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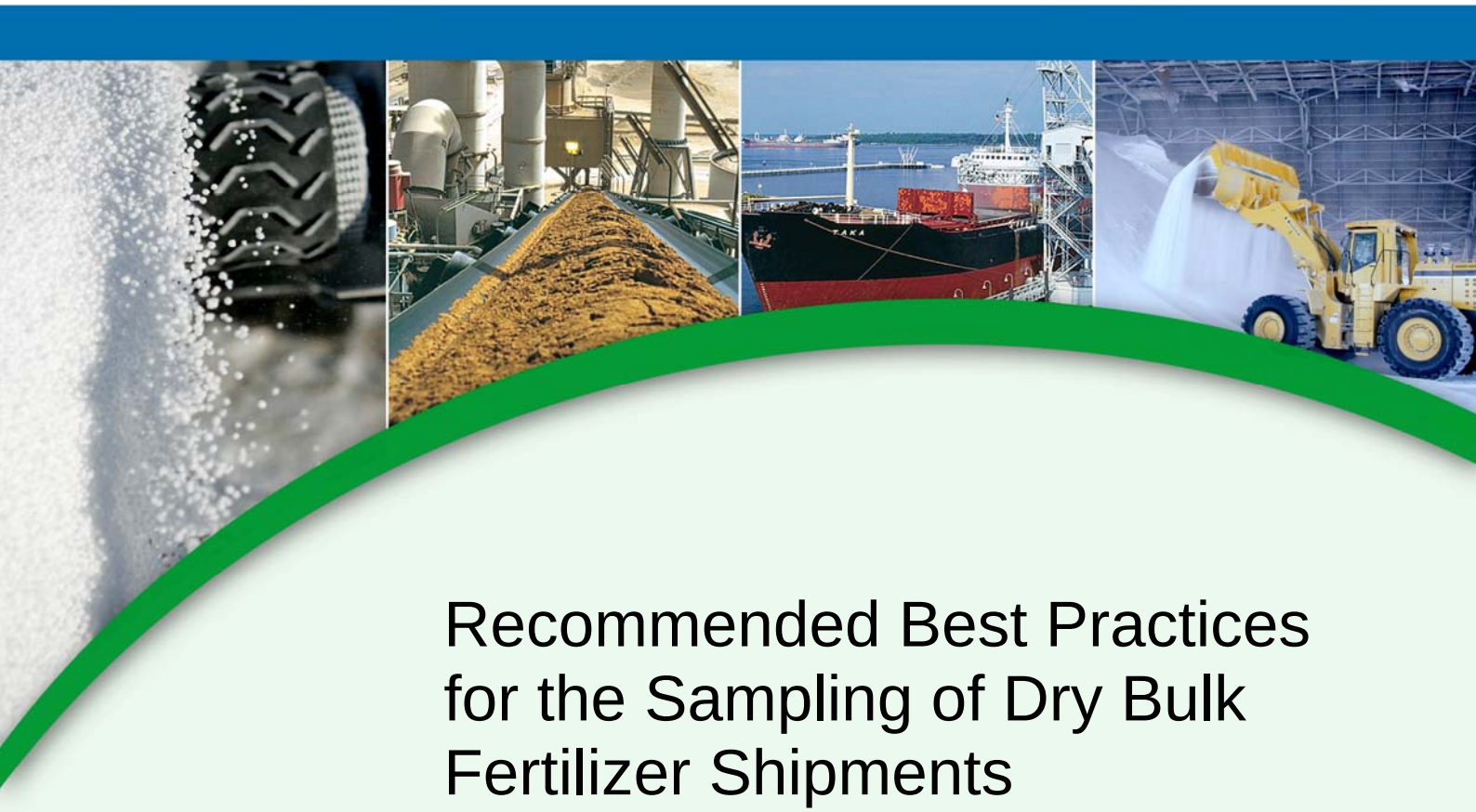

Recommended Best Practices for the Sampling of Dry Bulk Fertilizer Shipments

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This best practice recommendation was researched and prepared by the IFA Working Group on the Harmonization of Fertilizer Sampling and Methods of Analysis, which consists of representatives of the global fertilizer industry. This document is available to the general public and is a reference document for the international trade of fertilizer products. It should not be considered to be an international standard; nor does this document take precedence over existing national and regional regulations or standards.

This recommendation and its background documents are available to the general public on the IFA web site or through written request to the IFA Secretariat.

Contents

Terminology

Bias	The tendency to obtain a value that is either persistently higher or lower than the true value.
Consignment	The total quantity of product or shipment to be delivered.
Division	The process of decreasing the sample mass (without the modification of the particle size distribution) where a representative part of the sample is retained, while the remainder of the sample may be rejected.
Error	The procedures of sampling, sample preparation and analysis that are necessarily imperfect and that disperse experimental results.
Bulk / gross sample	A sample formed when all increments collected from a lot are combined for reduction in a laboratory.
Increment	The quantity taken by a single pass of the sampling device.
Lot (sample portion)	A quantity of fertilizer delivered at one time. The lot may be composed of one or more sampling units or sub-lots.
Manual sampling	The sampling by human effort using hand held devices to take increments forming sub-samples and gross samples.
Mass basis sample	The method of taking increments at uniform mass intervals throughout the sampling unit or lot.
Mechanical sampling	The operation of sampling when the increments forming sub-samples and gross samples are taken by a sampling machine.
Precision	A measure of the way in which a set of observations agree with each other.
Preparation	The process of preparing the sample for analysis or testing.
Reduced sample	Intermediary sample obtained after mixing and reducing the gross sample.
Sampling unit	The discrete units (railcar, sections of belt, daily production) that comprise the lot.
SGN (Size Guide Number)	The calculated diameter of the “average particle” expressed in millimeters to the second decimal and then multiplied by 100. It is the particle size which divides the mass of all particles in two equal halves, one having all the larger size particles and the other half having all smaller size particles.
Time basis sample	The method of taking increments at uniform time intervals throughout the sampling unit or lot.
Uniformity Index (UI)	The ratio of the sizes of “Small Particles” to “Large Particles” in the product. More precisely, it is the ratio, times 100, of the two extreme sizes in the range of particles retained at the 95% level and at the 10% level.

Executive Summary

Mechanical sampling systems (MSS) should be considered the preferred procedure, when available, to obtain gross fertilizer samples during dry bulk fertilizer shipments, as such systems have been determined to be the most reliable and consistent method for the extraction of representative samples. Furthermore, it is recommended that a sample taken by a mechanical sampling system should take precedence over samples obtained by other methods.

Mechanical sampling systems have several advantages over manual techniques:

1. Well-designed mechanical sampling systems allow for more reliable and cost-effective collection of primary increments from the full cross-section of a particular product stream in a single pass, thus providing samples that are representative of the entire lot. This aspect is particularly applicable within today's context of large lots and high-capacity flow rates, which may justify the additional investment capital for such a system.
2. Mechanical sampling may reduce health and safety risks to sampling personnel especially in cases where sampling is conducted from high-speed conveyor systems.

In the absence of a mechanical sampling system, manual sampling may be carried out on fertilizer material being transported along a conveyor belt and/or from a falling stream (from a belt or a chute). In-depth review by the IFA working group has shown that the Association of Fertilizer and Phosphate Chemists (AFPC) has adopted a suitable manual sampling scheme that is widely-used and recognized by the global fertilizer industry.

It is thus recommended that the AFPC VI.1D method, along with several procedural modifications described in this paper, should be considered a preferred alternative to mechanical sampling systems. Furthermore, during manual sampling of dry bulk fertilizer shipments, special consideration should be given to help minimize health and safety risks to personnel.

Recommended Best Practices for the Sampling of Dry Bulk Fertilizer Shipments

1. INTRODUCTION

With the rapid expansion of international trade, the global fertilizer industry has experienced an increasing number of contractual disputes due to the variable use of methods and procedures to sample and analyze international product shipments. In an IFA member-driven initiative, a broad-based international task force was formed to address this matter.

Accurate and representative sampling is a challenging operation that requires both understanding of the product as well as the correct application of the sampling process. Sampling methods should be applied with strict accuracy by specialized personnel with prior sampling experience. Moreover, a standardized approach should be adopted irrespective of the sampling location, shipment size or commodity.

The industry task force established that the following basic elements of the sampling process are important to a standard, systematic sampling plan:

a. Identify the consignment
b. Choose the sampling place
c. Choose the sampling time
d. Determine the number of increments
e. Determine the mass of increments
f. Determine the sampling interval
g. Determine the sampling tools
h. Sample preparation/division

Presently, on a global basis, the fertilizer industry is using various methods and procedures to perform dry bulk sampling operations. Following a global review, the task force determined that the current sampling procedures widely used in different markets can often lead to varying representativeness.

Sampling principles:

The frequency of sampling, sample size and sample processing are determined by the application of various theories and applied statistics, from which today's sampling standards have ultimately been derived.

Since it is important to collect increments from all parts of a particular lot, it is necessary that the total lot be accessible. In other words, it is of fundamental importance that all particles in the lot have the same probability of being included in the final sample, which is a key objective when performing such an operation. To achieve this goal, it is desirable to sample the lot while in a "dynamic" state or when particles are being evenly-distributed within a given mass or volume.

Observations from pilot sampling exercises:

The particle size variability with homogeneous product (product with low product size variability) was found to be minimal during the pilot exercises. However, for those products with higher particle size variability, the spread of results between various methods can reach 10% or more (an 8% spread was recorded in one exercise) This variance is not considered acceptable in commerce, and thus highlights the need for a more harmonized approach.

The procedures currently in practice throughout the industry vary widely and only a few methods noted have been adapted for modern bulk loading practices, such as for high speed conveyor belt transfer systems.

The pilot exercises demonstrate that methods are more reliable when a higher number of increments are included.

2. INDUSTRY BEST PRACTICES

2.1. Mechanical Sampling Systems (MSS)

Mechanical sampling systems (MSS) should always be the preferred procedure, when available, as these systems have been determined to be the most reliable and consistent means to extract representative samples during the shipment of dry bulk fertilizer material. Moreover, MSS samples should take precedent over samples obtained using other methods.

Mechanical sampling systems have several advantages over the manual techniques:

1. Well-designed mechanical sampling allows for a more reliable and cost-effective means to collect primary increments from the full cross-section of a particular product stream in a single pass - particularly applicable with today's large lots and high capacity flow rates - providing samples that are representative of the whole material.
2. This procedure reduces health and safety risks to sampling personnel as it avoids manual sampling from high-speed conveyor systems.

Mechanical sampling systems vary in complexity, the simplest consisting of a primary cutter that collects increments either by manual initiation or at pre-determined intervals by either a timer or weight meter. More complex sampling systems may include additional stages of sample division and various stages of particle size reduction.

The factors that may determine the type and size of mechanical sampling systems are:

- Belt capacity
- Material top size
- Lot size
- Sampling frequency
- Desired final sample mass

The most common types of mechanical systems used for bulk fertilizer shipments consist of one or two division stages. It is recommended that each sampling application should be considered thoroughly before making a selection of the system to be used. The ISO 11648 standard for bulk material sampling should provide the basis for the design of a MSS.

It is essential that sampling systems be regularly inspected and audited to ensure correct sampling and compliance to the original design and other applicable standards.

The following steps should be followed to ensure the proper functioning of mechanical sampling systems:

i. Critical inspection: inspect sampling systems regularly, observing the system under dynamic and static conditions, checking if system components conform to manufacturer design specifications. Critical inspection provides current operating information about the sampling system and provides useful information for the operation and ongoing maintenance.

ii. Bias testing: conduct a precision test to evaluate the performance of a sampling system by determining if samples collected and processed by the mechanical sampling system are being collected without bias. With respect to samples collected during a test, the ISO 13909-8 recommended interval should be used in bias testing.

iii. Statistical Process Control (SPC): in conjunction with periodic critical inspections, SPC is used to ensure that the bias test conditions actually do prevail throughout the ongoing operation of the mechanical sampling system.

2.2. Manual Sampling from a Falling Stream and/or a Conveyor Belt

In the absence of a mechanical sampling system, manual sampling may be conducted for material being transported along a conveyor belt and/or falling stream (from a belt or a chute). In-depth review has shown that the Association for Fertilizer and Phosphate Chemists (AFPC) has adopted a suitable manual sampling scheme that is widely used and accepted by the industry. Consequently, it is recommended that the AFPC VI.1D procedure, along with several procedural modifications, should be used as an alternative to a MSS.

The procedures below represent an IFA-recommended industry best practice for obtaining a gross sample of fertilizer when sampling from various capacity conveyor belts and/or falling streams.

Before embarking on a manual sampling operation, the establishment of an overall sampling plan is recommended. This undertaking would require an understanding of the shipment's contractual requirements as well as the proper execution of the sampling practice, the sample preparation and the measurement.

For sampling plans involving a single measurement - sampling, sample preparation, measurement should include:

1. The collection of increments.
2. The mixing of all increments together to form a gross sample.
3. The division and reduction to prepare a final measurement sample.
4. The measurement of a single sample representative of the entire cargo.

Sampling plans (involving testing of a lot or sub-lots):

An effective way of improving the precision of product testing is to conduct multiple measurements, and subsequently average the observed results. Before releasing the results of a cargo, the laboratory should take a weighted average of several independent determinations (however, the results should still refer to a single sample).

Statistically, the overall precision will be greatly improved if the testing is made on a number of samples rather than a single sample. This is achieved if testing is made on a series of samples representing parts of the consignment described as 'lots' or 'sub-lots'. In this case taking a weighted average of measurements on a number of lot or sub-lot samples will not only improve testing precision, but will also improve the sampling and sample preparation precision.

Furthermore, testing on a lot or sub-lot basis enables a more careful control of the critical or highly-variable characteristics of a cargo.

The use of lot or sub-lot sampling may substantially increase costs in the sample preparation and testing process, yet these costs should be balanced against the potential benefits resulting from the increased precision achieved.

Testing on a lot and a consignment basis:

Testing can be performed on a lot basis for certain parameters and on a consignment basis for others. The first step is to divide the consignment into a number of lots, usually on a weight basis (e.g. 100,000 tonne cargo might be divided into 10 x 10,000 tonnes). The samples would then be drawn for each of the ten lots by combining increments. Subsequently, the lot samples may be divided and the measurements of granulometry can be conducted at this stage. The samples can also be successively reduced and divided to make them suitable for other testing parameters. Portions of the prepared or semi-prepared samples can be blended to prepare new samples representative of the entire consignment. Before testing, this sample should be further mixed, reduced and divided.

This type of sample preparation scheme should be utilized for cargoes which have the granulometry or hardness reported on a lot basis, while the chemical analysis is conducted on a consignment basis.

Apparatus (conveyor belt sampling):

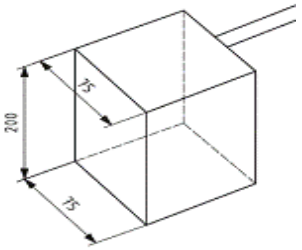
A sample ladle with a 500 ml sample cup and a three-foot (roughly 90 cm) handle, or similar device, should be the preferred tool for the sample collection. The long handle allows for greater leverage and for more effective penetration into the material running on the belt.

Apparatus (falling-stream sampling, higher-capacity belts):

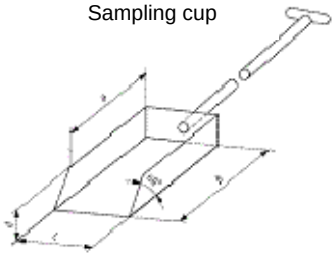
A sampling scoop of roughly 50 cm in length and 5-6 cm in width is recommended.

Apparatus (falling-stream sampling, lower-capacity belts):

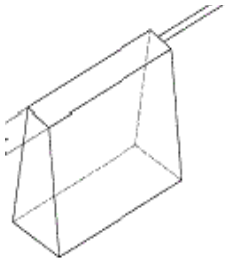
A stream sampling cup or manual cutter can be used. The sampling tool should have an opening at least three times the nominal top size. A permanent sampling system may be constructed by supporting the cup on a track of two steel rods such that the mouth of the cup is in the path of, and at right angles to, the stream flow - using guides attached to the cup and a handle on the side of the device.



Sampling cup



Scoop



Manual cutter

2.2.1. Falling stream sampling

Sample increments are taken based on systematic, time-based sampling procedures and will vary according to the total tonnage and load rate. The following sampling plan guideline is recommended as the minimum amount of increments necessary to ensure that the samples are ultimately representative of the entire lot.

Table 1. Sampling plan guideline.

		Average load rate in tonnes per hour (TPH)			
		400 TPH	800 TPH	1,000 TPH	1,400 TPH
Lot size	Minimum # of increments	Sampling intervals - in minutes			
2,000	90	3.5	2	1.5	1
2,500	120	3	1.5	1.5	1
3,000	120	4	2	1.5	1
3,500	120	4.5	2	2	1.5
4,000	120	5	2.5	2	1.5
4,500	120	6	3	2.5	1.5
5,000	120	6.5	3	2.5	2
7,500	230	5	2.5	2	1.5
10,000	230	6.5	3.5	3	2
12,000	230	8	4	3	2
15,000	230	10	5	4	3
18,000	300	9	4.5	4	2.5
20,000	300	10	5	4	3
25,000	300	12.5	6.5	5	3.5
30,000	300	15	7.5	6	4.5
35,000	350	15	7.5	6	4.5
40,000	350	17	8.5	7	5
45,000	350	19.5	6.5	7.5	5.5
50,000	350	21.5	10.5	8.5	6
55,000	400	20.5	10.5	8.5	6

This sampling plan guideline is based on the following equation:

Sampling interval = tonnage / load rate x 60 / # of increments

When sampling lower-speed belts (500 tonnes per hour or lower):

1. Select a position (e.g. belt transfer point) where the material is falling freely;
2. Take a sample by passing the stream sampling cup or manual cutter completely through the stream as the material drops from a transfer belt or spout;
3. Move the sample collector once across the full stream at an even speed. The long slot in the top of the sampling cup should be at a right angle to the falling stream;
4. Pass the cup through the complete stream at a uniform speed, such that the cup will collect approximately equal amounts during each pass, but will never overflow;
5. Empty contents of the cup from each pass into a suitable container. This is applicable for sampling material with uniform stream flow of three minutes or more, such as during transfer or shipment from a bin or large hopper, or for stream sampling from a continuous production unit;
6. When sampling from a conveyor belt, the belt must be in "full load" before an increment is collected;
7. Avoid sampling a trickle of fines or dust.

When sampling higher-speed belts (500 tonnes per hour or higher):

1. Crossing the product stream with the sampling cup or manual cutter may not be possible. In such situations, sub-increments should be taken with the sampling scoop through the whole width of the stream at a right angle at relatively equal intervals (approximately 10 to 15 seconds);
2. The number of sub-increments to form one increment should be at least five;
3. The number of total increments to be taken should be based on the total mass;
4. The sampling plan as defined in section 2.2.1 should be used.

2.2.2. Conveyor belt sampling

The sampling plan as defined in section 2.2.1 should be used.

It is important to verify the material to be sampled as well as to ensure that the belt system is clean and dry. Conveyor belts that transport solid materials are typically "U" shaped with the center of the belt at a lower point than at the edges. This has a channelling effect on the product and tends to reduce the stream into a line down the center of the belt. It is unlikely in manual sampling that an entire cross section (representative of material in full depth and width) can be taken in a single pass from one side of the belt. Increments should be taken in a planned sampling pattern from alternative sides and at central locations of the belt. In order to obtain a representative sample, the whole depth of the stream must be penetrated.

It is important to ensure that all sample increments are of the same size for a truly representative gross sample – and to stand in a position where you can safely reach the center of the product stream from a point where the product falls onto the conveyor belt. Increments should be collected by inserting the sample scoop down and into the center of the moving product stream, until the bottom of the belt is reached and the collection ladle is full.

At a strict minimum, the following safety precautions are recommended when sampling from a conveyor system:

- Facility representatives must be notified of the sampling plan and the sampling location prior to any sampling activity.
- Extreme care should be exercised since the moving product will force the scoop in the direction of the flow.
- Position oneself away from any moving parts including rollers.
- Access to the belt should be possible from either side with secure footing and guard rails.
- Never sample a coarse or lumpy material (top size more than 13 mm) from a moving belt.
- The correct sampling tool should be used with the appropriate capacity/particle size relationship, and its design should be such that it can be used and withdrawn with physical ease.
- Keep clothing, fingers, hair, and other parts of the body away from the conveyor.
- Do not climb, step, sit or ride on that conveyor belt at any time.
- Do not remove or alter the conveyor guards or the safety divides.
- Know the location and the function of all stop/start and alarm controls.
- Unless sampling, all personnel and equipment must be clear of the conveyor belt.
- The areas around the conveyors should be kept clear of obstructions.
- Adhere to the facility's safety requirements along with your company's safety requirements, and all unsafe practices should be reported to the site operator.

Disclaimer:

Manual sampling from a moving belt should only be carried out when no alternative sampling method or location is available. Conveyor belt manual sampling is a dangerous activity that should be considered only for lower- speed belts (less than 500 tonnes per hour) - and this, only when no mechanical sampling system and transfer points are available.

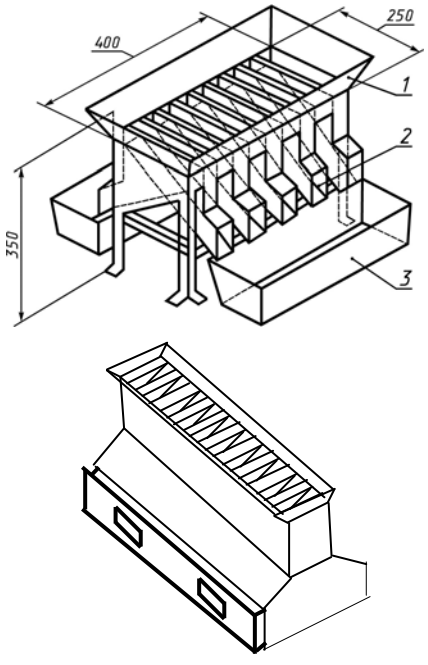
Conveyor belt manual sampling should only be performed in locations and facilities where such an activity is permitted in accordance with the local facility's health and security rules. Prior to performing such operations, it is highly recommended to obtain, in each instance, a written authorization from the facility owner or his representative. In the event of any doubt with respect to the procedure to be applied, to the local and facility rules, or to the authorization by the facility owner or his representatives, the conveyor belt manual sampling should not be performed.

While recognizing that conveyor belt sampling is a common practice in many locations, IFA does not accept any responsibility for any incident that may result from the execution of such sampling activities, nor for any of the other activities described in this paper.

2.3. Sample Preparation

It may be necessary to reduce the gross sample in a field setting prior to its submission to the laboratory for analysis. The sample reduction should be obtained using either a riffle divider, a mechanical divider, or by using the cone and quartering method. Also, the preparation and reduction equipment should be deemed appropriate for the material being sampled. It is equally important to ensure that the area that will be used to reduce the samples (including any equipment) is clean and dry.

Examples of such devices:



Riffle dividers

Division procedure:

In the reduction of an ungrounded sample using a riffle or sample splitter:

- Ensure that all equipment is clean;
- Set riffle level (not tilted in any direction) and place two empty pans beneath the riffle;
- Transfer the collected sample to one or two of the remaining pans, as required (each pan should not be more than two-thirds full);
- Level the surface of the pan before continuing;
- Rapidly tilt the pan to the hopper so that the material will flow evenly from the pan onto the riffle in one motion;
- Collect the entire sample in the pans beneath the riffle;
- When two pans are required for the original, take the second pan and transfer this material to the top of the riffle again;
- Repeat this procedure a minimum of three times for thorough mixing of the product, and subsequently reduce the product to the appropriate proportion required;
- Transfer the final sample to a moisture-proof container, and mark for identification.

Sample bags and containers:

The increments and the final samples should be placed in a clean, dry, moisture-proof container (MPC) with full labeling for final delivery and custody control. The quantity represented by each sub-lot should be specified on each sample label. Containers should protect the samples from any moisture and should be stored in order to maintain the overall integrity of the sample.

The recommended number of contractual samples (FOB, CIF, C&F contracts) should consist of the following for each sample set (unless specified otherwise):

1. Minimum of 2 kg per MPC – laboratory/analysis.
2. Minimum of 2 kg per MPC– retention/ arbitration.
3. Minimum of 2 kg per MPC – retention/arbitration.

References

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