Moisture determination. Mass loss on drying – Commercial sampling washed away

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What is this? Sampling washed away; "samplewashing?" Greenwashing is a known term nowadays, but is there such a thing as "Samplewashing" too? Yes and no: Greenwashing is the process of conveying a false impression or providing misleading information about how a company's products are more environ-mentally sound. ... Greenwashing is a play on the term "whitewashing," which means using misleading information to gloss over bad behaviour. And that is what this sampling paper is all about: presenting moisture results on samples of solid bulk materials where the theory of sampling was not applied… and therefore sampling errors are magnified by not only glossing over the representativeness of the process, but at the same time by watering-down the monetary profits of the trade for one whilst condensing them for the other party. It really is "Samplewashing" when it comes to moisture determination!

Introduction

Quantification of the moisture content is one of the main "value influencers" for traded solid bulk commodities where transactions are made and paid on the basis of its unofficial unit of measurement, or the "DMT" - Dry Metric Tonne. Googling for "what is DMT," we quickly run into the commodities glossary at IndexMundi for dmtu, or dry metric tonne unit[1]: "as the internationally agreed-upon unit of measure for iron ore pricing it has the same mass-value as a metric tonne, but the material has been dried to decrease the moisture level. A dry metric tonne unit consists of 1% of iron (Fe) contained in a tonne of ore, excluding moisture." Not much there ehh? Drying the whole tonne how; what temperature?

Decrease moisture to what level? Maybe commercial contracts provide some guidance as encountered on the Google quest to learn about moisture? Law Insider to the rescue with an appropriate example. Its Iron Ore Royalty Agreement[2] states: "Article 1.1 - Dry metric tons means the actual weight of Iron Ore calculated on a moisture-free basis. Methods of sampling, testing and calculating moisture-free weight shall conform to methods described in Article 5: Sampling, Assay, and Analysis." Excellent! We have just learned that the material should be in fact moisture-free and it also appears that calculation is done using testing on a sample.

Let's continue and find out how with referenced Article 5 of same agreement: "Article 5 - Sampling, Assay, and Analysis. Any determination of weight, volume, moisture content, or pay metal content, and any sampling and analysis required pursuant to this Article 5 shall be made in accordance with sound mining and metallurgical practices and standard sampling and analysis procedures prevailing in the Iron Ore mining and milling industry by an independent inspector mutually agreed to by IMG and the Agent. Upon the request of the Agent, but no more than three times in any 12-month period, IMG shall cause such inspector to take three samples and provide one such sample to IMG, a second sample to the Agent and retain a third sample in the event of a dispute among the Parties in respect thereof. All statements or reports wherein such independent inspector's assay of samples are set forth shall be conclusively presumed to be true and correct, unless, within ninety (90) days after such statements or re-ports are delivered to the Agent and IMG, either the Agent or IMG makes written objection thereto and demands an assay by an independent referee mutually agreed to by IMG and the Agent. Unless such objection and demand are made within such 90-day period, the independent inspector shall have no duty to preserve the third sample after the end of such 90-day period." Such absolute horror, but such a reality! If you do not see it yet, I will tell you why in this paper.

Pay metal content

This article will go over some key sentences of Article 5 of the Iron Ore Royalty Agreement mentioned above. These key sentences of Article 5 will be like the table of contents for of this document.

Whereas the Iron Ore Royalty agreement is a specific contract, its wording is very similar to trade contracts seen by the author. Taking things from the top of Article 5: Pay metal content. Pay Metal Content really is the culmination of all determinations that are required to have a commodity change ownership and decide on its price. It looks as if it is built up from weight, volume, moisture content and of course the analysis of the element of interest. Leaving the frivolous reference to volume aside, as this should not feature at all in a commercial contract covering solid bulk commodities, at this stage I would only want to point out the careless use of the term weight determination. As pointed out in one of my other papers (Vogel & Esbensen, 2021)[3] weighing deserves proper attention and clarification if used for commercial settlement transactions, the impact of errors in mass determination may overshadow all other sampling and testing errors.

Sound mining and metallurgical practices and standard sampling and analysis procedures prevailing in the Iron Ore mining and milling industry

Remembering that the referenced IndexMundi article is about DMT and also mindful that we are trying to find out its 'what' and 'how', then it is pretty disappointing to find ourselves with a set of undefined terms in the example Iron Ore Royalty Agreement. In the example agreement we find terms such as 'Sound mining' and 'metallurgical practices'. We can quickly agree that they refer to activities outside the scope of sampling and testing. But then, what does the article say next with respect to terms that do affect us?

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The Iron Ore Royalty Agreement – Article 5 further mentions: "standard sampling and analysis procedures pre-vailing in the iron ore mining and milling industry." Delegates of ISO Technical Committee 102 for Iron ore and direct reduced iron can surely voice their expert opinions on what could be considered as the appropriate standards for iron ore commodity trading. ISO (not an acronym) is the International Organization for Standardization that develops and publishes international standards that are designed to facilitate equal trade conditions. Unfortunately, here there is no reference in the Article to an ISO or other specific standard, or even to the Theory of Sampling. That would have helped the person that is tasked to do the sampling, moisture-testing and calculation of the DMT, but there is no further guidance in the agreement, not a commercial contract, there is nothing at all on the specific approach to follow when it comes to DMT and the associated sampling and testing. Is this just a bad example? Let's take another look in the repository of Law Insider for a reference to DMT and moisture. In a trade contract for a solid bulk commodity we find for a copper concentrate from a renowned Swiss trader[4]: "Article 9.02 - Determination of Dry Weight and Moisture. The dry weight and moisture as determined pursuant to Section 9.01 shall govern for the purpose of final settlement of the Total Compensation for each Parcel." Again, we are referred to another article in this Swiss trader contract to find the answers that we need, but here we go: "Article 9.01 - General Procedure. (a) With respect to Parcels shipped to Japan, South Korea, the Philippines, North America (including Mexico), or Western Europe (including Bulgaria) hereunder, the weighing, sampling and determination of moisture for each Parcel shall be carried out in accordance with internationally accepted industry standards at the Port of Discharge or at the ultimate receiving smelter in the country, whichever is customary. The Seller and the Buyer shall mutually agree on the person to supervise at these operations, and its costs shall be shared equally. (b) For shipments to China and India, the Seller and the Buyer shall mutually agree whether each ultimate receiving smelter: (i) has available reliable modern equipment; and (ii) applies accepted standards commonly recognized in the industry, for the purposes of weighing, sampling and determining moisture content of a Parcel. If the Seller and the Buyer agree that a smelter does not satisfy both of these requirements, the weighing, sampling and determination of moisture of each applicable Parcel shall be carried out in accordance with internationally accepted industry standards at the Port of Discharge. If, alternatively, the Seller and the Buyer agree that both of the foregoing requirements are satisfied, the weighing, sampling and determination of moisture shall be carried out in accordance with internationally accepted industry standards at the receiving smelter. In either event, the Seller and the Buyer shall mutually agree on the person to supervise at these operations, and its costs shall be shared equally."

Delegates to ISO Technical Committee 183 for copper, lead, zinc and nickel ores and concentrates would know too that there are specific standards that cover the weighing, sampling and moisture determination that can pro-vide a lot more guidance than the same undefined terms that we find in the Swiss contract. It was not just a coincidence: sales contracts very seldom actually specify the standards that will apply. And with more than 20 years of experience in traded commodity inspections the author can state that buyer and seller will seldom agree and actually specify a standard or a theory at any stage in their dealings. It is really left to the person that is appointed for sampling and moisture determination to make his/her own interpretation on what is an 'international standard', or what is a 'prevailing practice'. That is quite subjective unfortunately and it may stem from the insecurity or lack of expertise. It does explain another bold referenced sentence of Article 5… more on this later.

Three samples

Here we are not going into a quest for more contractual examples; please take my word for it that it is common again that, at least contractually, there is little to no meaningful guidance in commercial contracts on how many samples should be tested for moisture determination. Let alone a specification how many cuts or increments samples should be composited together. In fact, in most contracts the use of the term 'sample' has nothing to do with precision of sampling, but is only used to indicate the number of final split portions obtained by dividing an unspecified mass of sample material at an unspecified nominal top size. In this case the number of three samples corresponds to the parties to whom final samples are to be distributed:

- Seller
- Buyer
- Agent (umpire or referee sample)

Each party would receive only one sample each. This too is typical for sale and purchase agreements for solid bulk commodities where the financial settlement is finalized after both parties have tested their sample for metal content, or penalty elements… Such final samples should be the end result of a representative sampling process, they would be dried, crushed, homogenized and pulverized to a size of about 1/10 of a millimetre… But this is not meaningful at all for moisture determination!

Moisture – or mass loss on drying at a specified temperature in a specified atmosphere (e.g. air or inert gas) – should be determined on test portions that result from a representative sampling process that was done as close as possible to the time and place where increments (or cuts) were drawn from the bulk commodity. In practice it is not possible to pack and protect the integrity of divided sample material that is to be used for moisture determination. Not only is packing and transporting over long distances a concern with respect to loss of moisture, but it is also a cumbersome exercise with respect to the sample mass that is required for transport. In the examples of iron ore, the minimum mass of a single test portion is 1 kg (at 10 mm nominal top size) and as a minimum four test portions are required in case there is a single composite sample for the whole shipment. Therefore, the minimum mass of a divided sample to be dispatched – each of the 'three samples' – should already be 10 kg minimum as per ISO standards 3082 (Iron Ore)[5]. In the example of the copper concentrates where sub samples for each 500 tonnes were agreed the sample mass to be dispatched as per ISO 12743 [6] would have been minimum 46 kg (at 10 mm nominal top size of agglomerates) for a 10,000 tonnes shipment. Such sample weights may seem trivial in a world where millions of tons of solid bulk commodities are shipped, but the reality is that the logistics and costs of

shipping some tens of kilograms of sample material by mainstream courier companies is considered as burdensome due to red tape regulatory and documentary reasons. It is also considered expensive in relation to the cost of sampling and testing. All in all: not an option, moisture determination should be done on-site or near-site.

Figure 1. Samples in their non-absorbent air-tight bags awaiting drying soonest after sampling for best integrity

Unfortunately, in the contractual agreements for establishing dry metric tonnage, this is not properly addressed. Equally unfortunate is that inspection and laboratory management standards such as ISO 17020 for sampling, and ISO 17025 for testing, are not providing adequate help in my experience either. ISO 17020 and ISO 17025 accreditors often miss the true meaning of the commodity specific guidance that the subject matter experts working in ISO Technical Committees included to reduce risk of moisture bias and it happens that moisture samples of accredited sites still traverse quite a bit of distance and time before being tested. Advise: specify that your moisture samples are tested on site or near-site… and add KPI as maximum distance and time!

Retain a third sample in the event of a dispute

Retain a sample in the event of a dispute? Retaining a sample for longer periods for moisture determination? Following the last paragraph, where the objections of more or less immediate sample dispatch were already discussed, it is hopefully evident that a longer period of storage would be required here. The third sample will be retained pending testing for moisture by 1. the seller and 2. the buyer, plus a certain time needed for the unsuccessful dispute resolution; this will not do the sample integrity any good. But, for the sake of legal conformity as the contracts refer to international standards after all: we will check one such standard; using ISO 3082:2017 – Iron Ores – Sampling and preparation procedures. Again, just as an example of what specific standards would contain: "ISO 3082:2017 – 4 General considerations for sampling and sample preparation – Basic Requirements: … Moisture samples shall be processed as soon as possible and test portions weighed immediately. If this is not possible, samples shall be stored in non-absorbent airtight containers with a minimum of free air space to minimize any change in moisture content, but should be prepared without delay." ISO 3082:2017 – 10.2.4 Special procedure for moisture content: "Constitution and collection of moisture test samples for moisture determination shall be such that changes in moisture content are minimized, including measures to minimise the effects of weather (heat, humidity and rainfall) and time (evaporation), and may include the use of special containers and storage conditions. This will minimise bias and result in better overall precision (including sampling, sample preparation and moisture determination)…"

"You see!," the legal teams will now say, "…it is possible! Even the ISO international standard permits what we wrote in our contracts!" Admittedly, in a court of law or in arbitration proceedings it will require persuasion and expertise by looking further than the verbatim of the standard alone. Bear in mind too that the name ISO is derived from the Greek 'isos', meaning equal, and as such aims to ensure that its standards to describe the characteristics of the traded commodities provide confidence that the requirements for those commodities and testing have global relevance and are accepted worldwide. We must look a bit further and investigate context and meaning: ISO 3082:2017 – 10.2.4 "Special procedure for moisture content: …When it takes a long time for loading or unloading a lot, the lot shall be divided into parts corresponding to not more than 8 h periods. A moisture partial sample shall be constituted representing each part and a moisture determination shall

be carried out. The division into parts shall be subject to weather conditions, e.g. heavy rain or high temperature, and other conditions or circumstances at the time of loading or unloading. However, if the moisture sample containers and storage conditions prevent a change in the moisture content of the moisture partial samples, a moisture gross sample may be prepared for the whole lot…" The intention of ISO is clear, by reference to the 8 h periods it sets the expectations of moisture determination. It shall be done within hours after sampling. Not days or weeks that would be the case by courier logistics of the seller and buyer sample, testing in remote laboratories of seller and buyer, testing for moisture in both laboratories, exchange of results between seller and buyer, unsuccessful dispute resolution, courier dispatch of the third (umpire/referee) sample and ultimately testing at the umpire/referee lab.

If the legal teams would not give up yet and insist referring to text in ISO 3081 mentioning sample containers and storage conditions that can protect the integrity of such important moisture samples, then I want to refer to Ziegelaar and Fritz (2017)[7] that have studied a selection of sample containers. They concluded that well-sealed plastic bags, PVC jars with screw-top lids and even steel paint cans failed their test to preserve moisture… already for relatively short periods. Quantification of moisture losses is very difficult as there are so many variables that impact changes in moisture: ambient temperature and relative humidity just to name two. But, a difference of 0.1 per cent in moisture change is not hard to imagine for a loss over a period of a few weeks. Ziegelaar and Fritz even referred to errors from 0.5 to 1 per cent in moisture and calculated the impact of 0.1% for the Australian iron ore exports in 2017 to make a point. At current pricing levels of iron ore iron and an export tonnage of around 900 Mt/annum that would now convert to US$ 140M as cost for such a small error. Moisture determination needs to be done on-site, or near site. Immediately after sampling, or within hours after sampling but only provided that the containers and storage conditions are not introducing significant change in the moisture content in such period of hours as it not possible in practice to maintain sample integrity over longer time. It is practically impossible to preserve samples over longer periods of time without compromising accuracy of moisture determination. Consequently, the provisions in the contracts with respect to sampling and to samples have little real use when it comes to moisture determination and calculation of DMT.

All statements or reports wherein such independent inspector's assay of samples are set forth shall be conclusively presumed to be true and correct

Wow! Such high credit and praise to the independent inspector who appears to be all-knowing and with full empowering own discretion of deciding on a sampling plan and a way to test for moisture. A couple of grab increments at a random moment during loading or discharge taken by an ever so popular cast aluminium, round bottom, sugar/flour utility scoop would fill up a couple of moisture trays. Moisture trays with ore that are then put over a gas burner and stirred until the technician considers it as 'dried'.

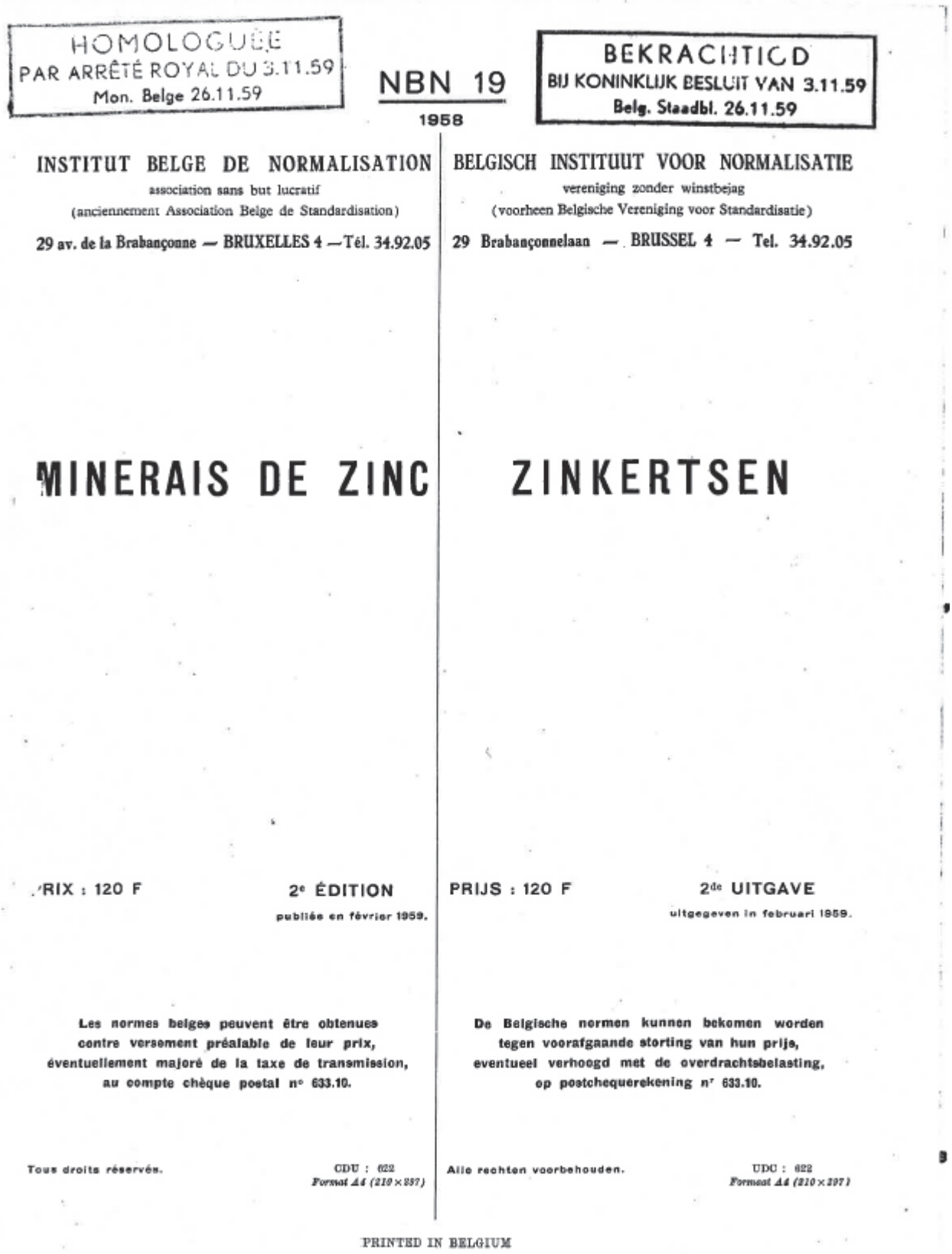

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Figure 2. Belgian standard from 1925, reissued in 1959 and still valid in 2022

"Surely that is not a standard practise for solid bulk minerals!", a reader may now exclaim in shock. "No?" Meet NBN 19:1958 [8], a Belgian standard for sampling and testing of Zinc Minerals which contents were first issued in 1925 but remains valid today as official standard for imported zinc ores and concentrates for the sampling and testing of the moisture content. Translated into English with smallest possible liberty and NBN-19:1958 states, bulletized:

- Take all 1 kg sub samples for each of 300 tonnes sublots and spread on a steel plate,
- Pound and mix twice, then form into a square,
- Extract 1 kg taking increments diagonally,
- Crush in a mortar till all passing a sieve with apertures between 2 mm and 3 mm,
- Place into a sample container and seal for transport to the laboratory,
- Moisture content shall be measured in the evening, or latest the next morning,

- Testing will be done by the buyer and with the help of the seller's representative,
- Open the container with the moisture sample [filled with 1 kg during sample preparation process] and transfer the sample to a large copper bowl,
- Spread out evenly,
- The operator accurately extracts a test portion of 100 g using a spatula and places it in a round porcelain enamel dish of 250 ml that was tare weighed earlier to nearest 0.05 g,
- Heat over a Bunsen burner,
- Monitor with a mercury thermometer that is kept in the bulk of the test portion during heating that the temperature will not exceed 100°C,
- Use the thermometer to stir and mix the test portion whilst making sure that no sample material is spilled; this can be checked by cleanliness of the working surface,
- Cut off the burner till pilot light when 100°C is reached and only ramp up again when temperature has dropped to 50°C,
- When the temperature has again reached 100°C the burner is turned off and a cold sheet of glass is placed over the moisture tray that should then not show any condensation, repeat heating process when condensation is noticed,
- Weigh the warm tray and test portion to nearest 0.05 g and heat for one more cycle,
- Re-weigh the warm tray and test portion, the weights should be identical; if not continue with heating and weighing cycles until two successive weighings are identical,
- Record mass of the gross dried test portion and the tray,
- Subtract 1/10 of a gram from the tare weight of the cold tray as this is the correction factor for weighing in hot condition,
- Subtract the corrected tare weight from the gross mass of the dried test portion and the tray in order to obtain the dry weight of the test portion,
- The difference between the 100 g that was initially placed in the tray and the dry weight represents the moisture that was in the sample and is used to calculated the moisture content of the sublot to nearest one-half of one-thousandth.

Using the above as example is very much for purpose of polarization, but it shows what might happen in an extreme case when a contract is followed to the letter and everything is left to the discretion of the inspector. In practice this kind of moisture determination is no longer observed in its entirety in Belgium on zinc concentrates. However, parts of the procedure such as pounding of the sample, mixing it and forming a square are for example. Also, for other materials such as ferro alloys, stainless steel scrap turnings and occasionally even on industrial minerals a similar heating procedure and drying over a Bunsen burner is still observed sometimes at locations without suitably equipped sample processing facilities, or where receivers do not mind preparation error of losing some sample material and inflating the moisture content. The point in case: specify the testing method! Define the mass of the test portion in relation to the particle size, state the temperature of drying, mention the atmosphere of drying and the endpoint of the test. This does not have to be too detailed. The following may suffice: "Test portions of 1 kg and sample material of max. 10 mm nominal top size shall be dried in air in a suitable oven at 105°C ±5° until constant mass is reached between two successive weight determinations and recordings of the dry mass; 4 hours apart. Constant mass is achieved when the two successive do not differ by more than 0.05% of the initial wet mass of the test portion." Without such definitions and agreements, it is a pretty big step to contractually agree that everything the independent inspector says is true.

Trueness of the reported moisture value will require that the tested sample is the output of a representative sampling process and that during the sample preparation noting is added to the sample… or removed from it. Sampling and its representative process is not given much attention in the example contract Iron Ore Royalty Agreement, neither in the copper concentrate contract of the Swiss trader. Nothing on the number of increments of cuts by the mentioned "available reliable modern equipment", we will have to hope for the best that the "sound mining and metallurgical practices and standard sampling and analysis procedures prevailing in the Iron Ore mining and milling industry" will deliver. That might be a bit too much to hope for unless each of such practices and standards find their foundation in the Theory of Sampling (TOS). In fact, would that not be the best provision in any contract: "Sampling shall be in compliance with TOS"? No need to refer to undefined approached and practices or to modern [what is it?] equipment, but to a comprehensive authority to resolve such uncertainties and undefined references. TOS is there to tackle all issues with respect to correct – and incorrect sampling, bias, precision and representativity; it is further detailed in four fundamental publications[9, 10, 11, 12]. Remember: TOS is not commodity specific! TOS can be applied across the board if implemented by a knowledgeable specialist who understands it as well as the commodity in question.

Back to "samplewashing" as this was the term used in the opening sentence of this paper. Let's for the purpose argument assume here that TOS was followed and that the sample is the end result of a representative sampling process. A process where heterogeneity and target precision were duly considered and where the airtight containers that were used, to preserve the sample integrity during transport and storage, were flawless. It was detailed already in this paper that maintaining the integrity of a moisture sample over longer time is not practically possible, but even if it were: then still all diligent efforts of sampling and packing samples can be washed away by incorrect sample preparation and testing.

We will go through the process steps sequentially, but first remember what the basic requirements for sample processing are. Most sampling standards that describe sample processing start with a variation of the below, this is from ISO 3082: "ISO 3082:2017 – 4 General considerations for sampling and sample preparation – Basic Requirements: The basic requirement for a correct sampling scheme is that all parts of the ore in the lot have an equal opportunity of being selected and becoming part of the sample for analysis (Gy; Pitard). Any deviation from the basic requirement can result in an unacceptable

loss of trueness and precision. An incorrect sampling scheme cannot be replied on to provide representative samples…"

First step, after sampling and storing moisture samples in non-absorbent airtight containers with a minimum of free air space, is to obtain a test portion from these samples. Therefore, the sample container must be emptied again. Often condensation is formed on the inside of the container during transport and storage. If not mixed back into the sample, or when not quantified by drying of the container itself after emptying it, then something – a mass of water – is excluded from the sample at that stage. This is preparation error, when it happens the reported moisture will likely be lower (than a situation without preparation error).

For some bulk ores, there can be a comminution step as the particle size is too large to accommodate oven drying. ISO 3082 states for iron ore that the nominal top size of the moisture test sample shall be 31.5 mm or less and that samples larger than 31.5 mm shall be crushed first. The standard does warn that the sample may be pre-dried – in its entirety – when it is difficult to conduct crushing and dividing owing to a sample being adhesive or excessively wet. "Oh, oh," we now even find vague, undefined conditions in the standard by the very ISO technical committee that I am part of myself. To completely avoid an unquantified loss of moisture due to heat generation of crushing, or wet adhesive particles left behind in the crusher pre-drying whilst weighing the wet and pre-dried sample: it really is the best approach to vigorously follow TOS and avoid all incorrect sampling errors. But it comes at a price, literary. To accommodate pre-drying, sample preparation facilities must have space, a lot of space to spread out uncrushed sample material and allow it to air-dry and equilibrate to the ambient conditions. The mass of the uncrushed sample that should be subjected to pre-drying is large: at 40 mm nominal top size table 4 in ISO 3082 gives an example of minimum 325 kg. If – as is the case in most sample processing facilities – multiple successive samples require processing in immediate, or short succession then this can become a serious bottleneck very quickly. With floorspace of sample preparation often not being much more than an ordinary office space in an available building, typically only one sample can be processed at once.

Figure 3. Sample immediately after crushing (during sample preparation), pre-drying was not possible and moisture loss has likely occurred

There is often no space for more samples, and it would be too much of a risk of cross contamination when successive samples would be spread out closely adjacent on the floor for airdrying and left uncovered for the time that such pre-drying requires. So, what is done in practice around the world? Here we inevitably come a point to consider what is available for sample processing on-site or near-site? The only option is to apply a best effort with the available drying space and processing equipment whilst using the wording such as "being adhesive or excessively wet" as found in ISO and some other standards as an escape clause and decide that samples are not too adhesive or too wet… and continue with crushing to 31.5 mm, or smaller, to a mass that will simply fit into the available drying space. When comminution is required on the sample that is used for moisture determination there is again risk that some sample with high moisture content is lost: this is preparation error and the reported moisture will likely be lower.

Except at limited locations where there are facilities with mechanical division, there will also be a step of mixing. This is an attempt to homogenise the sample and to aid extracting of test portions, because there will be segregation as a result of the compositing of the sample by individual cuts or increments, i.e. from the quality variation of the product itself. But there can also be segregation caused by the time and conditions of sample store in the container, referring to condensation again as perhaps the most imaginable one. But how to mix a sample of a bulk solid material without loss of moisture due to airflows

and evaporation? ISO 3082 will even prohibit mixing despite an obvious need in case of segregation; it states that mixing of moisture samples may result in moisture loss and hence bias, so moisture samples shall not be mixed prior to division. ISO 12743 is clear too by stating that moisture samples shall not be mixed prior to division, unless mixing is carried out in a closed container, such as a plastic bag. The differences between the two ISO standards are a result of the different commodities iron ore vs. non-ferrous concentrates. This is not because TOS provisions are different, but because the sampling and sample preparation conditions are different. The sampling and preparation conditions are designed towards the tonnage and volume processed at the place of sampling. ISO 3082 considers very large lots over 340,000 tons. ISO 12743 does not specifically consider the tonnage of shipments, but in practice a shipment is seldom more than 10,000 tons. The standards in the industry are designed by representatives from the industry and as a result include specificities of respective conditions. As industry standards and ISO are primarily there to accommodate equal trade it may thus happen that TOS takes the backseat. When it comes to moisture, that has such a significant impact on the money that changes hands: this is a potentially expensive oversight.

And then there is division, or extracting, into the actual test portions. As was covered above, it is practically impossible to pre-dry samples in their entirety, the same applies to 'full drying' and moisture determination on the entire sample. In most cases a sample must be divided into test portions that can be accommodated by the availability of moisture trays and fitted into the drying ovens. Like the installation of a sampling system is often an afterthought of engineers when designing a terminal, sample preparation facilities with appropriate drying facilities meet the same limitations. As a result, drying is not tailored to the conditions and properties of the samples, but it is the other way around and test portions are shaped in size and mass to fit into the oven space and maximum allowed time for drying. When it comes to moisture determination it is not only the weight of the test portion that may enable a sample preparation process to maintain its representativeness from correct sampling error perspective. Much more important is that there are no incorrect sampling errors introduced from division techniques where airflow causes evaporation.

Finally, after obtaining the test portions we are ready for the actual testing. That should be pretty much straightforward, "yes?" Weigh, stick it in the oven till dry, weigh again and express the loss of mass as percentage of the initial mass of the test portion… Well "yes" indeed, but mind your steps:

- moisture trays should pre-weighed clean and dry, made from non-corrosive material
- balance for weighing should be of sufficient size to support the moisture tray,
- test portion should be spread out as a thin layer of few or single particle thickness,
- drying temperature should be as agreed, or specified
- drying time should be sufficient, but not too long; especially when material may oxidize, sublimate, or lose volatile elements / hydrated water
- end-point should be defined, what is constant mass exactly?
- test portions shall weighed immediately after drying when they are still hot, or allowed to cool in a desiccator only
- balance for weighing should have a heat protective barrier so that loadcell(s) are not influenced by hot moisture trays
- preferably the same material tested for moisture should be used for further chemical analysis too (as common sample)

All in all quite a bit of detail ehh? And in the contract there is this: "All statements or reports wherein such independent inspector's assay of samples are set forth shall be conclusively presumed to be true and correct." Of course, the independent inspector is expected to be competent and suitably equipped with all the tools to perform its job without introducing bias or impacting precision. It is just that differences in comminution (or not), division techniques and to what mass, time of drying and end-point definition can have a significant effect on the trueness of the final moisture assay. Especially when considering the overall precision for moisture for the two example commodities of iron ore and copper concentrates when following the respective ISO standards.

Table 1. Target typical overall precision as absolute percentage for moisture βSPM or βT.

Commodity	Mass of lot t								
	Over 270 000	210 000 to 270 000	150 000 to 210 000	100 000 to 150 000	70 000 to 100 000	45 000 to 70 000	30 000 to 45 000	15 000 to 30 000	Less than 15 000
Iron Ore	0.34	0.35	0.37	0.38	0.40	0.42	0.45	0.49	0.55
Copper Concentrate									0.6

A target overall precision between 0.34% and 0.6% absolute… and that is for standards that are prescriptive when 'minding our steps!' For outdated yet arguably still legal, or regulatory standards such as NBN 19:1958 that will obviously not be the case; just to list a few reasons:

- evaporation during spreading and pounding = Preparation Error
- evaporation during mixing twice (how?) and spreading into a square = Preparation Error
- segregation after spreading into square = Quality Variation
- diagonal extraction of increments (how many and how much) into a 1 kg sample = Delimitation Error, Extraction Error & Preparation Error
- Fixed 'correction factor' of effectively 0.1% due to weighing hot sample trays…

Demanding an assay by an independent referee [in case of disputes]

Actually, this paper already touched on the futility of demanding a moisture re-test. Samples for moisture cannot be easily preserved over time and losses are likely to occur, especially over prolonged periods. It is expected that at least days have lapsed since the actual sampling and division of samples into moisture test portions. I will not dwell on this again at this stage. What is most interesting though, is that suddenly, the sample is considered absolutely true and representative of the cargo and material it was taken from!

Seller and buyer have a dispute on the moisture content, or DMT, but contractually look at the last little step of test-ing only. Albeit this last step is of course important and not devoid of its own problems as demonstrated above, the last step is what it is: then end of a process. In case of dispute on DMT not only the last step, but the whole process should be reviewed, the weighing and sampling first, then preparation and ultimately the test methodology itself. If a dispute is settled by just repeating the moisture test we can actually say with great confidence that TOS is thrown overboard: preparation errors are likely as moisture would have been lost since sampling and the most important scrutinizing of the sampling process is simply not considered at all. So: once a sample was generated, by whatever procedure deemed adequate, it becomes the contractual holy grail. Even when such process was flawed or erroneous, there is little contractual recourse possible. If that is not 'samplewashing' then I do not know what is! Mis-leading information on and of the sample is used to gloss over bad behaviour of non-TOS sampling and erroneous testing. Samplewashing!

Retrospectively it is not easy to repeat the process of weighing, sampling and moisture determination:

- it is difficult to demonstrate integrity of the material since it was weighed, sampled and stockpiled
- it is cumbersome and expensive
- and without TOS compliant standards… how?

For several reasons, but in practice there is only one time that DMT can be truly determined. That time is when the commodity changes ownership and the weighing sampling and moisture determination is performed right there and then. At that time and place the sampling should follow TOS and that is what should be contractually agreed to avoid disputes at later stage.

Conclusion

For commercial transactions of solid particulate commodities, the pay metal content is needed. It requires the moisture free mass, or dry metric ton basis. Which in turn requires sampling and testing for moisture determination. The problem is often that the procedure and theory for such sampling and testing are not contractually agreed or de-fined. Ultimately, in case of differences between interventions at different locations, there can be disputes. The disputes are most likely a result of a sampling methodology that is erroneous: where incorrect sampling errors were not completely eradicated and/or where correct sampling error was not sufficiently mitigated. However, in case of a dispute on DMT, in practice, only the moisture test is repeated on the erroneous sample. Even when flawed and stemming from bad behaviour, or when the sample integrity was compromised because moisture would have been lost, that sample still would be used to for commercial moisture determination and its result considered final and true. To glance over errors… and still use an outcome as true: that I can only call samplewashing.

The solution to this is the same message that you will have heard before. It is the same message when discussing installation of a sampling system when this is done as an afterthought in an existing port infrastructure. The message: do not forget to define a TOS compliant standard from the earliest stages! When designing a port, terminal, or warehouse and sampling is part of the workflow then it solves a lot of problems to set things up properly from the beginning. Ultimately the effort at the onset will result in cost savings for not having to make major modifications to logistics or sampling solutions. Or cost of being wrong, making ill-informed decisions, or trading on the wrong values. Perfectly in line with this reasoning is contractually agreeing on the moisture determination method itself and making sure that also here all incorrect sampling errors are removed and only correct measurement remains.

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