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The Importance of Cargo Sampling

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A grayscale background image showing a large port crane with the number '21' on its side, positioned over a large pile of granular cargo. The crane's boom extends diagonally across the upper right portion of the frame. The cargo pile is in the lower left foreground.

In virtually every dry and liquid bulk cargo, in many break bulk and container cargoes, sampling is critical and it is becoming increasingly important.

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Why do we sample cargoes?

Cargoes, whether dry or liquid, are stored and transported in large quantities and it is impractical and unrealistic to check every single portion of the cargo. Therefore, a smaller quantity is taken that has been shown to be statistically reliable estimate of the entire cargo. It is important to take a sample of a sufficient size, at a required frequency and often at different locations to properly represent the cargo quality. We will discuss the basics of statistics later with some examples.

Sometimes but less often, samples are taken in order to determine the causation which we will discuss briefly later.

Representative samples

Representative sampling methods are used on a daily basis in the grain industry. For example, the US Department of Agriculture has distinct grades for each of the commodities commonly traded. Samples are taken “live” during the loading process and the samples are usually taken from an automatic sampler prior to reaching loading bins. Those samples are combined in various ways to assess such wide-ranging parameters as odour, presence of insects and moisture content, but most critically they are used as a basis to assess the grade of the cargo. Grading parameters are commonly factors such as broken kernels, foreign matter and test weight, and since the value of the cargo is dependent on the grade, it is critical that the sampling is conducted correctly.

Grading of agricultural commodities may or may not be involved during loading of vessels, but almost certainly samples will be taken for contractual purposes between buyer and seller. Depending on the commodity, this will be GAFTA (grain and feed) or FOSFA (seeds) sampling rules, which recently have aligned themselves. Both give guidance on the sampling methods, frequencies and sample sizes. For example, GAFTA no.124 stipulates a sliding scale of sampling frequency depending on the consignment size. Please note this is the minimum frequency recommended:

Table1: Increment sampling - size of lots, number and size of consignments

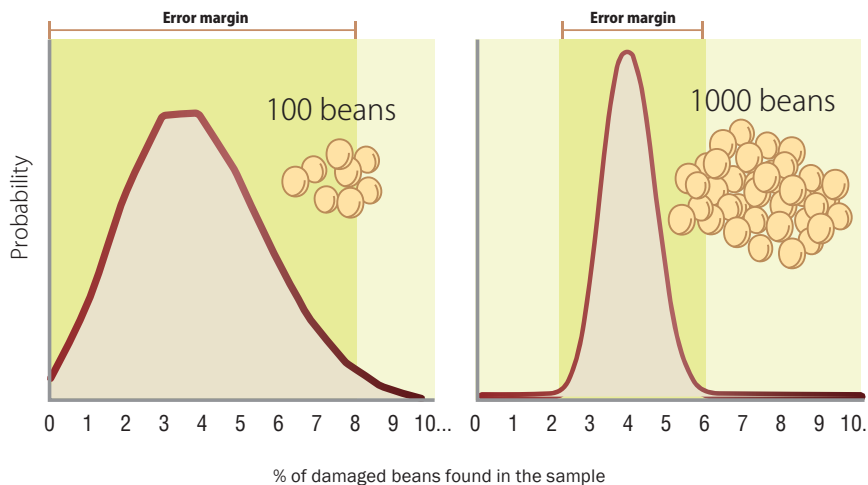
| Consignment size (Tonnes) | 0-5000 | 5001 – 10,000 | 10001-25,000 | > 25,000 |
|---|--------|---------------|--------------|----------|
| Lot size (Tonnes) | 500 | 1,000 | 2,500 | 5,000 |
| No.of increments per lot (number) | min 20 | min 30 | min 40 | min 50 |
| Min bulk aggregate sample per lot (Kilos) | 20 | 30 | 40 | 50 |
| Max weight of increments (Kilos) | 1 | 1 | 1 | 1 |

From GAFTA Sampling Rules No.124 Rules for sampling, Analysis Instructions, Methods of Analysis and Certification.

Virtually all international bulk cargo shipments will be at least 25000 tonnes and thus GAFTA stipulate the cargo is divided into lots of 5000 tonnes, each lot taking at least 50 individual samples and taking at least 50kg per lot but at most 1kg per sample.

Statistics

If only small quantities of samples are taken, then there is a higher margin of error. For example, the allowable level of heat-damaged beans in soya from Brazil is 4%. If we have a large quantity of, say, soya beans that definitely contains 4% heat damaged beans and 100 beans are selected at random, the chances of finding exactly 4% damaged beans are low. If, however, we take 1000 beans the probability of finding the actual level increases:



The margin of error decreases dramatically the more beans we take. This is the reason why as many samples as practically possible should be taken.

This example above is for a type of parameter that we call “non-continuous” variable. It is non-continuous in the sense that a bean can be either heat-damaged or not heat-damaged. Other types of parameters are called “continuous” variables and will have a range of values. For example, the moisture content will differ in different parts of an iron ore fines stockpile, but it is unlikely that the cargo will be very wet and/or very dry. Similarly, with parameters such as wet gluten in wheat samples - there will be a range of values in different parts of the cargo. Regardless of the type of variable, taking a higher number of samples will reduce the margin of error.

These above examples envisage a relatively even distribution of the parameter in the commodity but, in practical situations, there are factors at play by which cargoes can become heterogeneous (i.e. uneven) with respect to a factor, and in these cases the sampling method and sample handling are critical and need to be adjusted to suit their purpose.



Sampling methods

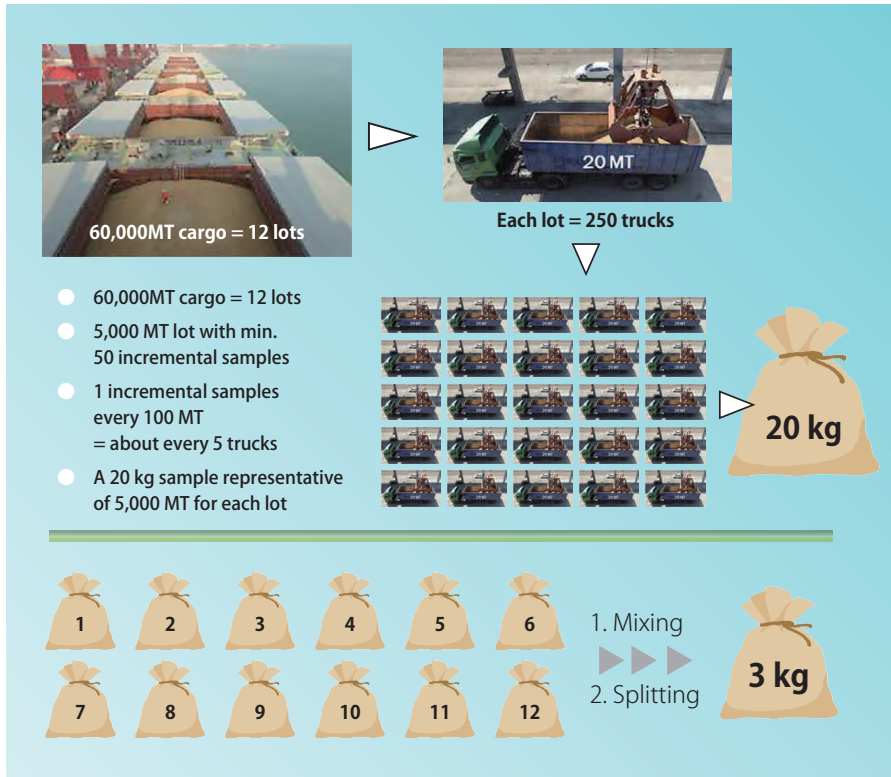
We mentioned earlier the GAFTA no.124 rules. In addition to stipulating the sample frequency, they also specify the following for bulk (for example) cargo samples:

- ✓ **Concurrently with loading**
- ✓ **At the nearest practical point to the vessel**
- ✓ **Throughout the consignment**
- ✓ **Uniformly**
- ✓ **Systematically**
- ✓ **At loading or at discharge from a moving stream**
(if taken from a conveyor, vehicle or port storage)
- ✓ **At loading from quay or barge**
(if loaded by grab)
- ✓ **At discharge from various parts of the hold**

All this is designed to create a reliable picture of the cargo condition throughout the consignment. If sampling is conducted to this regime, it is usually a reliable regimen.

However, cargoes are not always homogeneous and certain factors are notorious for this, mycotoxins being the classic example. Mycotoxins are highly toxic chemicals produced by mould. They are not always produced by mould – they are a defence mechanism that is intended to kill competing species. Some of these are also toxic to mammals and are strictly controlled nationally and internationally. For example, the most commonly encountered family of mycotoxins, aflatoxins, have a maximum residue level of 15ppb in some varieties of nuts intended for human consumption. The problem with aflatoxins

is that they are only produced by moulds under specific circumstances and therefore any affected cargo tends to have a highly sporadic distribution of the toxin, but where it is present, the levels will be very high. Such extreme unevenness has to be dealt with by adapting sampling methods (and sample handling, which we will discuss later). For example, for a typical 60,000 tonne grain shipment, GAFTA would require the cargo be split into 12 lots, each lot requiring 50 samples, so one sample every 100 tonnes:



Aflatoxin sampling is typically more frequent and involves taking larger samples. For example, Commission Regulation (EC) No 401/2006 of 23 February 2006 stipulates 500 tonne lots and taking 100g of sample every 5 tonnes to form 10kg samples. For a 60,000 tonne cargo that is a total of 12,000 incremental samples to form the 120 samples for analysis. The whole point of this is to try to lower the margin of error as much as possible.

What is important for operatives in the marine industry is that the above examples clearly demonstrate the importance of a sampling regime in cargo acceptance. We have had cases in the past where entire shipments have been rejected for aflatoxin contamination but on totally inappropriate sampling regimes. Some trivial sweat or wetting might result in mould growth and aflatoxin production in a small area, but the entirety of the lot must be taken into consideration.

Some cargoes are more predictably heterogeneous and sampling plans should take this into consideration. For example, many Group A ^❶ cargoes are typically formed into conveniently-sized stockpiles and are stored outside sometimes for extended periods, during which time the moisture will tend to drain. Samples taken from the upper surface will tend to have a lower moisture content than the samples taken from the base of the stockpile, so it is essential that all levels are sampled and tested. A recent addition to the IMSBC Code (section 4.4.8) states:

“For unprocessed mineral ores, the sampling of stationary stockpiles shall be carried out only when access to the full depth of the stockpile is available and samples from the full depth can be extracted.”

section 4.4.8

We would recommend this is conducted on individual samples in any case from various depths and regular points in nominated stockpiles to obtain a 3D map of each. This

❶ Group A cargoes

❶ Group A cargoes are those that are liable to liquefy. In order to be carried according to the IMSBC Code, the lowest moisture content at which the cargo is considered to achieve a flow state is measured on representative samples of the cargo in a laboratory. This is the Flow Moisture Point or FMP from which the Transportable Moisture Limit (TML) is calculated (90% of FMP). The moisture content of the cargo is also determined from samples – not necessarily the same samples. The moisture content in each cargo space must be lower than the TML.

is important: if a stockpile is nominally acceptable in terms of moisture content on average, the difference in moisture content can still be significant from the upper part of the stockpile to the lower part. As such, there is potential for the wetter part to be loaded in one cargo space which would be in contravention of section 4.3.5 of the IMSBC Code:

“When a concentrate or other cargo which may liquefy is to be loaded into more than one cargo space of a ship, the certificate or the declaration of moisture content shall certify the moisture content of each type of finely grained material loaded into each cargo space.”

section 4.3.5

If stockpiles have been properly mapped with respect to moisture content, this is straightforward to calculate and load safe cargo. Shippers have been reluctant in the past to go to these measures but it is often advantageous to them in that if the stockpile as a whole is not within the acceptable range, they might nominate not to load the wettest part. IMSBC Code section 4.4.4 states:

“Depending upon the results obtained in these tests, it may be necessary to reject those particular portions as unsuitable for shipment”.

section 4.4.8

Many stockyards use reclaimers which take layers from the surface of the stockpiles and it is straightforward to avoid any overmoist portions. The cargo is shipped and the Code requirements are satisfied.



Reclaimer loading iron ore from a stockpile

Following implementation of MSC.1/Circ.1454/Rev.1 of 15 June 2015 “Guidelines for developing and approving procedures for sampling, testing and controlling the moisture content for solid bulk cargoes which may liquefy” into IMSBC Code amendment 03-15, which is now mandatory, shippers are required to establish sampling procedures to ensure test samples are representative of the cargo to be shipped. The required procedures need to incorporate provisions such as:



Sampling methods

- ✓ Identifying the consignment to be sampled
- ✓ Describing the method of sampling
- ✓ Number of subsamples
- ✓ Location of subsamples
- ✓ Sampling equipment
- ✓ Evidence of staff training



This circular can be found in the 2016 Edition of the IMSBC Code on pages 561 to 566 and incorporates methods for analysis, guidelines for competent authority implementation and moisture control in addition to the sampling. We would encourage marine operatives from all involved parties to read and understand these sensible and comprehensive requirements. All the above sampling information should be readily available from shippers and we would recommend requesting it routinely in addition to the other important documentation such as moisture content, FMP and the laboratory worksheets.

We operate the same principles when sampling static grains because often quality and/or condition will change in agricultural commodities with depth in a stockpile. We discuss static sampling methods later.

One final point in this section is regarding recent issues with partial sampling of cargoes.

Occasionally, we encounter smaller but unusually high levels of bad quality cargo portions incorporated into an otherwise normal bulk. When such cargo is loaded in a hold, it will naturally flow into a layer shape on the stow surface. This is not easy to distinguish during loading by looking at the cargo in a moving stream, especially with dust generated by loading. Also the layer of highly affected cargo might be covered by a thin layer of normal cargo during a loading sequence which will totally obscure it. During offloading, however, the grab or vacuvator cuts through the layers and the contrast between the different qualities is obvious. Often in these instances, receivers will complain and segregate this darker layer as damaged cargo, sampling it during offloading and making a claim on any reduction in quality. However, the samples taken at load port will have incorporated both relatively good and relatively bad portions and the average is probably within specification, so the heterogeneity in this instance is not much of a problem in itself. Therefore, only sampling the “damaged” portion is ill-advised. While the “damaged” portion is more likely not to be in specification, the remainder of the cargo is most likely to be well above specification and, if taken as a whole – good and bad - is probably within specification. Our advice is to sample everything regardless of what segregation takes place. If the samples show that the cargo is within specification as a whole, it is disingenuous to claim for the worst part of it. We have successfully pre-empted claims by not allowing consignees to sample only the worst portion of the cargo.

Sample preparation

Obtaining a sample is an important step but often the sample is enormous and to properly prepare it for analysis is another critical and often overlooked step.

As highlighted earlier, there is a great deal of sample remaining following sampling and it is usually impractical to post large quantities of samples to laboratories. Typically samples have to be reduced in quantity in a sensible way that preserves the

characteristics of the material. For reasons we will not go into in this article, following typical transcontinental voyages we often see the upper part of cargo stows more heavily affected than lower parts. If sequential samples have been taken during offloading and placed in a large bag (as is the usual manner) this means the commodity in the lower part of the bag is probably worse than the top part. If the sample is split, say, into 4 portions just by decanting the contents into four separate bags then it is unlikely that these four samples will represent the same cargo.



Riffler

Usually, therefore, methods are used to obviate any variations and the two techniques commonly used are using a riffler or coning and quartering. A riffler is a contraption, typically metal, which has a series of chutes that direct cargo to alternate sides. These have been demonstrated to be effective in splitting samples to a reasonable statistical



Coning and quartering

standard. Other cargo splitters have different designs but use the same principle, cutting the sample stream in two and diverting it to split the sample.

If a riffler is not available, coning and quartering is another method shown to provide satisfactorily even splits. The sample is poured from a height at the same location, so it creates a cone, which is flattened and split into quarters, each quarter forming a sample. These can be further split if necessary.

These methods are acceptable for the usual grading parameters in cargoes, but for factors like mycotoxins, the sample preparation is even more critical. As discussed previously, the sample sizes are large and need to be reduced, but given the extreme heterogeneity of mycotoxin distribution, it is quite possible we obtain a situation where one infected kernel is containing the entirety of the contamination. This might be within specification in the much larger sample but if that sample is split into 2 or 4 subsamples, the offending kernel will only go into one of them and could render that sample off spec, which is not a true reflection of the real situation. Because of this, the recommended practice is to grind a large sample to make the particle sizes smaller and mix them. This makes a sample split more representative of the actual concentration when the sample is analysed.

Even if a cargo sample is taken correctly, if it is not processed correctly subsequently, then spurious results can be obtained, and this has in the past resulted in mass rejections. Marine operatives should ensure samples are being taken and processed properly to obtain the true picture of the contamination level. We would recommend owners and charterers pay special attention to this aspect because often local authorities will reject cargo on sketchy evidence and it is worth requesting full disclosure of sample preparation (in addition to the other sampling details and analysis).

Practical considerations

We hope the above gives some idea of the importance of sampling but also the sheer effort involved in obtaining samples. Often, we are asked “take samples” half-way through offloading or when offloading is nearing completion and there are many obstacles to that.

Static cargoes, i.e. in warehouses, barges and silos are not straightforward to sample.



Sampling sorghum with Pneumac

Spears are suitable for depths of up to 2m and we also have a pneumatic sampler – basically a powerful armoured vacuum cleaner – capable of sampling 6 m depths. Many sampling companies worldwide will possess such apparatus.

If the stockpile is higher than 6m or stored in a silo, it is not sampleable by static methods and normally we would suggest either stockpiles are constructed to allow the normal methods to be performed, or the silo contents are recirculated or passed to an empty silo. This is time-consuming and expensive in terms of manpower; it might also require the cargo custodian to rent another warehouse/silo.

It is much easier to sample cargoes as they are moving and the GAFTA/USDA rules envisage that system – it is relatively straightforward to sample from a moving stream.

Bagged cargoes are typically straightforward to sample if reasonable access is given in warehouses to reach the required stacks. Bags are typically stacked on wooden pallets in warehouses and pallets stacked atop each other, allowing access to a depth of cargo. Samples are taken by a small spear that can penetrate the bag weave without damaging it too much.

Either way, people should be aware



Sampling soybeans with spear



Bag sampling



250,000 tonne stockpile



Sampling nickel ore by excavator

of the physical reality of sampling. The pneumac above weighs 24kg and we typically double that when taking a sample as the chamber is filled. It is exhausting and realistically is a 2-3 person task to be done adequately. Most sampling needs to be conducted around the clock and for sampling to EU Mycotoxin requirements, this can only be done by a team of people taking shifts in order to keep up with the speed of offloading.

Sampling stockpiles of ore is only possible if they are of a reasonable size in accordance to Code stipulations as previously discussed. Often, we are asked to sample enormous quantities which is unreasonable and impossible. The following was a 250000 tonne 12m high cargo stockpile we were asked to sample:

Sampling of ores can be conducted with excavators and/or grabs but if the cargo is not very cohesive, the “pits” need to be dug very wide in order to get deep and this

takes a great deal more time than is often predicted.

Instructors of sampling companies need to be aware of the time and the effort required to take samples and to ensure it is properly funded with the appropriate manpower. Otherwise, these risk corners being cut.



Sampling bauxite in ship's hold by grab

Causation

Many operatives in the marine industry presume that the cause of damage of a given commodity is determinable by sampling. In most spoilage cases this is a misconception. In some instances, it can be useful to compare the properties of, say, an affected sample with the unaffected sample. The most commonly encountered instance of this is when seawater ingress is suspected, and tests are conducted on samples to see if seawater ions are present.

Liquid cargoes

Liquid cargoes are stored in tanks. Tanks can be static (i.e. shore tanks) or mobile (i.e. ship tanks or other tanks associated with other modes of transport such as rail, road or air, when the liquid is to be conveyed). Liquid cargoes are transferred from one tank medium to another via pipes and pumps. Pipes can be short, such as in a port when the cargo is being transferred from a shore tank into a ship, or long, as when oil is transferred across countries or continents. Short pipes can be solid and fixed or they can be flexible hoses. Pipes and tanks are often used to store or convey myriad types of liquid; they are often not dedicated to any particular cargo. Ownership of the liquid cargo often changes at the point of transfer and sampling is necessary at the point at

which custody handover takes effect so that quality and conformance to specification can be verified.

Unlike solid bulk cargoes, there are more opportunities for liquid cargoes to be contaminated and since liquid cargoes are typically much more sensitive to contamination, the ramifications are more serious. Liquid cargoes can be contaminated while in static storage or when being transferred from one storage tank to another via pipes, by picking up contaminants from the pipelines or pumps. Contamination can be due to pipes, pumps or tanks not being properly cleaned and prepared between differing cargo species. Cross contamination can also arise from pipes having valved branches to other adjacent pipes being improperly closed or secured.

Chain of Samples

In order to verify the condition of the cargo at any point in the transfer process, samples need to be taken at all possible locations at each relevant point along the line: at the originating storage tank before; at suitable points in the pipeline during; and in the receiving storage tank after the operation. This chain of samples from one location to another will be necessary in order to pinpoint the location of contamination, if it occurs, and, importantly, to help to apportion blame.

Dynamic sampling

Sampling can be static or dynamic. Static sampling is carried out from a body of liquid contained within a tank either before the start or after the completion of the transfer process. Dynamic sampling is carried out during the transfer process from the pipeline between the two storage locations.

Automatic dynamic sampling is often carried out by in-line samplers during the transfer

process. Ideally they take ‘bites’ of sample at flow proportioned intervals throughout the operation. The stored sample is then mixed and divided to give a number of identical portions. In-line sampling is usually used in bulk cargoes of crude oil when the static liquid may not be homogeneous due to the presence of water and sediments. Automatic in-line samplers usually offer the best chance of getting properly representative samples and they are therefore usually specified for this type of cargo. In-line samplers are sophisticated and expensive items and they are usually owned by the exporting or importing terminals and their operation is monitored by the independent petroleum inspectors. Ship’s crews seldom have access to such equipment but should be aware of it when in-line samples are presented to them as representing their cargoes.

The dynamic samples with which the crew should be more familiar are manifold samples usually taken at the start of loading or of discharging. These can be from shore or ship manifolds and are often used simply to verify the visual appearance of the cargo at that point in the line before valves are opened to allow it to flow on towards the receiving tank, either ship on loading or shore on discharging.

Manifold samples at discharge, for example, are important to verify that the ship’s lines and pumps are clean and free from water or other contaminants before cargo is released to shore. In some trades, such as in chemical and parcel tankers, it may be considered necessary to slop a small portion of cargo before it flows clean. This is more often the case with older ships which may have mild steel pipelines instead of stainless steel, or when the last cargo may not have been completely removed from the ship’s lines.

Manifold samples at loading are similarly important to check that the cargo is as it is supposed to be before it is allowed to flow into the ship’s tanks. Manifold samples are usually taken unilaterally by the ship’s crew. They will be of limited evidential value, however, unless they are properly registered, labelled and sealed, and preferably verified by an independent petroleum inspector.



Manifold sampling

Manifold samples can be taken at the very start of loading or discharging, as suggested above, or they can be taken at intervals as loading progresses. They might be taken, for example, after the delivery tanks are changed over, in order to confirm the ongoing condition of the cargo. Ship's manifolds, however, will often cause a "Venturi effect" at the sampling point such that there will be a suction into the sampling valve and no liquid will be forthcoming. This effect is most often caused during loading, when there is no back pressure at the manifold and a negative pressure is produced as the cargo drops into the ship's tanks. The effect can also be produced by liquid flow in the line passing across the opening that is the sampling point. The effect will cause outside air to be drawn in to the opened sampling cock so that no liquid comes out and no sampling is possible.

First foot sampling is often carried out with sensitive cargoes, as soon as the first foot

or so (300mm) of cargo has arrived in the ship's tanks. At that stage, loading may be stopped while a sample is taken and analysed. A running first foot may be taken without stopping just to confirm visual appearance. First foots will confirm the cleanliness and suitability of the ship's pipelines and give the opportunity to stop loading and to discharge if contamination is found, before too much cargo is affected. First foots taken in conjunction with manifold samples may help to identify where contamination occurred. In the absence of a clean manifold sample, it may not be possible to know whether the cargo actually arrived on board clean.

Static sampling

Static samples are usually taken through the upper openings of a tank: through the roof of a shore tank or the deck openings of a ship's tanks. They are usually comprised of either running samples or zone samples. Since liquids are mobile, it is far more straightforward to conduct reliable static sampling in them than in dry bulk cargoes.

Running samples are taken with the suitably weighed sampler, often a bottle with a restricted opening held in a cage, being lowered on a cord at a steady rate down through the liquid layer and then returned to the surface, again at a steady rate. The idea is to get the sampler to be filling evenly all the way down and then all the way back up. The sampler should not be full on completion, otherwise it cannot be known which portion of the liquid column has been represented and which has not. Running samples are usually used when it is thought that the liquid is reasonably homogeneous.

When the liquid cargo is not homogeneous, zone sampling may be considered more appropriate. A zone sampler is capable of being lowered through the liquid column to an appropriate height to capture a sample at that height. The captured sample from that zone is then recovered and transferred to a bottle. Zones can be at the top (near the surface), upper (upper third), middle (central third), lower (lower third), bottom (dead

bottom) or at any increment in between. Intermediate increments may be tailored to suit specific cargoes. For example, some fats and vegetable oils can become stratified with temperature changes and may exist in quite different forms at the various levels. They can even be damaged at lower levels in contact with the heating elements if heated too rapidly. Oil/water interfaces detected with water finding paste or electronic interface detectors may be used in order to inform the levels at which zone samples are to be taken.

A dead bottom sampler (often called a thief) usually has a valve at the lower end which is only opened when the device actually contacts the tank bottom, when it “steals” a liquid sample through the lower opening adjacent to the tank floor. Zone samplers have a device which allows them to be opened at any level or simple flap valves which allow flow through them going down but close to hold a sample going up.

Zone samples will often be proportionally blended in order to make up a composite of that tank. For example, a composite may be comprised of upper, middle and lower



Zone sampler

zones (U/M/L). However, bottom samples are usually kept out of a composite so as not to disproportionally represent the bottom (which may contain a lot of water in less dense liquids) in the finished sample. Bottom samples are considered to be for information purposes: they cannot be proportionally representative of the liquid existing higher up in the tank.



Dead bottom sampler

Zone samples mixed from different incremental heights within a liquid column will need to be proportioned according to the volume that they represent. For example, in a vertical cylindrical or square tank the proportions will be the same at each vertical zone. However, in a horizontally cylindrical or spherical tank, the central zones will represent a larger proportion of the total volume than the upper and lower zones.

Independent petroleum inspectors and retained samples

Most custody transfers (ship loading and discharging operations) in the petroleum industry are monitored and documented by accredited independent petroleum inspectors. The inspectors are generally appointed jointly by buyers and sellers of the cargo, one party is often the charterer. These inspectors will therefore act for cargo interests but being independent ought to act in the best interests of all parties, including the ship.

A ship's crew should be encouraged to cooperate with the independent inspectors but should also recognise that, as they act for cargo interests: in the event of there being a potential problem, a surveyor should also be appointed through the ship's P&I Club.

The ship's crew should ensure that the independent inspectors take proper and adequate

samples, and that these are sealed, labelled, receipted and distributed in accordance with normal procedures. The crew should ensure, for example, that duplicate sets of sealed samples, properly receipted, are retained on board for future reference.

Retained samples should be logged into the ship's own system and recorded in accordance with the SMS procedures. Samples retained on board should be housed in approved sample lockers where they can be kept dry, protected from sunlight, excessive heat and mechanical damage. They should be retained as long as necessary, usually until some months after the respective cargo has been discharged without comment, and then destroyed. A ship should avoid retaining old and irrelevant samples in the lockers. This just causes unnecessary clutter and confusion with current samples.

Sample handling

Regardless of whether the sample is a liquid or dry commodity, once the sample has been taken and processed, it will be sent or retained for analysis and therefore it is important to identify it properly. Labels stuck to the outside of samples often will go missing or become unreadable. We tend to double-bag all samples with clear polythene bags, with the label visible between inner and outer bag.

Sealing the sample with numbered seals is also recommended so there is less chance of interference. Many seals are available, but a higher security is obtained from using tamper-evident seals which show obvious signs of tampering if they are attempted to be reused.

Seals should be deployed properly and used to seal the actual sample. We have seen seals used around the neck of bottles – it would be a simple matter for devious workers to replace contents with that arrangement:

The label information is also critical. We recommend as a minimum:

The label information

| | |
|--|--|
| Ship/facility name | |
| Date | |
| Goods (e.g. wheat) | |
| Description (e.g. apparently sound) | |
| Place (port/city) | |
| Quantity | |
| Whether bulk/bag | |
| Sampler name and company | |
| Sampling method | |
| Identification mark (usually seal number) | |
| Origin of cargo | |
| Location of sampling (e.g. holds, truck, conveyor) | |



Using good quality ink that lasts at least several years is recommended.

The label information



Seal around bottle neck

We also take close-up photographs of the samples including close-up photographs of the seals and sealing arrangements. We have heard of unscrupulous parties 3-D printing new seals but when compared in detail with the originals, the structure is difficult to replicate in the necessary detail so clear detailed photographs can be useful to address such behaviour.

As part of the evidence trail we would also suggest taking time and date-stamped photographs of the samples with the new custodian whether that is a courier, a lab or surveyors so that evidence of sample location is retained.

Finally, samples should be stored in a clean, cool environment and checked on from time to time. Some samples need to be kept for 90 days or more as part of typical contractual agreements. We have in the past encountered samples totally destroyed by rats because the samples were stored in inadequate conditions.

Conclusions

1. Sampling of cargoes appropriately and comprehensively can prevent spurious claims or limit claims to realistic levels.
2. In some instances, for example Group A cargoes, proper sampling is critical for ship and crew safety.
3. Sampling is a time-consuming and exhausting undertaking and marine industry operatives should be aware of the efforts involved and adequately prepare for this.
4. All parties in the marine industry should be aware of the responsibility of creating statistically valid samples and relevant parties should be willing to demonstrate their veracity with supporting evidence.



Case study 1

A product tanker arrived at Barcelona with 30,000 tons of jet fuel to discharge. The arrival ullage survey, with water dips, showed there to be various amounts of free water in most of the ship's tanks. Samples taken at upper middle and lower levels showed that the cargo was on specification but the dead bottom samples revealed mostly water. Analysis of these samples showed a water that was neither fresh nor characteristically sea water: the sodium/chloride balance was not typical of sea water. Further investigation showed that the water was most likely to be refinery water and therefore from the loading port terminal.

With the cooperation of cargo interests, each cargo tank was de-bottomed to a slop tank on board. Suction was briefly made with the pumps from each tank in turn until all traces of free water had been removed. During this operation the ship's ballast tank contents was adjusted to give a list and trim which allowed for the most effective suction from each tank in turn. After de-bottoming, the cargo in the tanks was found to be of acceptable

quality with no significant free water. The ship's lines were stripped empty to remove the water and discharge was commenced.

At commencement of discharge the ship's manifold samples showed no significant water and the cargo was accepted without further comment.

The quantity of de-bottomed material drawn off was later discharged separately. No claim was made against the ship as the water was demonstrably not of ship board origin.

Effective sampling was the key to minimising loss in this case with a successful outcome for the ship.

Case study 2

A product tanker arrived at Djibouti to discharge separate parcels of jet fuel, gasoline and diesel, all loaded at Jeddah. Sampling prior to discharge showed that one of the two jet fuel tanks was off specification on flash point. Sampling and analysis showed that the tank had been contaminated with gasoline.

Investigation showed that the contaminated tank had been the first to receive jet fuel. The jet fuel was loaded after gasoline through the same ship's manifold and bottom line system. It was considered likely that the single tank had been contaminated by gasoline remaining in the ship's lines after that grade had been loaded first. The line was supposed to have been stripped dry between grades but the arrangement of the line showed that this was probably not possible due to the line having an undrainable belly.

No first foot samples of the jet fuel had been taken and no analysis had been carried out of the after loading samples at the loading port. However, sealed after loading samples were carried by the ship to

the discharge port. These included individual ship tank samples, being the composites of upper, middle and lower samples taken after loading. Analysis of the after loading sample from the contaminated tank showed that the contamination was already present after loading.

Discharge of the uncontaminated tank of jet fuel went ahead at Djibouti. However, the contaminated tank of jet fuel was retained on board, for later disposal elsewhere. In this way the flash point contamination was contained within the ship. Had discharge of all the jet fuel gone ahead in Djibouti, all the jet fuel would have been contaminated and the low flash point material would also have soiled the receiving shore terminal's dedicated storage facilities.

Proper sampling carried out at the discharge port allowed the damage to be restricted to just one of the two jet fuel tanks on board. However, a lack of proper sampling at the loading port had allowed the cargo to be loaded with one of two tanks being off specification, without this fact being identified at the time.

Case study 3

A bulk cargo of maize was delivered to receivers in Yemen. Heat leakage from settling tanks in way of hold no.5 aft bulkhead was insufficient to damage the maize, but it did initiate mould growth on the cargo surface in the vicinity of the aft bulkhead.

Samples taken by authorities in this area were found to have aflatoxins above the required level and the cargo was rejected.

Receivers were encouraged to take samples of the cargo according to international standard frequencies, also incorporated into Yemeni regulations. The new samples were more representative of the cargo and were all in specification, and the authorities allowed offloading to commence.

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