and be placed in the sample during the loading of the sample. One coupon should be half-buried in the top layer of the sample and be placed in the sample during the final stages of loading the sample. The final coupon should be suspended in the gas phase. Placement of the buried coupon should be such that its lower edge does not touch the bottom of the test vessel. The minimum distance between the upper edge of the fully buried coupon and the top of the test sample should be 10 mm. For some coarser samples, the fully buried coupon may need to be placed deeper to the sample to ensure it is held in place, vertically by sufficient particles.

.6 Atmosphere

The test vessel with steel coupons and sample should be closed with a fitted glass lid equipped with a reflux condenser. All remaining openings in the lid are to be closed using ground glass stoppers. The top of the condenser is to be set to represent the atmospheric carriage conditions of the cargo being tested. The lid is to be effectively sealed in order to ensure that no loss of moisture occurs during the test. Information on the as-shipped cargo space atmosphere should be provided to the laboratory by the shipper. For cargoes that react with oxygen and/or are shipped with an inert gas blanket, the test should be conducted with inert gas content and the top of the condenser closed. For cargoes where atmospheric conditions during shipping demonstrate strong oxygen depletion, the top of the condenser may be closed to better reflect the cargo carriage conditions. In all other cases and for all other cargoes, the test should be conducted with an open condenser to allow for air exchange.

Conducting of the test

5 The test temperature (i.e. the temperature of the sample in the test vessel) should be 55°C (55°C ±1°C). The test vessel should be heated using an appropriate, controllable method and the temperature of the test material monitored, checked and recorded regularly throughout the test. Heating should be achieved evenly through the sample material and the test temperature should, where practicable, be reached within one hour. The laboratory should document the details of the heating procedure.

Post-test guidance

6 At the completion of the test, the vessel should be allowed to cool to room temperature. The steel coupons should be carefully recovered and any excess sample material adhering to the coupons removed by hand, followed by inhibited pickling using a suitable procedure for corroded steel coupons in accordance with relevant international standards (e.g. ASTM G1-03). If necessary, inhibited pickling should be repeated, until coupon mass becomes constant at a low rate versus the blank coupon mass loss value, as per guidance in relevant international standards. Both coupon surfaces are to be photographed before and after inhibited pickling. After final cleaning with alcohol and acetone in an ultrasound bath and once dry, the test coupons should be weighed. Disposable plastic gloves should be worn at all times when handling the test coupons. The coupon weight should be used to determine the mass loss in grams and as a % of the original mass. Any inhibited pickling weight loss from blank coupons should be subtracted. The general corrosion rate should be calculated from the mass loss, the coupon dimensions and the exposure time.

Qualification of localized corrosion

7 After removal of corrosion products, coupons are to be analysed on both sides to identify the occurrence of localised corrosion. Localized corrosion identification and qualification is to be conducted using national and/or international corrosion standards (such as NACE/ASTM G193-12d).

Quantification of localized corrosion

8 When localized corrosion occurs in tandem with, or instead of, uniform corrosion attack of the surface, the depth of the deepest intrusion should be determined. The maximum measured loss of thickness (from the general corrosion rate measured by weight loss) is to be added to the depth of the deepest intrusion to determine the intrusion depth. The deepest intrusion is to be determined metallographically or using surface profilometry or other suitable methods, as outlined in relevant nationally or internationally accepted standards, such as ISO 11463:1995 and ASTM G46.