

Sampling and testing iron ore fines – Aide-mémoire

Guidance for shippers, shipowners, charterers, surveyors and ships' crews



These notes are for the guidance of, shippers, shipowners, charterers, surveyors, ships' crews and other parties involved in the sampling and testing of cargoes of iron ore fines so as to ensure that it is carried out in accordance with the IMSBC Code (the Code), which became mandatory worldwide on 1 January 2011. The relevant sections of the Code are Sections 4, 7 and 8.

Provision of information

The shipper must provide the master or his representatives appropriate information on the cargo sufficiently in advance of loading to enable precautions for proper stowage and safe carriage to be put into effect.

For iron ore fines (IOF) this includes:

- Cargo group (i.e. Group A – cargoes which may liquefy),
- Total quantity of cargo to be offered
- Stowage factor
- Need for trimming and trimming procedures
- Certificate of moisture content (MC) and its transportable moisture limit (TML)
- Likelihood of formation of a wet base in the holds due to moisture migration
- Identification of actual stockpiles for the cargo to be loaded.

Certificates of test

The shipper has to arrange for the cargo to be properly sampled and tested to obtain the information required above

For IOF (i.e. Group A cargo) the shipper shall provide the ship's master or his representative with a signed certificate of the TML and a signed certificate or declaration of the MC.

The certificate of TML shall contain, or be accompanied by, the result of the test for determining TML.

The declaration of the MC shall contain, or be accompanied by, a statement by the shipper that the MC is, to the best of his knowledge and belief, the average MC of the cargo at the time the declaration is presented to the master.

Section 4.3.3 of the Code states: When a cargo which may liquefy is to be loaded into more than one cargo space on a ship, the certificate or the declaration shall certify the moisture content of each type of finely grained material to be loaded into each cargo space. Notwithstanding this requirement, if sampling according to internationally or nationally accepted standard procedures indicates that the moisture content is uniform throughout the consignment, then one certificate or declaration of average moisture content for all cargo spaces is acceptable.

In other words, each type of material to be loaded should be sampled and tested to determine the flow moisture point (FMP), TML and MC.

Sampling procedures

General comments

A visual inspection of the consignment which is to form the ship's cargo should be carried-out. Any portions of material which appear to be contaminated or significantly different in characteristics or MC from the bulk of the consignment should be sampled and analysed separately.

Sampling should take into account the following factors:

- Type of material
- Manner in which the material is stored, in stockpiles, rail wagons or other containers and transferred or loaded by material-handling systems such as conveyors, loading chutes, crane grabs etc.
- The characteristics which need to be determined: MC, TML, bulk density/stowage factor, angle of repose etc.
- Variations in moisture distribution throughout the consignment which may occur due to weather conditions, natural drainage, e.g., to lower levels of stockpiles or containers, or other forms of moisture migration, and
- Variations which may occur following freezing of the material.

Where international or nationally accepted standard procedures are in-place for specific commodities, these should be followed. In the absence of such standards, the IMSBC Code makes the following recommendations in Section 4.6.4 of the Code;

“The number of sub-samples and sample size are given by the competent authority or determined in accordance with the following scale:

Consignments of not more than 15,000 t:

One 200g sub-sample is taken for each 125 t to be shipped.

Consignments of more than 15,000 but not more than 60,000 t:

One 200g sub-sample is taken for each 250 t to be shipped.

Consignments of more than 60,000 t:

One 200g sub-sample is taken for each 500 t to be shipped.”

In order to prevent loss of moisture from the samples between the time they were taken and time they were submitted to a laboratory for testing, each sample should be clearly labelled and sealed in thick polythene bags, preferably placed inside a second bag, or put into polythene bottles. The sealed sub-samples should be protected from

bright sunshine so as to minimise heating and moisture loss/condensation in the bags.

Testing for MC should then be conducted as soon as possible after drawing the sample so as to minimise loss of moisture from the samples. To facilitate this, MC determination should be conducted at a competent local laboratory so as to minimise the time taken to get the samples to the lab.

It is recommended that as many as possible of the sub-samples be tested for MC, such that a picture of the range of MC throughout the stockpile is obtained. Obviously, to conduct 200 such determinations for a shipment of 50,000MT is somewhat impractical under normal routine inspection where the cargo appears to be in satisfactory condition. It would be acceptable to reduce the number of sub-samples into a smaller number of sub-composites by compiling samples together, i.e. every two sub-samples could be composited to give 100 samples for MC determination or alternatively every four samples mixed to give 50 sub-composite samples.

The best method for sampling is invariably during movement of cargo – either during construction of a stockpile when ore is being delivered to the stack or during movement of ore from a stockpile for loading onboard a barge. However, often this is impractical for a number of reasons, such as the requirement for provision of information regarding the consignment prior to loading and/or due to exposure of the ore to rain whilst awaiting shipment.

Additionally, sampling from a conveyor line would normally be preferable during construction of a stockpile but, if the ore has subsequently been exposed to rain or been resident in the stockyard for more than seven days, then a further round of sampling and analysis will be required to demonstrate that the consignment is still fit for carriage. Sampling during movement on a conveyor line at the time of loading is unacceptable due to the requirement for the provision of a cargo certificate detailing the TML and MC prior to the commencement of loading.

Stockpiles

Establish the quantity of ore in the stockpile either directly from the shippers representatives or by estimating the length, width and height of the pile from which to calculate the overall volume and assume a density of about 2MT/M³.

From the quantity of ore, determine the number of sub-samples required from the scale above and then mentally divide the pile into separate smaller sections to simplify the process of sampling. Calculate the number of sub-samples to be drawn from the smaller sections of the pile and plan precisely from which parts each sub-sample will be taken.



Ideally the stockpile should be flattened such that all parts of the pile are accessible and can be sampled. An excavator should be employed to cut slots into the sides of the pile, and to dig pits into the top to access the core of the stockpile. If an excavator is totally unavailable, this should be reported to your principal so that an amended sampling method can be agreed.

The most likely method will be that the requisite number of pits are to be excavated into the surface of the pile to a depth of at least 50cm by hand and sub-samples drawn from the bottom of these pits. Care should be taken to ensure that the sample isn't cross-contaminated with surface material.

In order to be able to check on the variability of the MC within the pile, as many as possible sub-samples should be individually submitted for MC determination. In cases where this will lead to an excessively high number of such samples, i.e. more than 100 or so, it may be acceptable to combine sub-samples together so as to give a smaller number of 'sub-composites' for MC testing. For example, if dealing with a stockpile of 50,000MT, sampling in accordance with the Code requires 200 sub-samples to be drawn and although the overall composite sample for FMP testing should be composed of a portion of each sub-sample, every two or perhaps every four sub-samples could be combined to give either 100 or 50 sub-composite samples for MC determination.

If there is any doubt about the suitability of the stockpile for shipment or if there is an apparent significant variation in condition between one parcel and another, then it would be recommended to submit as large a number of sub-samples for MC determination as possible rather than to combine them.

A further portion of each sub-sample should be placed into a sack so as to give an overall composite sample for the entire pile for use in FMP testing. Obviously each sub-sample used to contribute towards the overall composite sample should be of the same size such that the final composite sample is truly representative of the stockpile as possible.

Barges

After establishing the quantity of ore onboard the barge, determine the total number of sub-samples required and divide the stow up into smaller sections. For each section, plan the sampling points so as to obtain the requisite number of sub-samples spread across the area concerned.

For the shallower sampling points around the peripheries of the stow,



it may be sufficient to manually excavate sampling pits to a depth of about 50cm. Where the stow is deeper, it is preferable to utilise the ship's gear with a grab to excavate deeper pits and to draw samples from these – providing of course that the barge is alongside a geared vessel.

If any areas of the cargo stow appear visually different/distinct from the rest of the cargo, then these should be sampled and analysed separately.

Cargo stows in ship's holds

Determine the required number of sub-samples on the basis of the quantity of cargo in the hold and the scale given above.

As a minimum, each sub-sample should be drawn from manually excavated pits of 50cm depth and care should be taken to ensure that no surface material is admixed. If the vessel is geared, then five pits should be excavated – four in the corners and one in the centre of the hold and samples drawn from each pit at varying levels from 50cm under the surface down to the level of the tank top if at all possible.

Can testing

The master should check for the possibility of flow in the material being loaded. The can test, set-out in Section 8 of the Code, is described as an '*auxiliary*' test method suitable for this.

The test is conducted using a can of about 1 litre capacity half-filled with the sample of material to be tested. After noting the appearance of the sample, the can is held in one hand and brought down sharply on a hard flat surface a total of 25 times from a height of about 20cm at one to two second intervals. The surface of the material is then examined for the presence of free moisture or fluid conditions, such as a glistening flat surface. If these signs are noted, then the Code states that further laboratory tests should be conducted on the material before it is accepted for loading.

It is important to note that the can test is not intended to be a substitute to the more accurate and reliable laboratory testing.

In our experience interpretation of can tests performed upon iron ore fines, particularly when the MC is around the level of the TML but not significantly above FMP, is difficult and requires considerable experience to interpret correctly. When the MC is above FMP, then the result is more clear-cut and easier to recognise.

Experience has shown that at the end of the test with a sample of ore with MC at or somewhat above TML loose, free-moving lumps may accumulate on top layers of the sample in the can, the presence of which may falsely be interpreted as a pass. However, if these are poured off, a solid, compacted lump of fines with a flat surface and free moisture glistening in that surface may sometimes be noted and this of course is indicative of a fail.

Additionally, when the lump is knocked out of the can the sides and bottom surfaces of the sample are often more visibly wet and sticky in appearance.

It is routine in Goa for barges of iron ore fines to be accepted and rejected on the basis of can testing conducted throughout the course of loading by local surveyors representing owners and charterers/ shippers. However, despite the care with which barges are checked using the can test, in a number of cases subsequent sampling in the holds and analysis has shown the loaded cargo to have MC above TML. This serves to illustrate the difficulty associated with interpretation of can tests performed upon this material and the risk of using it to approve cargoes as being fit for carriage in place of properly conducted laboratory testing.

Top left; clear fail: top right; clear pass: bottom left and right; difficult to interpret, recommend further tests in laboratory



Speedy moisture meters

The practice of using speedy moisture meters to monitor the MC of iron ore fines during loading appears to be becoming increasingly common, with a number of the Indian surveying companies now using these meters routinely for this purpose.

The device works by directly relating the pressure of acetylene gas produced by reacting calcium carbide with the moisture present in the sample.

However, there are serious limitations associated with these meters about which those using them should be aware since they severely restrict the suitability of these meters for the purpose to which they are now being used. Specifically these include, but are not limited to, the following:

- The test is performed on a very small sample of just six grams which is weighed using a small pan-balance. Such a sample size will not average-out variations in the composition of the iron ore arising due to the presence of fine grained particulates and larger lumps in any given sample.
- Intimate mixing of the sample with the calcium carbide is essential in-order to completely react with the moisture present in the sample and to generate the full amount of gas. Such mixing is possible with drier, powdery samples but becomes increasingly difficult with iron ore fines possessing MC close to or exceeding TML where the fines tend to agglomerate preventing full reaction and gas generation. As a consequence, with samples possessing MC close to TML, the meters will lead to an under estimation of the actual MC. This has been shown to be by as much as 2 to 3% in some cases.
- The inclusion of the mixing ball bearings during operation may compound the mixing problems by increasing the risk of sample agglomeration.
- The meters must be calibrated and maintained in good working order to ensure that leaks of gas through the reaction vessel/ screw-on-top seal is taken into account and that systematic errors associated with the pressure gauge itself are accounted for.

Even if the meters are cross-calibrated against oven dried readings on the same samples, the variability in the physical composition of the material, combined with the agglomeration problem encountered when MC approaches or exceeds TML, render these meters unsuitable for monitoring the MC of iron ore fines shipments during loading.

NOTE: These notes are not a substitute for the Code itself and if there is any doubt the Code should always be consulted. However these notes can be used as an aide-mémoire of the provisions contained in the Code.

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