INTERNATIONAL MARITIME ORGANIZATION

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PREPARATION OF DRAFT AMENDMENT 04-17 TO THE IMSBC CODE

New proposals of amendments to the Code, new individual schedules or amendments to existing ones

Update on research to define an appropriate test protocol for assessing the corrosivity of complex solids under the IMSBC Code

Submitted by the International Iron Metallics Association (IIMA)

SUMMARY

Executive summary:	This document provides an update on research underway to improve corrosivity test protocols for complex solid bulk cargoes
Strategic direction:	5.2
High-level action:	5.2.3
Output:	5.2.3.3
Action to be taken:	Paragraph 18
Related documents:	DSC 16/4/13 and CCC 2/5/31

Background

1 The amendment 02-13 of the IMSBC Code introduces criteria for determining Materials that are Hazardous only in Bulk (MHB). One of the MHB categories considered is "corrosive solids" – and it is noted that the UN Test C.1 is an acceptable test.

2 The test, however, was developed for liquids and solids that may become liquid during transportation and has never been validated for solids. The document DSC 16/4/13 concluded that with the presence of at least 10% moisture, the test could be used to assess MHB for solid cargoes.

3 At CCC 2, IIMA raised concerns that for complex solids such as mineral/metal concentrates, initial testing using this method resulted in anomalous and highly variable results. While delegates acknowledged the challenge, it was agreed that the test should be used until an alternative is defined.

4 The international mining and metals community has launched a research programme, coordinated by the International Council on Mining and Metals (ICMM) to create an improved test protocol for these cargoes. This document outlines work to date and the research that will be undertaken in 2016.

Initial testing: observations and concerns

5 The modified C.1 test as currently described in the IMSBC Code was applied as to a number of typical mineral cargoes including: coal (1 sample), copper concentrate (13 samples), lead concentrates (2 samples), molybdenum concentrate (3 samples), washed play sand (1 sample) and zinc concentrate (1 sample).

6 Even with the modification of 10% added moisture, there was significant concern with the reliability, repeatability and relevance of this test method when applied to these solid materials.

7 Several samples failed the test based on a single test coupon while duplicates in the same test pass, and variation in results for the same samples tested at different laboratories was also noticed. In summary:

.1 For mass-loss, the variability between triplicate coupons (tested with the same sample), calculated as % coefficient of variation = 100% *sample standard deviation/sample average, are higher than the usual acceptable limits: up to 22% for steel[i] and up to 52% for aluminium[ii]. In general, a variation in excess of 10% would be considered inappropriate for liquid samples using the same test[iii]. The inter-laboratory variation shows a factor of 1 to 3, but is based on very limited data.

.2 For localized corrosion, the level of variability noted between triplicate coupons exposed to the same sample also raises similar concern and inter-laboratory comparisons using the same cargo sample reveal a high degree of variability as shown in table 1.

Table 1

	Maximum pitting depth (steel) µm	
Coupon No.	Lab 1	Lab 2
HS 1	220	164
HS 2	None	136
FS 1	86	None
FS2	134	95

(Intra and Inter-laboratory variability for the results from "pitting depth" following tests 7 days exposure of steel coupons to one particular metal concentrate. (HS = half submerged coupons, FS= fully submerged coupons; HS1 and FS1 are the first replicate measurement; HS2 and FS2 are the second replicate measurement))

8 All laboratory experts involved in testing[iv] agreed that the C.1 test method as described in the UN Manual of test and criteria (modified by the IMSBC Code) needs refinement to increase its applicability to this type of solid. The lack of clarity and guidance on how to conduct the test is believed to be a major factor in the high level of variability seen in the test results – particularly for localized corrosion.

9 The following specific concerns related to the test and its relevance were identified during the initial test work and point to some areas of focus for the new protocol:

.1 samples of washed play sand, which would generally be considered inert, were seen to fail the test on the basis of exceeding the threshold for localized corrosion;

.2 for localized corrosion, the threshold of 120µm for the deepest intrusion is comparable to the size of the abrasive particles on the sandpaper used to prepare the coupons for testing (120 grit abrasive particles are approximately 125 µm). It is conceivable that "low spots" of greater than 120µm could therefore exist even before the test. Test coupon surfaces are not routinely analysed prior to exposure, test coupon treatments after testing solids need to be defined and no statistical analysis is prescribed in the test protocol;

.3 the IMSBC Code states that 10% moisture by volume is the minimum that should be contained in a test sample. This can mean making small additions of water to a cargo test sample, which is very difficult to do in practice and no guidance is given;

.4 the reflux condenser, included to avoid evaporation when testing liquid samples, results in water droplets falling onto the test material. This resulted in significant local heterogeneity in the test material in many tests – particularly near the Teflon rods that support the test coupons; and

.5 the test is conducted with the vessel open to air: indeed the C.1 protocol states that *"The entrance of air into the receptacle shall be ensured"*. Several solid bulk cargoes like metal sulphide concentrates are, however, shipped in closed holds that have been demonstrated to become oxygen depleted during transport.

Research programme coordinated by the International Council on Mining and Metals

10 The principal objective of the research programme, launched in late 2015, is to ensure that tests can be carried out in a suitable, consistent and repeatable way by adding details to the current test guidelines. This builds on the experiences of the labs that have carried out testing to date. We will investigate how varying key parameters affects the repeatability of the test for solids.

11 Three key focus areas have been identified based on the challenges and observations described above: sample preparation, test apparatus suitability and treatment/analysis of test coupons after testing.

Research planned for 2016

12 Step 1 is to create a detailed standard operating procedure (SOP) for the sample preparation and analysis. The rationale is to remove variation in the way that labs are conducting the test. It will take into account that the detailed procedures on pre and post-test treatment of test coupons; how to measure localized corrosion; and will check the ability of laboratories to make consistent analyses. An inter-laboratory exercise will be undertaken to ensure that procedures can be consistently applied in practice. This step will be completed in early 2016.

13 Step 2 will involve a "round robin" test to compare pit and mass loss corrosion results using the new SOPs and a range of solid cargoes. The rationale is to assess whether the improvements made in step 1 are effective in making the tests more repeatable across a range of solids.

14 Step 3 will be to optimize other test parameters that may not be relevant to complex solids as they are to liquids.

Conclusions

15 ICMM and its members are undertaking this work in partnership with the established physic-chemical testing laboratories in the United Kingdom and the United States and a recognized centre in Sweden on excellence for corrosion studies and testing.

16 The results of testing will be submitted to CCC 3 for consideration and, if sufficiently mature, consideration of an alternate test protocol for assessing corrosivity under the IMSBC Code.

17 ICMM will be pleased to keep interested delegations informed of the progress of test work as it proceeds and looks forward to future discussions at IMO.

Action requested of the group

18 The group is invited to note the information provided.

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[i] Range of variation (expressed as coefficient of variation (%) between fully submersed coupons) were Cu=5-22 (9 samples, 3 replicas per sample), Zn=20 (1 sample, 2 replicas), Pb=4-13 (2 samples, 2 replicas per sample), Sand=2-10 (2 samples, 2 replicas per sample), Mo=3-18 (2 samples, 3 replicas per sample)

[ii] Range of variation (expressed as coefficient of variation (%) between fully submersed coupons) were Cu=9-52 (9 samples, 3 replicas per sample), Zn=25 (1 sample, 2 replicas), Pb=**24-30** (2 samples, 2 replicas per sample), Sand=**28-47** (2 samples, 2 replicas per sample), Mo=**12-40** (2 samples, 3 replicas per sample)

[iii] The NACEASTM G13 test standard states that: "In laboratory immersion tests, corrosion rates of duplicate specimens are usually within ±10% of each other when the attack is uniform. If the rates exceed this variance, retesting should be considered. Occasional exceptions, in which a large difference is observed, can occur under conditions of borderline passivity of metals or alloys that depend on a passive film for their resistance to corrosion. When large disparities in measured corrosion rates occur, rather than reporting an average corrosion rate, the reason for the disparity should be investigated and reported. If the reason for the disparity cannot be found, retesting should be considered"

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